

Introduction to Process Engineering and Design

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Mr. Bhatt graduated in Chemical Engineering from the University Institute of Chemical Technology, University of Mumbai.

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Foreword

Dr. Jack Welch, CEO of General Electric, built its market cap by more than \$450 billion and established himself as the most admired business leader in the world. He is Doctorate in Chemical Engineering and is proud to be a Chemical Engineer. In his business autobiography he wrote, "I have always felt that chemical engineering was one of the best backgrounds for a business career because both the class-work and required thesis teach you one very important lesson: There are no finite answers to many questions."

A chemical process engineer's job is, therefore, tough. It is hard because he has to find not only a finite answer but to select the best answer for the situation from a variety of options. That is why process engineering is interesting and at the same time intriguing.

A process engineer has to design a plant or a part of it. He may have to design a unit operation or a unit process or a system. The important aspect is that it must work and operate reliably and smoothly. The unit is to be designed for a commercial purpose and, hence, it has to be economical and cost effective. A good process engineer has to have plant experience and, in addition, a good understanding of process economics and knowledge of current costs and trends.

Chemical process industry covers a very wide spectrum of industrial products. Apart from the chemicals they handle, the quantities and sizes also vary. This requires the process engineer to have flexibility of mind and vision to adopt itself to any scale. Selection and design of the equipment should therefore be capable to handle the capacities and instruments to measure the parameters for large chemical plants to micro units to manufacture enzymes.

Till 1973, fuel and energy costs were a small component of the manufacturing costs of a chemical product. Since then, the rising fuel prices have made it mandatory that the plant design and its operation be energy efficient. The approach to process design has undergone a paradigm change.

Bhopal gas leakage disaster and Chernobyl catastrophe made another impact on the minds of process engineers and changed the practices followed till then. Greater responsibility now rests on the process engineers for safer designs of the plants. HAZAN (Hazard Analysis) and HAZOP (Hazard and Operability) studies have become a part of process studies, attaching one more design aspect in the

minds of process engineers. Several lessons regarding storage and handling were learnt by the process engineer. He has to now respect environmental and ecological sustainability of the process and its design.

Software for the design of equipment such as heat exchangers, distillation columns, etc. are available. It has simplified piping design. Drawing and drafting has become easier, error free and faster. But without understanding the design principles, explained in this book, one cannot use them efficiently.

Yet, the process flow sheeting or the development of a process scheme still remains a brain teasing function. It can be explained and perhaps taught but cannot be standardized. Process engineer has to be innovative and creative. Preparation of a Process Flow Diagram is still a prominent and challenging domain of a process engineer.

It is more admirable that Mr. Bharat I. Bhatt and Prof. Shuchen B. Thakore decided to go deeper into some of these topics and write a book on Process Engineering and Design. They have done it with success. I had the opportunity of working with Mr. Bhatt. He is one of the best process engineers I have come across. The long industrial experience of Mr. Bhatt and extensive teaching experience of Prof. Thakore have made them eminently suitable to offer their knowledge to student fraternity and practicing engineers. I have no doubt that the book will find its place with most academicians involved in teaching the subject.

The book describes systematic methods for the process design with many illustrations, drawn from various sections of the industry. They have included several solved and unsolved problems with sufficient details. Authors' approach is user friendly and result oriented. In-depth study of these examples will boost confidence of the students in solving real life design and operating problems.

Large number of references, cited at the end of each chapter, indicates the extensive literature study made by the authors. Further, study of these references will provide more insight to process design aspects.

An exclusive chapter is devoted on development of Process Flow Diagrams (PFD) and Piping and Instrumentation Diagrams (P&ID) which is justified by my earlier comments. Sufficient emphasis is placed on 'green' engineering. These aspects have made this book unique.

Many recent topics like pinch technology, energy conservation in distillation, two-phase flow of fluids, design of loop reactors and many others are to be appreciated which are hard to find in other books on process design.

I have no doubt that the book will make the library of a student and practicing engineer richer.

Suhas M Mokashi
Ex-Director of Technology & Marketing
Kaevernar Powergas Pvt. Ltd.

has helped in deciding the appropriate coverage in the book for the course syllabus. Prior to his teaching experience, he worked as a process engineer in industries for five years. Thereafter he worked as a consultant for process design of various chemical equipment. Few problems covered in the book are derived from his actual assignments.

Bharat Bhatt spent more than 15 years in the Process Engineering Section of a large chemical plant. He also carried out the design of many chemical plant equipments, which were successfully commissioned. The present work reflects the experience gained by him during his tenure of service.

Features

- Only a limited number of unit operations are covered to highlight the process design aspects. In each of these operations, current developments are included which provide energy efficient, more reliable and safer designs. Networking of heat exchangers (pinch technology), two-phase fluid flow system, solids cooling, supercritical extraction, separation of azeotropic mixtures, energy conservation in distillation, design of jet reactors, handling of liquids saturated with gases and many other topics are covered in the book which would be of interest of the readers in process engineering and design.
- Use of correct property values in process design plays an important role. Property data are extracted from various literature sources which should help readers think of the various sources that are available for the specific data.
- Syllabus of many universities were gathered and a sincere attempt is made to cover most (if not all) topics of these syllabii.
- A number of ready to use software packages are available in the market. However, we believe that basic approach to process design should be clear to the chemical engineers. This knowledge should help the engineers to utilize the software in an efficient manner. Use of mathematical software (such as Mathcad[®], Maple[®], etc.) and spreadsheet software such as (Excel[®]) is made in solving many problems. Preparation of process diagrams with Autocad[®] or Visio[®] is encouraged to enhance the skills and speedy execution of the project.
- Every effort has been made to make the approach to the subject student friendly. Presentation is made in a lucid, logical and step-by-step manner. Writing style is kept relatively simple for easy understanding. Industrial examples are cited for each concept/principle so that the student can appreciate the concept.
- A large number of solved problems are drawn from the industry to make the book practical. The worked examples are regarded as part of the text and are clearly explained without expecting the student to fill in missing steps. They will give students a good understanding of the design steps and the confidence to solve problems themselves in real life.

- A large number of exercise problems are given at the end of each chapter. Design problems as well as problems with varying degree of difficulty are included.
- The approach adopted in the book will enable a student to progress from theory to practice smoothly. At the same time a practicing process design engineer may like to use the book as a ready reference.

Who will Benefit from this Book?

The text is an introduction to the vast subject of process design. It is intended to provide course material for a two-semester course in process design for undergraduate students of chemical engineering. It can be also used for the students pursuing higher studies for M. Tech. in Process Engineering and Design. It can serve as a reference text for other subjects like advanced separation techniques, advanced heat and mass transfer, etc.

The main prerequisites for the course using the text are chemistry, stoichiometry and unit operations. Although familiarity with computer and various software packages will be helpful, the user friendly methodology adopted in the book does not make knowledge of these mandatory.

Acknowledgements

We are greatly honoured by the Foreword written by Mr. S. M. Mokashi who is a well known process design expert. His industrial and consulting experience spans over 40 years. He has handled many large industrial projects. We are extremely thankful for his encouragement in our scholastic work.

We have been rendered help by many of our colleagues who were also a source of inspiration. We thank all of them for their indirect contributions. We are grateful to the Principal, Vishwakarma Government Engineering College, Gandhinagar, Gujarat for granting us the permission to publish this book. We are also thankful to the reviewers who read the original manuscript and offered valuable comments/suggestions. We would like to express our appreciation for loving support we had received from our wives; Dimple Thakore and Minaxi Bhatt.

We want to thank many at Tata McGraw-Hill for their overwhelming support. In particular, we wish to place on record our thanks to Ms Vibha Mahajan, Ms Shukti Mukherjee, Ms Mini Narayanan and Ms Anjali Razdan for their support and encouragement.

Suggestions and Comments

Suggestions and comments of the readers are welcome to improve the utility of the book in future. We can be reached through the publisher or directly at sbthakore@yahoo.co.in for Shuchen Thakore and b_bhatt26@hotmail.com for Bharat Bhatt.

**SHUCHEN B. THAKORE
BHARAT I. BHATT**

Chapter

1



Dimensions and Units

I.1 INTRODUCTION

Knowledge of dimensions and units is imperative in day to day dealings. While dimensions are recognized by our sensory perceptions, their use requires definite scales of measurement, known as units. When a solid is observed, one thinks about its volumetric and mass dimensions. Similarly a liquid is recognized by its volume and density measurements. A gas exerts definite pressure and temperature which are important for defining its state.

Various quantities, required to identify a substance and its state can be classified in two categories; namely fundamental and derived. Every quantity can be expressed as a product of a dimensionless number and a unit where the unit is a selected reference quantity in terms of which all quantities of the same kind can be expressed.

I.2 FUNDAMENTAL QUANTITIES OR DIMENSIONS

Fundamental quantities or dimensions are length, mass, time, thermodynamic temperature, electric current, amount of substance and luminous intensity. Units, representing these quantities are known as base units.

There are basically two systems of units fps and metric. The former is widely used in the USA and is also known as US Customary Units. It is based on foot, pound and second as standard measurements of length, mass and time, respectively.

Metric (mks) system is based on the unit of length, known as metre. The unit of mass and time in this system are kilogram and second. Its important feature is the decimal expression.

International system of units (or Le Système International d'Unités in French or simply SI Units) was adopted by the 11th General Conference of Weights and Measures in 1960. Base units of the mks system for length, mass and time are retained in the SI system. Base units of the other four fundamental quantities are defined in the SI system. Table 1.1 lists the fundamental quantities in different units.

The thermodynamic temperature (Kelvin), defined in SI, is accepted as the absolute temperature in the metric system. For definitions of all the fundamental quantities, references 1 to 4 may be consulted.

Table 1.1 Fundamental Quantities

Symbol	Fundamental quantity	System of units with symbolic abbreviation			Dimension
		SI	Metric	fps	
<i>l</i>	Length	Metre (m)	Metre (m)	Foot (ft)	L
<i>m</i>	Mass	Kilogram (kg)	Kilogram (kg)	Pound (lb)	M
<i>θ</i>	Time	Second (s)	Second (s)	Second (s)	θ
<i>I</i>	Electric current	Ampere (A)	Ampere (A)	Ampere (A)	I
<i>T, t or T'</i>	Temperature	Kelvin (K)	Celsius (°C)	Fahrenheit (°F)	T
<i>n</i>	Amount of substance	Mole (mol)			n
<i>I_v</i>	Luminous intensity	Candela (cd)			

In 1971 the concept of the amount of a substance was introduced as a fundamental quantity. It is defined as the amount of substance of system containing as many elementary entities as the number of atoms in 0.012 kilogram of carbon-12 element. This unit of quantity was called a *mole* (abbreviated as mol). Thus unified scale of mole gives value of the relative atomic mass (*m*). When a compound is represented by a chemical formula, then the total mass computed based on the formula is called **molar mass** (popularly known as molecular weight) and is represented by symbol *M*.

1.3 DERIVED UNITS

Derived units (including supplementary units) are expressed algebraically and in terms of base units by means of mathematical signs of multiplication and division. For example, the SI unit of work or energy is Joule which is defined as work done when one Newton (N) force acting on a body moves it by a distance of one metre (see Sec. 1.3.4).

Table 1.2 lists some of the important SI derived units used in Chemical Engineering with their special names while Table 1.3 lists frequently used derived units.

Table 1.2 SI Derived Units with Special Names, Including SI Supplementary Units

SI Derived Unit (Derived Quantity)	Special Name	Symbol	Expressed in Terms of SI Base Units and SI Derived Units
Plane angle	radian	α, β, γ	1 rad = 1 m/m = 1
Solid angle	steradian	Ω, w	1 sr = 1 m ² /m ² = 1
Frequency	hertz	f	1 Hz = 1 s ⁻¹
Force	newton	F	1 N = 1 kg · m/s ²
Pressure or stress	pascal	p, ρ	1 Pa = 1 N/m ²
Energy or work or Quantity of heat	joule	E, H	1 J = 1 N · m
Power	watt	P	1 W = 1 J/s
Radiant flux			
Electric charge or Quantity of electricity	coulomb	Q	1 C = 1 A · s

(Contd.)

Table 1.2 (Contd.)

SI Derived Unit (Derived Quantity)	Special Name	Symbol	Expressed in Terms of SI Base Units and SI Derived Units
Electric potential or Potential difference, or Tension or Electromotive force	volt	V	1 V = 1 W/A
Electric resistance	ohm	R	1 Ω = 1 V/A
Electric conductance	siemens	G = I/R	1 S = 1 Ω ⁻¹
Celsius temperature	degree Celsius ¹	t	1°C = 1 K

¹Degree Celsius is a special name for the unit Kelvin for use in stating value of Celsius temperature. $t/^\circ\text{C} = T/\text{K} - 273.15$.

Table 1.3 Additional Frequently Used SI Derived Units

Quantity	Symbol	SI Unit	Practical Units	Notes
Acceleration	a	m/s ²	m/s ² , cm/s ²	
Acceleration of free fall	g	m/s ²	m/s ²	
Isentropic exponent	κ			$\kappa = C_p/C_v$ (for ideal gas)
Ambient Pressure	p _{amb}	N/m ² , Pa	mbar, bar	
Angular acceleration	α	rad/s ²	rad/s ²	
Angular velocity	ω	rad/s	rad/s	
Area	A, A _s , S, a	m ²	m ² , cm ²	
Concentration of substance B	C _B	mol/m ³	mol/m ³ , mol/L	
Current density,	j	A/m ²	A/m ² , A/cm ²	
Density (mass concentration)	ρ	kg/m ³	kg/m ³ , g/cm ³ , kg/L	
Relative density / Specific gravity	d	ρ _{t₁} /ρ _{t₂}	—	Non-dimensional quantity
Diameter	d, D	m	cm, mm	
Diffusion coefficient	D	m ² /s	m ² /s, cm ² /s	
Efficiency	η	—	—	Non-dimensional quantity
Heat (enthalpy) of evaporation	λ	J/g, J/mol	kJ/kg, kJ/kmol	
Molar flow rate/ Volumetric flow rate	q _v	mol/s, m ³ /h	mol/s, m ³ /h, m ³ /s	
Gas constant (molar)	R	J/(mol · K)	$\frac{\text{mbar} \cdot \text{L}}{(\text{mol} \cdot \text{K})}$, (m ³ · bar)/(kmol · K)	

(Contd.)

Table 1.3 (Contd.)

Quantity	Symbol	SI Unit	Practical Units	Notes
Gas constant of substance B (specific)	R_B	J/(kg · K)	$\frac{\text{mbar} \cdot \text{L}}{(\text{kg} \cdot \text{K})}$, $(\text{m}^3 \cdot \text{bar})/(\text{kg} \cdot \text{K})$	
Heat capacity of a given mass	C	J/K	J/K, kJ/K	
Heat capacity, isobaric, molar	C_{mp}	J/(mol · K)	kJ/(kmol · K)	
Heat capacity, isobaric, specific	C_p, C_L	J/(kg · K)	J/(kg · K)	
Heat capacity, isochoric: molar	C_{mv}	J/(mol · K)	kJ/(kmol · K)	
Heat flow rate or heat flux	ϕ	W, J/s	kW, kJ/s, kJ/h	
Heat transfer coefficient	h	W/(K · m ²)	W/(K · m ²), kcal/(h · m ² · °C)	
Height	h	m	m, cm, mm	
Kinematic viscosity	γ	m ² /s	m ² /s, mm ² /s, cm ² /s, cSt	
Kinetic energy	E_k	J	J	
Leak rate (in a vacuum system)	q_L	N · m/s	mbar · L/s	
Length	L	m	m, cm, mm	
Liner expansion coefficient	α_l	m/(m · K),	m/(m · K), 1/K	
Mass flow rate	q_m, \dot{m}	kg/s	kg/s, kg/h, g/s	
Mass fraction	w_g, χ, \mathcal{Y}	kg/kg	%	ppm = parts per mega parts
Molality	m	mol/kg	mol/kg	—
Molarity	\mathcal{M}	mol/dm ³	mol/m ³ , mol/L	—
Molar mass	M	kg/kmol	kg/kmol, g/mol	
Molar volume	V_m	m ³ /mol	m ³ /mol, L/mol	
Number of revolutions	n	1/s	1/s, 1/min (or rpm)	
Partial pressure of component B	p_B	N/m ² , Pa	mbar, kPa, torr	
Period (periodic time)	θ	s	s, ms, μs	
Permeability (for gases)	μ_o	$\frac{\text{Nm}^3}{(\text{m}^2 \cdot \text{s} \cdot \text{Pa})}$	$\frac{\text{Nm}^3}{(\text{m}^2 \cdot \text{d} \cdot \text{bar})}$	
Pumping speed (rate)/volumetric flow rate	q_v, \dot{V}	m ³ /s	L/s, m ³ /h	
Quantity of gas	V	Nm ³	L	
Quantity of heat	Q, H, i	J	J, kJ, kWh, W · s	

(Contd.)

Table 1.3 (Contd.)

Quantity	Symbol	SI Unit	Practical Units	Notes
Radius (also of molecule)	r	m	cm, m, μm	
Vapour pressure	p_v	N/m ² , Pa	mbar, kPa, Pa, torr	
Saturation vapour pressure	p_s	N/m ² , Pa	mbar, kPa, Pa, torr	
Temperature difference	$\Delta T, \Delta t$	K	K, °C	
Thermal conductivity	k	W/(K · m)	W/(K · m), kW/(m · K), kcal/(h · m · °C)	
Thermal diffusivity	a	m ² /s	m ² /s	
Velocity	v	m/s	m/s, mm/s, km/h	
Viscosity (dynamic)	μ	Pa · s	mPa · s, cP	
Volume	V	m ³	m ³ , L, cm ³	
Volume (specific)	v	m ³ /kg	m ³ /kg, cm ³ /g	
Volume fraction of substance B	ϕ_B	m ³ /m ³	L/L, %, ppm	ppm = parts per mega parts
Wavelength	λ	m	nm, Å	
Weight (force)	G	N	kgf	
Specific/absolute humidity	H	kg /kg	kg/kg, g/g	
Relative humidity	RH	—	—	Non-dimensional quantity - do -
Saturation ratio	φ	—	—	

It will now be appropriate to elaborate a few of the important SI derived units for better understanding.

1.3.1 Force

According to the Newton's second law of motion,

$$F \propto m \times a \quad (1.1)$$

$$\text{or} \quad F = K m a \quad (1.2)$$

where, K = proportionality constant

Selecting K as unity, the unit newton is defined. Thus, when a force of one newton (N) is applied to a body having a mass of one kilogram it gives an acceleration of one m/s².

Definition of technical unit of force is obtained by selecting K equivalent to $1/g_c$ where g_c is called Newton's law conversion factor. Its value is 9.806 65 (kg · m)/(kgf · s²) and is equal to the acceleration due to gravity (g) at mean sea level. It may be noted that g_c is not equal g at all locations on the earth.

Kilogram-force (kgf) is the force which when applied to a body having a mass of one kilogram, gives it an acceleration of 9.806 65 m/s². It is the base unit in metric system of units.

$$1 \text{ N} = 0.101 972 \text{ kgf}$$

$$\text{or} \quad 1 \text{ kgf} = 9.806 65 \text{ N}$$

Although mass is the base unit, it nearly equals weight under gravitational acceleration (g). This is why ‘mass’ and ‘weight’ terms are commonly used without differentiation in general practice.

1.3.2 Pressure

Pressure is the force acting on a unit area, exposed to it.

$$p = \frac{F}{A} \quad (1.3)$$

Common units of pressure in SI and metric systems are N/m^2 (known as Pascal; symbol Pa) and kgf/cm^2 , respectively. Pressure, measured with the help of a gauge, is called gauge pressure or over pressure and the letter ‘g’ follows the unit. When (local) atmospheric pressure is added to the gauge pressure, it is called absolute pressure and the letter ‘a’ follows the unit. Standard atmospheric pressure (symbol atm) is measured at mean sea level and is assigned the value 101 325 N/m^2 or Pa or 1.033 kgf/cm^2 . Since Pascal is a very small unit of pressure, bar is chosen as a practical pressure unit in the SI system.

$$1 \text{ bar} = 10^5 \text{ Pa} = 1.019\,716 \text{ kgf/cm}^2 = 0.986\,923 \text{ atm}$$

$$1 \text{ atm} = 1.013\,25 \text{ bar}$$

Pressure is also expressed in terms of fluid heads; e.g. water column (WC), liquid column (LC), mercury column (Hg), etc.

$$\text{Fluid head} = \text{Pressure/density} = p/\rho \quad (1.4)$$

$$1 \text{ atm} = 760 \text{ mm Hg at } 0^\circ\text{C} \text{ or torr} = 10.33 \text{ m WC at } 4^\circ\text{C}$$

Vacuum refers to pressure below atmospheric pressure and hence absolute pressure of a vacuum system is derived by subtracting vacuum from atmospheric pressure.

1.3.3 Volume

Volume is the space occupied by a substance. It is measured in cubic metres and litres in SI and metric units, respectively. Approximately, $1 \text{ m}^3 = 1000 \text{ L} = 1 \text{ kL}$.

1.3.4 Work, Energy, Power and Heat

Work is defined as the product of force acting on a body and the distance travelled by the body.

$$W = F \times d \quad (1.5)$$

Units of work ($\text{kg} \cdot \text{m}^2/\text{s}^2$ or N · m or joule (symbol J) and $\text{m} \cdot \text{kgf}$ in SI and metric systems, respectively.

Internal energy (Symbol U or E) is a physical entity which is present in a system in different forms; mechanical, chemical, thermal, etc. Heat is the form of energy in transit that flows from higher temperature to lower temperature. Total **Enthalpy** (symbol H) of a substance is the total energy possessed by it under the given conditions.

$$H = U + pV \quad (1.6)$$

Unit of energy, heat and enthalpy are joule and calorie (symbol cal) in SI and metric systems, respectively.

$$1 \text{ calorie (International Steam Tables)} = 4.1868 \text{ J}$$

It may be noted that unit of work, energy, heat and enthalpy in SI unit is same, i.e. W (unlike metric system).

Power P is defined as the work done per unit time

$$P = \frac{W}{\theta} \quad (1.7)$$

Unit of power is Watt (i.e. J/s) and $\text{m} \cdot \text{kgf}/\text{s}$ in SI and metric systems, respectively. Practical unit of power in metric system is horsepower.

$$1 \text{ metric system horsepower} = 75 \text{ (m} \cdot \text{kgf})/\text{s} = 0.7355 \text{ kW}$$

1.3.5 Derived Electrical Units

Electric potential (V) is defined as

$$V = \frac{W}{I} \quad (1.8)$$

Its unit is volts.

Resistance of the conductor is defined as

$$R = \frac{V}{I} \quad (1.9)$$

Its unit is ohms Ω . Reciprocal of resistance is called conductance; symbol (G) and has units siemens (S). Quantity of electricity (symbol Q) is defined as the product of current and time.

$$Q = I \cdot \theta \quad (1.10)$$

Unit of quantity of electricity is coulomb (C).

$$1 \text{ Faraday (F)} = 96\,485.309 \text{ C/mol}$$

1.4 RECOMMENDED RULES FOR WRITING UNIT SYMBOLS

Following guidelines may be followed while using SI unit symbols.

- (i) SI prefixes are given in Table 1.4.
- (ii) An exponent attached to compound prefix-unit implies that the exponent refers to the entire compound unit and not just to the base symbol. 1 cm^3 means volume of a cube having one cm side.
- (iii) The product of two or more units may be indicated in any one of the following ways.
Correct: N m , or $\text{N} \cdot \text{m}$ Incorrect: Nm
- (iv) A solidus (oblique stroke), a horizontal line or negative powers may be used to express a derived unit, formed from two others by division, e.g.

$$\text{m/s}, \frac{\text{m}}{\text{s}}, \text{m} \cdot \text{s}^{-1} \text{ or } \text{m s}^{-1}$$

Table 1.4 SI Prefixes

Factor	Prefix	Symbol
10^{24}	yotta	Y
10^{21}	zetta	Z
10^{18}	exa	E
10^{15}	peta	P
10^{12}	tera	T
10^9	giga	G
10^6	mega	M
10^3	kilo	k
10^2	hecto	h
10^1	deca	da
10^{-1}	deci	d
10^{-2}	centi	c
10^{-3}	milli	m
10^{-6}	micro	μ
10^{-9}	nano	n
10^{-12}	pico	p
10^{-15}	femto	f
10^{-18}	atto	a
10^{-21}	zepto	z
10^{-24}	yocto	y

- (v) A solidus must not be repeated on the same line unless ambiguity is avoided by a parenthesis.

Correct Incorrect

m/s^2 or $m \cdot s^{-2}$ or $m\ s^{-2}$ $m/s/s$
 $W/(m^2 \cdot K)$ or $W\ m^{-2}\ K^{-1}$ $W/m^2 \cdot K$ or $W\ m^{-2}/K$

- (vi) Unit symbols do not change in the plural. For example, 5 metres should be abbreviated as 5 m and not as 5 ms.

- (vii) Unit symbols are not followed by a full stop (period) except at the end of a sentence.

Correct: 8 kg Incorrect: 8 kg.

- (viii) When numerical values fall outside the range of 0.1 to 1000, it is recommended that the numerals be separated into groups of three with a space replacing the traditional comma.

Recommended Not recommended

3 600 or 3600	3,600
19 625 725	19,625,725
0.001 625	0.001,625
0.046 89	0.046,89

In this book, four digits or decimals are grouped.

- (ix) Prefix symbols are printed without any space between the prefix symbol and the unit symbol.

Correct: 10 kW Incorrect: 10.5 k W

- (x) Compound prefixed formed by the juxtaposition of two or more SI prefixes are not to be used.
Correct: 6 GJ Incorrect: 6 MkJ
- (xi) A prefix should never be used alone.
Correct: $10^6/\text{m}^3$ Incorrect: M/m^3
- (xii) Although kilogram is the base unit in SI units, names of decimal multiples and sub-multiples of mass are formed by attaching prefixes to the word 'gram'.
Correct: 1 mg Incorrect: 1 μkg
- (xiii) Good practice recommends selection of a prefix which, whenever possible, provides a numerical value between 0.1 and 1000. Prefer expression 10.0 kPa over 0.01 MPa. However, when values are tabulated, they should be expressed in the same unit multiple even though their numerical value lies outside 0.1 to 1000 range.
 $12 \times 10^4 \text{ J}$ can be written as: 120 kJ.
1632 W can be written as 1.632 kW.
- (xiv) If the magnitude of the number is less than unity, the decimal sign should preferably be preceded by zero, e.g. .072 should be written as 0.072.
- (xv) The SI prefixed are not to be used with °C or K.

1.5 CONVERSION FACTORS

Appendix 1 lists the conversion factors in a direct usable form for conversion from one system of units to another system of unit.

REFERENCES

1. *The International System of Units (SI)*, National Institute of Standards and Technology (NIST) Publication No. 330, USA, 1991.
2. ISO 1000 : 1992/Amd. 1 : 1998 (E), *SI Units and Recomendations for the Use of their Multiples and Certain other Units (Amendment 1)*, International Organization for Standards, Switzerland.
3. Mills, I., T. Cvitas, K. Homann, N. Kallary and K. Kuchitsu, *Quantities, Units and Symbols in Physical Chemistry*, 2nd Ed., IUPAC Chemical Data Series, Blackwell Science Ltd., UK, 1993.
4. Bhatt, B. I. and S. M. Vora, *Stoichiometry*, 4th Ed., Tata McGraw-Hill Publishing Co. Ltd., New Delhi, 2004.

Chapter

2



Stoichiometry

2.1 INTRODUCTION

Stoichiometry is a branch of chemistry which deals with combining the proportions of the elements or compounds involved in chemical reactions. It is based on three concepts; conservation of mass, the relative masses of atoms and definition of a mole. While the last two concepts were introduced in Chapter 1, the law of conservation of mass is dealt in this chapter.

In any chemical reaction or in a unit operation, exchange of thermal energy also takes place. Therefore, heat balance is also important in stoichiometric calculations. Similar to mass balance, heat balance calculations are based on the law of conservation of energy (the first law of thermodynamics).

2.2 BASIC CHEMICAL CALCULATIONS

Before attempting mass and energy balance problems, knowledge of basic principles of chemical engineering calculations is necessary. A brief review of these calculations is presented as follows.

2.2.1 Atomic Mass and Molar Mass

Elements are basic entities which are combined (or reacted) to form a compound. As seen in Chapter 1, relative atomic masses for each element are presented on a unified scale which is known as Atomic Masses (Weights) of Elements¹.

For chemical compounds, a *mole* is defined as the amount of substance equal to its formula weight. Molar mass (M) is thus the total mass of elements, contained in the compound. Thus it is easy to calculate molar mass of a compound, knowing its chemical formula.

Example 2.1

Calculate molar mass of :

- (a) Sulphurous chloride (SOCl_2) and
- (b) 2,4,6 – trinitrophenol (also known as picric acid)

Solution:

(a) Molar mass of SOCl_2 = Atomic mass of sulphur + Atomic mass of oxygen + 2
 × Atomic mass of chlorine
 = $32.065 + 15.99\ 94 + (2 \times 35.453)$
 = 118.9704 (exact) kg/kmol

With rounded off values of atomic masses, molar mass of SOCl_2 = $32 + 16 + 2 \times 35.5 = 119$

(b) Chemical formula of picric acid = $\text{C}_6\text{H}_2(\text{NO}_2)_3\text{OH}$
 Molar mass of picric acid = $6 \times 12.0107 + 3 \times 1.007\ 94 + 3 \times 14.0067 + 7 \times 15.9994$
 = $72.0642 + 3.023\ 82 + 42.0201 + 111.9958$
 = 229.103 92 (exact) kg/kmol
 ≈ 229 (rounded off value)

In this book, rounded off values of atomic masses will be used for convenience.

2.2.2 Mole

In Chapter 1, mole is defined as a base unit in SI. It can be further defined as the amount of substance containing $6.022\ 1367 \times 10^{23}$ molecules (also known as Avogadro's number, N_A). In simple terms, number of moles can be found by dividing mass of the compound by its molar mass.

Example 2.2

Convert following into moles.

- (a) 1000 kg boron trichloride (BCl_3)
- (b) 1500 kg phenol ($\text{C}_6\text{H}_5\text{OH}$)

Solution:

(a) Molar mass of BCl_3 = $11 + 3 \times 35.5 = 117.5$ kg/kmol

$$1000 \text{ kg } \text{BCl}_3 \equiv \frac{1000}{117.5} = 8.5106 \text{ kmol}$$

(b) Molar mass of $\text{C}_6\text{H}_5\text{OH}$ = $6 \times 12 + 6 \times 1 + 16 = 94$ kg/kmol

$$1500 \text{ kg } \text{C}_6\text{H}_5\text{OH} \equiv \frac{1500}{94} = 15.9574 \text{ kmol}$$

2.2.3 Mass Fraction and Mole Fraction

In a mixture, mass fractions (w) or mole fractions (x, y) are expressed to represent its composition. Mass fraction is the ratio of the mass of a particular substance divided by total mass of the substance. Similarly, mole fraction is the ratio of the moles of a particular substance divided by total moles of all the substances. One can be calculated knowing the other.

Example 2.3

Aqueous calcium chloride brine contains 35% CaCl_2 by mass. Convert the same in mole %.

Solution:

Basis : 100 kg brine

Table 2.1 Composition of Brine

Component	Mass kg	Molar mass kg/kmol	Moles kmol	Mole % (x_i)
CaCl ₂	35	111	0.3153	8.03
H ₂ O	65	18	3.6111	91.97
Total	100		3.9264	100.00

$$\text{Average molar mass } (M) \text{ of the solution} = \frac{100}{3.9264} = 25.47 \text{ kg/kmol}$$

Example 2.4

Feed gas to ammonia synthesis loop has molar composition 24.75% N₂, 74.25% H₂, 0.7% CH₄ and 0.3% Ar. Calculate its mass composition, average molar mass and specific gravity (with respect to air).

Solution:

Basis : 100 kmol feed gas

Table 2.2 Composition of Feed Gas to Ammonia Synthesis Loop

Component	kmol	Molar mass kg/kmol	Mass kg	Mass %
N ₂	24.75	28	693.0	80.14
H ₂	74.25	2	148.5	17.17
CH ₄	0.70	16	11.2	1.30
Ar	0.30	40	12.0	1.39
Total	100.00		864.7	100.00

$$\text{Average molar mass } (M) \text{ of feed gas} = \frac{864.7}{100} = 8.647 \text{ kg/kmol}$$

Similarly, average molar mass of air can be calculated to be 28.968 (rounded off value to be 29).

$$\text{Specific gravity of feed gas} = \frac{8.647}{28.968} = 0.2985$$

While mass fractions are useful in establishing material balances, mole fractions are logical expressions in chemical reactions.

2.2.4 Density and Specific Gravity

Density of a compound in any state (solid, liquid or gas) is expressed as the ratio of mass per unit volume. It is normally expressed in g/cm³, kg/L or kg/m³.

Density of iron is 7.75 g/cm^3 at 20°C while that of 24% BaCl_2 solution at 40°C is 1.243 g/cm^3 .

Specific gravity of a liquid or solid is a dimensionless number and it is defined as the ratio of density of solid or liquid at a given temperature to the density of water at 4°C (which is nearly equal to 1.000 g/cm^3). For liquids, specific gravities are often expressed in terms of certain hydrometers. Among these, ${}^\circ\text{Twaddell}$ (${}^\circ\text{Tw}$) and ${}^\circ\text{Baumé}$ (${}^\circ\text{Be'}$) scales are well known. In petroleum industry, hydrometer scale called ${}^\circ\text{API}$ is used which is very useful in finding certain properties like molar mass, viscosity, heating value, etc. Specific gravity of 24% aqueous BaCl_2 solution at 40°C is $48.6 {}^\circ\text{Tw}$. Strength of certain important inorganic solutions such as that of caustic soda solutions, sulphuric acid solutions, etc. are tabulated on ${}^\circ\text{Tw}$ or ${}^\circ\text{Be}$ scale as hydrometers are convenient for use in industry.

Specific gravity of a gas is the parameter which indicates the heaviness of a gas as compared to that of air. One definition is the ratio of molar mass of gas to the molar mass of air. In Example 2.4, specific gravity of feed gas to ammonia synthesis loop is determined to be 0.2985, indicating that it is much lighter than air (both at same operating conditions).

Another definition of specific gravity of a gas is the ratio of the density of the gas at given conditions of pressure and temperature to the density of air at reference conditions. Although this is a more logical expression, molar mass ratio is chosen in this book to express heaviness of a gas as compared to the air.

2.2.5 Vapour Pressure and Boiling Point of Liquid

When a liquid is heated, it gets converted to vapour at a given temperature and pressure. For a pure liquid, since degree of freedom is one, it boils at a constant temperature for a given pressure. Conversely, for a pure liquid at a given temperature, its equilibrium pressure is fixed which is known as vapour pressure. This implies that when both phases exist in equilibrium, there is only one definite pressure (called vapour pressure) at a given temperature for a single component.

Boiling point of a pure liquid at standard atmospheric pressure (1 atm or 101.325 kPa) is called normal boiling point (NBP). At 1 atm, water boils at 100°C , ethyl alcohol boils at 78.4°C , methylene chloride boils at 40.1°C and refrigerant 134a (tetrafluoroethane) boils at -26.1°C . All are called normal boiling points.

Relationship of vapour pressure with its saturation temperature is expressed either empirically or in tabulated form. Variety of equations are known for calculating vapour pressure of a liquid. Among these, Antoine equation is well known and is expressed as follows:

$$\log p_v = A - \frac{B}{(T + C)} \quad (2.1)$$

where p_v is vapour pressure at temperature T (in K) while A , B and C are constants. Ref. 1 lists values of A , B and C for several liquids. Validity of equations for a given liquid is limited in a definite temperature range. Number of other equations are reported in literature for accurate prediction of vapour pressure.

Some of the fluids are extensively used in industry over a wide range of pressure and temperature. Their applications could vary from heating, cooling or processing. Steam and certain vapour phase heating fluids are used for heating. Similarly a variety of refrigerants are used for cooling. *n*-Hexane is used for extraction of oil from seeds. Nitrogen, oxygen etc. are also used as feed in various chemical reactions. Carbon dioxide, water, propane and butanes are used in their supercritical states for extraction and for carrying out certain chemical reactions. Thus, thermo-physical properties of all such fluids are required at different pressures and temperatures. For this reason, properties of these fluids are available in tabular form. These tables list temperature and its corresponding vapour pressure as one property. For example, steam tables (Ref. 2) are widely used in industry. Data, read from the table, permit quick calculations.

Several other properties of liquids and solutions are of importance in chemical engineering calculations. References 1 and 3 may be referred for the discussions on these properties.

2.2.6 Gases

Gaseous state of a pure component is defined by its pressure and temperature. However, when a mixture of gases is present, its composition is also required to be specified. In a conventional practice, mole % composition of a gaseous mixture is specified (as seen in Example 2.4).

Gases behave as ideal in a definite range of pressure and temperature such that they obey the well known ideal gas law.

$$pV = RT \quad (2.2)$$

where, p = absolute pressure of the gas, kPa

V = volume occupied by the gas, m³

R = Universal Gas Constant

= 8.314 51 J/(mol · K) or m³ · kPa/(kmol · K)

T = absolute temperature of the gas, K

When p is chosen as one standard atmosphere (101.325 kPa) and T is chosen as 273.15 K (i.e. 0°C), these are called normal conditions (i.e. NTP).

Molar volume at NTP of an ideal gas is 22.4136 m³/kmol.

Conversely an ideal gas is one which follows Eq. (2.2). Most gases under ordinary circumstances (such as ambient conditions) obey ideal gas law. However, real gases deviate significantly at high pressures and high temperatures. Real gas behaviours are described by equations of state (also called p - V - T relationship). Reference 4 may be referred for more information on various equations of state. p - V - T relations can be represented on phase diagrams. p - t diagrams for water and carbon dioxide are given in Fig. 7.22 and Fig. 7.23.

Critical conditions of a gas are those beyond which a gas cannot be liquefied. These conditions are defined by critical temperature, critical pressure and critical volume. By definition, critical temperature is the maximum temperature at which a gas can be liquefied. The critical pressure is the saturation (vapour) pressure corresponding to the critical temperature. The volume, occupied by the gas at critical point is the critical volume. At the critical point, densities (or specific

volumes) of gas and liquid (in equilibrium) are equal. Reference 1 lists the critical properties of number of compounds.

Physical state of a fluid above its critical conditions is known as supercritical state. In Chapter 7, extraction in supercritical fluids is discussed. Table 7.7 lists the critical properties of commonly used supercritical fluids.

When a mixture of gases is handled, its composition is specified in mole %. For an ideal gas mixture,

$$\text{mole \%} = \text{volume \%} = \text{pressure \%}$$

Partial pressure (or pure component pressure) of a component in the gas mixture is defined as total pressure of the system multiplied by the mole fraction of the component.

$$p_i = p \cdot y_i \quad (2.3)$$

where, p_i = partial pressure, kPa

p = total pressure, kPa

y_i = mole fraction of the component in vapour phase

For an ideal liquid-vapour mixture, equilibrium is represented by Raoult's law,

$$p_i = p \cdot y_i = p_{vi} \cdot x_i \quad (2.4)$$

where, p_{vi} = vapour pressure of the component at a given temperature, kPa

x_i = mole fraction of the component in liquid phase

Conversely an ideal liquid mixture is one which follows Raoult's law. Non-ideal behaviour of the liquid mixture is expressed in different ways for which a standard text book on thermodynamics may be referred (Ref. 4).

For certain liquid mixtures at a definite composition, liquid and vapour compositions are same at a given pressure. Composition of such a mixture is known as azeotropic composition. Ethyl alcohol-water, toluene-water and many multicomponent mixtures exhibit this property which is advantageously used in the industry to remove water from organic reaction mass.

2.2.7 Material Balances

A process represents a series of actions or operations or treatments that result in an end product. In chemical engineering, physical and/or chemical changes take place during the operations and the material flows and its inventory in a system are quantified during these operations which is known as material balance.

A system can be defined as the body or assemblage in which the process is set out for analysis. When a system is isolated from the surroundings, it is called a batch system. In other words, there is no transfer of materials from a batch during the time interval of interest. Contrary to this, in open system, material is transferred across the boundaries; i.e. from and to the surroundings. Thus it is important to define the system being analyzed.

Having defined the system, it is important to fix a basis; e.g. 100 kg feed, 100 kg reactant A, 100 kmol gas mixture, 1000 L liquid, etc. All computations are performed considering this basis. In a prolonged analysis of the system, it may become necessary to shift bases but interrelation of all the bases should be clear.

It is thus clear that material balance is a quantitative expression of law of conservation of matter. It can be expressed as

$$\text{Input} = \text{Output} + \text{Accumulation} \quad (2.5)$$

Equation (2.5) is always true for elements but often not true for compounds participating in chemical reactions. Because of its fundamental nature, material balance is one of the most useful tools in system analysis and therefore in process design.

2.2.7.1 Material Balances Involving No Chemical Reactions

In most unit operations, reactions do not take place. Material balances of such systems are worked out by making balances of a tie material or inert material. When two or more compounds in the system are simultaneously affected, material balance equations are solved by satisfying simultaneous equations.

Example 2.5

Soyabean seeds are batch extracted with *n*-hexane. Flaked seeds containing 19% oil, 69% solids and 12% moisture (by mass) are fed to the batch extractor. At the end of the extraction process, deoiled cake (DOC) is found to contain 1% oil, 88% solids and rest moisture (by mass). Find the percentage recovery of oil.

Solution:

Basis : 100 kg soybean seeds

Since solids do not take part in the extraction process, its balance can be made.

Solids in the feed = $100 \times 0.69 = 69$ kg

DOC contains 88% solids.

$$\text{Quantity of DOC} = \frac{69}{0.88} = 78.41 \text{ kg}$$

$$\text{Oil in seeds} = 100 \times 0.19 = 19 \text{ kg}$$

$$\text{Oil in DOC} = 78.41 \times 0.01 = 0.784 \text{ kg}$$

$$\begin{aligned}\text{Oil recovery} &= \frac{(19 - 0.784)}{19} \times 100 \\ &= 95.87\%\end{aligned}$$

Example 2.6

n-Butanol – water forms an azeotrope having composition of 57.5% *n*-butanol (by mass) at 93°C and 1 atm. When azeotropic vapour is condensed, and cooled to 20°C, the mixture separates in two layers. Top layer and bottom layer consist of 79.93% and 7.81% *n*-butanol, respectively (by mass) at 20°C. Calculate the proportions of top and bottom layers.

Solution:

Basis : 100 kg azeotropic vapour to condenser.

n-Butanol in vapours = 57.5 kg

Water in vapours = $100 - 57.5 = 42.5$ kg

In the liquid phase, the mixture separates in two layers. Since both components are affected, simultaneous equations need to be solved.

Let m_1 kg be top layer and m_2 kg be the bottom layer.

Overall balance : $m_1 + m_2 = 100$

n-butanol balance : $0.7993 m_1 + 0.0781 m_2 = 57.5$

Solving the equations, $m_1 = 68.9 \text{ kg}$, $m_2 = 31.1 \text{ kg}$

Solving simultaneous equations for a binary system could be easily done manually. However, when many components are present, use of a mathematical software such as Mathcad® will be advantageous.

2.2.7.2 Material Balances Involving Chemical Reactions

Material balance calculations of processes involving chemical reactions follow the same law of conservation of mass. However, moles are important rather than mass in chemical reactions. For material balance calculations, conversion and yield are required.

Conversion is defined as the fraction of the reactant in the feed which is converted to product(s). Conversion is normally defined on the basis of the limiting reactant which is present in the smallest stoichiometric amount. Yield is defined by the following equation.

$$\text{Yield} = \frac{\text{moles of desired product} \times \text{stoichiometric factor}}{\text{moles of specific reactant consumed}} \quad (2.6)$$

where, stoichiometric factor = stoichiometric requirement of the specific reactant (in moles) per mole of the desired product

Selectivity is the ratio of amount of limiting reactant that reacts to give the desired product to the amount that reacts to give undesirable product(s).

One more term used in reaction engineering is the excess of reactant which is defined as the reactant present in excess of stoichiometric requirement of the limiting reactant on mole basis.

While conversion is related to the degree of completion of reaction, yield and selectivity are related to the degree to which the desired reaction proceeds.

Study of oxidation reaction to produce nitrogen monoxide can be useful to understand various terms.

Following are the chief competing reactions taking place in the reactor in a nitric acid plant



Above reactions are carried out over a precious metal catalyst at high temperature. Aim is to maximize production of NO. Ammonia and oxygen (in the form of air) are the reactants. At the exit of the reactor, ammonia is completely consumed and hence it is the limiting reactant. Oxygen is fed in excess over the stoichiometric requirement (5/4 times ammonia feed rate) and is termed as the excess reactant. In this case conversion is 100% based on ammonia. Stoichiometric factor for reaction (1) is one, i.e. 1 mole NO is formed per mole of ammonia. Typical yield of NO is found to be 0.93 – 0.98 depending on operating temperature and catalyst activity. Take for example, yields of NO, N₂ and N₂O as 0.95, 0.02 and 0.03, respectively.

NH_3 consumed for desired product = 0.95 kmol

$$\begin{aligned}\text{NH}_3 \text{ consumed for undesired products} &= 0.02 \times 2 + 0.03 \times 2 \\ &= 0.1 \text{ kmol}\end{aligned}$$

$$\text{Selectivity of the catalyst} = 0.95/0.1 = 9.5$$

Combustion of a fuel is another oxidation reaction in which oxygen (i.e. air) is fed in excess to achieve complete combustion. Fuel is thus the limiting reactant. Oxygen is supplied in excess over the stoichiometric requirement in the form of air. Knowing the flue gas and fuel analyses, it is possible to calculate the excess air.

Example 2.7

Flue gas mixture from a waste incinerator is analysed and found to contain 12% CO_2 , 5.5% O_2 and rest N_2 (by mole) on dry basis. Assuming complete combustion, calculate the excess air and C:H mass ratio in fuel.

Solution:

Basis : 100 kmol dry flue gases

Table 2.3 Flue Gas Analysis on Dry Basis

Component	kmol	O_2 accounted kmol
CO_2	12	12
O_2	5.5	5.5
N_2	82.5	—
Total	100.0	17.5

Assume negligible nitrogen in the fuel (i.e. waste).

$$\text{O}_2 \text{ supplied in the form of air} = \frac{21}{79} \times 82.5 = 21.93 \text{ kmol}$$

$$\begin{aligned}\text{O}_2 \text{ consumed in reaction with hydrogen} &= 21.93 - 17.5 \\ &= 4.43 \text{ kmol}\end{aligned}$$

$$\begin{aligned}\text{Hydrogen reacted} &= 4.43 \times 2 \\ &= 8.86 \text{ kmol}\end{aligned}$$

$$\text{Carbon in waste} = 12 \times 12 = 144 \text{ kg}$$

$$\text{Hydrogen in waste} = 8.86 \times 2 = 17.72 \text{ kg}$$

$$\begin{aligned}\text{C:H ratio in waste} &= \frac{144}{17.72} \\ &= 8.13:1 \text{ by mass}\end{aligned}$$

$$\begin{aligned}\text{Total oxygen consumed for combustion} &= 12 + 4.43 \\ &= 16.43 \text{ kmol}\end{aligned}$$

$$\begin{aligned}\text{Excess oxygen or air} &= (5.5/16.43) 100 \\ &= 33.48\%\end{aligned}$$

2.2.7.3 Recycling and Bypassing Operations

Recycle stream is a process stream from exit or downstream from a unit back to the inlet or upstream of the same unit. Nature is a classical example in which nitrogen and oxygen are recycled. Animals inhale oxygen and exhale carbon

dioxide. The latter is used by plants for manufacture of food in presence of sun light. In this photosynthesis reaction, oxygen is released. In unit operations and in chemical reactions, recycling operations are performed for a variety of reasons. One of the chief reasons of recycling is to maximize the utilization of reactant(s) in reaction. Hydrogen is used in excess in many reactions. After the reaction, excess hydrogen is recycled from the separation unit back to the reaction unit. For recovery of volatile organic compounds (VOC) from vent gases, carbon adsorption units are commonly employed. Nitrogen is used for regeneration of the carbon bed from which VOC is separated. VOC laden nitrogen is recycled.

Other reasons for recycle are utilization of heat from the outgoing stream, to improve the performance of an equipment, to improve selectivity of a product, to improve safety of the process, to minimize waste generation, etc.

Chemical engineers are familiar with reflux in a distillation column. It is basically a recycle stream to maintain specific quality of the top product. In Chapter 8, number of examples are given for calculating reflux ratio which represent material balance in an unit operation.

In a loop reactor fraction of product stream (after cooling) is recycled back to the reactor to control the exothermicity of reaction.

2.3 ENERGY BALANCES

It is known that most physical and chemical changes are accompanied by enthalpy changes. It is therefore necessary to study the enthalpy balances.

Energy exists in different forms such as potential energy, kinetic energy, thermal energy, electrical energy, etc. In SI units, the unit of all types of energy is Joule (represented as J). Conversion of one form of energy into another is feasible. However, total energy of the universe is constant.

In this section, discussions will be limited to thermal energy and the terms energy, enthalpy and heat are essentially used for thermal energy. While enthalpy of a substance is associated with its state (i.e. pressure and temperature), the term heat is used for the enthalpy in transit. In physical and chemical changes, flow rate of heat (in W in SI units) is of interest to process engineers.

2.3.1 Sensible Heat Changes

Sensible heat changes in solid, liquid or gas means change in temperature of the substance. Increase in temperature is termed as heating while a decrease is termed as cooling. For evaluation of the heat change, mass, temperatures and heat capacity are required.

For any substance, heat capacity is defined as the heat required to effect the temperature change by 1°C of 1 kg substance. Thus, units of heat capacity are $\text{kJ}/(\text{kg} \cdot {}^{\circ}\text{C})$. If the heat capacity of one mole substance is specified, it is known as molar heat capacity and its units are $\text{kJ}/(\text{kmol} \cdot {}^{\circ}\text{C})$.

For gases, heat capacity depends on the route, followed for changing the temperature. One route is known as constant pressure path while another route is constant volume path. Majority of the processes follow constant pressure route and the heat capacity for the route is denoted by $C_{mp,i}$ and has units $\text{kJ}/(\text{kmol} \cdot {}^{\circ}\text{C})$.

This quantity is dependent on pressure and temperature. At 1 atm pressure, it is denoted by symbol C_{mp}° . A number of equations are developed, offering a relationship of C_{mp}° with temperature at 101.325 kPa. Among these, polynomial forms are used extensively.

$$C_{mp}^\circ = a + bT + cT^2 \quad (2.7)$$

$$\text{or} \quad C_{mp}^\circ = a + bT + cT^2 + dT^3 \quad (2.8)$$

Where a, b, c and d are constants (separate values for both equations) and T is temperature in K.

Values of C_{mp}° , derived from the equations, are accurate at 1 atm but they can be safely used for sub-atmospheric pressure and upto about 10 bar a. Correlations, graph and tables are available in literature for prediction of C_{mp} at high pressures (Ref. 1, 3, 4).

For mixture of ideal gases, sensible heat changes are calculated by calculating heat capacity of the mixture, using the following equation.

$$C_{mp\ mix}^\circ = \sum y_i \cdot C_{mpi}^\circ$$

where, $C_{mp\ mix}^\circ$ = heat capacity of gas mixture, kJ/(kmol · °C)

y_i = mole fraction of i th component

C_{mpi}° = heat capacity of i th component of gas mixture, kJ/(kmol · °C)

This equation is strictly valid for an ideal gas mixture but can be used for most practical purpose upto moderate pressures.

In case of liquids, heat capacities at constant pressure and at constant volume are nearly equal. Also variation of the heat capacity with pressure is insignificant for most liquids. Symbol for heat capacity of liquid is C_l . Polynomial equations are also developed for correlating C_l with T . For aqueous solutions, liquid mixtures, petroleum fluids, etc. graphical and tabulated data are available in literature. Heat capacity of an immiscible liquid mixture is given by

$$C_{l\ mix} = \sum x_i \cdot C_{li} \quad (2.9)$$

where, $C_{l\ mix}$ = heat capacity of liquid mixture, kJ/(kmol · °C)

x_i = mass fraction of i th component

C_{li} = heat capacity of i th component of liquid mixture, kJ/(kmol · °C)

Heat capacity of solids (C_s) are lower than that of liquids and increase with the increase in the temperature. However, variation of C_s with respect to T is generally insignificant.

2.3.2 Heat Changes During Phase Change

A study of $p-t$ diagram (Fig. 7.22 and Fig. 7.23) reveals interesting facts about phase changes. Above triple point, there are two phase change. When a solid is melted above its freezing point, latent enthalpy (or heat) of fusion is to be supplied. Water melts at 0.01°C at standard atmospheric pressure (101.325 kPa). Latent enthalpy of fusion at this temperature is 339.4 kJ/kg. In general, melting point of a pure fluid increases with increase in pressure while latent enthalpy of fusion decreases.

When heat is supplied to convert liquid to vapour at a constant temperature, it is called that latent enthalpy (heat) of vapourization. Water evaporates at 100°C

at 101.325 kPa when heat equivalent to 2256.9 kJ/kg is supplied. Saturation temperature of a pure fluid increases with increase in pressure while latent enthalpy of vaporization decreases and becomes zero at critical point.

Below triple point, a fluid sublimes from solid to vapour. Heat, supplied to change the solid phase to vapour phase of a pure substance, is called the latent enthalpy (heat) of sublimation. Sublimation temperature and latent enthalpy of sublimation decreases with decrease in pressure below triple point. Carbon dioxide sublimes at -78.46°C at 101.325 kPa and latent heat of sublimation is 571.0 kJ/kg. At triple point, latent heat of sublimation can be considered a total of latent heat of fusion and latent heat of vaporization. Triple point of carbon dioxide is -56.56°C at 5.18 bar. At this temperature, latent heat of fusion is 197.22 kJ/kg and latent heat of vaporization is 350.38 kJ/kg. Thus latent heat of sublimation at this temperature is 547.60 kJ/kg.

Above critical point, again sublimation of solid to vapour is experienced as there is no liquid phase. Critical temperature and critical pressure of water are 374.15°C and 221.2 bar, respectively. Water sublimes at -5°C at 600 bar and latent heat of sublimation is 319.92 kJ/kg at this temperature.

Various correlations are available in literature for prediction of latent heat of fusion and vaporization. However, latent heat of vaporization is most useful to a process engineer. Tables of thermodynamic properties (Ref. 2) are available in literature for large number of compounds. At standard atmospheric pressure, following equation is found to give latent heat of vaporization within 5% accuracy.

$$\frac{\lambda}{RT_B} = \frac{1.092 (\ln p_c - 5.6182)}{0.930 - T_{Br}} \quad (2.10)$$

where, λ = latent heat of vaporization at T_B , kJ/kmol

p_c = critical pressure, kPa

T_B = normal boiling point, K

T_{Br} = reduced temperature at $T_B = T_B/T_C$

$R = 8.314\ 51\ \text{kJ}/(\text{kmol} \cdot \text{K})$

Knowing latent heat of vaporization at one temperature, it can be calculated at another temperature, using the following Watson equation.

$$\frac{\lambda_1}{\lambda_2} = \left[\frac{T_c - T_1}{T_c - T_2} \right]^n \quad (2.11)$$

where, λ_1 and λ_2 refer to latent heat of vaporization at temperatures T_1 and T_2 (in K) respectively, T_c is critical temperature (in K) and n is characteristic constant, having a value of 0.38. Watson equation is simple and reliable in most cases.

For heat changes calculations during phase change, latent heat of appropriate phase change is to be taken into account.

2.4 ENTHALPY CHANGES ACCOMPANYING CHEMICAL REACTIONS

When any chemical reaction takes place, enthalpy changes are encountered. This is known as heat of reaction. For combustion reactions, it is termed as heat of

combustion. For calculation of heat of reaction, heat of formation data are required.

Standard heat of formation is defined as the isothermal enthalpy change in a synthesis reaction from the elements in their standard states. Standard heat of formation, denoted by ΔH_f° of large number of compounds, is available in literature¹ in tabular form. Standard states of compounds are pure crystalline solid at 25°C (298.15 K) and for pure liquid and pure gas are 1 bar a and 25°C (298.15 K). Since combustion reactions are easy for experiments, standard heat of combustion (ΔH_c°) data so obtained are used to calculate the standard heat of formation.

Standard heat of formation data are obtained for isothermal conditions. It varies with the temperature. Empirical relation of ΔH_f° with T is a polynominal equation, similar to that for heat capacity of gas [Eq. (2.7) or Eq. (2.8)].

Consider a chemical reaction between A and B to produce C and D . Stoichiometric relation can be written as

$$n_A + n_B = n_C + n_D$$

where, n_i = number of moles of i th component. Standard heat of reaction (ΔH_R°) for the above reaction can be calculated by the following equation.

$$\Delta H_R^\circ = \sum (n_i \cdot \Delta H_{fi}^\circ)_{\text{Products}} - \sum (n_i \cdot \Delta H_{fi}^\circ)_{\text{Reactants}}$$

where, ΔH_{fi}° = Standard heat of formation of i th component

Since ΔH_{fi}° will be at 25°C, ΔH_R° will be also at 25°C. However, if ΔH_{fi}° at some another temperature is taken (but all at one specific temperature), ΔH_R° will correspond to that temperature.

If heat of reaction is positive, the reaction is said to be endothermic while if the heat of reaction is negative, the reaction is termed exothermic.

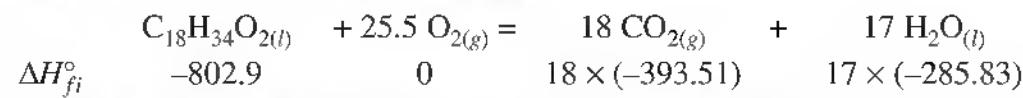
Example 2.8

Standard heat of formation of liquid oleic acid ($C_{18}H_{34}O_2$) is -802.9 kJ/mol at 25°C. Calculate its gross calorific value at 25°C.

Solution:

Basis : 1 mole liquid oleic acid

Gross calorific value or higher heating value is the heat liberated when a mole of the combustible compound is burnt with stoichiometric amount of pure oxygen at 25°C, product carbon dioxide is in gaseous form while product water is in liquid form. Combustion reaction of oleic acid can be written as



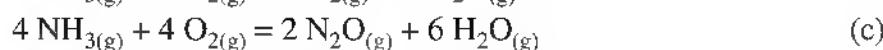
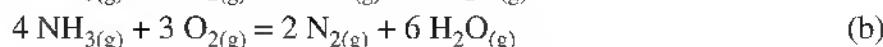
Standard heat of formation data for CO_2 and H_2O are taken from Ref. 1.

$$\begin{aligned} \Delta H_R^\circ &= 18(-393.51) + 17(-285.83) - (-802.9) \\ &= -11139.4 \text{ kJ/mol of liquid oleic acid} \end{aligned}$$

Literature reports ΔH_R° at 20°C for liquid oleic acid as -11154 kJ/mol.

Example 2.9

In the manufacture of nitric acid, ammonia is oxidized to nitric oxide on a Pt/Rh gauge (as catalyst) as per the following reactions.



Liquid ammonia at atmospheric pressure is pumped to 9.0 bar a. It is first vaporized with cooling water and subsequently superheated to desired temperature using low pressure saturated steam at 5 bar a. Oxygen in the form of air is supplied. Air is compressed to 9.3 bar a and 130°C in a two-stage compressor, driven by a steam driven turbine. Further it is heated to 260°C with the reactor exit stream. Superheated ammonia at the rate of 3266 Nm³/h and hot air at the rate of 29 394 Nm³/h are fed to the reactor. Ambient air has 0.016 kg moisture/kg dry air. In the reactor, ammonia is completely consumed. Yields of NO, N₂ and N₂O are 95%, 2% and 3%, based on ammonia conversion. Reactor exit gas stream temperature is observed to be 915°C at 7.5 bar g. Carry out the material and energy balances of the reactor system and preheating equipments.

Solution:

Basis :

$$\text{Ammonia flow rate to reactor, } q_{am} = 3266 \text{ Nm}^3/\text{h}$$

$$\begin{aligned} \text{Molar flow rate of ammonia, } q_{am} &= 3266/22.4136 \\ &= 145.71 \text{ kmol/h} \end{aligned}$$

$$\text{Volumetric flow rate of moist air, } q_a = 29 394 \text{ Nm}^3/\text{h}$$

$$\begin{aligned} \text{Molar flow rate of moist air, } q_a &= 29 394/22.4136 \\ &= 1311.44 \text{ kmol/h} \end{aligned}$$

Assume dry air molar composition to be 21% O₂, 1% Ar and 78% N₂.

$$\begin{aligned} \text{Average molar mass of dry air} &= 0.21 \times 31.9988 + 0.01 \times 39.948 + 0.78 \times 28.0134 \\ &= 28.9697 \end{aligned}$$

$$\begin{aligned} \text{Moisture in dry air} &= 0.016 \text{ kg/kg dry air} \\ &= 0.016 \times 28.9697/18.0153 \\ &= 0.0257 \text{ kmol/kmol dry air} \end{aligned}$$

$$\text{Moist air} = 1.0257 \text{ kmol/kmol dry air}$$

$$\begin{aligned} \text{Moisture, entering with air} &= 1311.44 \times 0.0257/1.0257 \\ &= 32.86 \text{ kmol/h} \end{aligned}$$

$$\begin{aligned} \text{Dry air flow rate to reactor} &= 1311.44 - 32.86 \\ &= 1278.58 \text{ kmol/h} \end{aligned}$$

$$\text{O}_2 \text{ supply to reactor} = 1278.58 \times 0.21 = 268.50 \text{ kmol/h}$$

$$\text{Argon supply to reactor} = 1278.58 \times 0.01 = 12.79 \text{ kmol/h}$$

$$\begin{aligned} \text{N}_2 \text{ supply to reactor} &= 1278.58 - 268.50 - 12.79 \\ &= 997.29 \text{ kmol/h} \end{aligned}$$

Table 2.4 Thermodynamic Data¹

Component	State	Molar mass kg/kmol	ΔH_f° kJ/mol	Heat Capacity Equation Constants			
				a	$b \times 10^3$	$c \times 10^6$	$d \times 10^9$
NH ₃	gas	17.0305	-45.94	25.6503	33.4806	0.3518	-3.0832
O ₂	gas	31.9988	0	26.0257	11.7551	-2.3426	-0.5623
N ₂	gas	28.0134	0	29.5909	-5.141	13.1829	-4.968
Ar	gas	39.948	0	20.7723	—	—	—

(Contd.)

Table 2.4 (Contd.)

Component	State	Molar mass kg/kmol	ΔH_f° kJ/mol	Heat Capacity Equation Constants			
				a	$b \times 10^3$	$c \times 10^6$	$d \times 10^9$
NO	gas	30.0061	90.25	29.4867	-2.0524	11.3379	-4.8195
N ₂ O	gas	44.0128	82.05	23.2082	63.6695	-42.3402	10.4061
NO ₂	gas	46.0055	33.18	23.5804	53.5944	-31.4901	6.5394
H ₂ O	gas	18.0153	-241.82	32.4921	0.0796	13.2107	-4.5474
H ₂ O	liquid	18.0153	-285.83				
HNO ₃	liquid	63.0128	-174.10				

Source of data: Ref. 1

Conversion = 100%

Yield of NO = 95%

NH₃ converted to NO = 145.71×0.95

$$= 138.42 \text{ kmol/h}$$

NO produced

$$= 138.42 \text{ kmol/h}$$

NH₃ converted N₂

$$= 145.71 \times 0.02$$

$$= 2.91 \text{ kmol/h}$$

N₂ produced

$$= 2.91 \times 2/4$$

$$= 1.457 \text{ kmol/h}$$

NH₃ converted to N₂O

$$= 145.71 - 138.42 - 2.91$$

$$= 4.38 \text{ kmol/h}$$

N₂O produced

$$= 4.38 \times 2/4$$

$$= 2.19 \text{ kmol/h}$$

Total O₂ consumed

$$= 138.42 (5/4) + 2.91 (3/4) + 4.38 (4/4)$$

$$= 173.025 + 2.183 + 4.38$$

$$= 179.588 \text{ kmol/h}$$

O₂ in reactor exit stream

$$= 268.50 - 179.588$$

$$= 88.912 \text{ kmol/h}$$

Total H₂O produced

$$= 145.71 \times 6/4$$

$$= 218.565 \text{ kmol/h}$$

Total H₂O in reactor exit stream

$$= 218.565 + 32.86$$

$$= 251.425 \text{ kmol/h}$$

Total N₂ in reactor exit stream

$$= 1.457 + 997.29$$

$$= 998.747 \text{ kmol/h}$$

Table 2.5 Reactor Ingoing Stream

Component	kmol/h n_i	Mole % (wet)	kg/h \dot{m}_i	Heat Capacity Equation Constants			
				$n_i \cdot a_i$ $\times 10^3$	$n_i \cdot b_i$ $\times 10^3$	$n_i \cdot c_i$ $\times 10^6$	$n_i \cdot d_i$ $\times 10^9$
NH ₃	145.71	10.00	2481.51	3737.5	4878.5	51.3	-449.3
N ₂	997.29	68.44	27 937.75	29 510.7	-5127.1	13 147.2	-4954.5
O ₂	268.50	18.43	8591.68	6987.9	3156.2	-629.0	-151.0
Ar	12.79	0.87	510.93	265.7	-	-	-
H ₂ O	32.86	2.26	591.98	1067.7	2.6	434.1	-149.4
Total	1457.15	100.00	40 113.85	41 569.5	2910.2	13 003.6	-5704.2

Average molar mass of reactor ingoing stream
 $= 40\ 113.85/1457.15 = 27.53 \text{ kg/kmol}$

Table 2.6 Reactor Exit Stream

Component	kmol/h n_i	Mole % (wet)	kg/h \dot{m}_i	Heat Capacity Equation Constants			
				$n_i \cdot a_i$ $\times 10^3$	$n_i \cdot b_i$ $\times 10^3$	$n_i \cdot c_i \times$ $\times 10^6$	$n_i \cdot d_i$ $\times 10^9$
N ₂	998.747	66.92	27 978.30	29 553.8	-5134.6	13 166.4	-4961.8
O ₂	88.912	5.96	2845.08	2314.0	1045.2	-208.3	-50.0
Ar	12.790	0.85	510.93	265.7	-	-	-
NO	138.420	9.27	4153.44	4081.5	-284.1	1569.4	-667.1
N ₂ O	2.190	0.15	96.39	50.8	139.4	-92.7	22.8
H ₂ O	251.425	16.85	4529.50	8169.3	20.0	3321.5	-1143.4
Total	1492.484	100.00	40 113.64	44 435.1	-4214.1	17 756.3	-6799.5

Average molar mass of reactor exit stream
 $= 40\ 113.64/1492.484 = 26.88 \text{ kg/kmol}$

It may be noted that mass flow rates of ingoing and outcoming streams are equal which should be checked during every material balance calculations.

Energy Balance:

For carrying out energy balance calculations, reference (or base) temperature need to be fixed. Since standard heat of formation data are based on 25°C, selection of 25°C as the reference temperature is logical.

$$T_0 = 25 + 273.15 = 298.15 \text{ K}$$

Reactor exit gas temperature is desired at 915°C.

$$T_s = 915 + 273.15 = 1188.15 \text{ K}$$

Heat flow rate of reactor exit stream

$$\phi_8 = \int_{298.15}^{1188.15} (44\ 435.1 - 4214.1 \times 10^{-3} T + 17\ 756.3 \times 10^{-6} T^2 - 6799.5 \times 10^{-9} T^3) dT$$

$$= 43\ 156\ 523 \text{ kJ/h} \equiv 11\ 987.92 \text{ kW}$$

Standard Heats of Reaction:

For reaction (a) :

$$\begin{aligned} \Delta H_{R_1}^\circ &= [4(90.25) + 6(-241.82)] - [4(-45.94) + 5(0)] \\ &= 361 - 1450.92 + 183.76 \\ &= -906.16 \text{ kJ/4 mol NH}_3 \text{ reacted} \\ &\equiv -226.54 \text{ kJ/mol NH}_3 \text{ reacted (exothermic)} \end{aligned}$$

For reaction (b) :

$$\begin{aligned} \Delta H_{R_2}^\circ &= [2(0) + 6(-241.82)] - [4(-45.94) + 3(0)] \\ &= -1450.92 + 183.76 \\ &= -1267.16 \text{ kJ/4 mol NH}_3 \text{ reacted} \\ &\equiv -316.79 \text{ kJ/mol NH}_3 \text{ reacted (exothermic)} \end{aligned}$$

For reaction (c) :

$$\begin{aligned} \Delta H_{R_3}^\circ &= [2(82.05) + 6(-241.82)] - [4(-45.94) + 4(0)] \\ &= 164.1 - 1450.92 + 183.76 \end{aligned}$$

$$\begin{aligned}
 &= -1103.06 \text{ kJ/4 mol NH}_3 \text{ reacted} \\
 &\equiv -275.765 \text{ kJ/mol NH}_3 \text{ reacted (exothermic)}
 \end{aligned}$$

Total heat liberated during reaction,

$$\begin{aligned}
 \phi_7 &= (226.54 \times 138.42 + 316.79 \times 2.91 + 275.765 \times 4.38) \times 1000 \\
 &= 33\,487\,377 \text{ kJ/h} \equiv 9302.05 \text{ kW}
 \end{aligned}$$

Heat input to reactor (i.e. for the reactants' stream)

$$\begin{aligned}
 \phi_6 &= 43\,156\,523 - 33\,487\,377 \\
 &= 9\,669\,146 \text{ kJ/h} \\
 &\equiv 2685.87 \text{ kW}
 \end{aligned}$$

Let temperature of reactants' stream be T_4 .

$$\begin{aligned}
 \phi_6 &= \int_{298.15}^{T_4} (41\,569.5 + 2910.2 \times 10^{-3} T + 13\,003.6 \times 10^{-6} T^2 - 5704.2 \times 10^{-9} T^3) dT \\
 &= 9\,669\,146 \text{ kJ/h} \equiv 2685.87 \text{ kW}
 \end{aligned}$$

Solving by trial and error or by Mathcad, $T_4 = 515.22 \text{ K}$ or 242.07°C

Among the two reactants, air is introduced at 260°C or 533.15 K (T_3).

Heat input of air,

$$\begin{aligned}
 \phi_5 &= \int_{298.15}^{533.15} (37\,832 - 1968.3 \times 10^{-3} T + 12\,952.3 \times 10^{-6} T^2 - 5254.9 \times 10^{-9} T^3) dT \\
 &= 91\,42\,364 \text{ kJ/h} \equiv 2539.55 \text{ kW}
 \end{aligned}$$

Heat input by superheated ammonia,

$$\begin{aligned}
 \phi_4 &= 9\,669\,146 - 9\,142\,364 \\
 &= 526\,782 \text{ kJ/h} \equiv 146.33 \text{ kW}
 \end{aligned}$$

If temperature of superheated ammonia is T_2 ,

$$\begin{aligned}
 \phi_4 &= \int_{298.15}^{T_2} (3737.5 + 4878.5 \times 10^{-3} T + 51.3 \times 10^{-6} T^2 - 449.3 \times 10^{-9} T^3) dT \\
 &= 526\,782
 \end{aligned}$$

Solving by Mathcad,

$$T_2 = 395.41 \text{ K} \text{ or } 122.26^\circ\text{C}.$$

Ammonia is fed at -33.33°C (saturation temperature at 101.325 kPa) as liquid. Assume that in the evaporator, liquid ammonia is vaporized and heated at 9.0 bar a to its saturation temperature of 21.5°C with cooling water.

$$T_1 = 21.5 + 273.15 = 294.65 \text{ K}$$

From ammonia tables (Ref. 2)

Enthalpy of liquid ammonia, $h = 49.08 \text{ kJ/kg}$

Enthalpy of vapour ammonia at 9 bar a and 21.5°C , $H_1 = 1480.78 \text{ kJ/kg}$

Note that the values of both the above enthalpies are based on reference temperature of 0°C in ammonia tables which is different from original reference temperature of 25°C . Nevertheless, since difference in only two enthalpy values is required, it does not matter.

Heat duty of ammonia evaporator,

$$\begin{aligned}
 \phi_1 &= 145.71 \times 17.0305 (1480.78 - 49.08) \\
 &= 3552\,784 \text{ kJ/h} \\
 &\equiv 986.88 \text{ kW}
 \end{aligned}$$

Cooling water at 32°C is used to evaporate liquid ammonia. Assume a drop in cooling water temperature by 5°C.

$$\text{Cooling water flow, } \dot{m}_{CW} = \frac{3552\ 784}{5 \times 4.1868}$$

$$= 169\ 714 \text{ kg/h}$$

$$\equiv 169.7 \text{ m}^3/\text{h}$$

Enthalpy of ammonia gas at 9.0 bar a and 122.26°C, $H_2 = 1740.15 \text{ kJ/kg}$

$$\text{Heat duty of superheater, } \phi_2 = 145.71 \times 17.0305 (1740.15 - 1480.18)$$

$$= 643\ 630 \text{ kJ/h}$$

$$\equiv 178.79 \text{ kW}$$

If heat capacity data (at 1 atm) for ammonia, are used for calculations of ϕ_2 ,

$$\phi_2 = \int_{294.65}^{395.41} (3737.5 + 4878.5 \times 10^{-3} T + 51.3 \times 10^{-6} T^2 - 449.3 \times 10^{-9} T^3) dT$$

$$= 544\ 914 \text{ kJ/h}$$

$$\equiv 151.37 \text{ kW}$$

$$\text{Error} = \frac{(643\ 630 - 544\ 914)}{643\ 630} \times 100$$

$$= 15.34\% - \text{significant}$$

Error indicates justification of tabular data, particularly at high pressures.

Saturated steam at 5 bar a is used for superheating ammonia vapours from 21.5°C to 122.26°C.

Latent heat of vaporization of steam at 5 bar a, $\lambda = 2107.4 \text{ kJ/kg}$ (Ref. 2)

Steam consumption in superheater,

$$\dot{m}_s = \frac{643\ 630}{2107.4}$$

$$= 305.4 \text{ kg/h}$$

Heat duty of air preheater,

$$\phi_3 = \int_{403.15}^{533.15} (37\ 832 - 1968.3 \times 10^{-3} T + 12\ 952.3 \times 10^{-6} T^2 - 5254.9 \times 10^{-9} T^3) dT$$

$$= 5\ 098\ 328 \text{ kJ/h} \equiv 1366.16 \text{ kW}$$

2.5 HEAT CHANGES IN MIXING PROCESSES

When a solid or gas is dissolved in a solvent, heat change takes place. This is known as heat of solution. Also when two liquids are mixed, a heat change takes place which is termed as heat of mixing. These heat changes are measured at constant temperature, say at 25°C (298.15 K) and at standard atmospheric pressure. Units of the heat changes are kJ/mol solute, kJ/kg solution, etc. depending on user's convenience.

When solids like caustic soda, caustic potash, sodium carbonate, etc. are dissolved in water, heat is evolved. Similarly when acids are mixed (a mixture of

$\text{HNO}_3 + \text{H}_2\text{SO}_4 + \text{H}_2\text{O}$), heat is released. Dissolution of ammonia or formaldehyde in water is exothermic. These heat changes should be considered while making heat balance calculations.

Heat of mixing data are extensively covered in many handbooks. Heat of formation data for large number of compounds and their solutions is published by National Institute of Standards and Technology, USA which permits heat of mixing calculations. Reference 1 may be consulted for more discussions on the subject.

Following example will illustrate the method of calculations relating to heat of mixing in gas–liquid system.

Example 2.10

Refer Example 2.9, reactor exit stream is cooled in various heat exchangers to 40°C and introduced in an absorber alongwith the condensate (formed in the coolers), operating at 1.5 bar g. Secondary air at the rate of 8145 Nm³/h is also introduced at the bottom of the absorber at 40°C which converts NO to NO₂. Fresh demineralized water is introduced from the top at 25°C which scrubs NO₂ to produce HNO₃. Absorption of NO₂ in water is exothermic and therefore cooling coils are provided in the absorber. Tail gas, leaving the absorber at 0.1 bar g and 50°C, is found to contain 0.2% NO (on dry basis). Aqueous 58% acid (by mass) leaves absorber at 50°C from the bottom. Make material and energy balance of the absorber. Find cooling water requirement for the absorption system assuming availability of cooling water at 32°C and a rise of 5°C.

Solution:

Basis : Same as that of Example 2.9

Pressure of gas at the inlet of absorber, = 1.5 bar g

$$= 251.325 \text{ kPa a}$$

Vapour pressure of water at 40°C = 7.375 kPa (Table 6.8 of Ref. 1)

$$\text{Moisture in ingoing gas mixture} = \frac{7.375(1492.484 - 251.425)}{(251.325 - 7.375)}$$

$$= 37.519 \text{ kmol/h at } 40^\circ\text{C}$$

$$\equiv 675.92 \text{ kg/h}$$

Liquid water, entering absorber = 251.425 – 37.519

$$= 213.906 \text{ kmol/h at } 40^\circ\text{C}$$

$$\equiv 3853.58 \text{ kg/h}$$

Pressure of outgoing mixture = 0.1 bar g

$$= 111.325 \text{ kPa a}$$

Vapour pressure of water at 50°C = 12.335 kPa (Table 6.8 of Ref. 1)

$$\text{Water content of tail gas} = \frac{12.335}{(111.325 - 12.335)}$$

$$= 0.1246 \frac{\text{kmol}}{\text{kmol dry gas}}$$

Secondary air flow = 8145 Nm³/h

Molar flow rate of wet air = 8145/22.4136

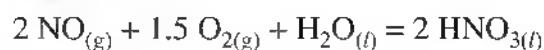
$$= 363.4 \text{ kmol/h}$$

Moisture, entering with air	$= \frac{363.4 \times 0.0257}{1.0257}$
	$= 9.11 \text{ kmol/h}$
Dry air flow rate	$= 363.4 - 9.11$
	$= 354.29 \text{ kmol/h}$
O ₂ entering the absorber	$= 354.29 \times 0.21$
	$= 74.40 \text{ kmol/h}$
Argon entering the absorber	$= 354.29 \times 0.01$
	$= 3.54 \text{ kmol/h}$
N ₂ entering the absorber	$= 354.29 - 74.40 - 3.54$
	$= 276.35 \text{ kmol/h}$

Table 2.7 Gas Stream Entering Absorber

Compo- nent	kmol/h <i>*n_i</i>	Mole % (wet)	kg/h <i>m_i</i>	Heat Capacity Equation Constants			
				<i>n_i · a_i</i>	<i>n_i · b_i × 10³</i>	<i>n_i · c_i × 10⁶</i>	<i>n_i · d_i × 10⁹</i>
N ₂	1275.097	77.66	35 719.80	37 731.3	-6555.3	16 809.5	-6334.7
O ₂	163.312	9.95	5225.79	4250.3	1919.7	-382.6	-91.8
Ar	16.330	0.99	652.35	339.2			
NO	138.420	8.43	4153.44	4081.5	-284.1	1569.4	-667.1
N ₂ O	2.190	0.13	96.39	50.8	139.4	-92.7	22.8
H ₂ O	46.629	2.84	840.04	1515.1	3.7	616.0	-212.0
Total	1641.978	100.00	46 687.81	47 968.2	-4776.6	18 519.61	-7282.8

Average molar mass of gas mixture, entering absorber = $\frac{46\,687.81}{1641.978} = 28.43$

Reaction:

Let x kmol/h NO is reacted by the above reaction.

$$\text{O}_2 \text{ consumed} = 1.5x/2$$

$$= 0.75x \text{ kmol/h}$$

$$\text{NO in tail gas} = 138.42 - x \text{ kmol/h}$$

$$\begin{aligned}\text{Dry tail gas} &= 1641.978 - x - 0.75x - 46.629 \\ &= 1595.349 - 1.5x \text{ kmol/h}\end{aligned}$$

Tail gas contains 0.2% NO.

$$\frac{(138.42 - x)100}{1595.349 - 1.5x} = 0.2$$

$$x = 135.63 \text{ kmol/h}$$

$$\begin{aligned}\text{Dry tail gas} &= 1595.349 - 1.5 \times 135.63 \\ &= 1391.904 \text{ kmol/h}\end{aligned}$$

$$\begin{aligned}\text{Moisture in tail gas} &= 0.1246 \times 1391.904 \\ &= 173.431 \text{ kmol/h}\end{aligned}$$

Table 2.8 Absorber Exit (Tail Gas) Stream

Compo- nent	kmol/h <i>n_i</i>	Mole % (dry)	kg/h <i>m_i</i>	Heat Capacity Equation Constants			
				<i>n_i · a_i</i>	<i>n_i · b_i × 10³</i>	<i>n_i · c_i × 10⁶</i>	<i>n_i · d_i × 10⁹</i>
N ₂	1275.097	93.90	35 719.80	37 731.3	-6555.3	16 809.5	-6334.7
O ₂	61.590	4.54	1970.81	1602.9	724.0	-144.3	-34.6
Ar	16.330	1.20	652.35	339.2	—	—	—
NO	2.790	0.20	83.72	82.3	-5.7	31.6	-13.4
N ₂ O	2.190	0.16	96.39	50.8	139.4	-92.7	22.8
H ₂ O	173.431	—	3124.41	5647.3	13.8	2296.1	-790.4
Total	1531.428	100.00	41 647.41	45 453.8	-5683.8	18 900.2	-7150.3

Average molar mass of exit gas stream = 41647.41/1531.428 = 27.20

Reference temperature, $T_0 = 298.15\text{ K}$

Heat input to absorber (of gas stream)

$$\phi_9 = \int_{298.15}^{313.15} (47 968.2 - 4776.6 \times 10^{-3} T + 18 519.6 \times 10^{-6} T^2 - 7282.8 \times 10^{-9} T^3) dT$$

$$= 720 459 \text{ kJ/h}$$

$$\equiv 200.13 \text{ kW}$$

Heat in tail gas stream

$$\phi_{10} = \int_{298.15}^{323.15} (45 453.8 - 5683.8 \times 10^{-3} T + 18 900.2 \times 10^{-6} T^2 - 7150.3 \times 10^{-9} T^3) dT$$

$$= 1132 458 \text{ kJ/h}$$

$$\equiv 314.57 \text{ kW}$$

Heat of reaction (Ref. 1):

$$\Delta H_R^\circ = 2 (-174.10) - [2 (90.25) + 1.5(0) + (-285.83)]$$

$$= -348.2 - 180.5 + 285.83$$

$$= -242.87 \text{ kJ/2 mol NO reacted}$$

$$\equiv -121.435 \text{ kJ/mol NO reacted (exothermic)}$$

Total heat generated during reaction:

$$\phi_{11} = 121.435 \times 1000 \times 135.63$$

$$= 16 470 229 \text{ kJ/h}$$

$$\equiv 4575.06 \text{ kW}$$

$$\begin{aligned} \text{Water evaporated in absorber} &= 173.805 - 46.629 \\ &= 127.176 \text{ kmol/h} \\ &\equiv 2291.1 \text{ kg/h} \end{aligned}$$

Assume average condensation temperature of 45°C.

λ at 45°C = 2394.9 kJ/kg (Ref. 2)

Heat absorbed during evaporation,

$$\begin{aligned} \phi_{12} &= 2291.1 \times 2394.9 \\ &= 5486 955 \text{ kJ/h} \equiv 1524.15 \text{ kW} \end{aligned}$$

Strength of aqueous acid at the bottom of the absorber = 58%

$$\begin{aligned} \text{Total HNO}_3 \text{ produced} &= 135.65 \text{ kmol/h} \\ &= 8547.69 \text{ kg/h (100%)} \end{aligned}$$

$$\text{Aqueous acid produced} = \frac{8547.69}{0.58}$$

$$= 14737.39 \text{ kg/h}$$

$$\begin{aligned}\text{Water in the acid} &= 14737.39 - 8547.69 \\ &= 6189.7 \text{ kg/h}\end{aligned}$$

Fresh demineralized water input

$$\begin{aligned}&= 6189.7 + (135.63/2) 18.0153 - (3853.58 + 840.04 - 3124.41) \\ &= 5842.2 \text{ kg/h} \\ &\equiv 5.84 \text{ m}^3/\text{h}\end{aligned}$$

Since demineralized water is added at 25°C, its heat input is zero.

$$\phi_{13} = 0$$

When HNO₃ dissolves in water, heat change takes place (Ref. 1).

$$\Delta H_f^\circ \text{ of 58% HNO}_3 \text{ solution} = -197.0 \text{ kJ/mol HNO}_3$$

$$\Delta H_f^\circ \text{ of 100% HNO}_{3(l)} = -174.2 \text{ kJ/mol HNO}_3$$

$$\text{Heat change} = -197.0 - (-174.2)$$

$$= -22.8 \text{ kJ/mol HNO}_3 \text{ (exothermic)}$$

Heat evolved by dissolution of HNO₃ in water at 25°C,

$$\begin{aligned}\phi_{14} &= 22.8 \times 1000 \times 135.63 \\ &= 3092\ 364 \text{ kJ/h} \\ &\equiv 858.99 \text{ kW}\end{aligned}$$

Heat capacity of 58% HNO₃ = 2.64 kJ/(kg · K) (Ref. 1)

Enthalpy of liquid 58% HNO₃ over 298.15 K

$$\begin{aligned}&= 2.64 (313.15 - 298.15) \\ &= 39.6 \text{ kJ/kg at 313.15 K}\end{aligned}$$

Heat of 58% acid at 40°C over 25°C

$$\begin{aligned}\phi_{15} &= 8547.69 \times 39.6 \\ &= 338\ 488 \text{ kJ/h} \\ &\equiv 94.02 \text{ kW}\end{aligned}$$

Heat of secondary air at 40°C

$$\begin{aligned}\phi_{16} &= \left(\frac{8145}{29\ 394} \right) \int_{298.15}^{313.15} (37\ 832 - 1968.3 \times 10^{-3} T + 129\ 523 \times 10^{-6} T^2 - 5254.9 \times 10^{-9} T^3) dT \\ &= \frac{8145 \times 574\ 358}{29\ 394} = 159\ 153 \text{ kJ/h} \\ &\equiv 44.21 \text{ kW}\end{aligned}$$

Enthalpy of condensate, entering absorber:

$$\begin{aligned}\phi_{17} &= 3853.58 (40 - 25) 4.1868 \\ &= 242\ 013 \text{ kJ/h} \\ &\equiv 67.23 \text{ kW}\end{aligned}$$

Heat to be removed by cooling water

$$\begin{aligned}&= \phi_9 + \phi_{11} + \phi_{13} + \phi_{14} + \phi_{16} + \phi_{17} - \phi_{10} - \phi_{12} - \phi_{15} \\ &= 200.13 + 4575.06 + 0 + 858.99 + 44.21 + 67.23 - 314.57 - 1524.15 - 94.02 \\ &= 3812.88 \text{ kW}\end{aligned}$$

Cooling water is supplied to internal coils at 32°C and leaves it at 37°C.

Required cooling water flow rate, \dot{m}_{CW}

$$\begin{aligned} &= \frac{3812.88 \times 3600}{5 \times 4.1868} \\ &= 655\,697 \text{ kg/h} \\ &\equiv 655.7 \text{ m}^3/\text{h} \end{aligned}$$

An example relating to absorption of formaldehyde in water to produce 37% aqueous solution is solved as Example 9.5 in Chapter 9. Both these examples refer to absorption with heat of absorption.

EXERCISES

- 2.1 A cylinder is filled with 6 Nm³ hydrogen. Its pressure and temperature are 150 bar g and 35°C. If the actual specific volume of hydrogen at these conditions is 0.1847 m³/kmol, what is the geometric capacity of the cylinder? Instead of using actual specific volume data, if ideal gas law is used for calculations, what will be the % error?
- 2.2 The chloride of some element contains 90.8% chlorine (by mass). What is the formula of the chloride compound ?
- 2.3 A mixture, amounting to 18.3 kg of a trivalent metal and its oxide, is dissolved in 32.0 kg sulphuric acid having 98% strength. The mixture reacts with stoichiometric amount of sulphuric acid and forms metal sulphate. There is no excess sulphuric acid leftover. Calculate the composition of the mixture.
- 2.4 Complete combustion of an alkane produces water vapour, equivalent to the oxygen consumed (on mole basis). Find the alkane.
- 2.5 Nitration product of a hydroxybenzene [C₆H_(6-a)(OH)_a] contains 49% oxygen by mass. Determine the structure of the compound.
- 2.6 Reduction of nitrobenzene is to be carried out in a batch reactor. At the end of the reaction, aniline is steam distilled and collected in a receiver at 40°C. Distilled mass is separated in two layers. Top layer is decanted in another vessel and measured to be 910.2 kg. Bottom layer is measured to be 1089.8 kg. It is known from the literature⁵ that top aniline rich layer contains 94.3% aniline while the bottom aqueous layer contains 3.83% aniline by mass at 40°C.
 - Calculate total aniline distilled.
 - Assume that all aniline, produced during reaction, is distilled out. If 1200 kg nitrobenzene was charged to the reactor and conversion is 100%, what is the yield of aniline?
- 2.7 Ethylene chlorohydrin and sodium bicarbonate are reacted in a continuous stirred tank reactor. Reaction takes place as given in the following equation at 110 kPa a and 82°C.

$$\begin{array}{ccc} \text{CH}_2\text{OH} & + & \text{NaHCO}_3 \\ | & & \\ \text{CH}_2\text{Cl} & & \end{array} = \begin{array}{c} \text{CH}_2\text{OH} \\ | \\ \text{CH}_2\text{OH} \end{array} + \text{NaCl} + \text{CO}_2$$

Aqueous solution of 30% of ethylene chlorohydrin and aqueous solution of 15% NaHCO₃ (both by mass) are fed to the reactor in equimolar proportion. Conversion of the reactants is 95% while the yield of ethylene glycol is 100%. Calculate the mass % and mole % compositions of the product stream.

Carbon dioxide, produced during the reaction, is vented out at 110 kPa a and 82°C. Calculate the concentrations of carbon dioxide, water vapour, ethylene chlorohydrine and ethylene glycol in the vent gas stream. Assume ideal behaviour of the liquid mixture.

Data :

- (a) Vapour pressures of water, ethylene chlorohydrine and ethylene glycol at 82°C are 51.329 kPa, 17.065 kPa and 0.79 kPa, respectively.
- (b) Lowering of vapour pressure of water due to the presence of inorganic salts ($\text{NaCl} + \text{NaHCO}_3$) having less than 10% concentration (by mass) in water at 82°C is 2.62 kPa.

- 2.8 Effluents generate at various locations in a composite textile unit. Reject water from reverse osmosis plant for production of water for boiler feed (BF) make-up purpose, boiler and cooling tower blowdowns, dye-house effluents, fabric washing liquor, etc. collect in a pit. Raw water from an underground source and combined effluent analyses are given in Table 2.9.

Table 2.9 Analysis of Raw Water and Combined Effluent

Parameter	Raw water	Combined Effluent
pH	7.8 – 7.9	7.5 – 7.9
Dissolved solids (DS), mg/L	1250	12 600
Total hardness as CaCO_3 , mg/L	230	500
Permanent hardness as CaCO_3 , mg/L	Nil	Nil
Calcium hardness as CaCO_3 , mg/L	170	375
Total alkalinity as CaCO_3 , mg/L	310	1190
Chlorides as Cl, mg/L	320	6260
Sulphates as SO_4 , mg/L	156	210
Silica as SiO_2 , mg/L (max.)	3	30
Suspended solids, mg/L (max.)	Nil	100
Chemical oxygen demand (COD), mg/L (max.)- dichromate value	Nil	800
Biological oxygen demand (BOD), mg/L (max.)	Nil	75
Oil and grease, mg/L (max.)	Nil	3
Colour	None	Dark

At first, the textile unit adopted primary and secondary treatments for removal of oil, grease, suspended matter (< 30 mg/L), COD (< 250 mg/L), BOD (< 30 mg/L) and colour. Effluents with nearly 13 000 mg/L DS after these treatments used to go to solar evaporation lagoons. Capacity of the plant was 100 m³/d.

Shortage of water of underground source over a period forced the textile unit to consider integrated additional treatments for recovery of water for reuse. The unit requires about 75 m³/d of raw water for BF Make-up purpose for passing through the reverse osmosis (RO) plant.

In the newly designed treatment facility (see Fig. 2.1) effluent from the existing treatment plant is first passed through an ultrafilter in which multicapillary hollow fibre membrane is used. This filter acts as a polishing filter in which suspended solids, organic impurities, colloidal silica, etc. are removed. Clear effluents with nearly same composition (in terms of inorganic compounds) and 12 250 mg/L DS

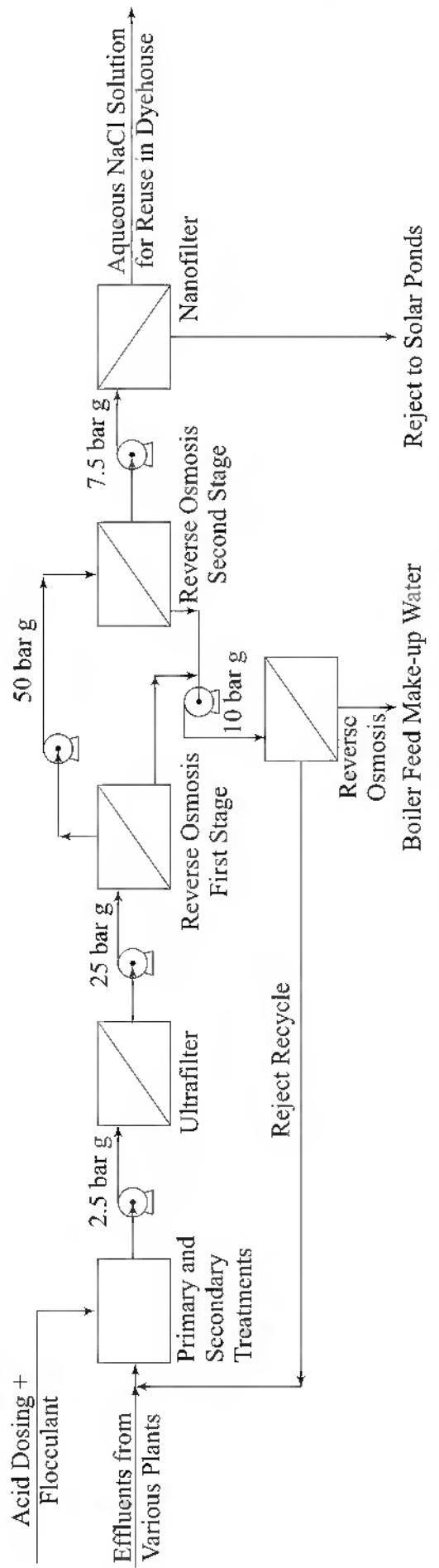


Fig. 2.1 Proposed Effluent Treatment Facility for a Composite Textile Unit

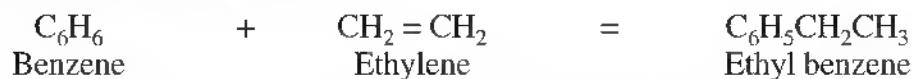
is passed through two stage RO units. In the first stage, spirally wound membranes for brackish water are housed. Effluents enter at 25 bar g to this unit. Recovery of 62% is achieved as permeate which contains 520 mg/L DS and 275 mg/L chlorides (as Cl). Reject from the first unit is pumped to 50 bar g and passed through the second stage of the RO unit. This unit houses spirally wound membranes, designed for high solids brackish water desalination. Recovery of 28% is achieved from the second stage as permeate which contains 960 mg/L DS and 486 mg/L Cl. Permeates from both the stages are collected, mixed and passed through the existing reverse osmosis plant at 10 bar g to produce BF make-up water.

Reject from the second stage RO plant is pumped again to 7.5 bar g and passed through a nanofilter. In this unit, special spiral wound membranes are housed which selectively removes chlorides in the permeate. Recovery from the nanofilter is 90% in which 95% of the chlorides, present in the feed, are dissolved. Reject from the nanofilter is sent to solar evaporation lagoons.

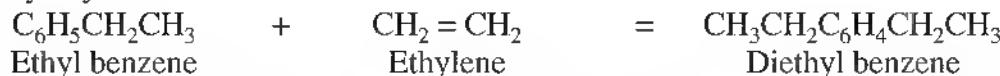
- Calculate the total permeates, available from two stages of RO system. Check whether this quantity is adequate for production of BF make-up water.
- Brine (i.e. permeate with high chlorides from the nanofilter) is reused for production of 12% NaCl solution for dyehouse. Calculate the flow rate and concentration of brine.
- Calculate the reduction in final effluent, discharged to solar evaporation lagoons.

Hint : Assume chlorides as NaCl at all points in the treatment plant.

- 2.9 Ethyl benzene is an important intermediate for the manufacture of styrene. This intermediate can be manufactured by reacting benzene with ethylene over a catalyst in vapour phase. Patented Mobil-Badger process uses Zeolite as catalyst. Chief reaction can be written as under.



Alkylation reaction does not stop at monoethyl benzene but a part of ethylbenzene is further alkylated to diethyl benzene, triethyl benzene, etc., collectively referred to as polyethyl benzenes.



A high benzene to ethylene ratio in the feed gives a low polyethyl benzenes to ethyl benzene ratio in the product stream. Excess benzene is recovered from the product gas stream and recycled to the feed.

While pure ethylene stream can be used as a feed, dilute gas stream, such as treated off gas mixture from Fluid Catalytic Cracking (FCC) unit of a petroleum refinery with low ethylene concentration, can also be used as the feed.

Figure 2.2 is a simplified flow diagram of the process. Fresh benzene is vaporized and mixed with recycled benzene vapours. Ethylene in the form of offgas mixture from a FCC unit of refinery, having molar composition of CH₄: 37%, C₂H₆: 19%, C₂H₄: 19%, H₂: 9%, N₂: 13% and CO: 3%, is compressed to 12 bar g and fed to the reactor after heating in a heater, using molten salt.

Reactor has three 3 catalyst beds and operates at 12 bar a. Offgases are heated and sent to first bed. An overall molar ratio of 10 : 1 of benzene to ethylene is used for the reaction. Of the total benzene, 40% quantity is fed to the first bed at 350°C. Assume 50% conversion of ethylene in the first bed with 90% yield to ethyl benzene. Offgas temperature is so controlled that the product gas mixture from the first bed is maintained at 420°C. A definite quantity of benzene at 300°C is mixed into

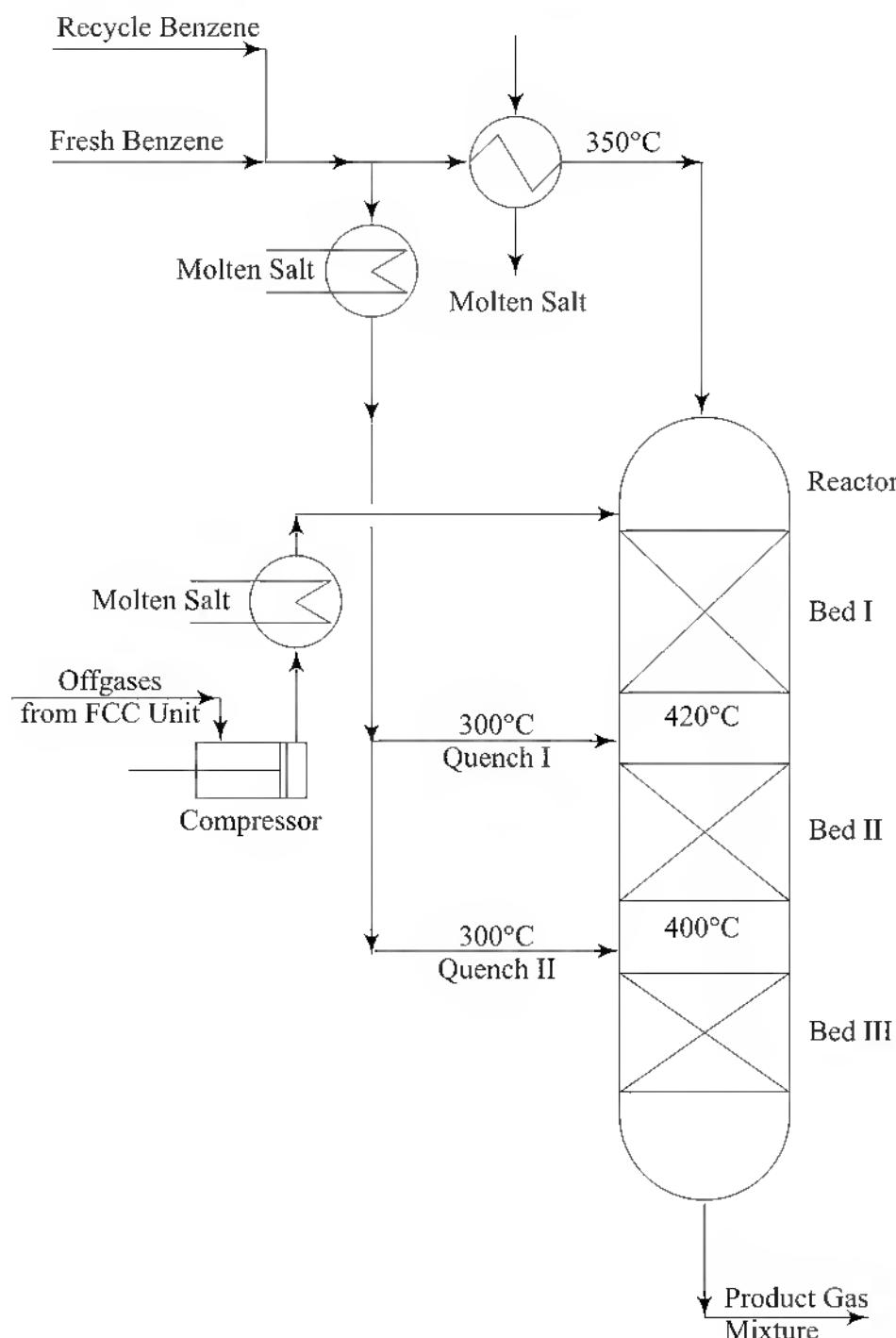


Fig. 2.2 Manufacture of Ethyl benzene

the production gas mixture from the first bed. In the second bed additional 25% conversion of ethylene (on total feed basis) is achieved with 90% yield to ethyl benzene. Temperature of product gas from the second bed is maintained at 400°C. The product gas mixture from the second bed is quenched with balance quantity of benzene at 300°C. In the third bed, rest 25% conversion takes place with 90% yield to ethyl benzene. All polybenzenes may be assumed as diethyl benzene for calculation purpose.

Table 2.10 Data on Gaseous Components

Component	Formula	ΔH_f° , (gas) kJ/mol at 1 bar & 25°C	Gaseous Heat Capacity Equation Constants at 1 bar			
			a	b $\times 10^3$	c $\times 10^6$	d $\times 10^9$
Benzene	C ₆ H ₆	82.93	-37.99	490.42	-321.39	79.36
Methane	CH ₄	-74.52	19.25	52.11	11.97	-11.32
Ethane	C ₂ H ₆	-83.82	5.41	178.09	-67.37	8.71
Ethylene	C ₂ H ₄	52.50	4.13	155.02	-81.55	16.98
Hydrogen	H ₂	0	28.61	1.02	-0.15	0.77
Nitrogen	N ₂	0	29.59	-5.14	13.18	-4.97
Carbon monoxide	CO	-110.53	29.12	0.25	-2.07	5.90
Ethyl benzene	C ₈ H ₁₀	29.92	-36.72	671.12	-422.02	101.15
Diethyl benzene	C ₁₀ H ₁₄	22.27	-37.41	867.09	-556.0	141.10

Source of data: Ref. 1

Calculate: (a) Required temperature of the offgas mixture, entering the first bed. (b) Benzene fed (in % of total) to the second bed as quench and (c) Temperature of product gas stream, leaving 3rd bed.

Note: Assume that enthalpy of formation and heat capacity data of 1 bar are valid at 12 bar a.

REFERENCES

1. Bhatt, B. I. and S. M. Vora, *Stoichiometry*, 4th Ed., Tata McGraw-Hill Publishing Co. Ltd., New Delhi, 2004.
2. Bhatt, B. I., *Design Databook; Properties of Steam, Selected Refrigerants, n-Hexane and Brines*, CBS Publishers & Distributors, New Delhi, 2006.
3. Himmelalue, D. M., *Basic Principles and Calculations in Chemical Engineering*, 5th Ed., Prentice-Hall of India Pvt. Ltd., New Delhi, 1994.
4. Smith, J. M., H. C. Van Ness, M. M. Abbott and B.I. Bhatt, *Introduction to Chemical Engineering Thermodynamics*, 6th Ed., Tata McGraw-Hill Publishing Co. Ltd., New Delhi, 2003.
5. *International Critical Tables*, Vol. 3, McGraw-Hill, USA, 1928, p. 389.
6. Chopey, N. P., *Handbook of Chemical Engineering Calculations*, 2nd Ed., McGraw-Hill, Inc., USA, 1994.
7. Kapoor, K. L., *Numerical Problems in Chemistry*, Tata McGraw-Hill Publishing Co. Ltd., New Delhi, 1991.
8. Reklaitis, G. V. and D. R. Schneider, *Introduction to Material and Energy Balances*, John Wiley & Sons, USA, 1983.
9. Hougen, R. A., K. M. Waston and R. A. Ragatz, *Chemical Process Principles*, Part-I, CBS Publishers & Distributors, New Delhi, 1995.
10. Fedler, R. M. and R. W. Rousseau, *Elementary Principles of Chemical Processes*, 2nd Ed., John Wiley & Sons, USA, 1986.

Chapter

3



Process Engineering

3.1 INTRODUCTION

A process is simply a method by which products can be manufactured from the raw materials. Selection of process steps for the manufacture of the product to the required specifications is the job of a process engineer. There could be number of methods to manufacture the same product. The best method for manufacturing a product has not yet been discovered nor can it be said that a job can be performed in only one way. The process engineer faces alternative solutions to manufacturing problems. This possibility comes out of a combination of factors such as information gained by him from the past processes, imagination, his own knowledge of known processes, etc. However, economy of manufacture is a factor that frequently decides the best route to be followed. Thus, it is safe to say that innovation, imagination and economy are the key factors in process engineering.

3.2 EXAMPLES OF ALTERNATIVE ROUTES IN PROCESS ENGINEERING

Process engineering and design can be explained best with the following illustrations.

1. Consider treatment of water for various end uses. End use of treated water is a chief governing factor in deciding the process steps to be selected. Production of drinking water demands different steps as compared to production of high quality water for boiler feed (BF) make-up purpose.

Drinking water requirements can be met by different water treatment steps. If water contains less dissolved solids, say less than 500 mg/L, clarification and disinfection by chlorination could be satisfactory. However, if raw water has high dissolved solids, dual filtration followed by desalination by reverse osmosis and pH adjustment would be necessary. These are relatively simple process steps.

In case of BF make-up water requirements for high pressure boilers, number of routes of the process are possible. Clarification, activated carbon treatment and demineralization by ion exchange is one route. However, this route may not be economical if raw water has high dissolved

solids (say more than 1500 mg/L). In the latter case pre-treatment, reverse osmosis treatment, followed by ion exchange treatment would become necessary. The final process route to be followed will depend on raw water quality, operating pressure of the boiler, effluent disposal problem, etc.

2. In another case, consider the classic example of production of anhydrous (100%) ethanol. Ethanol produced by fermentation of molasses can be first distilled to produce azeotropic mixture (containing 95.6% alcohol by mass) which can be further dehydrated to 100% by use of an entrainer such as benzene in azeotropic distillation. New development of pervaporation process can be used in place of azeotropic distillation. Similarly, drying of azeotropic mixture by using molecular sieve beds can be also employed. Innovations thus have a strong impact on process design alternatives.
3. Partially hydrogenated edible oils are extensively used for variety of applications. It is normally manufactured by hydrogenation of an edible oil in presence of a nickel catalyst under pressure (say at 2 bar g) and at 140 to 160°C in an autoclave. In a batch process, the reactor can have an anchor agitator with convolute at the top of the liquid level for proper mixing of hydrogen with the oil. This age old technology is replaced by installing a gas induced agitator (may be a tube type) which can result in a better product in a shorter period. This innovation has several advantages over the conventional design. Further research has demonstrated that use of carbon dioxide along with hydrogen at high pressure results in a product with stringent specifications (e.g. with less *trans* fatty acids).
4. Modern ammonia plants are large size plants (1800 t/d or more) which are continuous and reliable. These are quite compact than those originally developed to operate at very high pressures. Also, newer plants are safe and highly energy efficient proving the economy of the scale.

Several examples can be cited where technological developments have totally changed the manufacturing methods. This can be attributed to newer technologies, efficient equipment designs, use of alternate raw materials, etc. Process engineer's job is, therefore, to evaluate various alternatives available to him and decide over the best method to be adopted. Batch type hydrogenation plants are still preferred over continuous one due to many reasons such as relatively small capacities, different feedstocks for different batches, safety in handling, etc. Design of ammonia plants are currently more guided by available raw material and specific energy consumption. Thus it cannot be said that the best method for manufacturing a product can be performed in only one way. The possibility of alternate process operations comes as combination of information gained from the past, imagination and the process engineer's knowledge of the known processes.

3.3 GENERALIZED APPROACH TO THE CHEMICAL PLANT DESIGN

Design of chemical plant involves chemical technology, unit operations, instrumentation/controls, use of specialized equipments, safety considerations, materials of construction, etc. Chemical technology is the branch of chemical engineer-

ing dealing with chemical changes and physical methods involved in industrial process. It is necessary to supplement the knowledge of unit operations with considerations of chemical technology in order to obtain a concept of the complete plant.

From the above discussions, it is clear that before attempting process engineering design it is necessary to fix the product specifications. Capacity of the plant can then be decided based on demand and/or investment. Evaluation of available technologies will have a say on investment. In order to arrive at consumption figures of raw materials and energy, preliminary stoichiometric calculations would be necessary. This would give some rough idea of cost of production due to inputs which normally accounts for higher proportion in the cost.

Following sequence might be followed in implementation of the project right from conception of the idea to construction and operation of the plant.

- | | |
|--------------------------------------|-------------------------------|
| (i) Process Research | (iv) Process Development |
| (ii) Preliminary Process Design | (v) Final Process Design |
| (iii) Preliminary Process Evaluation | (vi) Final Process Evaluation |

3.3.1 Process Research and Pilot Studies

Chemically feasible process is fundamentally characterized by process research. It is an accepted fact that process research is a basic necessity of the process industry. Orderly research and development has become an integral part of all leading industrial organizations and is synonymous with the progress. Basic operating conditions are explored and sufficient information is obtained from process research for preparing a preliminary material and energy balance flow sheet.

In many instances, development of a pilot plant is a necessity, particularly where laboratory scale data are not sufficient to scale-up or safety aspects are not well known. It is better to commit blunders on a small scale and make profits on a large scale. Such pilot plants can result in eliminating production of the off-spec product, good yields, reduced cost of manufacturing, etc. by fixing best operating conditions, installation of corrosion resistant plants, deployment of best equipment, reduced recycles due to better yields and so on.

Need for a pilot plant is justified in any manufacturing (Refs 1, 2 and 3). Take for example, manufacture of bulk drugs. Manufacture of antibiotics involve reactions (of exothermic nature) at very low temperatures; at times at cryogenic temperatures. Use of ice or dry ice suffice at laboratory scale but at production level it is necessary to deploy industrial refrigeration which can be simulated in a pilot reactor. Design of a reactor changes significantly with industrial refrigeration in which possibly use of liquid nitrogen may be necessary. Similarly, solvent to reactant ratio used at laboratory scale may not be economically viable at the plant scale and it may be necessary to reduce the ratio to an optimum by experiments on a pilot scale.

As seen in the earlier section, hydrogenation of edible oil is carried out in a slurry comprising of a catalyst. Selection of agitator is crucial. Pilot studies are valuable in the development of an agitator ideally suited for the reaction. Similarly, ozonation is a dangerous reaction and is very much dependent on intensive

diffusion of gas in the liquid. Use of a static mixer with liquid circulation and simultaneous injection of ozone in it could prove to be advantageous. However, such designs should be supported by pilot studies for its efficacy and control of reaction. Design of a seal for agitator can become an important design factor when dealing with high pressure gas reactions or with dangerous gases such as chlorine, ozone, etc. Viscosity of reactant mass, operating pressure and temperature, suspended solids concentration, heterogeneous reaction mixture, etc. can have a strong bearing on reactor design and process research with a pilot plant could therefore be fully justified.

Pilot scale studies may be warranted for other unit operations. Its requirement can be decided based on the data available in literature or from the lab scale experiments. For example, if sufficient vapour-liquid equilibrium data are available for separation of a liquid mixture, a distillation equipment can be designed with certainty and pilot study may not be required. Similarly, mutual solubility data, if available, extraction equipment design can be carried out with known principles of unit operations. In the event of a specific design of the equipment, pilot studies are recommended. For example, for carrying out high vacuum distillation of a heat sensitive product, specialized equipment such as short path distillation or a wiped film evaporator is recommended. Manufacturers of such equipments provide pilot study on a small scale equipment with reasonable scale up confidence.

Pilot scale study can provide more reliable flow sheet than the one obtained from lab scale study. Versatile equipments which can be procured off the shelf are available for pilot studies. Alternately process engineers can design a tailor made pilot scale equipment for the study.

Pilot plant can be used for ongoing improvements of the existing process or development of a next generation process. One of the objectives of such a study is substitution of a raw material. Improvement in product quality, reduction in process costs, safety and minimizing the pollution effects are other objectives of the pilot study.

Introduction of a new product adds a dimension of risk relative to the existing product. Manufacturing a small volume of the new product in a pilot plant can be valuable in supplying it for commercial testing to a customer whose evaluation is crucial before introducing the product on a commercial scale. In many instances, when the customer demands some product specifications change, it could be achieved by process research on a pilot plant.

It can, therefore, be said that a pilot plant is an intermediate sized plant, modeling the process of interest, constructed and operated so as to test the model in a convenient, less expensive and safe manner.

Results obtained in the pilot plant builds confidence in management and help them in taking commercial, technical and financial decisions, there by focusing their attention and allocating the resources accordingly.

Cost of a pilot plant could be significant but risks are relatively low and payoffs could be substantial.

Another important consideration in pilot plant operation is the selection of manpower. A project leader (a chemical engineer), familiar with the chemical

process and the equipments, is chosen for the job. He should be assisted by one or more chemists, depending on the size and complexity of the pilot plant. Careful observations during the operation and interpretation of data are the skills desired from the personnel. Effective communication among the staff is crucial. Cost of such talented staff would be high but is rewarding. Motivation to the staff enhances self satisfaction and increases dedication towards the work.

Logging of data can range from visual examinations to instrument readings. Relatively inexpensive data acquisition system based on electronic instruments is available which can be installed and connected to a computer to record the data, generate trend analysis, know critical events, find temperature or pressure spikes, etc.

Instrumentation needs for the pilot plant depends on its complexity. A reasonably straight forward process may not justify complex instrumentation/control system but a high pressure and hazardous pilot plant will call for an expensive instrumentation package. Take for example, a pilot plant for the manufacture of alkyd resins or antibiotics require normal instrumentation. However pilot plants for hydrogenation, ozonation, supercritical extraction with carbon dioxide, carbonylation with carbon monoxide, etc. may call for full automatic operation with data logging systems.

Pilot plant can be used as a training tool to plant operating personnel. It is well known that aeroplane pilots are trained on a simulator. Pilot plant can be considered as a simple version of ‘simulator’ of the main plant.

Project leader will be able to prepare a preliminary operating manual of the main plant based on his study of the pilot plant. Pre-startup testing, startup, normal operation and shutdown, emergency shutdowns, etc. can be well visualized by the project leader. The manual can be subsequently updated by the plant personnel based on their operating experience.

A process flow diagram can be offered by a team leader for further process design based on pilot plant studies.

3.3.2 Process Design and Development

After carrying out process research and pilot plant study, the process details are now available to the process engineer which can be scaled up with sufficient confidence level to a commercial plant. Immediately the process engineer is not concerned with detailed design of pieces of equipment but is concerned with the overall process covering following major items.

- (a) Preparation of a block diagram for presentation to the management for preliminary feasibility of the project.
- (b) Carryout overall and individual material balances showing the yield and quality of all materials to and from all pieces of equipment.
- (c) Heat balances over each piece of equipment.
- (d) Prepare a Process Flow Diagram based on data to steps (b) and (c).
- (e) Establish flow quantities and suction and discharge conditions of all rotating equipments.
- (f) Size reactors and storage tanks.

- (g) Separation process calculations with optimum operating conditions to effect the desired separation.
- (h) Estimate utility requirements such as steam, cooling water, refrigeration, electricity, thermic fluid, fuel, etc.
- (i) Prepare a Piping and Instrumentation Diagram so that requirement of instrumentation/control system is assessed.
- (j) Develop a tentative layout of the plant that should take into considerations of safety requirements.
- (k) Estimate preliminary capital investment and production cost.

Preliminary process design is necessary to find economic viability of the project and focus on important practical aspects. Take for example carbonylation of ethanol to propionic acid. This is high pressure and high temperature catalytic reaction of ethanol and carbon monoxide. Toxic nature of carbon monoxide dictates prominently in deciding over the safety requirements and practical operational aspects over the economic viability of the project.

Preliminary design will show the preferred process and whether further research and development (R & D) work is necessary. This additional R & D efforts will be specifically targetted to obtain missing information. Many a times physical properties of intermediates, corrosion data, heat of reaction, etc. are not available in literature and may have to be obtained by R & D efforts.

Duration of completion of project can be predicted based on the preliminary design. Based on this information, marketing strategy of the product can be developed.

Development of PFD and P & ID are dealt in detail in Chapter 4 with specific examples.

After completion of the preliminary process design and its evaluation, detailed process design work is taken up. For the detailed design, variety of 'tools' are used in calculations. Estimation of properties by group contribution methods, literature search for correlations for heat transfer, pressure drop and mass transfer calculations, mixing, etc. are the tools available to a process engineer. Many a times empirical sizing of equipment is carried out based on experience or literature survey. Numerous process design examples are worked out in Chapters 5 to 10 relating to fluid flow, heat transfer and separation processes.

Instrument engineer is simultaneously consulted in selecting the final control strategy. Suggestions/comments of the project leader of the pilot plant are quite valuable on the preliminary PFD and are incorporated as far as possible.

On completion of all the above steps, final PFD and P & ID are prepared by the process engineer and handed over to the project manager with equipment datasheets (containing process specifications) for further execution of the project.

During project execution stage also, role of the process engineer cannot be undermined. He will have an advisory role to ensure that process will function smoothly as anticipated during design stage.

3.3.3 Batch vs Continuous Operations

When designing processes or facilities for some applications, process engineer

must approach the task differently than he would for a project in other chemical process industries. Bulk drugs, pharmaceutical intermediates, dyes intermediates, fine chemicals, etc. are the products in which batch operations dominate. Take the pharmaceutical industry sector. In this sector, lots of manufactured product are required to be segregated. In this way waste is minimized and regulations could be easily conformed to. Secondly, most pharmaceutical processes deal with relatively smaller amounts of material than with typical chemical processing operations. For this reason, batch processing equipment is often the preferred choice. Many a times, an equivalent continuous based unit operation that is small enough to accommodate the volume of material to be processed is not available. Further, most pharmaceutical processes come out of the lab where operations are predominately in batches. These batch type processing techniques migrate to the larger production scale.

Although one would not propose changing batch processes to continuous process for small volumes, many operations could be made continuous. A continuous unit operation can be used to process a tank, full of material. For example, a continuous filtration method, such as a rotary drum filter can be used instead of batch method such as a plate and frame filter.

Another example of making continuous unit operations work involves two phase extraction process. The traditional batch approach is to use a simple mixer-settler that mixes two phases together in a tank before they are allowed to separate. In a continuous analog, a small extraction column could be used in which two phases flow past one another continuously to facilitate phase contact. This continuous operation can be performed in a much smaller equipment. It also provides numerous ‘stages’ of phase contact resulting in more efficient mass transfer operation. Similar examples can be cited for fermentation, precipitation, crystallization, drying, etc.

Conversion from batch processing to continuous processes would thus allow smaller equipment with lower capital cost. Continuous equipment could be designed to be enclosed and cleaned easily, allowing the use of less costly production space. Many of these continuous equipment would operate at ‘steady state’ and it would be easy to automate them, reducing labour cost.

Batch operations and the equipment that facilitate them are by no means obsolete. Continuous equipment by its nature must be designed around a standard process that will be repeated for a significant period. For products that are made infrequently it is impractical to design continuous operations to support them. Processes where the input material varies over time also exist. These applications require an adjustment to the processing techniques to accommodate new variables. In many such cases only batch operations are flexible enough to handle these situations. Further some processes handle materials that are so valuable or hazardous that the transients that might occur in a continuous process are unacceptable. In these cases, only batch processes offer the control and containment, required to make them successful.

Continuous extraction of oil from oil-rich cake with *n*-hexane is practiced in industries on large scale. However, when residual oil is to be extracted from

bleaching earth before its disposal, a batch operation is normally performed as its quantity is quite small.

Bleaching of edible and non-edible oils was performed traditionally in batch equipment. Recent development of continuous bleachers give better quality of bleached oil with less bleaching earth/activated carbon and eliminate need for intermediate storage of bleached oil before its passage to continuous deodorizer. Also, the size of a continuous bleacher is much smaller than a batch bleacher.

Many a times, solvent to reactants ratio is kept high (may be in excess of 15) in a batch reactor based on lab studies. Many of such reactions could be relatively fast and endothermic. At the end of the reaction, solvent is required to be distilled out. Such a process lends itself for conversion to continuous operation. A mixed stream of solvent and reactants (possibly with much lower ratio) can be heated in a standard shell and tube heat exchanger, passed through a mixing equipment (such as a static mixer) and sent to a reactive distillation column where sufficient reaction time can be provided and simultaneously solvent is distilled out. Such a conversion can result in substantial energy savings apart from reduced time cycle of production.

Large capacity chemical plants are continuous in nature. Manufacture of ammonia, methanol, acetic acid, carbon black, formaldehyde and many others are the examples where continuous plants are designed and operated. Consistent quality, optimum energy input, lower cost of installation of plant per unit (say kg) of product, etc. are inherent benefits of continuous plants.

The process engineer must evaluate both the operations before deciding the best operation, suited for the product. As explained in previous paragraphs, batch operations should be restricted to a minimum so that economy of scale, a feature of continuous operations, can be availed at a large.

Reference 4 deals exclusively on batch process design.

3.4 PREPARATION OF OPERATING INSTRUCTIONS MANUAL

One of the less appreciated tasks of a process engineer is preparation of an Operating Instructions Manual (also known as Operation Manual) — OIM of the plant. When a calculator is purchased, a leaflet is provided alongwith the calculator which describes its functioning. In a similar fashion, OIM is prepared for safe, smooth and efficient operation of the plant. Well prepared OIM serves as a tutor to the operating personnel. It is also a valuable reference tool in day to day operation.

Safe operation of the plant is the prime objective of preparation of the manual. Therefore, safe operating limits are prescribed clearly for each equipment and process. Dangers, if any, of crossing the critical parameters must be underlined.

Start-up and shut-down procedures are outlined in the manual. These include purging, slow pressure build-up, well defined heating procedure, etc.

Normal operating procedures are covered for achieving optimum performance of the plant. These procedures cover desired operating parameters' range, generation of minimum effluent quantity, optimum utility consumptions, etc.

Emergency handling procedures are well covered in the manual. Power failure, leakages, utility failure, fire, etc. are the emergencies which must be well thought of and a plan of each emergency must be described.

PFDs and P&IDs are included in OIM. Wherever required, additional sketches are included to clear the operational aspects. Calibration charts of tanks, reactors, dosing systems, etc. are included in OIM. For example, capacity of a screw feeder with its speed is an essential requirement of an operator.

Process datasheets and manufacturers' datasheets are normally compiled as Equipment Datasheets and it is a separate manual by itself. Instrument specifications are also included in this manual.

Useful guidelines are drawn from the pilot plant operation for the preparation of OIM. Equipment and instrument supplier catalogues provide their own operating instructions. OIM should cover comprehensively operating instruction of the manufacturers.

In many instances, standby pumps, filters, etc. are provided. Periodic operation of standby equipments is to be included in OIM. Similarly, periodic calibration of instruments is needed to ensure that they are functioning in order. For example, in case of pressure gauges, zero error is checked periodically. In case of temperature measuring instruments, calibration with a standard instrument is recommended periodically.

Preparation of OIM is normally undertaken by the process engineer during project execution. Draft copy of OIM is circulated to operating personnel and project leader of pilot plant. Suggestions received from them should be critically reviewed and if found useful should be included in the final OIM.

Over a period of time, OIM needs upgradation as more experience is gained in actual operation. Also the changes in OIM should cover the modifications that are carried out over the years. Thus upgradation is a never ending process.

Preparation of log-sheets for manual logging of data is normally the job of operating personnel. Nevertheless, the process engineer can review the draft of the log-sheets and may offer suggestions. For medium scale and small scale plants, these are also known as batch sheets.

The process engineer should study the log-sheets periodically and offer suggestions to the operating personnel. If necessary, OIM may require upgradation on account of review of the log-sheets.

3.5 CONDUCTANCE OF PERFORMANCE TEST RUNS

On installation of the plant, process engineer is required to conduct a performance test of an equipment or entire plant. When a centrifugal pump is commissioned, its capacity, head and power consumption are checked with the datasheet. When a water treatment plant; say a reverse osmosis plant, is commissioned, its performance is checked for permeate quality (*vis-a-vis* feed water quality), permeate/feed ratio and power consumption. In the same manner, performance of cooling tower is checked for achieving anticipated cooling water temperature for the prevalent wet bulb temperature of ambient air and for its power consumption.

In large chemical plants, performance guarantees are well specified in the contract. These include product quality specification, raw materials consumptions

and utility consumptions. All these parameters are averaged over a specified continuous run of the plant, say for 120 hours or so. Average values are compared with those guaranteed.

Conductance of performance test run requires specialized skills. It is carried out by the process engineer in coordination with the plant personnel and contractor personnel. Before starting the test run, instruments are calibrated. Any specific deviation from normality is documented in the logsheet.

Test run report comprises logsheets, observation sheets, analytical reports and summary of findings. It is used as Acceptance Report for the equipment or the plant.

3.6 TROUBLESHOOTING

Troubleshooting is yet another task of the process engineer. During commissioning or normal operation, many operational problems are experienced. Some of them are related to the process. Process engineer is required to study the problem, discuss with operating personnel and present a solution.

Take an example in which cooling water cools a fluid. If cooling is less effective than design, reasons for the same are to be determined. Less flow of cooling water or high supply cooling water temperature or inadequate heat transfer surface could be the problem for a new system. Material and energy balance across the cooler will reveal the problem and corrective action can be taken. During the course of operation, if the cooler performance deteriorates, fouling of cooling surface could be anticipated.

Take another example of a separator in which organic liquid is separated from water. While studying the performance of the separator, check moisture content of the organic layer and chemical oxygen demand (COD) of the water layer. If the results are not satisfactory in terms of separation, check operating temperature of the separator or its design.

In a distillation column, if the distillate quality is not maintained, one can check the reflux ratio and other parameters.

3.7 COST CUTTING MEASURES

In a normal running plant, process engineer has to consider cost cutting measures in the plant. This is a never ending exercise as there exists an opportunity for improvement in each plant. For finding the opportunity, indepth study of the plant is recommended.

Consider a chilling plant in which brine at -10°C is produced using refrigerant R-22. It is a known fact that in this temperature range Coefficient of Performance (COP) of ammonia refrigeration cycle is better than that of R-22 cycle. Thus replacement of the refrigeration skid of R-22 system by ammonia system will consume less power. However, fresh investment for the ammonia refrigeration system will be needed. A cost benefit analysis will have to be carried out by process engineer.

Take another case of a plant in which hot oil at $150\text{--}175^{\circ}\text{C}$ is used to heat reaction mass and carry out the reaction at around 105°C . Alternative system

with saturated steam at 3.5 bar g could be equally or more effective. Both economical and practical aspects need be examined before any change in the plant is considered.

In large chemical plants such as ammonia plants many opportunities exist for cost reduction. With methane and ambient air as feedstocks at 25°C, theoretical energy consumption is 20.618 GJ/t ammonia. Actual ammonia plants consume energy equivalent to 27 to 42 GJ/t ammonia, depending on the feedstock and technology. This indicates a potential for reduction in energy consumption and thereby in cost of production. Similar opportunities can be found in each plant.

Newer technologies and innovations can contribute significantly to cost reduction. Process engineering plays a vital role in their implementation.

3.8 GREEN ENGINEERING

A set of 12 guiding principles of ‘green chemistry’ were established by Dr. P. Anastas and J. C. Warner in 1998 for creating safer chemicals and designing for degradation to save the environment. Further to these guidelines, a multidisciplinary group of engineering disciplines met in May 2003 in Florida, USA to draft the principle of Green Engineering. The conference focused on the definition of the scope and tone of green engineering relative to all engineering disciplines; including chemical, mechanical, civil, electrical and environmental.

The objective of green engineering is to transform existing engineering disciplines and practices to those that promote sustainability. It incorporates the development and implementation of technologically and economically viable products, processes and systems to promote human welfare while protecting human health and elevating the protection of the biosphere as a criterion in engineering solutions. To implement these solutions, engineers are advised to use the following principles⁵.

- (i) Engineer processes and products holistically, use systems analysis and integrate environmental impact assessment tools.
- (ii) Conserve and improve natural ecosystems while protecting human health and well-being.
- (iii) Use life-cycle thinking in all engineering activities.
- (iv) Ensure that all material and energy inputs and outputs are as inherently safe and benign as possible.
- (v) Minimize depletion of natural resources.
- (vi) Strive to prevent waste.
- (vii) Develop and apply engineering solutions while being cognizant of local geography, aspirations and cultures.
- (viii) Create engineering solutions beyond current or dominant technologies; improve, innovate and invent (technologies) to achieve sustainability.
- (ix) Actively engage communities and stakeholders in development of engineering solutions.

Process engineer can play a vital role in achieving the objectives of green engineering. At every stage of process engineering, the engineer must consider and review whether the design adheres to the principles of green engineering. A

few examples will demonstrate implementation of principles of green engineering in practice.

- (a) In solvent extraction plants, *n*-hexane is used to extract oil from oil-rich cake. It is known that *n*-hexane is not totally safe for human beings. New super critical extraction process based on carbon dioxide as an extracting agent is found to be eco-friendly, safe for human beings and also gives good quality oil.
Water can also be used as supercritical fluid to produce biocrude from organic waste. In this application, water is a safe extracting agent while it helps in safe ‘disposal’ of organic (including hazardous) waste.
- (b) Hydrogenated edible oils are used extensively in bakery and other food products. Presence of trans-fatty acids in the hydrogenated fat is known to be unfit for human consumption. Technological developments should be targeted to produce the fat with low trans-fatty acids. One such development is hydrogenation in presence of carbon dioxide at low temperature. Another development relates to the use of noble metal catalyst (instead of nickel) for hydrogenation.
- (c) Methyl tertiary butyl ether (MTBE) is known an oxygenating product for improvement of octane number of the gasoline. Research has revealed the health hazards of MTBE when autovehicles emit gases to atmosphere. Addition of anhydrous ethanol (a renewable product) is said to improve octane number without creating health hazards.
- (d) Trans esterification of fatty acids with methanol can produce biodiesel which is considered an eco-friendly fuel for DG sets or for autovehicles. Fatty acids can be obtained from edible and non-edible oils which are renewable resources.
- (e) Ozone depleting substances are threats to human beings. Montreal protocol, 1987 bans the use of certain chlorofluoro hydrocarbons which are established to have very high ozone depleting potential, apart from their low degradability. Classical example is dichlorodifluoromethane (CFC-12) which is banned for use as a refrigerant, aerosole agent, etc. Engineers are advised to use ammonia (R-717) or chlorodifluoromethane (CFC-22) in place of CFC-12. Eventually CFC-22 is also to be phased out.
- (f) In a classical Bechamp process, nitro-compound is converted to a corresponding amine by using iron power and water mixture. Reaction takes long time. At the end of the reaction, total mass is required to be filtered out of the iron sludge (fine in nature). Later the mass is separated in two layers. Water, so separated, has high chemical oxygen demand (COD) and pose an effluent-problem.
In new developed processes, hydrogen is used in presence of a catalyst under pressure. Use of gas-induced agitator, nickel or noble metal catalysts, etc. are the developments which result in reduced time of reaction and also do not generate effluents. Thus, it is an eco-friendly process.
- (g) While methanol is used as a solvent in many chemical plants, it is toxic to human beings. Its use is considered unsafe in pharmaceutical and food

processing. Its higher homolog ethyl alcohol is safe for use in the industry as a solvent.

3.9 PROCESS INTENSIFICATION

Process intensification can be defined as improving productivity of a system by changing operational parameters and/or by carrying out desirable modifications. In a process plant, improvement in productivity means product quality improvement, yield/production rate increase, reduction in energy consumption, reduction in other inputs (such as raw materials), safer operation, or reduction in pollution load of the plant. Process intensification differs from troubleshooting (Sec. 3.6) as the latter mainly focuses on solving the problems, faced by operating personnel. Cost cutting measures (Sec. 3.7) can be considered as one of the objectives of process intensification.

Process engineer can contribute significantly in process intensification; either at the process design stage or in the running plant. Scientific approach to the design and critical analysis and review of the operating data are the keys to process intensification.

At the process design stage, a number of options are available to the process engineer. Consider design of a multiple-effect evaporator system. Apart from the process parameters; such as concentration of ingoing/outcoming solution, heating medium and its conditions (e.g. pressure and temperature), etc., process engineer can design the evaporators with equal or unequal heat transfer area (HTA) in the stages. Optimization techniques such as Evolutionary Operation (EVOP) or dynamic programming can be used to find HTA for the both the alternatives, thereby cost of the installed systems for both the alternatives can be worked out. Optimum design of the system can then be selected.

A cascading type steam balance offers an interesting example of utility system optimization as a part of process intensification. Modern heavy chemical plants such as ammonia plant, methanol plant, ethylene manufacturing, petroleum refinery, etc. have cascading steam systems which can be optimized to consume minimum fuel in the boiler and also reduce load on cooling tower. Reference 6 has given several steam balances for a given plant offering an opportunity to a process engineer to select the best steam system.

In a simpler case, selection of an agitator in a reactor can result in substantial energy saving-with improved agitation.

Linear programming is another tool which helps in optimizing the proportions of components in a reaction mixture. Consider manufacture of alkyd resins by reaction of polyols (such as glycerol) with fatty acids by azeotropic distillation method. Fatty acids could be either distilled (pure) fatty acid or in the form of an oil. Similarly, a variety of alcohols such as glycerol, sorbitol, etc. are used. Theoretical calculations can be made with the help of linear programming to achieve desired properties of the product with best possible yield which can later be confirmed in laboratory trials.

Innovations and adoption of newer technologies are integral to process intensification. Adoption of a standard operating practice in one industry into another

industry should be considered for a similar situation. A fluid bed cooler is used in large urea plant to produce urea of uniform (narrow range particle size distribution) size prills at low temperature so that urea prills do not agglomerate to lumps on storage in a silo which produce dust on breaking them. Similar problems are also experienced in a carbon black plant. Adoption of the fluid bed cooler should be considered in a carbon black plant.

Use of a static mixer in mixing an organic compound with a mixed acid ($\text{HNO}_3 + \text{H}_2\text{SO}_4$) to form nitro compound is found to be very effective in controlling side reactions with efficient temperature control in a batch reactor.

Several examples can thus be cited to demonstrate that process intensification at the design stage can result in a plant with lower operating cost, possibly lower initial cost, ease of operation and safer operation.

In an operating plant, critical review of operational parameters can result in substantial savings. Use of statistical methods are invaluable in this exercise. In fact ISO 9001 gives stress on statistical methods for improvement in quality control which is advised as a continuous improvement process.

In a similar manner, it is advised that variation in process parameters and its effect on plant performance be studied which can reflect the best and safer operating conditions.

Modifications in the plant can be aimed at reduction in energy consumption or reduction in raw materials consumptions. In ammonia plants, purge gas from synthesis loop was being vented or used a fuel in the furnace in many grass root plants. Adoption of purge gas recovery plant to recover and recycle hydrogen from the purge gas could result in increase in production by about 5% and simultaneously reduce energy consumption of the entire production by about 2%.

In a bulk drug plant, some reactions are carried out at very low temperatures (around -80°C). Injection of liquid nitrogen is quite common for achieving such low temperature in the industry. This method is rather inefficient and expensive. Instead, a cascading type mechanical refrigeration system with R-23 and R-22/R-717 in tandem can be considered for adoption to achieve the same temperature at much lower investment and operating cost. Also the space requirement is less for the refrigeration system. Adoption of cascading refrigeration system may bring down the refrigeration cost by about 50% as compared to liquid nitrogen injection system for a given plant.

Use of aqueous calcium chloride brine was common in refrigeration. However, numerous operating problems were experienced with it such as slimy spills, chocking of pipelines due to freezing, difficulty in maintaining brine concentration, etc. Aqueous monoethylene glycol (MEG) is found safe and convenient substitute to aqueous calcium chloride brine, thereby design of brine circulation system is simplified. Use of aqueous methanol brine is made for low temperature applications (-20 to -50°C) but the brine solution is flammable in nature and requires specific care in design. Thus adoption of new systems is a part of process intensification.

In large chemical plants, the network of heat exchangers can be reworked in line with pinch technology (Sec. 6.13). Such an exercise can result in substantial energy savings.

3.10 CONCLUSION

Process engineering is the heart of any process design. While adequate attention at the design stage can pay rich dividends, adoption of process engineering principles in a running plant is always paying.

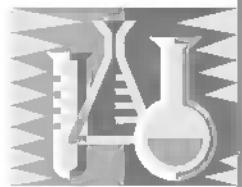
EXERCISES

- 3.1 Give brief answers of the following.
 - (a) Name ten biofuels that are used for steam generation.
 - (b) Differentiate petroleum based lube oils and eco-friendly lube oils.
 - (c) An innovative hydrogenation process for edible oil is to be developed which is based on supercritical principle. Carbon dioxide and propane are considered as alternative supercritical fluids (SCFs) for the purpose. Study the advantages and disadvantages of each for the purpose.
 - (d) Name five ‘green’ solvents for use in chemical synthesis.
 - (e) An aqueous *iso*-propyl alcohol mixture, containing 50% alcohol (by mass), is to be separated to a stream of 100% alcohol. Suggest process steps with reasonings.
 - (f) Nitrogen stream at atmospheric pressure and dew point of -60°C is used for deriming a cold box of air liquefaction plant. If argon is used instead of nitrogen, what should be the dew point on equivalent moisture content basis?
 - (g) For relatively small capacity storage of liquefied gases (such as ammonia, LPG, etc.), a sphere construction is selected. Why?
 - (h) As a rule of thumb for most of the distillation cases, an increase in column pressure means: (a) decrease or (b) increase in the column capacity and (i) better or (ii) work fractionation, given other parameters.
 - (i) Opening stock of a fuel oil took was calculated as $600 \text{ kL} \times \text{SG of } 0.94 = 564 \text{ t}$, and the closing stock as $550 \text{ kL} \times \text{SG of } 0.94 = 517 \text{ t}$ in a day, i.e. transfer of 47 t to the boiler. Temperature variations were ignored thinking that the variation in SG is about 1% and should, for all practical purposes, be acceptable. In reality, the temperatures of the fuel oil at the time of measurements of opening and closing stocks were such that corresponding SG were 0.94 and 0.95, respectively. It really meant an error of about as much as (a) 5.5, (b) 3.5, (c) 1.5, (d) 0.15 or (e) 0.05 t.
- 3.2 Chlorination of an organic compound is to be carried out with chlorine. Hydrogen chloride gas with some chlorine are vented out from a batch reactor at near atmospheric pressure. Consider the various options, available for trapping hydrogen chloride gas from the reactor and develop process flow diagrams for each of them.
- 3.3 Study a batch reactor operation. Write start-up, normal operating and shutdown procedures for the reactor, including safety requirements. Also prepare a log-sheet for the batch operation with a view to collect consolidated information for review and comments.
- 3.4 In a chemical plant, a cooling tower of 1000 kW capacity is operated. An operator reports in summer that the production is limited due to cooling tower. How will you check the efficiency of the cooling tower?

- 3.5 In a batch reactor, reaction is carried out under reflux conditions for long hours with a condenser using cooling water at 32°C. Solvent used in the vessel is methanol. Plant data indicate substantial loss of methanol to atmosphere during the reaction. Analyze the situation and suggest remedial measures for loss prevention with reasonings.

REFERENCES

1. Jones, J., W. Asher, J. Bomben, D. Bomberger, C. Marynowski, R. Murray, R. Phillips, D. Robers, K. Semaru, R. Swidler, C. Witham and J. Zuegel, *Chem. Engg.*, **100** (4), April 1993, p. 138.
2. Lowenstein, T. G., *Chem Engg.*, **92** (25), December 9, 1985, p. 62.
3. Frye, A. L., *Ind. Engg. Chem.*, **50** (4), 1958, p. 577.
4. Sharrat, P. N., Ed., *Handbook of Batch Process Design*, Kluwer Academic, USA, 1999.
5. AIChEExtra, *Chem. Engg. Progr.*, **99** (9), 2003, p. 78.
6. Bhatt, B. I. and S. M. Vora, *Stoichiometry*, 4th Ed., Tata McGraw-Hill Publishing Co. Ltd., New Delhi, 2004.
7. Kletz, T., *Process Plants : A Handbook for Inherently Safer Design*, Taylor and Francis, USA, 1998.
8. Ludwig, E. E., *Applied Process Design for Chemical and Petrochemical Plants*, Vol. 1–3, 3rd Ed., Gulf Publishing Co., USA, 1999–2001.
9. Elvers, B., S. Hawkins and G. Schulz, Ed., *Ullmann's Encyclopedia of Industrial Chemistry*, 5th Ed., Vol. B1 to B4, VCH Verlagsgesellschaft, Germany, 1988–1992.
10. McKetta, J. J. and A. W. Cunningham, *Encyclopedia of Chemical Processing and Design*, Vol. 1 to 68, Marcel Dekker, Inc., USA, 1976–1999.
11. Evans, L., Jr., *Equipment Design Handbook for Refineries and Chemical Plants*, Vol. 1 and 2, Gulf Publishing Co., USA, 1971.



Importance of Process Diagrams in Process Engineering

4.1 INTRODUCTION

Engineering Diagrams are vital for understanding of a process to chemical engineers, of equipments to mechanical engineers, of electrical circuits to electrical engineers and so on. These diagrams are prepared in many different ways to suit the specific requirements. Process Flow Diagrams (PFD) and Piping and Instrumentation Diagrams (P&ID) are basic requirements of process engineering and are prepared before attempting any detailing. Steps followed in preparation of these diagrams and interpretation of the same are covered in this chapter.

4.2 NEED FOR PFD AND P&ID

When any chemical manufacturing project is conceived, immediate information is sought on the requirements of raw materials and utilities. This information can be calculated using the Laws of Conservation of Mass and Energy, covered in the previous chapter on Stoichiometry. When these data are presented in a sequential manner with symbolic representation of equipments, it is known as PROCESS FLOW DIAGRAM (PFD) or PROCESS FLOW SHEET. It can be said that PFD is a schematic illustration of the system. It shows relationships between the major components in the system. It also tabulates the process design values for the components.

PIPING & INSTRUMENTATION DIAGRAM (P&ID) is different from PFD. While PFD is a quantitative representation of the process, sequential presentation of flow, piping with instrumentation and controls that are required for smooth operation of the process are given on a P&I diagram. It can therefore be said that P&ID is a schematic illustration of functional relationship of piping, instrumentation and system equipment components. It shows all the piping including the physical sequence of branches, fittings, equipments, instrumentation and control interlocks.

PFD is a useful diagram in effective communication of design information. It helps an operator in adjusting his operating parameters, a shift supervisor in

checking the plant operation, management in discussions across the table and project engineers in comparison and evaluation of different processes and their quantitative requirements.

Modern plants have extensive instrumentation and controls such that a minimum attention is required of an operator. Apart from controlling the plant variables, these plants give better productivity in terms of yield and quality. Also, less manpower is required to operate the plant with increased safety.

Process engineers are involved in preparation of both these types of diagrams.

4.3 BLOCK DIAGRAMS

Simplest flowsheets are block diagrams. Process engineer begins the process design with a block flow diagram in which only the feed and product streams are identified. Such input-output diagrams are not very detailed and as such are most useful in early stages of process development.

Figure 4.1 is a block diagram of the process to manufacture carbon dioxide from flue gas of power generation plant using electrochemical membrane separation technique.

Absorption of carbon dioxide from the flue gas mixture using an aqueous amine solution or caustic potash solution is difficult because of presence of oxygen. Electrodialysis cell has an edge over the absorption technology in which presence of oxygen is not a hinderence to the recovery of carbon dioxide. The block diagram is useful in understanding overall process and also gives rough quantitative picture of raw materials and utility consumptions. These data can help in working out preliminary variable cost.

The processes, described in the block diagram, are then broken down into basic functional elements such as reaction and separation sections. Process engineer can also identify the recycle streams and additional unit operations to achieve the desired operating conditions. Identification of basic elements help in performing material and energy balance calculations which in turn help in preparation of the PFD.

4.4 DEVELOPMENT AND UTILITY OF PROCESS FLOW DIAGRAMS

Any product or process development starts with stoichiometric calculations. At first, required capacity of the final product is fixed. The product can normally be manufactured by inputting different raw materials and following different process routes. For example, manufacture of ammonia is possible with different hydrocarbon feed stocks such as natural gas, naphtha, fuel oil, coal, etc. Process route for each feed stock is different. For example, reforming is a preferred route for natural gas or naphtha while partial oxidation is the route followed for fuel oil or coal. Nitrogen for the ammonia manufacture is invariably obtained from air but it may be fed as air or nitrogen is separated from air by liquefaction and it is fed to the process. PFD (Process Flow Diagram) for each continuous process is different.

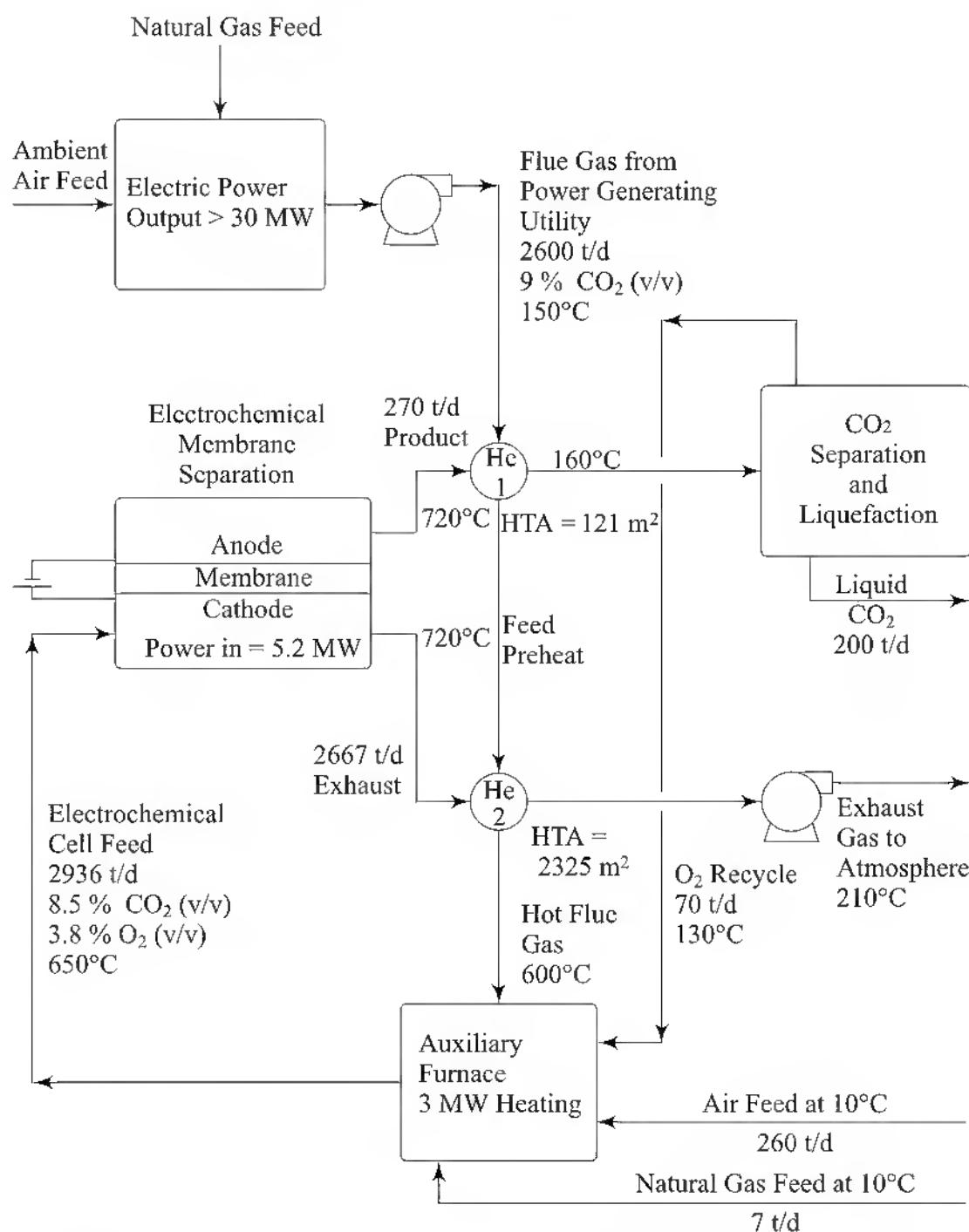


Fig. 4.1 Block Diagram for Manufacture of 200 t/d CO₂ by Electrochemical Membrane Process¹
(Reproduced with the Permission American Institute of Chemical Engineers, USA)

Another example is reduction of nitro aromatic compounds to corresponding amines. In a classical Bechamp process, nitro compound is mixed with water and fine iron powder in a batch reactor. The reaction mixture is moderately heated in presence of a catalyst to initiate the reaction (basically in an acidic medium). Hydrogen is generated in situ and reduces the nitro compound to corresponding amine. Reaction is exothermic and temperature is controlled by cooling. Reaction takes long time to complete. Product mixture is separated in organic and aqueous layers. While aqueous layer is drained out, organic layer is siphoned and purified by filtration and distillation under vacuum.

In newer version of the process, hydrogen gas is bubbled in the organic liquid under pressure. Nickel or noble metal based catalyst is used. Gas dispersion through the liquid is improved by different methods such as use of static mixer, use of gas induced agitator, liquid jet ejector system, etc. Such a system is quite efficient and the reaction can be completed in relatively short period. Both the process routes are used in industry. PFDs for both the routes are different.

Above examples involve chemical reactions. However, there are PFDs which involve unit operations but no chemical reactions. For example, separation of components of natural gas in a distillation column by cryogenic means, removal of acid gas by absorption–desorption, removal of Volatile Organic Compounds (VOC) by adsorption–desorption, etc., do not involve a chemical reaction. Each PFD is, therefore, to be treated on its own requirements.

A Process Flow Diagram generally includes following information.

- (a) Flow rate of each stream in case of continuous process or quantity of each reactant in case of a batch process.
- (b) Composition of streams.
- (c) Operating conditions of each stream such as pressure, temperature, concentrations, etc.
- (d) Heat added or removed in a particular equipment.
- (e) Flows of utilities such as steam, cooling water, brine, hot oil, chilled water, thermic fluid, etc.
- (f) Major equipment symbols, names and identification.
- (g) Any specific information which is useful in understanding the process. For example, symbolic presentation of a hazard, safety precautions, sequence of flow, etc.

PFD of a laboratory scale process could be somewhat different from that of plant scale PFD. In laboratory, more emphasis is given on safety aspects. Also some utilities could be different from those used on a plant scale. For example, in laboratory use of ice and dry ice are common for achieving required chilling. On a plant scale, chilled water or chilled brine at different temperatures are used. Similarly, once through raw water system is common for cooling in condensers in the laboratory while closed circuit of cooling water is a practice in the industry. Batch distillation is commonly employed in the laboratory while in plants, batch or continuous distillation columns are employed with specific controls to achieve desired purity of the distilled materials.

Process Flow Diagram of a laboratory experimental unit could be simple and different from that of a pilot plant. Thodos *et al*² have described a PFD for the study of reaction kinetics to synthesize ethyl chloride from ethylene and hydrogen chloride gas. This is a gas phase endothermic catalytic reaction, studied in the pressure range of 8–11.5 bar and temperature range of 150°C–300°C. A 100 mm steel pipe reactor with electric heating element, wrapped over it, was used. Dry ice trap, following the reactor, removed ethyl chloride while excess HCl was scrubbed in a water trap. Thus a continuous experimental set-up provided useful data for determining reaction rate equation.

When the above process is to be studied on a pilot plant, the reactor could still be a pipe (bigger in size) with jacket in which thermic fluid (hot oil) circulation is

provided. For separation of ethyl chloride, heat exchanger with chilled brine can be considered. Scrubbing of unreacted HCl can be performed in an efficient packed tower.

Prugh³ has developed a laboratory scale PFD for the manufacture of phenylethyl alcohol by reacting ethylene oxide and benzene in presence of aluminium chloride catalyst. Such a PFD is helpful in transferring information from the research chemist to the process engineer, engaged in designing a pilot plant or in developing a PFD for a commercial plant. Sufficient stress is laid on safe scale-up. Effectiveness of the research efforts is thereby improved in designing a plant.

In the laboratory, many hazardous reactions are carried out such as chlorination, hydrogenation, ozonation etc. Some of them are not only explosive but also prone to health hazards. During scale-up of such processes, these aspects need special attention so that the plant is safe in operation and environmental pollution problem is not created. For example, venting of hydrogen containing gas mixture must ensure that the gas mixture is outside flammability limits. Also a right type of flame arrestor is to be installed on the vent. Similarly chlorine to be vented is to be scrubbed in aqueous caustic solution. These details form a part of process (although not a part of reaction) and therefore PFD must include these equipments.

PFD of a commercial plant covers material and energy balance details extensively. In PFDs of batch processes, quantities of inputs and outputs are specified with normal operating conditions. For a continuous process plant, a good practice is to give process stream flow rates and compositions in a tabular form at the bottom of PFD. Reference 4 gives several PFDs for batch as well as continuous processes.

Development of PFD of a process plant starts with conceptual design. At first the process flow sequence is established with the help of symbols of equipments or blocks. While symbols of equipments give better idea of the type of equipment to be deployed, block diagrams are satisfactory to start with. End product and capacity of production are defined which are entered at the end of PFD. Then an overall theoretical material balance is established to find the theoretical raw materials requirements. These quantities are entered at appropriate places in PFD where the specific raw materials are to be introduced. Thus a preliminary PFD is prepared. Now start establishing material balance and energy balance across each equipment taking into conversion limitations, catalyst activity, equipment limitations, etc. It is well known that conversions are dictated by kinetic considerations and hence it is not possible to achieve 100% conversion every time. These calculations are performed for each equipment with actual conversions and results are entered in the PFD. With these revised data, PFD becomes more like a working PFD. To this, add utilities and safety aspects. Final PFD is now ready for presentation.

Material and energy balance calculations for the manufacture of formaldehyde from methanol based on Formox process are well covered in Ref. 4. Using this information, its PFD is developed for a plant having methanol feed rate of 4000 kg/h (Fig. 4.2). In this diagram, process flow rates and operating conditions are given along the process lines. Heat duties of various equipments are also specified near the respective equipment. Stream compositions are given in tabular form at the bottom of the PFD.

The above PFD is quite useful to a process engineer in detail design of the equipments, selection of right equipment for the duty, pipe sizing, selection of right instrumentation and control system, etc. Further, PFDs, submitted by different vendors at the quotation stage, help the project engineer in comparison and evaluation of alternative process routes. Competitive processes can be evaluated from different angles such as ease of operation, minimum energy requirement, less hazardous operation, etc. Such an evaluation becomes useful in discussions with the management across the table thereby the technologically best plant can be selected.

PFD of an operating plant provides design data at 100% plant load. Such a diagram helps the operator in adjusting his process parameters and the shift supervisor in checking/controlling the plant operation to optimize the production. Manufacture of formaldehyde is a catalytic process. Comparison of actual operating data with the design conditions (such as those given in Fig. 4.2) can provide a trend analysis thereby a decision can be taken on when to change the catalyst. However, if the supervisor finds that productivity of the plant is not commensurate with the anticipated catalyst life he would look for other factors like poisoning of catalyst, fouling of coolers, etc. responsible for the same and corrective actions can be taken.

In Fig 4.3, PFD for the production of liquid oxygen from air at low pressure (~ 10 bar g) is given. In this process only physical separation takes place unlike chemical reaction, described in Fig. 4.2. This PFD was developed to compare the low pressure process with the high pressure ($\sim 135\text{--}140$ bar g) process. It does not give the process flows and stream compositions and hence it is not directly useful to plant operating personnel. Nevertheless it is useful to the project engineer in preliminary evaluation.

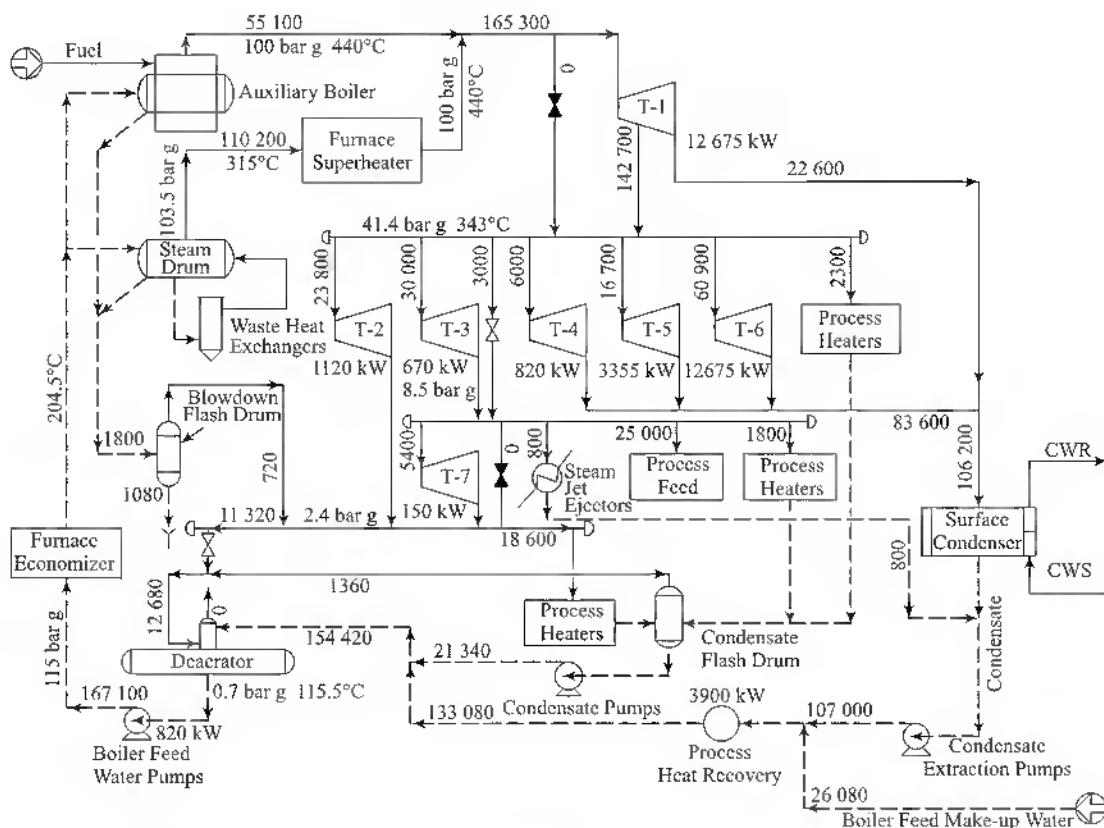
PFD of a utility system is somewhat different from the PFD of the process plant. Steam generation, steam distribution and utilization, cooling water circuit, thermic fluid circuit, instrument/plant air system, nitrogen (inert) gas system, refrigeration system, etc. fall under this category. Figure 4.4 is a typical cascade steam balance of a large chemical plant. This is also a PFD of the steam system. Development of an efficient steam system can pay rich dividends in terms of optimum energy input.

In Ref. 4, several steam balances are worked out for the same plant and corresponding PFDs are presented to exemplify the alternatives available to a process engineer during the design stage.

Similar PFDs can be developed for other utility systems. Liu *et al*⁷ have presented PFDs for raw water in a chemical plant. Reduction in raw water consumption as much as 50% could be achieved by optimizing such a PFD.

Cooling water is yet another utility for which an efficient distribution network can be developed. In Fig. 4.7, cooling water network is demonstrated.

Process flowsheeting can be carried out with the help of a computer. Steady state material and energy balance calculations can be performed on a spreadsheet or with the help of mathematical software which also can generate block diagrams. Specialized software such as Microsoft Visio, Aspen Plus, etc. are available for



Note: All flows are in kg/h

Fig. 4.4 Steam Balance in a Large Chemical Plant⁶ (Reproduced with the Permission from Chemical Engineering by Access Intelligence, USA)

the preparations of PFDs. References 8–10 are useful in developing computerized flow sheets.

4.5 DEVELOPMENT OF PIPING AND INSTRUMENTATION DIAGRAMS

Having understood the requirements of PFD, it is rather easy to understand the requirements of P&ID. Emphasis is changed from process flows to process control. Controls are not possible without measurements of variables; such as pressure, temperature, concentration, etc. for which instruments are required. A diagram incorporating piping, instruments and controls is known as P&ID.

In simple plants minimum instruments are installed. They are just adequate for manual control. However, it is needless to emphasize that installation of adequate instruments justify the cost in terms of productivity and safety. Many batch processes are being controlled in manual manner. But as the scale of plant is getting bigger and bigger, role of instrumentation and control (I&C) is becoming important. This I&C hardware not only results in smoother and efficient plant operation but also gives safer operation and high onstreams factor. Modern chemical plants incorporate sophisticated I&C, thereby centralized operation from a control room is possible. PLC (Programmable Logic Control) and DCS (Distributed Control System) have become common terminology in I&C. Touch screen operation of the panel gives a wealth of information to the operator at his finger tips.

In addition to I&C, P&ID also incorporates other details such as pipeline details, location of valves and fittings, safety valves' locations, etc. In major chemical engineering projects, piping and fittings are classified to cover specific range of pressure and temperature for each fluid. This classification lists material of construction, thickness, types of fittings required, etc. for piping. This classification is mentioned on each pipe symbol on P&ID in addition to the size of the pipe. Many times, fluid flowing in the pipe, is indicated by symbolic letters; e.g. NG for natural gas, S for steam, etc. This helps project engineer to specify the piping material with fittings for procurement with ease.

Apart from instrumentation and control details, many other details are also covered on P&ID. Cold and hot insulation are marked. At times elevation is mentioned when relative height of a specific equipment is important. For example, barometric condenser is installed at 10.5 m height from hotwell water level as standard atmospheric pressure equals 10.33 metres of water column. Similarly, when a pump take suction from a vacuum system, NPSH requirements are to be met which means positive suction is required depending on the vacuum. Deaerator for boiler feed water (BFW) is installed at a height to provide sufficient NPSH to BFW pump. When complete draining of a vessel is desired, slope of specific pipeline is desired which can be indicated on P&ID in a symbolic manner. Man-entry and maintenance requirements are kept in mind while preparing P&ID. For this purpose, purging of vessels with inert gas/compressed air is necessary although this requirement is not indicated on PFD. Location of sample point and their arrangements are also shown on P&ID.

Symbolic presentation on the diagram should be easily understood by the engineer. There are standard symbols for such presentation. All engineering companies have their own standard symbols which are supplied alongwith P&ID.

However, standards are prevalent for the graphical symbols. Among them, following are prominently followed by many.

Indian Standards:

IS: 3232 Graphical Symbols for Process Flow Diagrams

IS: 9446 Graphical Symbols for Pipelines and Instrumentation Diagrams

US Standards:

ANSI Y32.2.3 Graphical Symbols for Pipe Fittings, Valves and Piping

ANSI Y32.2.11 Graphical Symbols for Process Flow Diagrams

ISA 5.5 Graphical Symbols for Process Displays

British Standards:

BS: 1646 1 to 4 Symbolic Representation for Process Measurement, Control Functions and Instrumentation

German Standards:

DIN 19227 P1 to P3 Graphical Symbols and Identifying Letters for Process Measurement and Control Functions

Figures 4.5 (a) and (b) give recommended symbols for equipments while Figs 4.6 (a) to (c) give recommended symbols for piping, instruments and controls which will be generally followed in this book.

Bartow *et al*¹¹ have presented a P&ID for a pilot plant to carry out research and development work relating to manufacture active pharmaceutical ingredients

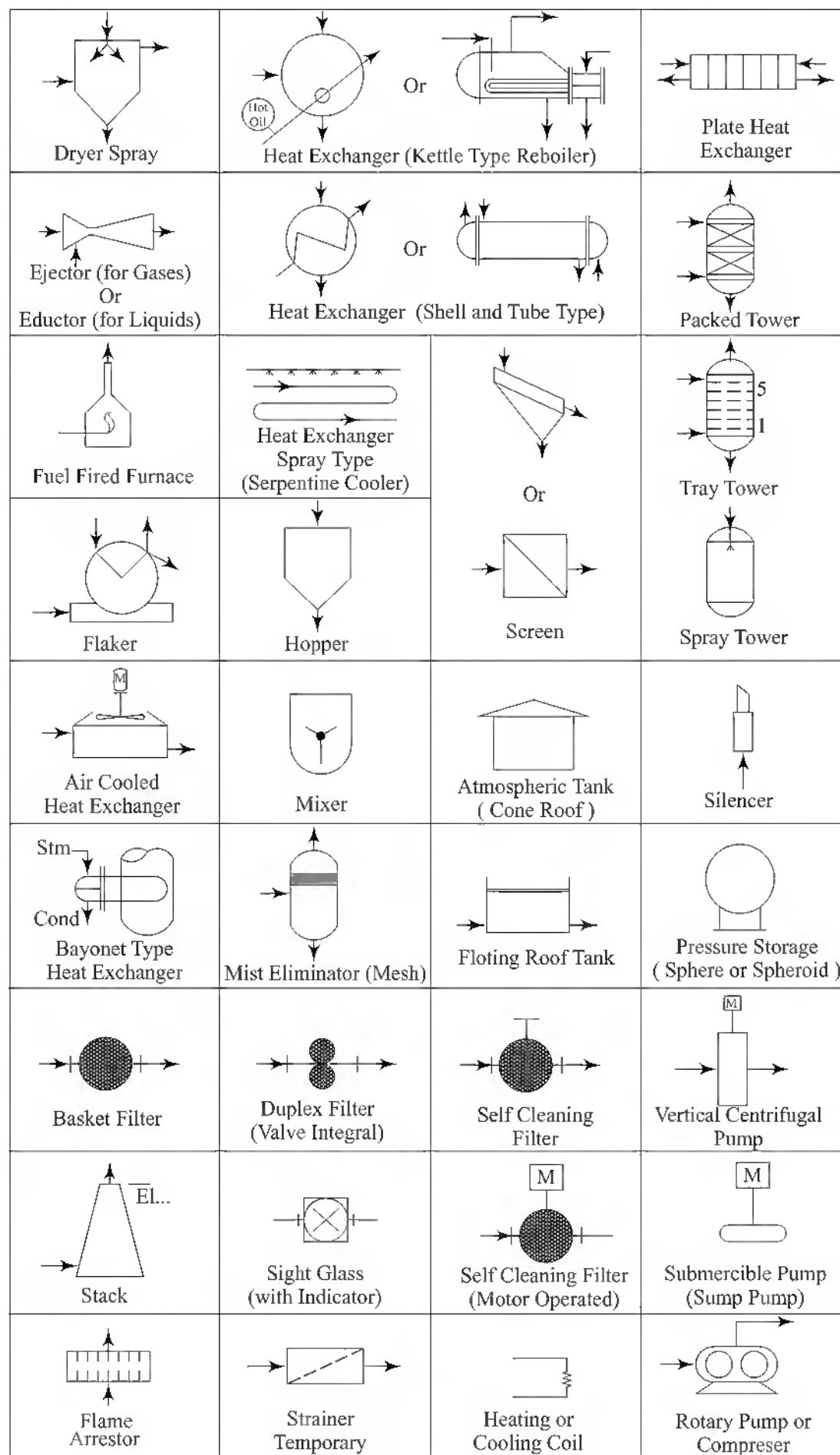


Fig. 4.5(a) Symbols for Equipments

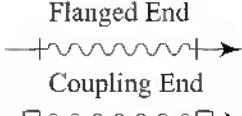
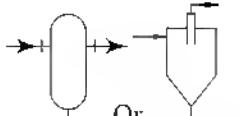
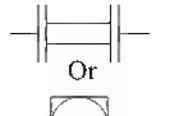
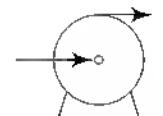
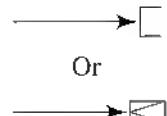
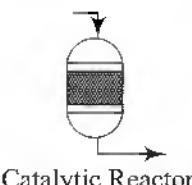
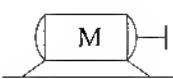
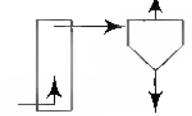
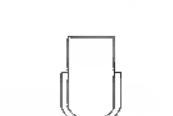
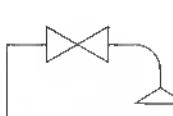
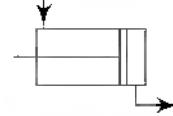
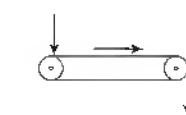
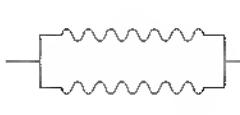
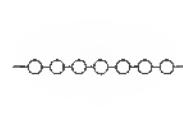
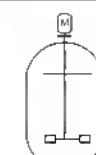
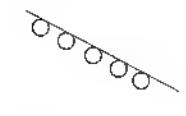
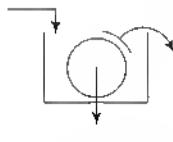
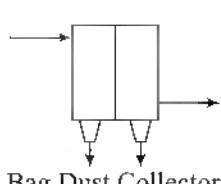
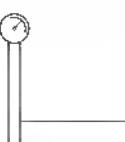
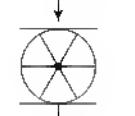
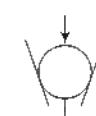
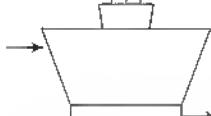
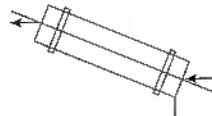
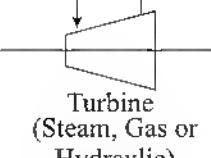
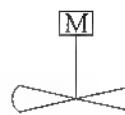
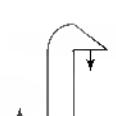
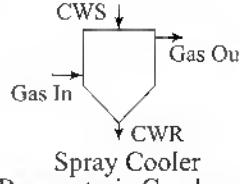
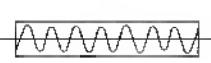
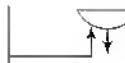
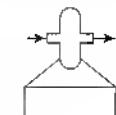
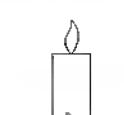
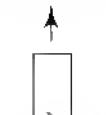
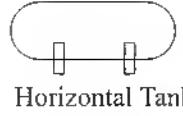
 Hoses	 Or Separator (Steam, Oil or Water)	 Or Sight Glass Plain	 Centrifugal pump Or compressor
 Or Couplings Plain	 Catalytic Reactor	 Motor	 Compressor or Positive Rotary Pump
 Pneumatic Conveyor	 Jacketed Vessel	 Safety Shower	 Reciprocating Compressor or Pump
 Belt Conveyor	 Bellow	 Perforated Pipe	 Agitator (Turbine)
 Roller Conveyor	 Open Rotary Vacuum Filter	 Bag Dust Collector	 Weigh Scale
 Rotary Feeder	 Crusher	 Autoclave	 Drain Point (Closed)
 Cooling Tower	 Rotary Dryer	 Turbine (Steam, Gas or Hydraulic)	 Mechanical Stirrer
 Conveyor (Bucket)	 Gas In Gas Out CWS CWR Spray Cooler (Barometric Condenser)	 Screw Conveyor or Static Mixer	 Eye Wash Fountain
 Gear Pump	 Hot Flare	 Cold Flare	 Horizontal Tank

Fig. 4.5(b) Symbols for Equipments

	Gate Valve Butt Welded Gate Valve Flanged		Diaphragm Operated Butterfly Valve
	Globe Valve Butt Welded		Electric Motor Operated Valve
	Plug Valve		Flush Bottom Tank Valve
	Three Way Plug Valve		Needle Valve
	Ball Valve		Direct Float Operated Valve
	Angle Valve		Piston Operated Valve (Air or Oil)
	Damper Valve		Pressure Reducing Valve
	Diaphragm Valve		Slide Valve
	Butterfly Valve	(a)	Rupture Disc (a) In Line. (b) On Line
	Air Motor Operated Valve		Hose Connection
	Bellows Seal Valve		Vent or Drain With Blind
	Combination of Safety Relief Valve & Rupture Disc Assy		Y-Type Strainer
	Safety Relief Valve with Hand Lever		Valve Closed
	Control Valve		Vent or Drain Plugged
	Diaphragm Operated Control Valve		Check (Non-Return) Valve
	Diaphragm Operated Valve (Angle Type)		Solenoid Operated Gate Valve
	Diaphragm Operated Control Valve (Three Way Type)		Solenoid Operated Gate Valve with Manual Reset
	Main Line		Spectacle Blind (Normally Closed)
	Auxiliary Line		Spectacle Blind (Normally Open)
	Piping Under Ground		Drain
	Insulated Piping		Trap Assembly
	Jacketed Piping		Restriction Orifice and Flange
	Tracing (Process)		Union

Fig. 4.6(a) Symbols for Piping

	Electrically Traced Piping		Reducer (concentric)
	Piping Crossover		Reducer (eccentric)
	Piping Junctions (distinguished from Crossover by Displacement)		Elbow 90° Short Radius ($R = 1 D$)
	Flow Direction (Arrows at Change at Routing)	(a) (b)	Elbow 90° Long Radius ($R = 1.5 D$) (a) Butt Weld (b) Screwed/sw
	Piping Flange		Elbow 45° Screwed or Socket Welded
	Piping Flanged with Blind		Alteration in Pipe Size (only Shown where Essential to Understand Process Requirement)
	Pipe Termination with Cap/BW		Self Actuated Control Valve
	Pipe Termination with Screwed/SW	Piping Identification (Typical) Example: S-3-100-CS1 	
	Pipe Specification Change		
	Boundary by Indication (suitably Closed) Owner Contractor	Note: Process engineer can develop his own piping identification.	

Fig. 4.6(a) Symbols for Piping

A. Instruments	
B. Instruments Signal Lines	
	Pneumatic (Air) Signal
	Electric Signal
	Instrument Impulse Line
	Instrument Capillary Tube
	Radioactive, Sonic or Light Signal
E/P Potential to Pneumatic Converter	
I/P Current to Pneumatic Converter	
E/I Potential to Current Converter	

Fig. 4.6(b) Symbols for Instrumentation and Controls

C. Instrumentation Symbols			
	Locally Mounted Instrument		
	Main Panel Mounted Instrument		
	Local Panel Mounted Instrument		
D. Process Variable Symbols			
	Instrument Mounted Behind Panel		
	Instrument with Two Functions		
 Examples (a) (b) (c) (d)	Instrument Identification Pressure Indicating Controller No. 51 High and Low Temperature Alarms High and Low Level Alarms and Low Low Level Trip Main Panel Mounted Analyser Indicator and Recorder for Oxygen		
E. Instrument Function Symbols			
Indicator	I	Element	E
Controller	C	Gauge	G
Alarm	A	Well	W
Differential	D	Recorder	R
F. Common Service Fluid Codes			
Instrument Air	IA		
Plant Air	PA		
Process Air	PRA		
Fuel Gas	FG		
Inert Gas/Nitrogen	IG/N		
Vent	V		
Blow Down	BD		
Effluent	EFF		
Drain	DR		
Raw Water	RW		
Demineralized Water	DMW		
Process Water	PW		
Cooling Water	CW		
Hot Water	HW		
Chilled Water	CHW		
Brine	BR		
Steam	S		
Refrigerant	R		
Hot Oil	HO		

Fig. 4.6(c) Symbols for Instrumentation and Controls

(API) in a bulk drug plant. Different types of reactions can be carried out in such a pilot plant with heating and cooling, varying from cryogenic conditions (upto -100°C) to hot conditions (upto $+250^{\circ}\text{C}$). Process engineer can develop P&ID for such an universal facility.

Figure 4.7 is P&ID for formaldehyde manufacturing, described in Fig. 4.2 with recycle option. It is developed to ensure safe and smooth operation of the plant in an efficient manner.

Types of control system, shown in Fig. 4.7 can vary from one process engineer to another. For example, air flow to the reactor can be controlled by a control valve on feed air line (A-3-750-CS1). However, in Fig. 4.7, vent controls the feed air flow. Such a control system is found more reliable and accurate in industries. Also, discharge pressure of the blower could be lower in the suggested scheme. Flow of demineralised (DM) water to the absorber may not be a critical requirement. Manual control could therefore be adequate and costly I&C system for its flow control may not be warranted. Thus, preparation of P&ID provides an opportunity to the process engineer to demonstrate his skills. Further, the diagram has to take care of start-up requirements, emergency response, purging, etc.

In certain plants, universal facilities are required for the manufacture or processing of similar products. For example, manufacture of different amino and alkyd resins call for such an universal facility. Lanson¹² has presented a P&ID for the resin manufacturing either by azeotropic distillation process or by vacuum process. An edible oil refinery in which edible oils of different seeds can be refined also represents a similar case.

Several softwares are available for preparation of P&ID also. Microsoft Visio, Aspen Plus, Smart Sketch, etc. can be used to reduce the time in such preparation.

4.6 CONCLUSION

PFD and P&ID are indispensable as transmitters of information, essential in the design stages. They can also be considered as the instruments of communication. In the final analysis, process diagrams communicate an astonishing amount of engineering information about many things to many groups for a variety of uses.

It is not surprising that preparations of PFD and P&ID have assumed great importance in chemical process industry. Process engineers can produce innovative designs which can be well presented by these diagrams. Time spent on preparation can easily result in cost savings, efficient plant layout and safer plant operation.

EXERCISES

4.1 Prepare P&I diagrams for following cases with safety measures.

- Hydrogen from a bullet at 10 bar g and 40°C is let down to 2 bar g for feeding to edible hydrogenation reactor.
- Carbon dioxide is to be cooled from 110°C to 40°C using cooling water in an intercooler at 68 bar a. Cooling water is supplied at 32°C. Critical pressure and critical temperature of carbon dioxide are 73.77 bar and 31°C, respectively.
- A heat sensitive product is concentrated in a wiped film evaporator (refer Chapter 8) at absolute pressure of 1×10^{-3} Pa and 180°C. Ultra high vacuum is achieved with the help of dry vacuum pump and 3-stage ejector system (with

barometric condenser) in series. A cold trap is provided with brine/steam utilities for knocking off organic vapours, being carried to the vacuum system. Temperature in the evaporator is controlled by thermic fluid, entering at 240°C in the jacket and leaving at 220°C from the jacket. Adequate instrumentation and controls are to be provided for smooth operation.

- (d) Visit a nearby liquefied gas (such as ammonia, carbon dioxide, LPG, etc.) storage facility, study the same and prepare a P&I diagram of the system, including storage tank and holding refrigeration system.
- 4.2 A pilot plant reactor (SS 316L) of 250 L geometric capacity is to be designed for conducting sponsored research at any temperature, ranging from –80°C to +220°C. For this purpose, it is desired to use a specialty synthetic (silicone polymer based) heat transfer medium, such as Syltherm XLT® of Dow Chemical Co., USA. Typical properties of Syltherm XLT are as under.

Chemical: Dimethyl polysiloxane

Average molar mass: 317

Flash point: 54°C

Auto ignition temperature: 350°C

Freezing point: Less than –110°C

Density @ 25°C: 0.852 kg/L

Gross heat of combustion at 25°C: 32 800 kJ/kg

The reactor will be suitable for maximum operating pressure of 25 bar g. It will be provided with a turbine agitator with a variable speed drive for speed control in the range, 80–200 rpm. A flame proof electric motor of 3 kW capacity, operating on $415 \pm 10\%$ V, 3 phase, 50 ± 2 Hz power will be used as a drive. Agitator will be sealed with a mechanical seal with cooling arrangement with cooling water circulation.

The reactor will also be suitable for operation under vacuum upto 700 torr. Reflux arrangement with two condensers; one using cooling water at 32°C and another vent condenser, using brine at –10°C, will be provided.

Heat transfer medium (HTM) will be circulated in the jacket with the help of a gear pump. A degassing tank of 100 L capacity will be provided at 2 m elevation relative to the outlet nozzle of the reactor.

- (a) HTM will be cooled from –40°C to –100°C with liquid nitrogen in a welded plate heat exchanger (PHE).
- (b) HTM will be cooled with the help of refrigerant R–22 in the temperature range –40°C and +40°C in a welded PHE.
- (c) HTM will be cooled with cooling water at 32°C in the temperature range +40°C and +80°C in a shell and tube heat exchanger.
- (d) HTM will be heated upto +160°C with saturated steam at 7 bar g in a shell and tube heat exchanger.
- (e) HTM will be heated beyond +160°C upto +220°C with the help of thermic fluid, supplied at +280°C in a shell and tube heat exchanger.

Heat transfer duty will be 5 kW for each case.

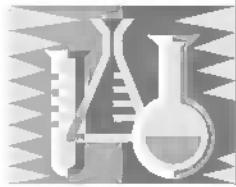
Based on above information, develop a P&I diagram of the above system.

- 4.3 Prepare a PFD for the manufacture of nitric acid based on stoichiometric calculations, given in Examples 2.9 and 2.10.

REFERENCES

1. Winnick, J., *Chem. Engg. Prog.*, **86** (1), 1990, p. 41.
2. Thodos, G. and L. F. Stutzman, *Ind. Engg. Chem.*, **50** (3), 1958, p. 413.
3. Prugh, R. W., *Chem. Engg. Prog.*, **70** (11), 1974, p. 72.
4. Bhatt, B. I. and S. M. Vora, *Stoichiometry*, 4th Ed., Tata McGraw-Hill Publishing Co. Ltd., New Delhi, 2004.
5. Guccione, E., *Chem. Engg.*, **72** (4), 1965, p. 144.
6. Slack, J. B., *Chem. Engg.*, **79** (20), 1972, p. 107.
7. Liu, Y. A., B. Lucas and J. Mann, *Chem. Engg.*, **111** (1), Jan. 2004, p. 30.
8. Westerburg, A.W., H. P. Hutchison, R. L. Motard, P. Winter, *Process Flowsheeting*, Cambridge University Press, UK, 1980.
9. Turner, J., *Chem. Engg. Progr.*, **88** (8), 1992, p. 59.
10. Julian, F.M., *Chem. Engg. Progr.*, **81** (9), 1985, p. 35.
11. Bartow, M. and E. Spark, *Chem. Engg.*, **111** (1), Jan. 2004, p. 49.
12. Swaraj Paul, *Surface Coating; Science & Technology*, 2nd Ed., John Wiley & Sons, USA, 1985, p. 127.

Chapter



5

Process Design of Piping, Fluid Moving Devices and Flow Meters

5.1 INTRODUCTION

A fluid is a substance which when in static equilibrium cannot sustain tangential or shear forces. This property of action under stress distinguishes it from the ‘solid’ state of the matter. Fluids are commonly divided into two substances; liquids and gases. A liquid occupies a definite volume, independent of the dimensions of its container while a gas tends to expand to fill any container in which it is contained. A vapour differs from a gas by being readily condensable to a liquid on cooling.

Strictly speaking all fluids are compressible. However, gases are frequently regarded as compressible while liquids are as incompressible as the latter undergoes small changes in the properties under compression. Ambient air is treated as incompressible fluid as its density changes insignificantly with minor changes in atmospheric pressure and temperature. When compressed, air is treated as a compressible fluid.

In a pump, a fan or a compressor, mechanical work is transformed into fluid energy. A turbine is a machine for transforming fluid energy into mechanical work at the rotating shaft. The word ‘pump’ is normally used for a machine handling liquid. In a fan the density change is so small, due to relatively low pressure difference, that the gas is regarded as incompressible for evaluation. In a compressor, the density change is appreciable.

This chapter deals with fluids, its transportation through various moving devices and various flow measuring instruments.

5.2 PROCESS DESIGN OF PIPING

Design of piping system incorporates all major disciplines of engineering like chemical engineering, mechanical engineering, civil engineering, instrumentation and control engineering, electrical engineering, etc.

Process design of pipe is a balance between size or diameter of pipe and pressure drop in the pipe. For a given flow rate of fluid if larger size of pipe is selected then it gives lesser pressure drop. Larger size of pipe increases the fixed cost of pipe and lesser pressure drop means lesser power consumption or lesser operating cost of pipe. Lesser pressure drop in pipe may reduce the size of flow moving device like pump and thereby it may reduce the fixed cost of pump. Thus capital cost of pipe increases with diameter, whereas pumping or compression cost decrease with increasing diameter. Ideally one should find the pipe size which gives the sum of amortized capital cost plus operating cost a minimum or one should find the optimum pipe size.

5.2.1 Optimum Pipe Size

Several equations and nomographs are available to estimate the optimum pipe diameter. But these equations or nomographs are not reliable as optimum pipe diameter depends on the current cost of material, cost of power, rate of interest at particular place and at particular time. It changes with place and time. For example, optimum pipe diameter equation, derived in USA, cannot be used to find the same in India. One such equation¹ derived for carbon steel pipe and turbulent flow is

$$d_{\text{opt}} = 293 G^{0.53} \rho^{-0.37} \quad (5.1)$$

where, d_{opt} = Optimum pipe size, mm

G = Flow rate, kg/s

ρ = Density of fluid, kg/m³

5.2.2 Recommended Fluid Velocities in Pipe

The velocities, recommended in Table 5.1, are only for approximate calculations of pipe diameter which can be used for the quick calculation of short distance pipe line or for estimating pipe size as a starting point for pressure drop calculations.

5.2.3 Pressure Drop in Pipe

Relation between pressure drop and pipe diameter is given by Fanning or Darcy equation. It is derived for steady flow in uniform circular pipes running full of liquid under isothermal conditions.

$$\frac{\Delta p}{L} = \frac{2f v^2 \rho}{g_c \cdot D_i} = \frac{32f G^2}{\pi^2 \rho g_c D_i^5} \quad (5.2)$$

For SI units, g_c = Newton's law conversion factor = 1

where, Δp = Pressure drop, Pa

L = Length of pipe, m

G = Mass flow rate of fluid, kg/s

ρ = Density of fluid, kg/m³

D_i = Pipe inside diameter, m

v = Velocity of fluid, m/s

f = Fanning friction factor

Table 5.1 Recommended Fluid Velocities

Fluid		Recommended Velocity, m/s
Water	Pump suction line	0.3 to 1.5
	Pump discharge line	2 to 3
	Average service	1 to 2.5
Steam	0 to 2 atm g, saturated	20 to 30
	2 to 10 atm g, saturated	30 to 50
	Superheated below 10 atm g	20 to 50
	Superheated above 10 atm g	30 to 75
	Vacuum lines	100 to 125
Air	0 to 2 atm g	20
	> 2 atm g or above	30
Ammonia	Liquid	1.8
	Gas	30
Organic liquids and oils		1.8 to 2
Natural gas		25 to 35
Chlorine	Liquid	1.5
	Gas	10 to 25
Hydrochloric acid	Liquid	1.5
	Gas	10
Inorganic liquids		1.2 to 1.8
Gas and vapours		15 to 30

The Fanning friction factor is a function of the Reynolds number (Re) and the roughness of inside surface (ϵ).

Values of ϵ for various materials are given in Table 5.2.

Table 5.2 Values of Surface Roughness for Various Materials

Material	Surface Roughness ϵ , mm
Commercial steel or Wrought iron	0.045 72
Galvanized iron	0.152
Cast iron	0.259
Concrete	0.305 – 3.05
Riveted steel	0.914 – 9.14
Brass, Lead, Glass, Cement and Bituminous Linings	0.001 524

A plot of fanning friction factor as a function of Reynolds number (Re) and relative roughness, ϵ/D_i is given as Fig. 5.1.

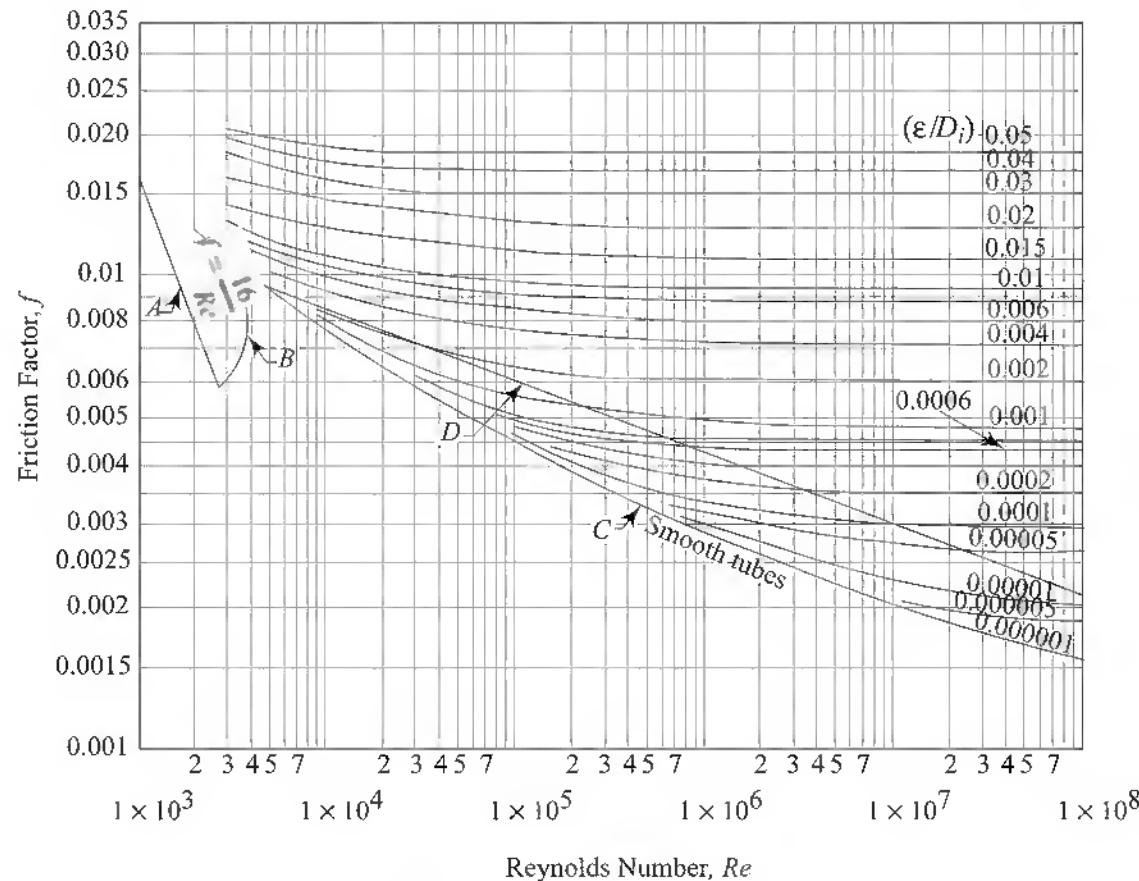


Fig. 5.1 Fanning Friction Factors²
(Reproduced with the Permission of McGraw-Hill Education, USA)

For turbulent flow in commercial steel pipe, $f = 0.0394 Re^{-0.16}$ (5.3a)

Above equation gives the value f on higher (safe) side. One of the more accurate relationships between f and Re for turbulent flow (Ref.: 3)

$$\frac{1}{\sqrt{f}} = -1.7372 \ln \left(\frac{Re}{1.964 \ln(Re) - 3.8215} \right) \quad (5.3b)$$

$$\text{where, } Re = \frac{D_i v \rho}{\mu} = \frac{4G}{\pi D_i \mu} \quad (5.4)$$

μ = Viscosity of fluid

From Eqs. (5.2), (5.3a) and (5.4),

$$\frac{\Delta p}{L} = \frac{32}{\pi^2} \times \frac{0.0394 Re^{-0.16} G^2}{\rho g_c D_i^5}$$

$$\frac{\Delta p}{L} = 0.1277457 \left(\frac{4G}{\pi D_i \mu} \right)^{-0.16} \left(\frac{G^2}{\rho D_i^5} \right)$$

$$\frac{\Delta p}{L} = 0.122\ 9024\ G^{1.84}\ \mu^{0.16}\ D_i^{-4.84}\ \rho^{-1}$$

If G in kg/s, μ in kg/(m · s) or in N · s/m², D_i in m, ρ in kg/m³, L in m, then Δp is in Pa or in N/m²,

Normally pipe diameters are expressed in mm. If D_i is in mm then

$$\frac{\Delta p}{L} = 0.122\ 9024 \times 1000^{4.84}\ G^{1.84}\ D_i^{-4.84}\ \rho^{-1} \cdot \mu^{0.16}$$

$$\frac{\Delta p}{L} = 4.07 \times 10^{13}\ G^{1.84}\ \mu^{0.16}\ D_i^{-4.84}\ \rho^{-1}$$

If Δp is in kPa or in kN/m², then

$$\frac{\Delta p}{L} = 4.07 \times 10^{10}\ G^{1.84}\ \mu^{0.16}\ D_i^{-4.84}\ \rho^{-1} \quad (5.5)$$

Example 5.1

Estimate the optimum pipe diameter for a flow of dry chlorine gas of 10 000 kg/h at 6 atm a and at 20°C through carbon steel pipe using Eq. (5.1).

Solution: For carbon steel pipe, optimum pipe diameter

$$d_{\text{opt}} = 293\ G^{0.53}\ \rho^{-0.37} \quad (5.1)$$

$$G = 10\ 000\ \text{kg/h} = 2.7778\ \text{kg/s}$$

$$\rho = \frac{pM}{RT} = \frac{pM}{T} \times \frac{T_s}{p_s V_s} = \frac{6 \times 71}{(273 + 20)} \times \frac{273}{(1 \times 22.4136)}$$

$$\rho = 17.71\ \text{kg/m}^3$$

$$d_{\text{opt}} = 293(2.7778)^{0.53}(17.71)^{-0.37} \\ = 173.855\ \text{mm}$$

Use next higher size of standard pipe, i.e. 200 mm (8 in) NB standard pipe.

5.2.4 Standard Pipes

Standard pipes are available from 3 mm (1/8 in) to 600 mm (24 in) size. Standard pipes are specified with three different diameters, namely inside diameter, outside diameter and nominal diameter. For standard pipes having diameter more than 300 mm (12 in), nominal diameters are equal to the actual outside diameter but for smaller pipe there is no relation between nominal diameter and inside diameter or outside diameter.

The wall thickness of standard pipe is indicated by the schedule number as per the US standards.

$$\text{Schedule Number, SCH} = \frac{p_{sf} \times 1000}{\sigma_s} \quad (5.6)$$

where, p_{sf} = Safe working pressure, N/mm²
 σ_s = Safe allowable stress, N/mm²

Standard pipes are available with variety of schedule numbers; 5, 10, 20, 30, 40, 60, 80, 100, 120, 140, 160. With carbon steel pipe, schedule 40 upto 150 mm (6 in) and with stainless steel pipe, schedule 10 are used for general purposes. Thickness of standard pipe increases with increases in schedule number. Seamless standard pipes do not have welding joint. Fabricated pipes are fabricated by rolling the plates followed by joining the ends of plate by welding which are also known as Electric Resistance Welded (ERW) pipes.

In metric system (including those specified in Indian Standards), thickness of a pipe is specified in mm.

Thickness of pipe, subjected to internal pressure, is determined by equation.

$$t = \frac{pr_i}{\sigma E - 0.6p} + CA = \frac{pr_o}{\sigma E + 0.4p} + CA \quad (5.7)$$

where, t = Thickness of pipe, mm

p = Internal design pressure, N/m²

r_i = Inside radius of pipe, mm

r_o = Outside radius of pipe, mm

σ = Allowable stress of pipe material at design temperature, N/m²

E = Joint efficiency; for seamless standard pipe $E = 1$

CA = Corrosion allowance, mm

5.2.5 Pressure Drop in Fittings and Valves

In addition to pipes, piping system contains fittings and valves. These fittings and valves offer the additional frictional loss or additional pressure drop. This additional frictional loss of a fitting or of a valve is expressed either as an equivalent straight pipe length (L_e) or as a number of velocity heads (K), lost in pipe of same size and of same material.

5.2.5.1 Equivalent Length of Pipe (L_e)

Equivalent length of a valve or of a fitting is the length of straight pipe of same size creating the same friction loss as the fitting or valve in consideration. Nomographs for finding equivalent length of pipe for various fittings and valves are given in various books (Ref. 4, 5 and 6).

Often, L_e is expressed in terms of inside diameter of pipe. Then $L_e = \frac{L_e}{D_i} D_i$

where, D_i = Inside diameter of pipe.

Values of L_e/D_i for few valves and fittings are given in Table 5.3.

Table 5.3 Equivalent Lengths of Fittings and Valves

Valve or fitting	$\frac{L_e}{D_i} = \frac{\text{Equivalent length}}{\text{Inside diameter}}$
Gate valve (fully open)	7 to 10
Gate valve (3/4 closed)	800 to 1100
Gate valve (1/2 closed)	190 to 290
Globe valve (fully open)	330 to 480
Angle valve, (fully open)	165 to 220
Plug valve, (fully open)	18
90° elbows (standard radius)	30
45° elbows (long radius)	5.8
45° elbows (short radius)	8.0
Return bend (medium radius)	39 to 56
Coupling or union	Negligible
Tee, straight through	22

Another way of calculating pressure drop through the fittings and valves is the use of factor K .

“Number of velocity heads (K) lost in pipe” for fittings or valves is defined by equation

$$\frac{\Delta F}{v^2/2g_c} = \frac{\Delta p/\rho}{v^2/2g_c} \quad (5.8)$$

$$\text{or } \Delta p = \frac{K\rho v^2}{2g_c}, \text{ in SI system, } g_c = 1 \quad (5.9)$$

where, ΔF = Additional frictional loss, J/kg or N · m/kg

Δp = Additional pressure drop, N/m²

v = Average fluid velocity, m/s

Values of K for normally used fittings and valves are given in Table 5.4.

Table 5.4 Equivalent Velocity Heads of Fittings and Valves

Type of fitting or valve	Equivalent number of velocity heads (K) (applicable only for turbulent flow)
Gate valve (open)	0.17
Globe valve, bevel seat (open)	6.0
Plug valve (open)	9.0
Diaphragm valve (open)	2.3
Angle valve (open)	2.0
Check valve (swing)	2.0
Foot valve	15
Coupling	0.04
90° elbows (standard)	0.75
90° elbows (long radius)	0.45

(Contd.)

Table 5.4 (Contd.)

45° elbows (long radius)	0.2
45° elbows standard	0.35
Tee straight through	0.4
Butterfly valve ($\alpha = 5^\circ$)	0.24

Simpson⁷ has given values of K for variety of other fittings, such as miter bends, different types of reducers, different bends, etc.

Example 5.2

Carbon dioxide is to be conveyed from the top of the stripper of ammonia plant to urea plant. Calculate the pipe size required based on following data.

Flow rate of CO₂ = 1000 t/day

Total length of pipe = 800 m

Available pressure at inlet of pipe = 24 kPa g

Discharge pressure of CO₂ from pipe required = atmospheric

No. of 90° elbows in pipe line = 8

No. of butterfly valve = 1

No. of flow nozzle = 1

Temperature of gas = 60°C

Viscosity of CO₂ gas = 0.016 mPa · s or cP

Solution:

Density of carbon dioxide (CO₂) gas:

$$\rho = \frac{pM}{RT}$$

$p = 1$ atm, Molar mass of CO₂, $M = 44$

$$R = 0.0821 \frac{\text{atm} \cdot \text{m}^3}{(\text{kmol} \cdot \text{K})}, T = 273 + 60 = 333 \text{ K}$$

$$\rho = \frac{1 \times 44}{0.0821 \times 333} = 1.6094 \text{ kg/m}^3$$

$$\text{Mass flow rate of CO}_2, \dot{m} = \frac{1000 \times 10^3}{24 \times 3600} = 11.574 \text{ kg/s}$$

Let the velocity of gas through pipe, $v = 20 \text{ m/s}$

$$\begin{aligned} \frac{\pi}{4} D_i^2 &= \frac{\dot{m}/\rho}{v} \\ &= \frac{11.574/1.6094}{20} = 0.3596 \text{ m}^2 \end{aligned}$$

$$D_i = 0.677 \text{ m}$$

Selecting fabricated pipe having inside diameter, $D_i = 677 \text{ mm}$

Reynolds number

$$Re = \frac{D_i v \rho}{\mu} = \frac{0.677 \times 20 \times 1.6094}{0.016 \times 10^{-3}}$$

= 1361 955 > 10 000 (i.e. turbulent flow)

Pressure drop in straight steel pipe for turbulent flow

$$\begin{aligned}\frac{\Delta p}{L} &= 4.07 \times 10^{10} G^{1.84} \mu^{0.16} D_i^{-4.84} \rho^{-1} \\ &= 4.07 \times 10^{10} (11.574)^{1.84} (0.016 \times 10^{-3})^{0.16} (677)^{-4.84} (1.6094)^{-1} \\ &= 7.8046 \times 10^{-3} \text{ kPa/m} \equiv 7.8046 \text{ Pa/m}\end{aligned}\quad (5.5)$$

Pressure drop in butterfly valve:

$$\begin{aligned}\Delta p_{E1} &= \frac{K_i \rho v^2}{2} = \frac{0.24 \times 1.6094 \times 20^2}{2} \\ &= 77.25 \text{ Pa}\end{aligned}$$

Pressure drop in 90° elbow (standard):

$$\begin{aligned}\Delta p_{E2} &= \frac{K_i \rho v^2}{2} = \frac{0.75 \times 1.6094 \times 20^2}{2} \\ &= 241.41 \text{ Pa}\end{aligned}$$

Pressure drop in the flow nozzle is considered negligible.

Total pressure drop in pipe line

$$\Delta p_t = 7.8046 \times 800 + 77.25 + 8 \times 241.41 = 8252 \text{ Pa} \equiv 8.25 \text{ kPa}$$

Available pressure is 24 kPa. Hence, to decrease the fixed cost, smaller pipe can be considered. Similar calculations for smaller pipe sizes give the following results.

Table 5.5 Comparison of Pressure Drop Calculations

Nominal Size	Schedule No.	ID of Pipe, mm	Δp , kPa
677 mm	—	677	8.25
600 mm (24 in)	20	590.55	15.558
500 mm (20 in)	20	488.95	37.525

Select 600 mm, SCH-20 pipe size for the given duty.

Example 5.3

Calculate the pipe size based on following data. Fluid flowing through pipe is carbon monoxide. Discharge pressure of carbon monoxide required from the pipe is atmospheric.

Available pressure at inlet of pipe = 50 kPa g

Length of pipe = 4 km

Flow rate of CO = 1500 kg/h

Temperature of gas = 50° C

No. of gate valves in pipe line = 2

No. of 45° elbows = 3

Nos. of 90° elbows = 6

Viscosity of CO = 0.018 mPa · s or cP

Solution:

Density of carbon monoxide (CO) gas:

Molar mass of CO, $M = 28$

$$\rho = \frac{pM}{RT}$$

$$\begin{aligned} &= \frac{pM}{T} \times \frac{T_s}{p_s V_s} = \frac{1 \times 28}{(273 + 50)} \times \frac{273}{(1 \times 22.414)} \\ &= 1.0558 \text{ kg/m}^3 \end{aligned}$$

Pressure drop in straight pipe for steel material and for turbulent region:

$$\frac{\Delta p}{L} = 4.07 \times 10^{10} G^{1.84} \mu^{0.16} D_i^{-4.84} \rho^{-1} \quad (5.5)$$

For the first trial calculations let the nominal size be 12 in NB (300 mm), SCH-40 pipe having inside $D_i = 304.8 \text{ mm}$.

$$\begin{aligned} \frac{\Delta p_1}{L} &= 4.07 \times 10^{10} \left(\frac{1500}{3600} \right)^{1.84} (0.018 \times 10^{-3})^{0.16} \times (304.8)^{-4.84} \times (1.0558)^{-1} \\ &= 1.2724 \times 10^{-3} \text{ kPa/m} \equiv 1.2724 \text{ Pa/m} \end{aligned}$$

$$\begin{aligned} \text{Reynolds numbers, } Re &= \frac{4\dot{m}}{\pi D_i \mu} = \frac{4 \times \left(\frac{1500}{3600} \right)}{\pi \times 0.3048 \times 0.018 \times 10^{-3}} \\ &= 96\,697 > 10\,000, \text{ i.e. CO is flowing in turbulent region} \end{aligned}$$

$$\text{Velocity of gas, } v = \frac{\dot{m}}{\rho \times \frac{\pi}{4} D_i^2} = \frac{1500/3600}{1.0558 \times (\pi/4)(0.3048)^2} = 5.4 \text{ m/s}$$

For gate valve,

$$\Delta p_{E1} = \frac{K_i \rho v^2}{2} = \frac{0.17 \times 1.0558 \times 5.4^2}{2} = 2.617 \text{ N/m}^2 \text{ per gate valve}$$

$$\begin{aligned} \text{Similarly for } 45^\circ \text{ elbows, } \Delta p_{E2} &= \frac{K_i \rho v^2}{2} = \frac{0.35 \times 1.0558 \times 5.4^2}{2} \\ &= 5.388 \text{ N/m}^2 \text{ per } 45^\circ \text{ elbow} \end{aligned}$$

$$\text{For } 90^\circ \text{ elbows, } \Delta p_{E3} = \frac{K_i \rho v^2}{2} = \frac{0.75 \times 1.0558 \times 5.4^2}{2} = 11.545 \text{ N/m}^2 \text{ per } 90^\circ \text{ elbow}$$

Total pressure drop in pipe line

$$\Delta p_t = 1.2724 \times 4000 + 2.617 \times 2 + 5.388 \times 3 + 11.545 \times 6$$

$$\Delta p_t = 5180.27 \text{ Pa} \equiv 5.18 \text{ kPa} \equiv 0.0511 \text{ atm}$$

For 300 mm NB SCH-40 pipe, pressure drop in piping is very low. Hence, to reduce the fixed capital investment, smaller pipe diameter can be considered. Similar calculations of smaller pipe diameters gives the following results.

Table 5.6 Comparison of Pressure Drop Calculations

Nominal Size	Schedule No.	ID of Pipe, mm	Δp_f , kPa	Remarks
100 mm (4 in)	SCH-40	102.26	1012.38	Very high
150 mm (6 in)	SCH-40	154.05	139.75	Higher
200 mm (8 in)	SCH-40	202.72	37.10	Reasonable
300 mm (12 in)	SCH-40	304.80	5.18	Very low

Considering fixed capital investment, operating cost and safety margin in design, 200 mm (8 in) SCH-40 pipe can be selected for the given application. For this size of pipe, velocity of gas through pipe is equal to 12.23 m/s which is acceptable (refer Table 5.1).

Example 5.4

100 000 kg/h of water is to be transferred from canal to reservoir by gravity. Maximum height of water level above the discharge of pipe in reservoir will be 2 m. Difference between minimum level of water in canal and maximum level of water in reservoir is 8 m.

Length of pipe = 3000 m

Equivalent length of pipe for fittings and valves = 200 m

Maximum temperature of water = 40°C

Density of water at 40°C = 993 kg/m³

Viscosity of water at 40°C = 0.67 mPa · s or cP

Surface roughness for carbon steel, ϵ = 0.0457 mm

Surface roughness for concrete, ϵ = 1.2 mm

Material of pipe is carbon steel. Determine the suitable pipe size. If material of pipe is concrete will there be any change in pipe size required?

Solution:

Maximum allowable pressure drop in pipe = 8 – 2 = 6 m WC

Δp_{\max} = 6 m WC = 58.84 kPa = 0.58 atm

Let the velocity of water through pipe v = 0.5 m/s (For first trial calculation)

$$v = 0.5 \text{ m/s} = \frac{\dot{m}/\rho}{(\pi/4) D_i^2} = \frac{(100\ 000)/3600}{993 \times (\pi/4) D_i^2}$$

$$D_i = 0.2669 \text{ m} \equiv 266.9 \text{ mm}$$

Select 300 mm NB, SCH-40 carbon steel pipe.

ID of pipe, D_i = 304.8 mm, μ = 0.67×10^{-3} kg/(m · s)

$$\text{Reynolds number, } Re = \frac{4G}{\pi D_i \mu} = \frac{4 \times (100\ 000/3600)}{\pi \times 0.3048 \times 0.67 \times 10^{-3}} = 173\ 188$$

$$\frac{\epsilon}{D_i} = \frac{0.0457}{304.8} = 1.499 \times 10^{-4}$$

From Fig. 5.1, $f = 0.0042$

$$\text{Pressure drop in pipe, } \Delta p = \frac{32f G^2}{\pi^2 \rho g_c D_i^5} \times L \quad (5.2)$$

$$\Delta p = \frac{32 \times 0.0042 \times (100\,000/3600)^2 \times (3000 + 200)}{\pi^2 \times 993 \times 1 \times 0.3048^5}$$

$$\Delta p = 12\,871.2 \text{ Pa} \equiv 12.871 \text{ kPa} < \Delta p_{\max}$$

For the next smaller size of pipe, i.e. for 250 mm NB, SCH-40 pipe, $D_i = 254.5 \text{ mm}$, $Re = 207\,417.3$,

$$\frac{\epsilon}{D_i} = 1.7956 \times 10^{-4}, \quad f = 0.0041 \text{ and } \Delta p = 30.959 \text{ kPa} < \Delta p_{\max}$$

Use 250 mm NB SCH-40 carbon steel pipe.

If pipe material is concrete, then for the same size

$$\frac{\epsilon}{D_i} = \frac{1.2}{254.5} = 4.715 \times 10^{-3} \text{ and } Re = 207\,417.3$$

From Fig. 5.1, $f = 0.0077$

Pressure drop $\Delta p = 58.142 \text{ kPa} \equiv \Delta p_{\max}$

Thus calculated pressure drop is nearly equal to the maximum allowable pressure drop. For safer design, next higher size of concrete pipe, i.e. 300 mm (12 in) pipe, should be used.

Example 5.5

An organic liquid is discharged at the rate of 5000 kg/h from a reactor to a storage tank at 50°C as shown in Fig. 5.2. Reactor is under pressure at 600 kPa g. Density of the organic liquid is 930 kg/m³ and viscosity is 0.91 mPa · s or cP. Assume no flashing of the organic liquid across the control valve.

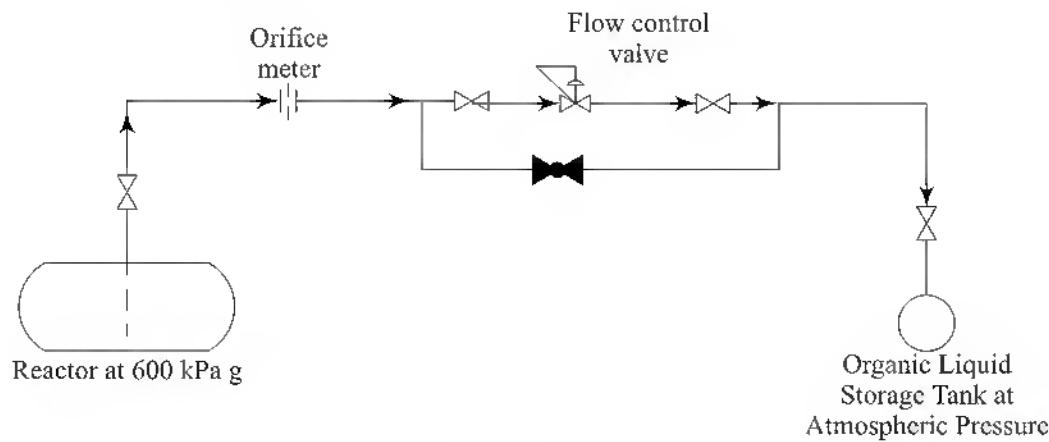


Fig. 5.2 Liquid Flow System

Piping System Details:

Linear length of straight pipe = 50 m

No. of 90° elbows of standard radius = 6 (All are not shown in Fig. 5.2.)

No. of Tees = 2

Pressure drop in orifice meter = 40 kPa

No. of gate valves = 4

No. of globe valve = 1

No. of flow control valve = 1

Determine the pipe size. Assume it to be uniform throughout. Also find the residual pressure drop that must be taken by the flow control valve.

Solution:

Flow rate of liquid, $\dot{m} = 5000 \text{ kg/h}$

Volumetric flow rate of liquid

$$q_v = \frac{\dot{m}}{\rho} = \frac{5000/3600}{930}$$

$$= 1.4934 \times 10^{-3} \text{ m}^3/\text{s}$$

Based on Table 5.1, select velocity of liquid to be 1.8 m/s.

Inside diameter of pipe required based on selected velocity

$$D_i = \sqrt{\frac{4 \times q_v}{\pi \times v}}$$

$$= \sqrt{\frac{4 \times 1.4934 \times 10^{-3}}{\pi \times 1.8}} = 0.0325 \text{ m} \equiv 32.5 \text{ mm}$$

Selected 40 mm (1.5 in) NB, SCH-40 pipe having $D_i = 40.894 \text{ mm}$

$$\text{Reynolds number, } Re = \frac{D_i v \rho}{\mu} = \frac{0.040894 \times 1.8 \times 930}{0.91 \times 10^{-3}} = 752.27 \text{ (i.e. turbulent flow)}$$

For the turbulent flow in commercial steel pipe

$$\begin{aligned} \frac{\Delta p}{L} &= 4.07 \times 10^{10} G^{1.84} \mu^{0.16} D_i^{-4.84} \rho^{-1} & (5.5) \\ &= 4.07 \times 10^{10} \left(\frac{5000}{3600} \right)^{1.84} (0.91 \times 10^{-3})^{0.16} \times (40.894)^{-4.84} \times (930)^{-1} \\ &= 0.41365 \text{ kPa/m} \end{aligned}$$

Linear length of pipe = 50 m

Table 5.7 Equivalent Lengths of Fittings and Valves

Type	No.	Pipe ID, mm	L_e , m (From Table 5.3)
90° Elbows	6	40.894	$6(30D_i) = 7.36$
Tees	2	40.894	$2(22D_i) = 1.8$
Gate valves	4	40.894	$4(10D_i) = 1.636$
Total			10.796

Total pressure drop

$$\begin{aligned} \Delta p_t &= 0.41365 (50 + 10.796) + \text{Pressure drop in orifice meter} \\ &= 0.41365 (50 + 10.796) + 40 = 65.15 \text{ kPa} \end{aligned}$$

Pressure balance:

Pressure in Reactor $-\Delta p_r - \Delta p_{\text{control valve}}$ = Pressure in storage tank

$$(600 + 101.325) - 65.15 - \Delta p_c = 101.325$$

$$\Delta p_c = 534.85 \text{ kPa}$$

This residual pressure drop should be offered by the control valve. Specifications of the control valve should be prepared accordingly.

5.3 PROCESS DESIGN OF FLUID MOVING DEVICES

It includes process design of pump, fan, blower and compressor.

5.3.1 Process Design of Pump

Pump is a device used in flow system of liquid to increase the mechanical energy of the flowing liquid. Pumps are broadly divided into two types; (i) centrifugal pumps and (ii) positive displacement pumps. The following terminologies are used with pump.

- (i) Capacity of pump : Flow rate (q_v) of fluid created by the pump is known as capacity of pump. In SI units, capacity is expressed in m^3/h or L/s.
- (ii) Total Dynamic Head : Total dynamic head H of a pump is the difference between total discharge head h_d and total suction head h_s .

$$H = h_d - h_s \quad (5.10)$$

Before installation of pump it is possible to estimate the total discharge head h_d by using following equation.

$$h_d = h_{sd} + h_{fd} \quad (5.11)$$

where, h_{sd} = Static discharge head = $p' \pm Z'$ (5.12)

p' = Absolute pressure over free surface of liquid in receiver

Z' = Vertical distance between free surface of liquid in receiver and centre line of pump placed horizontally (for vertical pump Z' is distance between free surface of liquid and eye of suction of impeller)

$h_{sd} = p' + Z'$, if pump is below the level of free surface of liquid in receiver and $h_{sd} = p' - Z'$, if it is placed above the free surface of liquid in receiver.

h_{fd} = Discharge friction head = frictional loss in discharge line.

During operation, total discharge head h_d can be determined by following equation.

$$h_d = h_{gd} + \text{atm pressure} + h_{vd} \quad (5.13)$$

where, h_{gd} = Discharge gauge pressure measured by pressure gauge. If pressure is below atmospheric, vacuum gauge reading is used for h_{gd} in Eq. (5.13), but with negative sign.

h_{vd} = Velocity head at the point of gauge attachment in discharge line

Before installation of pump, total suction head can be estimated by following equation

$$h_s = h_{ss} - h_{fs} \quad (5.14)$$

where, h_{ss} = Static suction head = $p \pm Z$

p = Absolute pressure over free surface of liquid at source

Z = Vertical distance between free surface of liquid at source and center line of horizontal pump (or suction eye impeller for vertical pump)

$h_{ss} = p + Z$, if pump is going to be installed below the free surface of liquid

$h_{ss} = p - Z$, if pump is going to be installed above the free surface of liquid

h_{fs} = Friction loss in suction line

In existing installation, total suction head h_s can be determined by following equation.

$$h_s = \text{atm pressure} + h_{gs} + h_{vs} \quad (5.15)$$

where, h_{gs} = suction gauge pressure,

h_{vs} = velocity head at the point of gauge attachment.

(iii) NPSH

Net positive suction head is the excess of sum of velocity head and pressure head in suction line over the vapour pressure head of liquid. When pump installation is designed, available net positive suction head ($NPSH_A$) must be equal to or greater than net positive suction head required; ($NPSH_R$).

$(NPSH)_R$ is normally specified by pump supplier, while based on the installation of pump, $(NPSH)_A$ should be calculated and specified by the process engineer. Theoretically $(NPSH)_A$ should be greater than zero to avoid cavitation.

$(NPSH)_R$ depends on properties of liquid, the total head, pump speed, capacity and impeller design. Practical curves of $(NPSH)_R$ vs capacity and speed of pump are supplied by pump manufacturer. Figures 5.3 and 5.4 can be used as a guideline to find the value of $(NPSH)_R$, for centrifugal pump handling water at temperatures below 100°C and above 100°C, respectively.

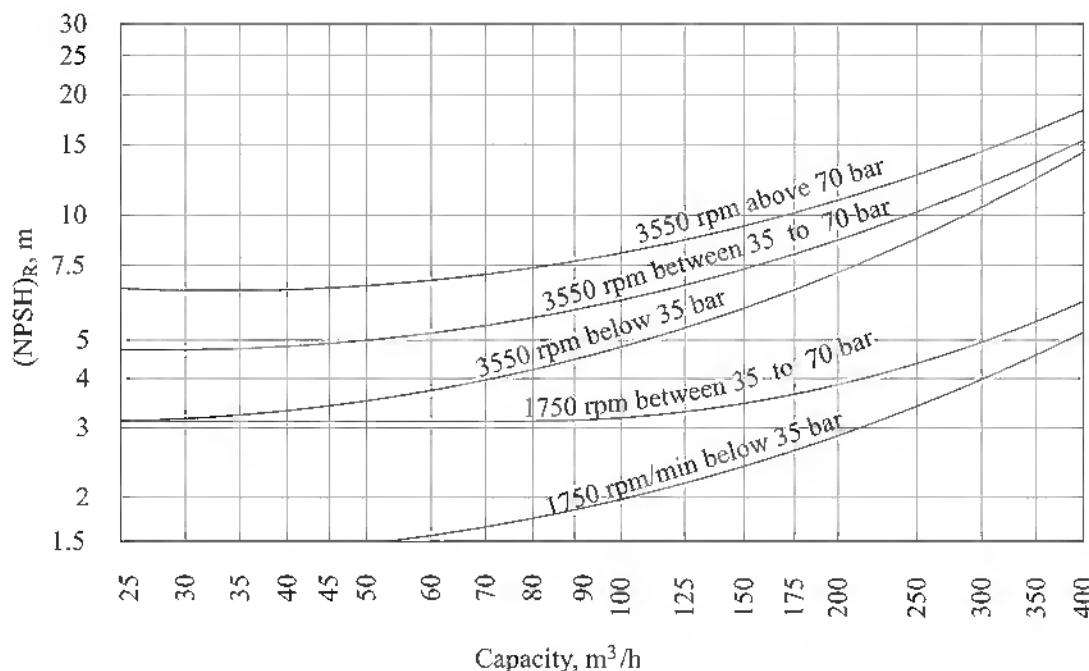


Fig. 5.3 Net Positive Suction Head for High Pressure Centrifugal Hot-Water Pumps⁸
(Reproduced with the Permission of Hydraulic Institute, USA)

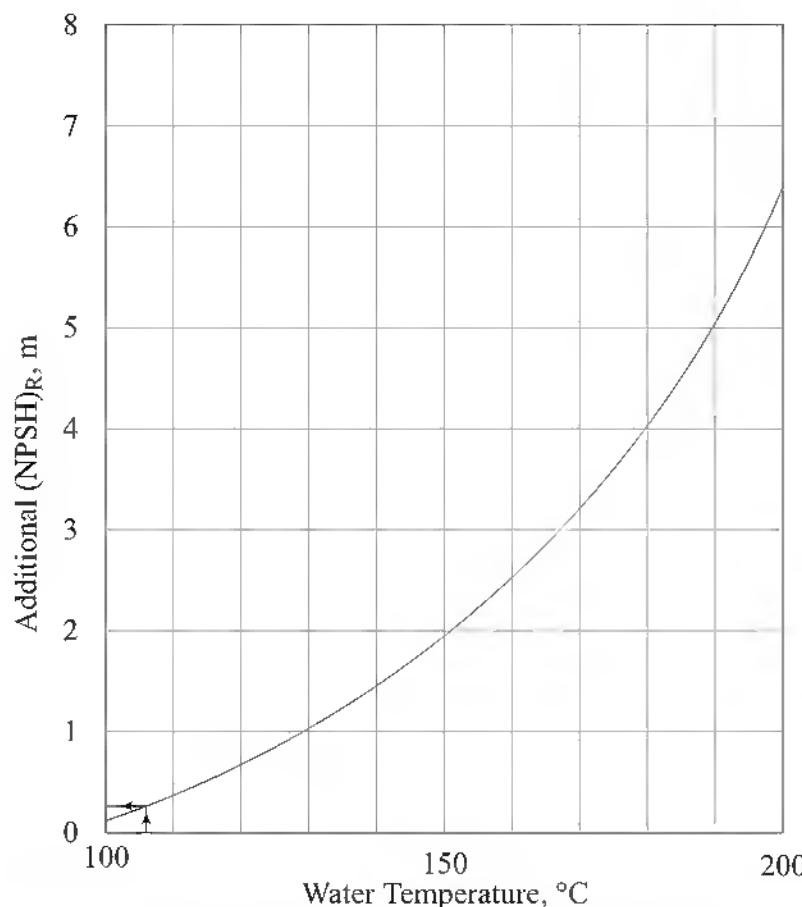


Fig. 5.4 Temperature Correction Chart for Net Positive Suction Head Requirement for Centrifugal Hot Water Pumps⁸
 (Reproduced with the Permission of Hydraulic Institute, USA)

When a pump installation is being designed, the available net positive suction head (NPSH)_A can be calculated by following the equation.

$$(NPSH)_A = h_{ss} - h_{fs} - p_v \quad (5.16)$$

where, h_{ss} = Static suction head, $m = p \pm Z$

h_{fs} = Friction loss in suction line, m of liquid column

p_v = Vapour pressure of liquid at suction temperature expressed in m of liquid column

For existing installation, (NPSH)_A can be determined by following equation

$$(NPSH)_A = \text{atm pressure} + h_{gs} - p_v + h_{vs} \quad (5.17)$$

where, h_{gs} = suction gauge pressure, m of liquid column

p_v = vapour pressure of liquid at suction temperature expressed in m of liquid column

h_{vs} = Velocity head at the point of gauge attachment, m of liquid column.

As a general guide (NPSH)_A should be above 3 m for pump capacities upto a flow rate of 100 m³/h, and 6 m above this capacity.

5.3.2 NPSH Requirement for Liquids Saturated with Dissolved Gases

In many situations, the liquid to be pumped is saturated with gases, which have definite solubilities in the liquid. When a suction system for such a liquid is

to be designed for a centrifugal pump, $(NPSH)_A$ calculations differ from Eqs. (5.16) or (5.17).

Pumping of cooling water (saturated with air), pumping of condensate from a knock-out drum of a compressor, pumping of solution from an absorber, etc. can be cited as examples of such situations where the liquids are saturated with gases.

Dissolved gases start desorbing when the pump is started and suction is generated at the pump eyes. Normally a pump can tolerate 2 to 3% flashed gases at the pump eye without encountering cavitations. If the design of the suction system is made to restrict about 2.5% flash, it is considered safe for the pump operation.

In Eq. (5.16) suction source pressure is the system pressure (p) minus the vapour pressure (p_v) for a normal liquid having practically no dissolved gases. For a liquid saturated with dissolved gases, p_v is replaced by p_{va} which is called artificial liquid vapour pressure. For evaluation of p_{va} , following procedure is recommended.

- Calculate molar mass of the gas mixture, dissolved in the liquid.
 - Calculate mass fraction (w_o) of the dissolved gas mixture.
 - Calculate psuedo critical properties of the dissolved gas mixture, if system pressure is high.
 - Calculate specific volume of the dissolved gas mixture (V_{Ga}) at the operating conditions.
- Steps (a) to (d) can be avoided if the solubility of the gas mixture in the liquid (such as air in water) is known from the literature.
- Calculate volume fraction of the dissolved gas (GVP) in a hypotheoretical gas-liquid mixture. Considering one unit mass of the liquid in which the gas mixture is dissolved. If GVP is less than or equal to 2.5%, Eq. (5.16) can be safely used to calculate $(NPSH)_A$ using vapour pressure (p_v) of the liquid at the operating temperature. If higher than calculate volume fraction (a) of flashed gas mixture (as pressure is lowered) over the liquid, saturated with the dissolved gas mixture, using following equation⁹.

$$a = \frac{1}{\sqrt{\left[\frac{\left(\frac{p}{p_0} - \frac{p_v}{p_0} \right)^2 \left(1 - \frac{p_v}{p_0} \right)}{\left(\frac{V_{Ga}}{V_L} \right) \left(\frac{p}{p_0} \right) \left(1 - \frac{p}{p_0} \right)} + 1 \right]}} \quad (5.18)$$

where, p = liquid pressure at pump eye, kPa

p_v = vapour pressure of liquid at the operating temperature, kPa

p_0 = system pressure, kPa

V_{Ga} = specific volume of the dissolved gas mixture, m^3/kg [see step (d) above]

V_L = specific volume of the liquid at the operating conditions, m^3/kg

Equation (5.18) assumes that the dissolved gas mixture follows ideal gas law, Dalton's law and Henry's law.

- (f) Calculate a for different value of p . Draw a graph of a vs p . Read corresponding to $a = 0.025$ which is called p_{va} . Alternately by trial and error, calculate p_{va} , for $a = 0.025$.
- (g) Use Eq. (5.16) and insert p_{va} in place of p_v and calculate $(NPSH)_A$. The above procedure is well exemplified in example 5.10.

5.3.3 Power Required for Pumping

Power required for pumping an incompressible fluid is given by equation:

$$P = \frac{Hq_v \rho}{3.67 \times 10^5 \times \eta} \quad (5.19)$$

where, P = Power required, kW

H = Total dynamic head, m of liquid column

ρ = Fluid density, kg/m³

η = Efficiency of pump

q_v = Capacity, m³/hr

Example 5.6

A centrifugal pump draws benzene from an overhead tank. Operating pressure in the tank is 700 torr a. Vertical distance between free surface of liquid in tank and centerline of pump is 12 m. Maximum operating temperature is 50° C. Vapour pressure of benzene at 50°C is 280 torr. Density of benzene at 50°C is 870 kg/m³. Frictional loss in suction line of pump is 1 m of benzene column. Calculate the $(NPSH)_A$ of centrifugal pump.

Solution:

$$(NPSH)_A = h_{ss} - h_{fs} - p_v \quad (5.16)$$

$$h_{ss} = p + Z$$

$$p = 760 - 700 = 60 \text{ torr a}$$

$$1 \text{ atm} = 10.33 \text{ m water column}$$

$$p = \frac{60}{760} \times 10.33 \times \frac{\text{Density of water}}{\text{Density of benzene at } 50^\circ\text{C}} = \frac{60}{760} \times 10.33 \times \frac{1000}{870} = 0.937 \text{ m}$$

$$p_v = \frac{280}{760} \times 10.33 \times \frac{1000}{870} = 4.37 \text{ m}$$

$$(NPSH)_A = p + Z - h_{fs} - p_v = 0.937 + 12 - 1 - 4.37 = 7.567 \text{ m}$$

Example 5.7

A centrifugal pump is drawing methanol from an underground tank. Operating pressure in the underground tank is 1 atm. Vertical distance between centre line of pump and free surface of liquid in the tank is 3 m. Maximum operating temperature is 50° C. Vapour pressure and density of methanol at 50° C are 400 torr and 785 kg/m³, respectively. Friction loss in suction line is 1 m of liquid column. Calculate the $(NPSH)_A$ of centrifugal pump.

Solution:

$$(NPSH)_A = h_{ss} - h_{fs} - p_v = p - Z - h_{fs} - p_v \quad (5.16)$$

$$p = 10.33 \times \frac{1000}{785} = 13.16 \text{ m LC}$$

$$p_v = \frac{400}{760} \times 10.33 \times \frac{1000}{785} = 6.926 \text{ m LC}$$

$$(NPSH)_A = 13.16 - 3 - 1 - 6.926 = 2.234 \text{ m LC}$$

Selection of the pump is critical. As a general guideline $(NPSH)_A$ should be greater than 3 m for a centrifugal pump.

Example 5.8

Installation of pump is to be decided for the following application. Pump will handle hot water at a temperature of 176.7°C and a suction pressure of 1.034 MPa g. Capacity of pump will be 10 m³/h. It is planned to use a centrifugal pump with single suction for this applications. Speed of its impeller will be 2900 rpm.

Find the minimum distance that must be kept between free surface of liquid at source and centre line of horizontal centrifugal pump.

Solution:

$$(NPSH)_A \geq (NPSH)_R$$

From Figs 5.3 and 5.4, data for centrifugal pump having a speed of 3550 rpm are given. Assume these data to be applicable for 2900 rpm (a safer design). For 3550 rpm, 10 m³/h, 176.7°C and $p < 35$ bar:

$$(NPSH)_R = 3.048 + 3.6576 \text{ m (additional)}$$

$$(NPSH)_R = 6.7 \text{ m}$$

$$(NPSH)_A \geq 6.7 \text{ m}$$

$$p \pm Z - h_{fs} - p_v \geq 6.7 \text{ m}$$

$$p = 1.034 \text{ MPa g} = 1.034 \times 10^6 \text{ Pa g}$$

$$1 \text{ atm} = 101.325 \text{ kPa}$$

$$p = (1.034 \times 10^6 + 101.325 \times 10^3) \text{ Pa}$$

$$p = 1.135325 \times 10^6 \text{ Pa}$$

Vapour pressure of hot water at 176.7°C = 0.928 MPa

$$p_v = 0.928 \times 10^6 \text{ Pa}$$

Density of water at 176.7°C, $\rho = 890 \text{ kg/m}^3$

$$p - p_v = 0.207325 \times 10^6 = \frac{0.207325 \times 10^6}{\rho \times g}$$

$$= \frac{0.207325 \times 10^6}{890 \times 9.81} = 23.746 \text{ m of WC}$$

Select the pipe size and piping system such that $h_{fs} \leq 1 \text{ m}$.

$$-Z - 1 + 23.746 \geq 6.7 \text{ m}$$

(if pump is installed above the free surface of liquid at source)

or $Z \leq 16.046 \text{ m}$

Pump can be installed maximum up to an elevation 16.046 m above the free surface of liquid at source to avoid cavitation. Pump can be installed at any distance, if it is to be installed below the free surface of liquid at source.

Example 5.9

In the previous example, if hot water is saturated at 176.7°C temperature at source, what will be the minimum height that must be kept between free surface of liquid at source and centre line of selected pump? Type, speed and capacity of pump are same as that for previous example.

Solution:

For the single suction centrifugal pump having speed 2900 rpm and capacity 10 m³/h

$$(NPSH)_R = 6.7 \text{ m}$$

$$(NPSH)_A \geq (NPSH)_R \text{ or } (NPSH)_A \geq 6.7 \text{ m}$$

Here hot water is saturated at 176.7°C at source.

$$p = p_v$$

In this case pump must be installed below the free surface of liquid at source.

$$(NPSH)_A = p + Z - h_{fs} - p_v = Z - h_{fs} \geq 6.7 \text{ m}$$

Let the velocity of water in suction line = 1 m/s

$$q_v = v \times (\pi/4)D_i^2 = 10 \text{ m}^3/\text{h} \equiv 2.7778 \times 10^{-3} \text{ m}^3/\text{s}$$

$$D_i = \sqrt{\frac{4 \times q_v}{\pi \times v}} = \sqrt{\frac{4 \times 2.7778 \times 10^{-3}}{\pi \times 1}} = 0.05947 \text{ m}$$

From the table of standard pipe select 62.5 mm NB SCH-40 steel pipe having $D_i = 62.71 \text{ mm}$

$$Re = \frac{D_i v \rho}{\mu}$$

$$\rho \text{ of water at } 176.7^\circ\text{C} = 890 \text{ kg/m}^3$$

Velocity based on $D_i = 62.71 \text{ mm}$, $v = 0.9 \text{ m/s}$

Viscosity of water at 176.7°C and at saturation pressure, 9.08 atm a must be determined.

Effect of pressure on viscosity of liquid is insignificant, up to about 50 atm.

For reduced temperature $T_r < 0.75$, relation between viscosity of liquid and temperature is given by equation

$$\ln \mu_L = \frac{B}{T + C}, T \text{ in K}$$

where $C = 17.71 - 0.19 T_b$, T_b in K

For water, $C = 17.71 - 0.19 (100 + 273) = -53.16$

From Appendix 14 of Ref. 10:

$$\mu_{149^\circ\text{C}} = 0.185 \text{ cP}, \mu_{115.6^\circ\text{C}} = 0.242 \text{ cP or mPa} \cdot \text{s}$$

$$\ln (0.185) = A + \frac{B}{[(149 + 273) - 53.16]}, \ln (0.242) = A + \frac{B}{[(115.6 + 273) - 53.16]}$$

$$A + 0.0027112 B = -1.6874, A + 0.002981 B = -1.4188$$

$$A = -4.3865, B = 995.55$$

$$\ln \mu_w = \frac{995.55}{[(176.7 + 273) - 53.16]} - 4.3865$$

$$\mu_w = 0.1532 \text{ cP or mPa} \cdot \text{s at } 176.7^\circ\text{C}$$

$$\mu_w = 0.1493 \text{ mPa} \cdot \text{s at } 180^\circ\text{C from JSME Steam Tables}$$

$$Re = \frac{0.06271 \times 0.9 \times 890}{0.1532 \times 10^{-3}} = 327876.7 \text{ (i.e. turbulent)}$$

For turbulent flow through commercial steel pipe

$$\frac{\Delta p}{L} = 4.07 \times 10^{10} \dot{m}^{1.84} \mu^{0.16} D_i^{-4.84} \rho^{-1} \quad (5.5)$$

$$q_v = 10 \text{ m}^3/\text{h} = 2.7778 \times 10^{-3} \text{ m}^3/\text{s}$$

$$\dot{m} = \text{mass flow rate} = q_v \times \rho = 2.7778 \times 10^{-3} \times 890 = 2.4722 \text{ kg/s}$$

$$\frac{\Delta p}{L} = 4.07 \times 10^{10} \times (2.4722)^{1.84} \times (0.1532 \times 10^{-3})^{0.16} \times (62.71)^{-4.84} \times (890)^{-1}$$

$$\frac{\Delta p}{L} = 0.1186 \text{ kPa/m}$$

For 6.7 m elevation,

let L = actual length of pipe = 7 m

Assuming three 90° elbows and one globe valve in the suction line.

Table 5.8 Equivalent Lengths of Fitting and Valve

Valve or Fitting	No.	L_e
90° elbows (standard)	3	3 (30 D) = 5.64 m
Globe valve (open)	1	350 D = 21.95 m

$$L \approx 7 + 5.64 + 21.95 \approx 34.59 \text{ m}$$

$$\Delta p = 4.102 \text{ kPa} = \frac{4.102 \times 10^3}{\rho \times g} \text{ m Liquid Column (i.e. WC at } 176.7^\circ\text{C)}$$

$$h_{fs} = \Delta p = \frac{4.102 \times 10^3}{890 \times 9.81} = 0.47 \text{ m of WC}$$

$$Z \geq (6.7 + 0.47) \text{ m or } Z \geq 7.17 \text{ m}$$

Minimum elevation required between free surface of water at source and centre line of pump = 7.17 m

Example 5.10

Carbon dioxide gas (> 99.5% pure) from ammonia plant is washed with cooling water in a packed column before compression in urea plant at 1.01 atm and 40°C. Cooling water from bottom of the column is pumped back to cooling tower. Solubility of carbon dioxide in water is 0.0973 kg/100 kg water at 1 atm and 40°C. Find the volume fraction of carbon dioxide over cooling water. Also calculate artificial liquid vapour pressure.

Solution:

Molar mass of carbon dioxide, $M = 44.0095$

From JSME Stream Tables, vapour pressure of water at 40°C,

$$\begin{aligned} p_v &= 0.07375 \text{ bar} \\ &= 0.0728 \text{ atm} \end{aligned}$$

Mass fraction of carbon dioxide in the liquid

$$\begin{aligned} w_0 &= \frac{0.0973 \times 1.01}{(100 + 0.0973)} \\ &= 9.8177 \times 10^{-4} \end{aligned}$$

$$\begin{aligned} V_{Ga} &= \frac{9.8177 \times 10^{-4} \times 0.08206 \times 313.15}{44.0095 \times 1.01} \\ &= 5.676 \times 10^{-4} \text{ m}^3/\text{kg H}_2\text{O} \end{aligned}$$

From JSME Steam Tables

$$V_L = 1.0078 \text{ m}^3/\text{kg H}_2\text{O at } 40^\circ\text{C}$$

$$\begin{aligned} \text{GVP} &= \frac{5.676 \times 10^{-4} \times 100}{(1.0078 + 5.676 \times 10^{-4})} \\ &= 0.0563\% \end{aligned}$$

Thus, volume per cent of carbon dioxide is only 0.0563% (< 2.5%) over cooling water and there is no danger of pump cavitation.

$$p_o = 1.01 \text{ atm}$$

$$a = \sqrt{\left[\frac{\left(\frac{p}{1.01} - \frac{0.0728}{1.01} \right)^2 \left(1 - \frac{0.0728}{1.01} \right)}{\left(\frac{5.676 \times 10^{-4}}{1.0078} \right) \left(\frac{p}{1.01} \right) \left(1 - \frac{p}{1.01} \right)} + 1 \right]} \quad (5.18)$$

By trial and error,

For $p = 0.145 \text{ atm}$, $a = 0.0247$

Hence, $p_{va} = 0.145 \text{ atm}$

Thus, if vapour pressure of water (0.0728 atm) is used instead of p_{va} (i.e. 0.145 atm), $(NPSH)_A$ would have been erroneous by 0.75 m H_2O .

Above calculations indicate that at higher temperature and lower suction pressure, p_{va} could have significantly affected $(NPSH)_A$.

Example 5.11

Benzene at 37.8°C is pumped through the system at a rate of $9.09 \text{ m}^3/\text{h}$ with the help of a centrifugal pump. The reservoir is at atmospheric pressure. Pressure at the end of discharge line is 345 kPa g. The discharge head is 3.05 m and the pump suction head is 1.22 m above the level of liquid in reservoir. The friction loss in suction line is 3.45 kPa and that in the discharge line is 37.9 kPa. The mechanical efficiency of the pump (η) is 0.6. The density of benzene is 865 kg/m^3 and its vapour pressure at 37.8°C is 26.2 kPa. Calculate (a) $(NPSH)_A$ and (b) power required by centrifugal pump.

Solution:

$$(a) (NPSH)_A = h_{ss} - h_{fs} - p_v = p - Z - h_{fs} - p_v$$

$$p = 1 \text{ atm} = \frac{101.325 \times 10^3}{865 \times 9.81} = 11.94 \text{ m LC}$$

$$Z = 1.22 \text{ m}$$

$$h_{fs} = 3.45 \text{ kPa} = \frac{3.45 \times 10^3}{865 \times 9.81} = 0.4066 \text{ m LC}$$

$$p_v = 26.2 \text{ kPa} \equiv 3.088 \text{ m LC}$$

$$(NPSH)_A = 11.94 - 1.22 - 0.4066 - 3.088 = 7.2254 \text{ m of LC}$$

(b) Power required by centrifugal pump

$$P = \frac{H q_v \rho}{3.67 \times 10^5 \times \eta} \quad (5.19)$$

$$p' = \text{Pressure at discharge} = 345 + 101.325$$

$$p' = 446.325 \text{ kPa} = \frac{446.325 \times 10^3}{865 \times 9.81} = 52.6 \text{ m LC}$$

$$Z' = 3.05 - 1.22 = 1.83 \text{ m}$$

$$h_{sd} = 52.6 + 1.83 = 54.43 \text{ m LC}$$

$$h_{fd} = 37.9 \text{ kPa} = \frac{37.9 \times 10^3}{865 \times 9.81} = 4.47 \text{ m LC}$$

$$h_d = 58.9 \text{ m LC}$$

$$h_s = h_{ss} - h_{fs} = p - Z - h_{fs} = 11.94 - 1.22 - 0.4066 = 10.31 \text{ m}$$

$$H = 58.9 - 10.31 = 48.59 \text{ m}$$

$$P = \frac{48.59 \times 9.09 \times 865}{3.67 \times 10^5 \times 0.6} = 1.735 \text{ kW}$$

Example 5.12

It is proposed to pump 10 000 kg/h of saturated toluene at 114°C and 1.1 atm a from the reboiler of a distillation tower to a second distillation unit without cooling. If the friction loss in the line between the reboiler and pump is 7 kPa and the density of toluene is 866 kg/m³, how much liquid level in the reboiler be maintained to give a net positive suction head of 2.5 m? Calculate the power required to drive the pump if the pump is to elevate the toluene to 10 m to a second unit at atmospheric pressure. Assume friction loss in the discharge line to be 35 kPa. Pump efficiency is 62 %.

Solution:

In this case the pump is installed below the liquid level.

$$(NPSH)_A = p + Z - h_{fs} - p_v = 2.5 \text{ m}$$

Liquid in reboiler is saturated liquid at reboiler conditions. Therefore, $p = p_v$

$$Z - h_{fs} = 2.5 \text{ m}$$

$$h_{fs} = 7 \text{ kPa} = \frac{7 \times 10^3}{\rho \times g} = \frac{7000}{866 \times 9.81}$$

$$= 0.824 \text{ m LC}$$

$$Z = 2.5 + 0.824 = 3.324 \text{ m}$$

Pump must be installed at a level 3.324 m below the free surface of liquid in reboiler.
Power required to drive the pump

$$P = \frac{H q_v \rho}{3.67 \times 10^5 \times \eta} \quad (5.19)$$

$$p' = 1 \text{ atm} = 101.325 \text{ kPa} = \frac{101325}{\rho \times g} \text{ m LC}$$

$$= \frac{101325}{866 \times 9.81} = 11.927 \text{ m LC}$$

$$Z' = 10 \text{ m}$$

$$h_{sd} = 10 + 11.927 = 21.927 \text{ m}$$

$$h_{fd} = 35 \text{ kPa} = \frac{35000}{866 \times 9.81} = 4.12 \text{ m LC}$$

$$h_d = 21.927 + 4.12 = 26.047 \text{ m}$$

$$\text{Total suction head } h_s = h_{ss} - h_{fs} = p + Z - h_{fs} \quad (5.14)$$

$$p = 1.1 \text{ atm} = \frac{111.4575 \times 10^3}{866 \times 9.81} = 13.12 \text{ m LC}$$

$$h_s = 13.12 + 3.324 - 0.824 = 15.62 \text{ m LC}$$

$$H = h_d - h_s = 047 - 15.62 = 10.427 \text{ m LC}$$

$$q_v = \frac{10000}{866} = 11.5473 \text{ m}^3/\text{h}$$

$$P = \frac{10.427 \times 11.5473 \times 866}{3.67 \times 10^5 \times 0.62} = 0.4582 \text{ kW}$$

Example 5.13

Hexane at 37.8°C is pumped through the system at a rate of 9.09 m³/h. The tank is at atmospheric pressure. Pressure at the end of discharge line is 345 kPa g. The discharge head is 3.05 m and the suction lift is 1.22 m above the level of liquid in the tank. The friction loss in suction line is 3.45 kPa and that in the discharge line is 37.9 kPa. The mechanical efficiency of the pump is 0.6. The density of hexane is 659 kg/m³ and its vapour pressure at 37.8°C is 33.71 kPa.

Calculate (a) (NPSH)_A and (b) power required by centrifugal pump.

Solution:

$$(a) (NPSH)_A = h_{ss} - h_{fs} - p_v = p - Z - h_{fs} - p_v$$

$$p = 1 \text{ atm} = \frac{101.325 \times 10^3}{659 \times 9.81} = 15.67 \text{ m LC}$$

$$Z = 1.22 \text{ m}$$

$$h_{fs} = 3.45 \text{ kPa} = \frac{3.45 \times 10^3}{659 \times 9.81} = 0.534 \text{ m LC}$$

$$p_v = 33.71 \text{ kPa} = 5.214 \text{ m LC}$$

$$(NPSH)_A = 15.67 - 1.22 - 0.534 - 5.214 = 8.702 \text{ m of LC}$$

(b) Power required by centrifugal pump

$$P = \frac{H q_v \rho}{3.67 \times 10^5 \times \eta} \quad (5.19)$$

$$p' = \text{pressure at discharge} = 345 + 101.325 \text{ kPa} = 446.325 \text{ kPa a}$$

$$p' = 446.325 \text{ kPa} = \frac{446.325 \times 10^3}{659 \times 9.81} = 69.04 \text{ m LC}$$

$$Z' = 3.05 - 1.22 = 1.83 \text{ m}$$

$$h_{sd} = 69.04 + 1.83 = 70.87 \text{ m LC}$$

$$h_{fd} = 37.9 \text{ kPa} = \frac{37.9 \times 10^3}{659 \times 9.81} = 5.86 \text{ m LC}$$

$$h_d = 70.87 + 5.86 = 76.73 \text{ m LC}$$

$$h_s = h_{ss} - h_{fs} = p - Z - h_{fs} = 15.67 - 1.22 - 0.534 = 13.916 \text{ m}$$

$$H = 76.73 - 13.916 = 62.814 \text{ m}$$

$$P = \frac{62.814 \times 9.09 \times 659}{3.67 \times 10^5 \times 0.6} = 1.71 \text{ kW}$$

Example 5.14

A centrifugal pump is drawing water from an overhead tank, exposed to atmosphere. Vertical distance between free surface of liquid in the tank and center line of pump is 10 m. Capacity of centrifugal pump is 10 000 kg/h. Maximum operating temperature is 50°C. Vapour pressure of water at 50°C is 92.51 torr. Total length of suction pipe 10.5 m, having two 90° elbows. Determine the size of suction pipe and $(NPSH)_A$ of centrifugal pump. Material of pipe is carbon steel. Density of water = 1000 kg/m³, Viscosity of water = 0.558 mPa · s (or cP).

Solution:

In Table 5.1, velocity of water in suction line v is recommended in the range of 0.3 to 1.5 m/s

Let velocity of water in suction line, $v = 1 \text{ m/s}$

$$\frac{\pi}{4} D_i^2 = \frac{q_v}{v} = \frac{10 \times (1/3600)}{1}$$

$$D_i = 0.0595 \text{ m}$$

Select 62.5 mm NB SCH-40 pipe, $D_i = 62.7 \text{ mm}$

$$v = \frac{q_v}{\frac{\pi}{4} D_i^2} = \frac{(10000/3600)(1/1000)}{\frac{\pi}{4} \times (0.0627)^2} = \frac{2.778 \times 10^{-3}}{\frac{\pi}{4} \times (0.0627)^2}$$

Actual velocity, $v = 0.9 \text{ m/s}$ through the selected pipe.

$$Re = \frac{D_i v \rho}{\mu} = \frac{0.0627 \times 0.9 \times 1000}{0.558 \times 10^{-3}}$$

$$= 101129$$

Surface roughness of carbon steel, $\epsilon = 0.0457 \text{ mm}$

$$\frac{\epsilon}{D_i} = \frac{0.0457}{62.7} = 0.000729$$

From Fig. 5.1, $f = 0.005$

$$\frac{\Delta p}{L} = \frac{32f G^2}{\pi^2 \rho g_c D_i^5} = \frac{32 \times 0.005 \times (10000/3600)^2}{\pi^2 \times 1000 \times 1 \times 0.0627^5} = 129 \text{ Pa/m}$$

$$(5.2)$$

For 90° Elbows, additional pressure drop

$$\Delta p_E = \frac{K \rho v^2}{2 g_c}$$

$$(5.9)$$

$$\Delta p_E = \frac{0.75 \times 1000 \times (0.9)^2}{2} = 303.75 \text{ Pa per elbow}$$

Total pressure drop in suction line

$$\Delta p_t = 129 \times 10.5 + 303.75 \times 2$$

$$\Delta p_t = 1962 \text{ Pa} \equiv 1.962 \text{ kPa} \equiv \frac{1.962 \times 10^3}{1000 \times 9.81} \equiv 0.2 \text{ m WC}$$

For 62.5 mm NB SCH-40 pipe,

$$(NPSH)_A = h_{ss} - h_{fs} - p_v = p + Z - h_{fs} - p_v$$

$$(5.16)$$

$$p = 1 \text{ atm} = \frac{101.325 \times 10^3}{1000 \times 9.81} = 10.33 \text{ m WC}$$

$$p_v = \frac{92.51}{760} = 0.1217 \text{ atm} \equiv 0.1217 \times 10.33 \equiv 1.26 \text{ m WC}$$

$$(NPSH)_A = 10.33 + 10 - 0.2 - 1.26 = 18.87 \text{ m}$$

Example 5.15

A centrifugal pump is used to transfer hot water at 110°C from a deaerator to boiler as shown in Fig. 5.5. Hot water is saturated liquid in the deaerator at 110°C. Capacity of pump is 16.5 m³/h and its efficiency is 60%. Speed of the impeller is 2900 rpm. Pipe line to the boiler contains one control valve and five 90° bends. Its straight length is 25 m.

Calculate:

- (a) Recommended size of suction line
- (b) Minimum vertical distance Z required between lowest liquid level in the deaerator and center line of pump and
- (c) Recommended size of pipe line to boiler.

Solution:

$$(NPSH)_A \geq (NPSH)_R$$

For the single suction centrifugal pump having speed 2900 rpm and capacity 16.5 m³/h

$$\text{rpm} \times \sqrt{q_v} = \text{constant}$$

$$2900 \times \sqrt{16.5} = 3550 \times \sqrt{x}$$

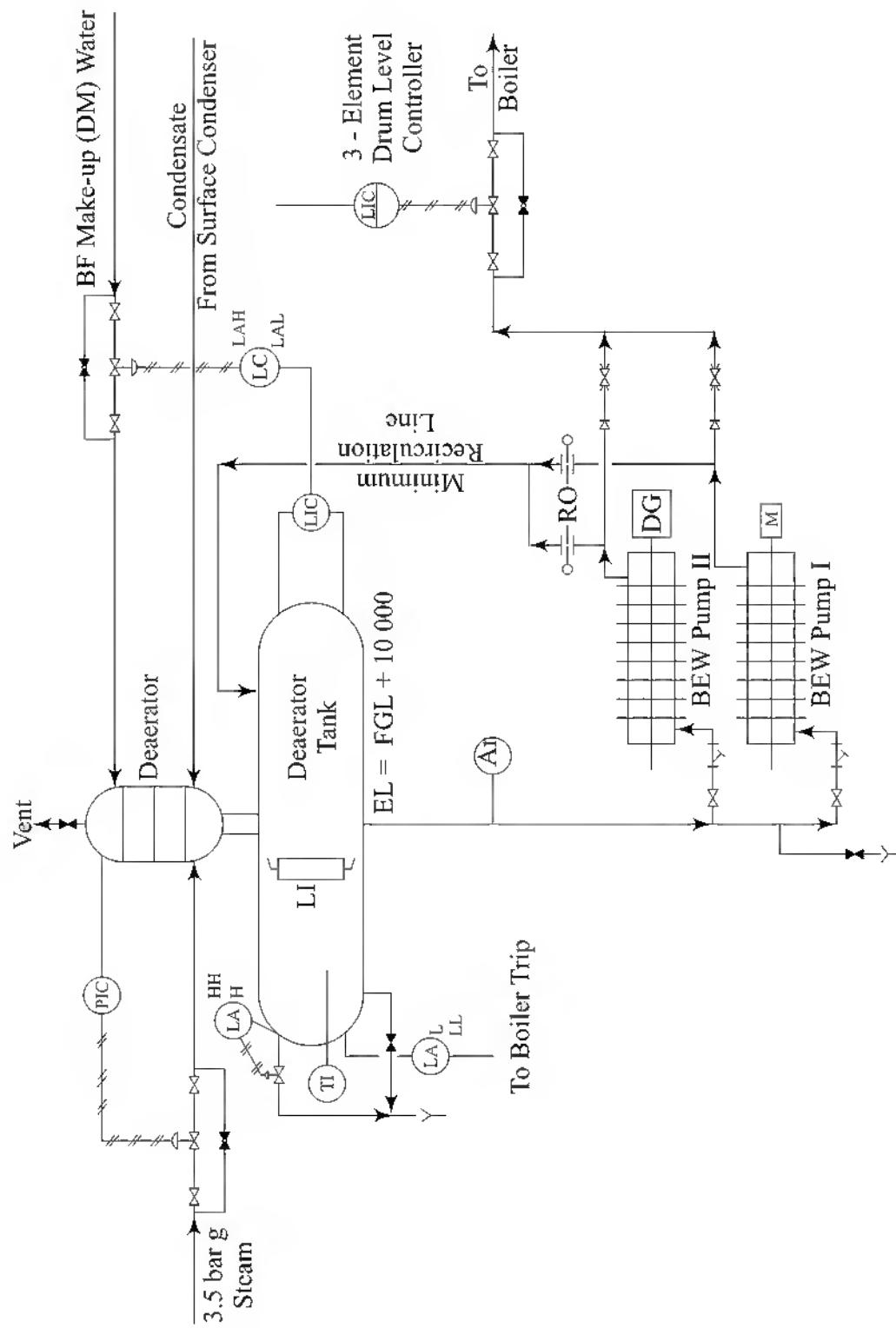


Fig. 5.5 Typical Boiler Feed Water System

$$x = 11 \text{ m}^3/\text{h}$$

Operating pressure, $p = 0.147 \text{ MPa g}$ (From JSME Steam Tables corresponding to the 110°C saturation temperature.)

$$p = 0.147 \text{ MPa g} = 248.325 \text{ kPa} = 2.48 \text{ bar} < 35 \text{ bar}$$

From Figs 5.3 and 5.4, $(\text{NPSH})_R = 3.048 + 0.4572 = 3.5052 \text{ m}$, say 3.5 m

$$(\text{NPSH})_R = 3.5 \text{ m}$$

$$(\text{NPSH})_A = p + Z - h_{fs} - p_v \geq 3.5 \text{ m}$$

In the deaerator, water is saturated, hence $p = p_v$

$$Z - h_{fs} \geq 3.5 \text{ m}$$

Let $h_{fs} = 1 \text{ m}$ (assumed) $Z \geq 4.5 \text{ m}$

$$Z_{\min} = 4.5 \text{ m}, \text{ Suggested } Z = 5 \text{ m}$$

Ans. (b)

Size of suction line:

Let velocity of hot water in suction line = 1 m/s (Table 5.1)

$$\frac{\pi}{4} D_i^2 = \frac{q_v}{v} = \frac{16.5/3600}{1} = 4.5833 \times 10^{-3} \text{ m}^2$$

$$D_i = 0.0764 \text{ m}$$

Select 75 mm NB, SCH-40 MS pipe

$$D_i = 77.92 \text{ mm}$$

$$\text{Reynolds number } Re = \frac{D_i v \rho}{\mu}$$

Velocity of water based on 77.92 mm diameter, $v = 0.961 \text{ m/s}$

Viscosity water at 110°C , $\mu = 0.256 \text{ mPa} \cdot \text{s}$ (or cP), ρ at $110^\circ\text{C} = 950.12 \text{ kg/m}^3$

$$Re = \frac{0.07792 \times 0.961 \times 950.12}{0.256 \times 10^{-3}} = 277914.3$$

Surface roughness of steel pipe = 0.04572 mm (Table 5.2)

$$\frac{\epsilon}{D_i} = \frac{0.04572}{77.92} = 0.000587$$

From Fig. 5.1, $f = 0.0045$, $G = 16.5 \times \frac{1}{3600} \times 950.12 = 4.3547 \text{ kg/s}$

$$\frac{\Delta p}{L} = \frac{32f G^2}{\pi^2 \rho g_c D_i^5} \quad (5.2)$$

$$= \frac{32 \times 0.0045 \times 4.3547^2}{\pi^2 \times 950.12 \times 1 \times 0.07792^5} \\ = 101.38 \text{ Pa/m}$$

For 90° elbows, additional pressure drop

$$\Delta p_E = \frac{K \rho v^2}{2 g_c}$$

$$\Delta p_E = \frac{0.75 \times 950.12 \times 0.961^2}{2} = 329.05 \text{ Pa per elbow}$$

Total pressure drop in suction line,

$$\Delta p_t = 101.38 \times 5 + 1 \times 329.05 = 835.95 \text{ Pa}$$

$$= \frac{835.95}{950.12 \times 9.81} = 0.09 \text{ m WC}$$

Corrected value of $Z_{\min} = 3.59 \text{ m}$, say 3.6 m

The recommended size of suction line is 75 mm NB, SCH-40 pipe and minimum vertical distance Z_{\min} required between lowest liquid level in deaerator and center line of pump is 3.6 m.

Ans. (a) and (b)

Size of discharge line to boiler:

Let the velocity of water in discharge line, $v = 2 \text{ m/s}$

$$\frac{\pi}{4} D_i^2 = \frac{q_v}{v} = \frac{16.5/3600}{2}$$

$$D_i = 0.054 \text{ m}$$

Select 50 mm (2 in) NB, SCH-40 MS pipe for which $D_i = 52.5 \text{ mm}$

Velocity of water based on 52.5 mm inside diameter $v = 2.12 \text{ m/s}$

$$Re = \frac{D_i v \rho}{\mu} = \frac{0.0525 \times 2.12 \times 950.12}{0.256 \times 10^{-3}}$$

$$= 413,079.5$$

Surface roughness of steel pipe $\epsilon = 0.045 \text{ } 72 \text{ mm}$

$$\frac{\epsilon}{D} = \frac{0.045 \text{ } 72}{52.5} = 8.7 \times 10^{-4}$$

From Fig. 5.1, $f = 0.0047$

$G = 4.3547 \text{ kg/s}$

For the turbulent flow through steel pipe,

$$\frac{\Delta p}{L} = \frac{32f G^2}{\pi^2 \rho g_c D_i^5} = \frac{32 \times 0.0047 \times 4.3547^2}{\pi^2 \times 950.12 \times 1 \times (0.0525)^5}$$

$$= 762.6 \text{ Pa/m}$$

Table 5.9 Equivalent Velocity Heads of Fittings and Valves

Fittings/valves	No. (n)	K
Gate valve	1	0.17
Controll valve	1	6
90° Bends	5	0.75
Check valve	1	2

Total pressure drop in discharge line

$$\Delta p_t = \frac{\Delta p}{L} \times \text{straight length} + (\sum K_i n_i) \frac{\rho v^2}{2 g_c}$$

$$= 762.6 \times 25 + \frac{(0.17 + 6 + 0.75 \times 5 + 2) \times 950.12 \times 2.12^2}{2 \times 1}$$

$$= 19065 + 25450.5 = 44515.5 \text{ Pa} \equiv 44.515 \text{ kPa}$$

This value of pressure drop is within permissible limit.

Hence size of discharge line to boiler is recommended to be 50 mm NB, SCH-40.

Example 5.16

Crude dichlorobenzene is pumped from a storage tank to a distillation column. The tank is blanketed with nitrogen and the pressure above the liquid surface is held constant at 0.1 bar g. The minimum depth of liquid in the tank is 1 m.

The distillation column operates at a pressure of 500 torr a. The feed point to the column is 12 m above the base of the tank. The tank and column are connected by a 50 mm internal diameter commercial steel pipe, 200 m long. The pipe, running from the tank to the column, contains the following valves and fittings: 20 No. of standard radius 90° elbows, two gate valves (fully open), an orifice plate and a flow control valve.

If the maximum flow rate required is 20 000 kg/h, calculate the power required by pump. Pump efficiency is 70%. Pressure drops across the control valve is 0.5 bar. Density of the dichlorobenzene is 1300 kg/m³ and viscosity is 1.4 m Pa · s (or cP).

Flow meter/Valve/Elbows	Equivalent number of velocity heads, K
(i) Standard radius 90° Elbows	0.75
(ii) Gate valve (Fully open)	0.17
(iii) Orifice meter	10

Solution:

Power required by pump is calculated by following equation

$$P = \frac{H q_v \rho}{3.67 \times 10^5 \times \eta} \quad (5.19)$$

$$q_v = \frac{20000}{1300} = 15.3846 \text{ m}^3/\text{h}$$

$$\eta = 0.7$$

$$H = h_d - h_s = (p' + Z' + h_{fd}) - (p + Z - h_{fs})$$

p' = Operating pressure in distillation column

$$p' = 500 \text{ torr a} = 66661.184 \text{ Pa}$$

To convert p' in m of liquid column,

$$p' = \frac{66661.184}{\rho \times g} = \frac{66661.184}{1300 \times 9.81} = 5.227 \text{ m LC}$$

$$Z' = 12 \text{ m}$$

$$p = 0.1 \text{ bar g}$$

$$\text{or } p = 10\ 000 + 101\ 325 = 111\ 325 \text{ N/m}^2 \text{ a}$$

$$= \frac{111\ 325}{1300 \times 9.81} = 8.7293 \text{ m LC}$$

$$Z = 1 \text{ m}$$

h_{fs} = Friction loss in suction line $\equiv 0$ m LC

h_{fd} = Total friction loss in discharge line in m of liquid column

$$h_{fd} = \frac{\Delta p}{\rho \times g}$$

where, Δp = Total pressure drop in discharge line

Velocity of dichlorobenzene in discharge line

$$v = \frac{q_v}{\frac{\pi}{4} D_i^2} = \frac{15.3846 \times (1/3600)}{\frac{\pi}{4} \times (0.05)^2} = 2.1765 \text{ m/s}$$

$$Re = \frac{D_i v \rho}{\mu} = \frac{0.05 \times 2.1765 \times 1300}{1.4 \times 10^{-3}} = 101\ 052 > 4000$$

Flow is turbulent.

For the turbulent flow of fluid through commercial steel pipe

$$\begin{aligned} \frac{\Delta p}{L} &= 4.07 \times 10^{10} G^{1.84} \mu^{0.16} D_i^{-4.84} \rho^{-1} \\ &= 4.07 \times 10^{10} \left(\frac{20\ 000}{3600} \right)^{1.84} (1.4 \times 10^{-3})^{0.16} \times (50)^{-4.84} \times (1300)^{-1} \\ &= 1.535\ 73 \text{ kPa/m} \end{aligned}$$

$$\begin{aligned} \Delta p_t &= \left(\frac{\Delta p}{L} \right) \times L + \Delta p \text{ of control valve} \\ &\quad + \Delta p \text{ across } 90^\circ \text{ elbows, gate valves and orifice meter} \end{aligned}$$

$$\begin{aligned} &= 1.535\ 73 \times 10^3 \times 200 + 0.5 \times 10^5 + (20 \times 0.75 + 2 \times 0.17 + 1 \times 10) \times \frac{\rho v^2}{2} \\ &= 307\ 146 + 50\ 000 + \frac{25.34 \times 1300 \times 2.1765^2}{2} \\ &= 435\ 171.63 \text{ Pa} \equiv 435.172 \text{ kPa} \end{aligned}$$

$$h_{fd} = \frac{435\ 171.63}{1300 \times 9.81} = 34.123 \text{ m LC}$$

$$\begin{aligned} H &= (5.227 + 12 + 34.123) - (8.7293 + 1 + 0) \\ &= 41.62 \text{ m LC} \end{aligned}$$

$$\begin{aligned} P &= \frac{41.62 \times 15.3846 \times 1300}{3.67 \times 10^5 \times 0.7} \\ &= 3.24 \text{ kW} \end{aligned}$$

5.4 EVALUATION OF CENTRIFUGAL PUMP PERFORMANCE WHEN HANDLING VISCOUS LIQUIDS

When centrifugal pumps, designed and tested for handling water are used for viscous liquids, significant increase in power consumption and decrease in head and capacity occur.

Figures 5.6 and 5.7 provide means of determining the performance of a conventional centrifugal pump handling a viscous liquid when its performance on

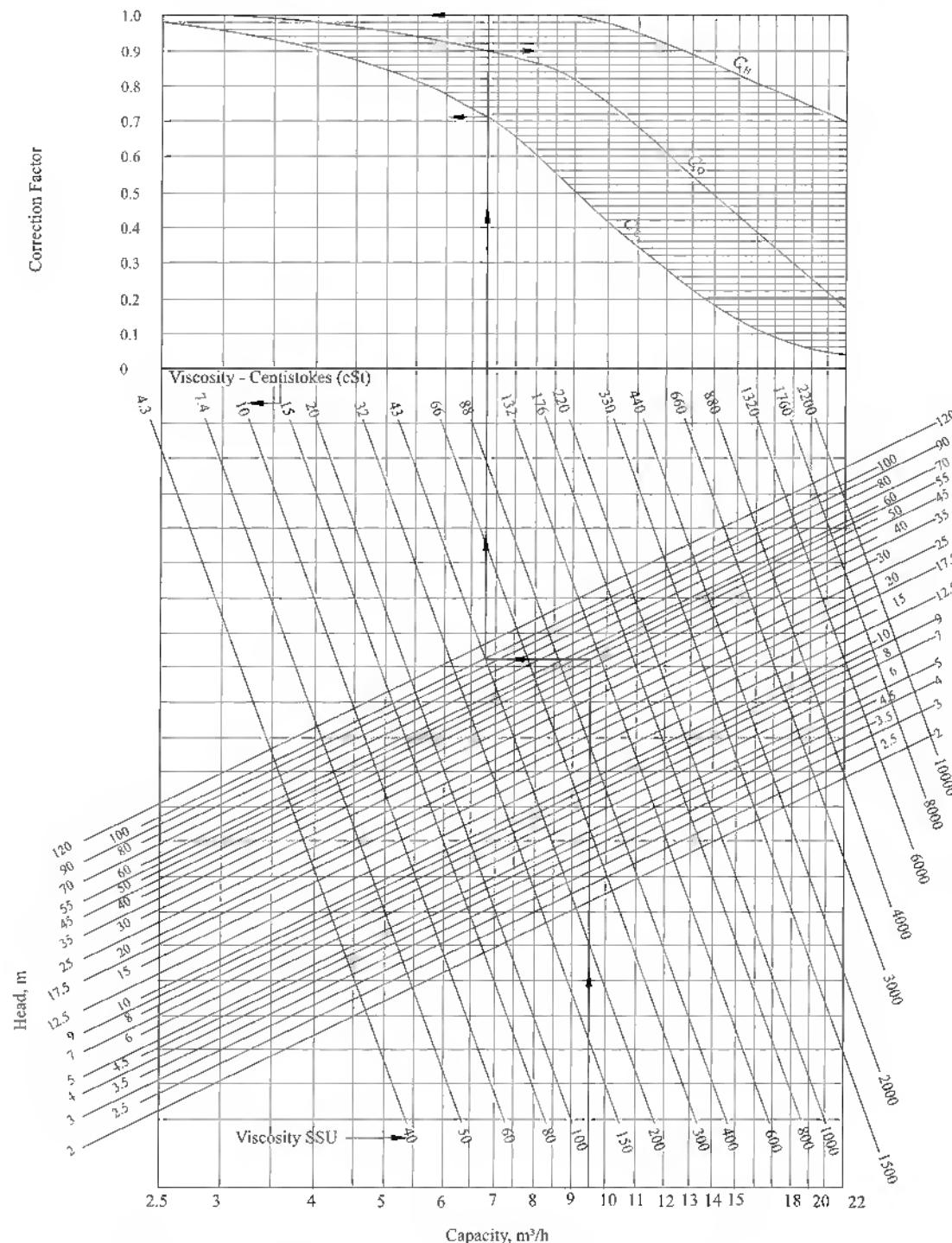


Fig. 5.6 Viscosity Correction Chart⁸
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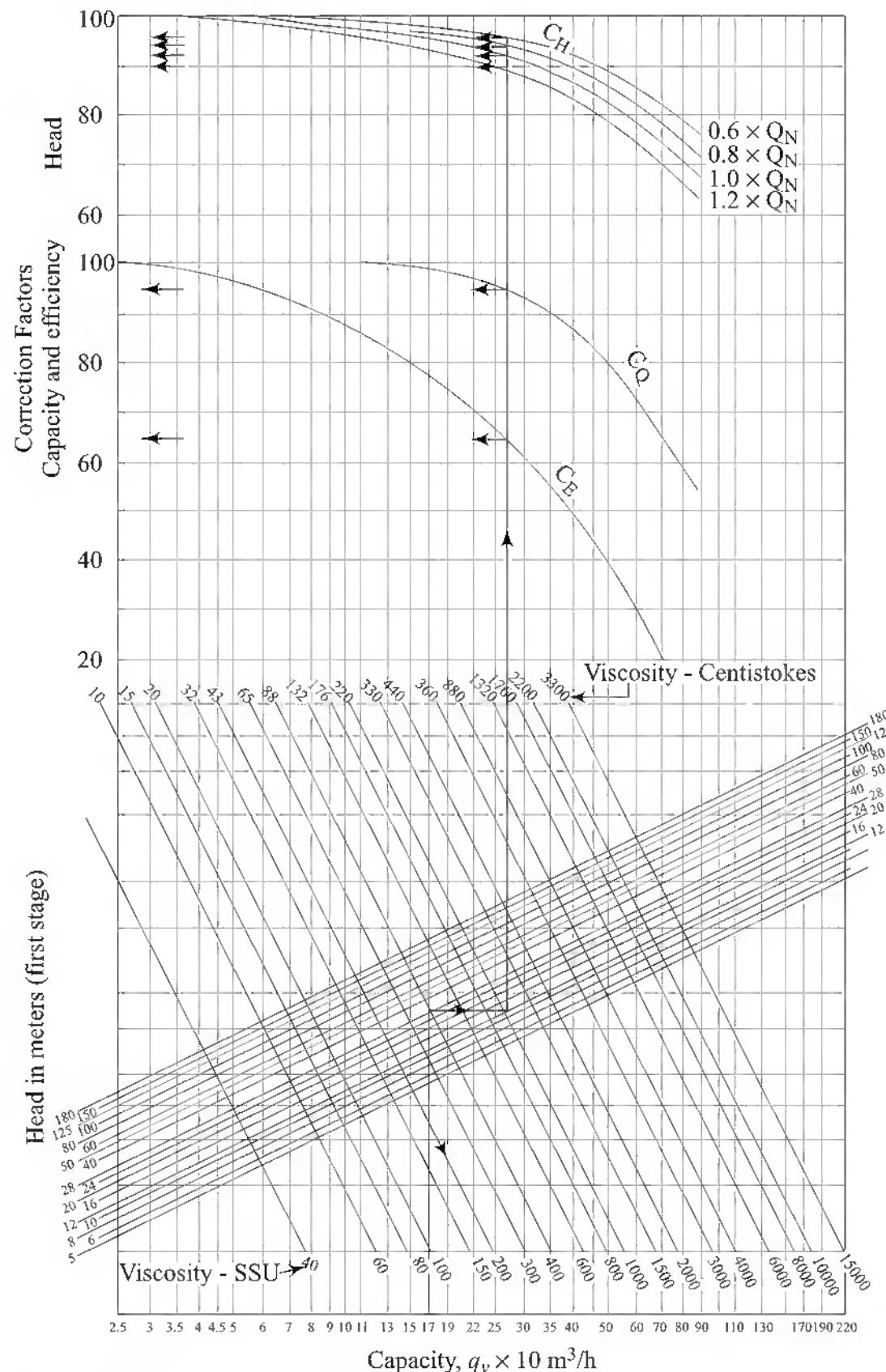


Fig. 5.7 Performance Correction Chart⁸
(Reproduced with the Permission of the Hydraulic Institute, USA)

water is known. The same can also be used as an aid in selecting a pump for a given application.

Limitations on use of viscous liquid performance correction chart (Figs 5.6 and 5.7):

- (i) Extrapolation is not recommended.
- (ii) Use only for pumps of conventional hydraulic design in the normal operating range with open or closed impellers. Do not use for mixed flow or axial flow pumps
- (iii) Use only for Newtonian liquids. Do not use for gels or slurries or other non-Newtonian liquids.

Example 5.17

Performance of a centrifugal pump on water is known. It can deliver $9.5 \text{ m}^3/\text{h}$ of water at 50 m total head of water with 80% efficiency. By using Figs 5.6 and 5.7 predict the performance of this pump if it is used to deliver lubricating oil with $43 \times 10^{-3} \text{ m}^2/\text{s}$ (or 43 cSt) viscosity at 40°C. Determine the percentage change in Head, Capacity and Power required by the pump. Density of lubricating oil at 40°C is 869 kg/m³ at the operating temperature.

Solution:

Power required by centrifugal pump for delivering $9.5 \text{ m}^3/\text{h}$ of water at 50 m total head with 80% efficiency

$$P = \frac{H q_v \rho}{3.67 \times 10^5 \times \eta} \quad (5.19)$$

(Density of water at 40°C, $\rho = 992.215 \text{ kg/m}^3$ from Table 3.28 of Ref. 2)

$$P = \frac{50 \times 9.5 \times 992.215}{3.67 \times 10^5 \times 0.8} = 1.60525 \text{ kW}$$

Enter the graph (Fig. 5.6, viscosity correction chart) with $9.5 \text{ m}^3/\text{h}$ capacity, go up to 50 m head, over to 43 cSt viscosity, and then up to the correction factors.

$$C_Q = 0.9$$

$$C_H = 1$$

$$C_E = 0.71$$

Capacity of centrifugal pump when it handles the lubricating oil

$$Q_{\text{oil}} = C_Q Q_W = 0.9 \times 9.5 = 8.55 \text{ m}^3/\text{h}$$

Head developed by pump

$$H_{\text{oil}} = C_H \times H_W = 1 \times 50 = 50 \text{ m}$$

Efficiency of pump $E_{\text{oil}} = C_E \times E_W = 0.71 \times 0.8 = 0.568$

Note that there is a significant drop (29%) in the efficiency.

Power required by pump when it handles the lubricating oil,

$$P' = \frac{H_{\text{oil}} \times Q_{\text{oil}} \times \rho_{\text{oil}}}{3.67 \times 10^5 \times E_{\text{oil}}} = \frac{50 \times 8.55 \times 869}{3.67 \times 10^5 \times 0.568} = 1.7821 \text{ kW}$$

$$\% \text{ decrease in capacity of pump} = \frac{(9.5 - 8.55)}{9.5} \times 100 = 10\%$$

% decrease in head developed of pump = 0% (i.e. no change)

$$\% \text{ increase in power required by pump} = \frac{(1.7821 - 1.60525)}{1.60525} \times 100 = 11.02\%$$

Example 5.18

Figure 5.8 shows the performance curve of a centrifugal pump operated on water. If the same pump is used for handling oil with a specific gravity of 0.9 and a viscosity of 1000 SSU (220 cSt) at the pumping temperature, develop the performance curve of the same pump for oil.

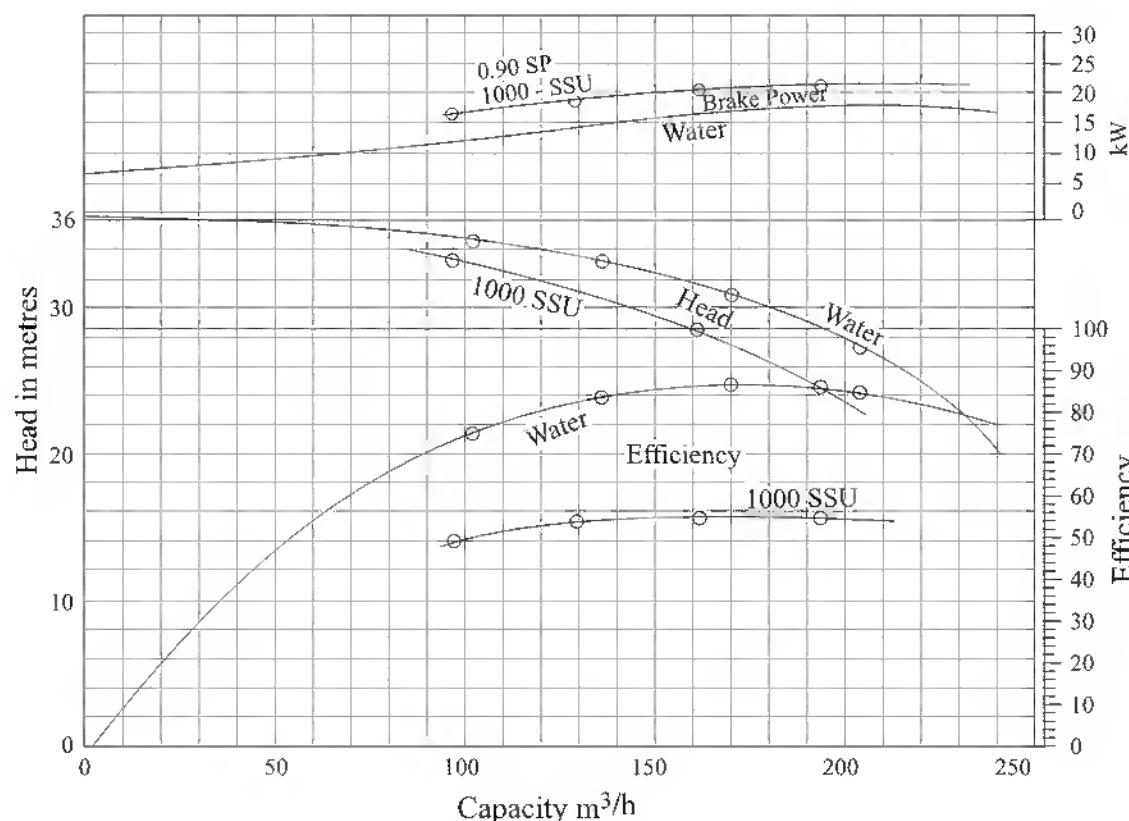


Fig. 5.8 Sample Performance Chart

Solution:

On the performance curve for water, locate the best efficiency point. In this example from Fig. 5.8 (from the efficiency vs capacity curve for water), it is 170 m³/h. For water with 170 m³/h capacity, efficiency of pump is maximum (86%). Tabulate capacity, head and efficiency for 0.6 × 170, 0.8 × 170 and 1.2 × 170 m³/h flow rates. By using Fig. 5.8, for 170 m³/h, 31 m of head is developed by the pump when it handles the water (from head vs capacity curve for water).

Enter the graph (Fig. 5.7, viscosity correction chart) with 170 m³/h capacity, go upto 31 m head, over to 1000 SSU (220 cSt) viscosity and then up to correction factors.

$$C_E = 0.645$$

$$C_Q = 0.95$$

$$C_H = 0.895 \text{ (for } 1.2 \times Q_N\text{)}$$

$$C_H = 0.92 \text{ (for } 1 \times Q_N\text{)}$$

$$C_H = 0.94 \text{ (for } 0.8 \times Q_N\text{)}$$

$$C_H = 0.96 \text{ (for } 0.6 \times Q_N\text{)}$$

Sample calculations for $0.6 \times Q_{NW}$ capacity:

$$Q_{NW} = 170 \text{ m}^3/\text{h}$$

$$0.6 \times Q_{NW} = 102 \text{ m}^3/\text{h} = Q_W$$

$$H_W = 34.62 \text{ m (from head vs. capacity curve for water in Fig. 5.8)}$$

$$\eta_W = 0.76 \text{ (from efficiency vs. capacity curve for water in Fig. 5.8)}$$

Viscosity of oil, $\mu = 220 \text{ cSt}$

$$C_Q = 0.95 \quad Q_{oil} = C_Q \cdot Q_W = 0.95 \times 102 = 96.9 \text{ m}^3/\text{h}$$

$$C_H = 0.96 \quad H_{oil} = C_H \cdot H_W = 0.96 \times 34.62 = 33.235 \text{ m}$$

$$C_E = 0.645 \quad \eta_{oil} = C_E \cdot \eta_W = 0.645 \times 0.76 = 0.4902$$

Here again, note the drop (24%) in efficiency.

Power required by pump when it handles the oil

$$P = \frac{H_{oil} \times Q_{oil} \times \rho_{oil}}{3.67 \times 10^5 \eta_{oil}} = \frac{33.235 \times 96.9 \times 900}{3.67 \times 10^5 \times 0.4902} = 16.11 \text{ kW}$$

Calculation for obtaining the performance curve for oil is given in Table 5.10. Same data are presented in Fig. 5.8.

Table 5.10 Revised Performance of Centrifugal Pump for Handling Oil

Capacity	$0.6 \times Q_{NW}$	$0.8 \times Q_{NW}$	$1.0 \times Q_{NW}$	$1.2 \times Q_{NW}$
$Q_w \text{ m}^3/\text{h}$	102	136	170	204
$H_W \text{ m}$	34.62	33.21	21	27.3
η_W	0.76	0.84	0.86	0.85
C_Q	0.95	0.95	0.95	0.95
C_H	0.96	0.94	0.92	0.895
C_E	0.645	0.645	0.645	0.645
$Q_{oil} = C_Q \times Q_W$	96.9	129.2	161.5	193.8
$H_{oil} = C_H \times H_W$	33.325	31.217	28.52	24.434
$\eta_{oil} = C_E \times \eta_W$	0.4902	0.5418	0.5547	0.5483
$P \text{ kW}$	16.11	18.26	20.363	21.18

5.5 POWER REQUIRED IN FAN, BLOWER AND IN ADIABATIC COMPRESSOR

Fans, blowers and compressors are used to increase the mechanical energy of gases. Discharge pressure of a fan is less than 3.45 kPa g (350 mm WC). If discharge pressure required is higher than 3.45 kPa g then a blower is used. Maximum discharge pressure of blower is 1 atm g. If the discharge pressure required is more than 2 atm a then a compressor is used. Discharge pressure of compressor ranges from 2 atm a to thousands of atmospheres.

5.5.1 Power Required by Fan

Fans are of two types. (i) centrifugal fans and (ii) axial flow fans. Efficiency of a

fan ranges from 40 to 70%, depending on its specific speed. Power input to a fan is given by equation.

$$P_0 = 2.72 \times 10^{-5} q_v p_d \quad (5.20)$$

where, P_0 = Power required by fan, kW

q_v = Capacity of fan, m³/h

p_d = Discharge pressure of fan, cm WC

5.5.2 Power Required in Blower and in Adiabatic Compressor

Blowers are always operated in an adiabatic manner. Many compressors are also operated in adiabatic manner (Example: air compressors upto 10 bar g). In adiabatic compressor, jacketed cooling is not provided. While in other types; polytropic and isothermal compressors, jacketed cooling is provided around the compressor suction. In isothermal compressor inlet temperature of gas is equal to outlet temperature of gas. If both temperatures are not same even after providing the jacket cooling, it is called polytropic compressor.

For adiabatic compression

$$\frac{p_2}{p_1} = \left(\frac{V_1}{V_2} \right)^k, \quad \frac{T_1}{T_2} = \left(\frac{V_1}{V_2} \right)^{k-1}, \quad \frac{p_2}{p_1} = \left(\frac{T_2}{T_1} \right)^{\frac{k}{k-1}} \quad (5.21)$$

where, $k = C_p/C_v$

k = ratio of specific heat at constant pressure to specific heat at constant volume

p_2, p_1 = outlet/inlet pressure of gas, kPa

V_2, V_1 = outlet/inlet volume of gas , m³

T_2, T_1 = outlet/inlet temperatures of gas , K

Power required in single stage blowers or in single stage adiabatic compressor,

$$P_o = 2.78 \times 10^{-4} \left(\frac{k}{k-1} \right) q_{v1} p_1 \left[\left(\frac{p_2}{p_1} \right)^{\frac{k-1}{k}} - 1 \right] \quad (5.22)$$

where, P_0 = Power required, kW

P_1 = Absolute inlet pressure, kPa

q_{v1} = Volumetric flow rate of gas based on inlet condition, m³/h

Discharge temperature of gas from the blower or single stage adiabatic compressor,

$$T_2 = T_1 \left(\frac{p_2}{p_1} \right)^{\frac{k-1}{k}} \quad (5.23)$$

Value of k ranges from 1.39 to 1.41 for air and perfect diatomic gases. For $k = 1.395$, value of bracketted expression in Eq. (5.22) is known as X factor

$$X = \left[\left(\frac{p_2}{p_1} \right)^{\frac{k-1}{k}} - 1 \right] = r^{\frac{k-1}{k}} - 1 \quad (5.24)$$

where, $k = 1.395$ for air or a diatomic gas

r = Compression ratio

Power required for blower and adiabatic compressor

$$P_0 = 9.81 \times 10^{-4} q_{v1} p_1 X \quad (5.25)$$

Other gases are having value of k in the range of 1 to 1.4. Power required by blower or adiabatic compressor, for the gases having value of k other than $k = 1.395$, is given by following equation.

$$P_0 = 6.37 \times 10^{-4} q_{v1} p_1 \frac{X_G}{d} \quad (5.26)$$

where, $d = 2.292 ((k - 1) / k)$

Values of $\left(\frac{X_G}{X} \right)$ and d can be obtained from Fig. (5.9) for the different values of k and for different values of r .

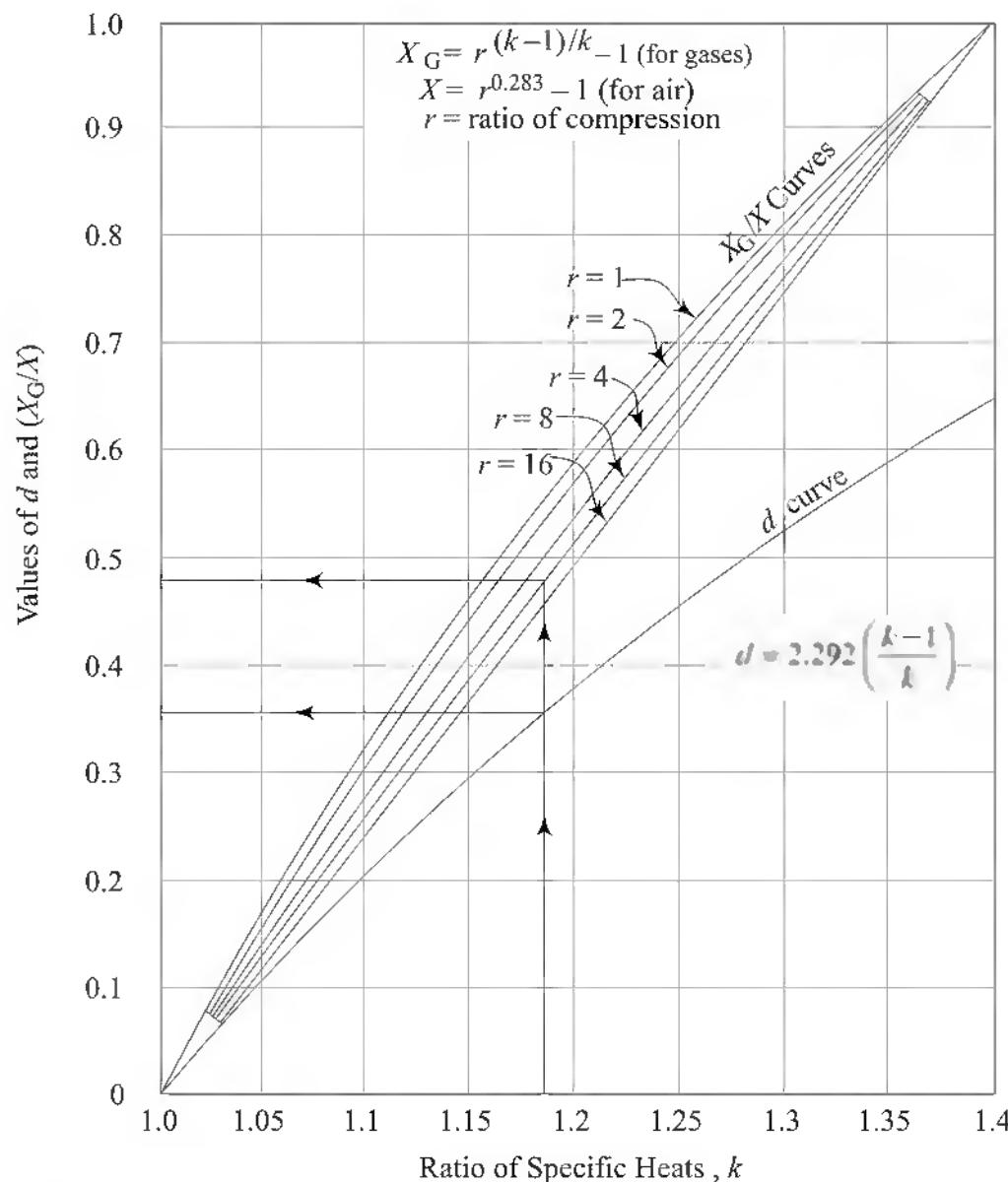


Fig. 5.9 (X_G/X) vs k and d vs k^2

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For a multistage compressor with N stages of compression in which

- (a) adiabatic compression takes place in each stage,
- (b) near equal work done in each stage (i.e. equal compression ratio in each stage, r) and
- (c) intercooling is provided between each two stages of compression to same inlet gas temperature (T_1),

Power requirement can be calculated by the following equation.

$$P_o = \frac{6.37 \times 10^{-4} N q_{v1} p_1}{d} \left(\sqrt[N]{X_G + 1} - 1 \right) \quad (5.27)$$

For the multistage compressor, discharge temperature from the last stage can be predicted by the following equation.

$$T_2 = T_1 \left(\sqrt[N]{X_G + 1} \right) \quad (5.28)$$

where, N = number of stages

Example 5.19

Find out the power required for a turbo blower for the following duty.

Fluid = Atmospheric air

Capacity = 1000 Nm³/h

Discharge pressure = 2 atm a

Also find the discharge temperature of air.

Solution:

Power required for turbo blower

$$P_o = 2.78 \times 10^{-4} \frac{k}{k-1} q_{v1} p_1 \left[\left(\frac{p_2}{p_1} \right)^{\frac{k-1}{k}} - 1 \right] \quad (5.22)$$

For air, $k = 1.395$

q_{v1} = Volumetric flow rate based on inlet condition in m³/h

$$\frac{p_1 q_{v1}}{T_1} = \frac{1 \times 1000}{(273 + 25)}$$

$$q_{v1} = \frac{1000 \times 1}{298} \times (273 + 50) = 1083.9 \text{ m}^3/\text{h}$$

$$P_o = 2.78 \times 10^{-4} \frac{1.395}{(1.395 - 1)} \times 1083.9 \times 101.325 \times (2^{(1.395-1/1.395)} - 1)$$

$$P_o = 23.38 \text{ kW}$$

Discharge temperature of Air

$$T_2 = T_1 \left(\frac{p_2}{p_1} \right)^{\frac{k-1}{k}} \quad (5.23)$$

$$T_2 = 323 \left(2 \right)^{\frac{(0.395)}{1.395}} = 393.04 \text{ K}$$

Discharge temperature of air, $t_2 = 120^\circ\text{C}$

Example 5.20

A three stage reciprocating compressor is used to compress $306 \text{ Sm}^3/\text{h}$ of methane from 0.95 atm a to 61.3 atm a. The inlet temperature is 26.7° C .

Specific heat ratio of methane, $k = 1.31$

Calculate (a) Power required for compression, if mechanical efficiency is 80 percent and

(b) Discharge temperature of gas after 1st stage

Solution:

For a multistage compressor total power required is minimum if each stage consume the same power or same compression ratio in each stage.

For same compression ratio in each stage,

$$\text{Compression ratio in each stage } r = \left(\frac{61.3}{0.95} \right)^{1/3} = 4.01$$

Power required for each stage

$$P_0 = \frac{2.78 \times 10^{-4}}{\eta} \frac{k}{k-1} q_{v1} P_1 \left[\left(\frac{p_2}{p_1} \right)^{\frac{k-1}{k}} - 1 \right] \quad (5.22)$$

$$k = 1.31, \quad n = 0.8$$

q_{v1} = Volumetric flow rate based on inlet condition m^3/h

$$\frac{P_1 q_{v1}}{T_1} = \frac{P_s q_{vs}}{T_s} = \frac{1 \times 306}{273}$$

$$q_{v1} = \frac{306}{273} \times \frac{(273 + 26.7)}{0.95} = 353.6 \text{ m}^3/\text{h}$$

$$P = \frac{2.78 \times 10^{-4}}{0.8} \frac{1.31}{1.31-1} \times 353.6 \times 0.95 \times 101.325 \left((4.01)^{\frac{0.31}{1.31}} - 1 \right)$$

$$= 19.45 \text{ kW}$$

If power loss in allied piping and intercoolers is neglected, then total power required for three stages,

$$P'_0 = 3 \times 19.45 = 58.35 \text{ kW}$$

(b) Discharge temperature after 1st stage

$$T_2 = T_1 \left(\frac{p_2}{p_1} \right)^{\frac{k-1}{k}}$$

$$T_2 = (273 + 26.7)(4.01)^{(0.31/1.31)}$$

$$T_2 = 416.3 \text{ K}, t_2 = 143.3^\circ \text{C}$$

or

for $k = 1.31$ and $r = 4.01$, from Fig. 5.9

$$d = 0.54, (X_G/X) = 0.8$$

$$X = r^{\frac{0.395}{1.395}} - 1 = 4.01^{\left(\frac{0.395}{1.395}\right)} - 1 = 0.4818$$

$$X_G = 0.8 \times 0.4818 = 0.3854$$

Power required for each stage

$$P_0 = 6.37 \times 10^{-4} \frac{q_{vl} p_1}{\eta} \frac{X_G}{d} \quad (5.26)$$

$$P_0 = \frac{6.37 \times 10^{-4} \times 353.6 \times 96.2587}{0.8} \times \frac{0.3854}{0.54} = 19.34 \text{ kW}$$

Total power required $P'_0 = 3 \times 19.34 = 58.03 \text{ kW}$
(Neglecting pressure loss in the intercoolers and allied piping)

5.6 FLOW METERS

A variety of flow meters are in use in chemical process industry. One category is the traditional differential pressure (DP) type volumetric flow meters. In many applications the volumetric flow rate is of direct interest to the user. Because of its accuracy, simplicity and relatively lower cost, these flow meters are popular in the chemical industry. By multiplying the flow rate with the actual density, mass flow rate can be obtained. Pitot tube, orifice meter, venturi meter and flow nozzles fall under this category. Among these, orifice meter is by far the most popular in the industry. Figure 5.10 shows these meters schematically.

Less commonly used flow meter is vortex meter which works on Von Karman effect. Flowing fluid separates on two sides of shedder bar face. Vortices form behind the face and cause alternating differential pressures (DP) around the back of the shedder.

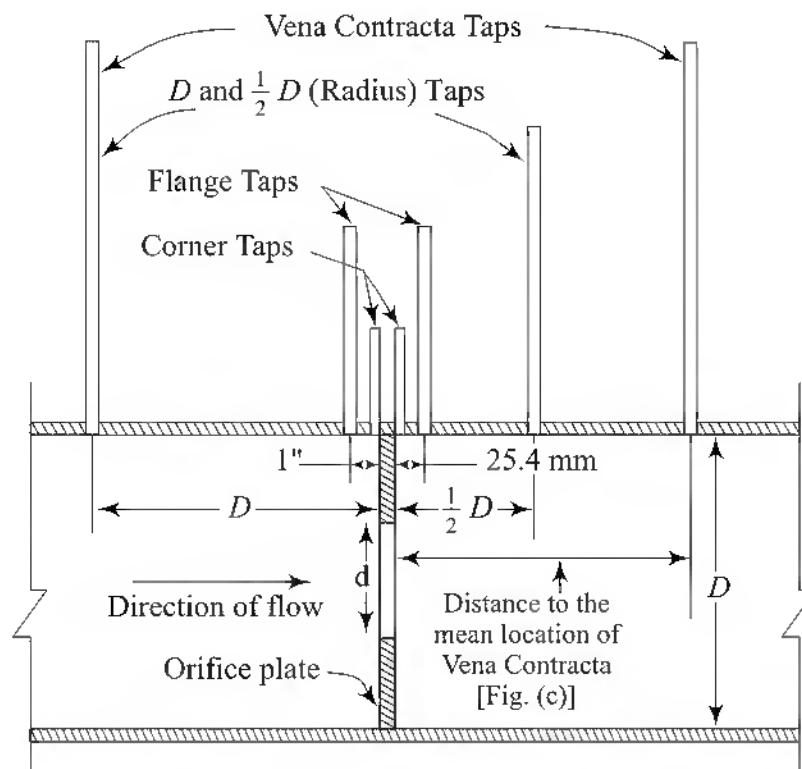
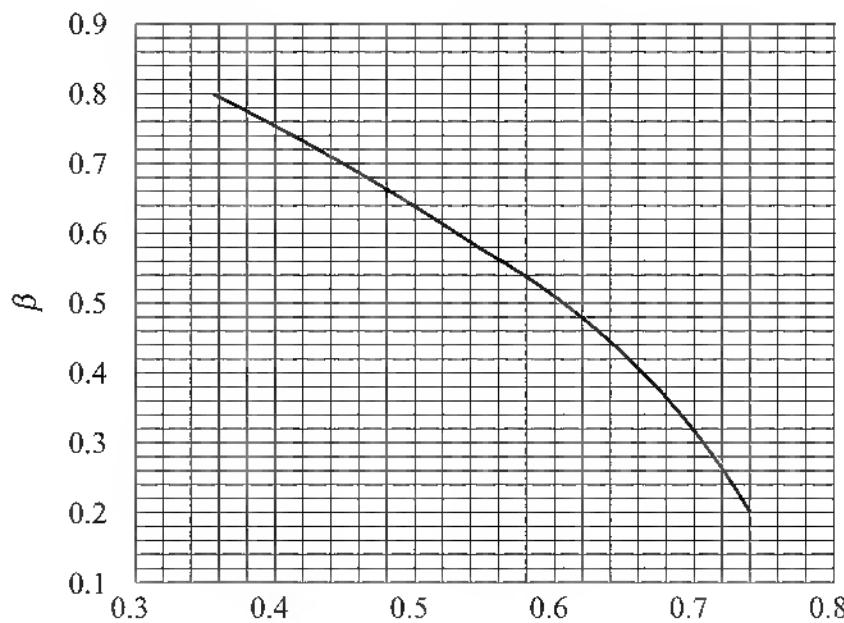
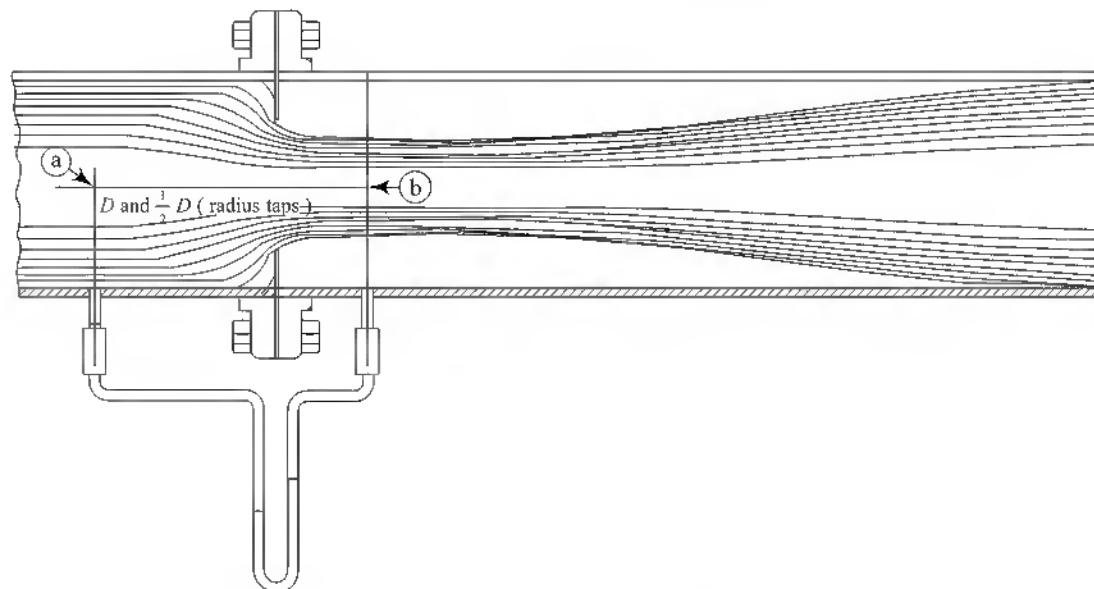


Fig. 5.10(a) Various Tap Locations for Orifice Meter



Pipe Diameters from Inlet Face of Orifice Plate
Extracted from the ASME Flow Meter Computation Handbook, 1961
(b)

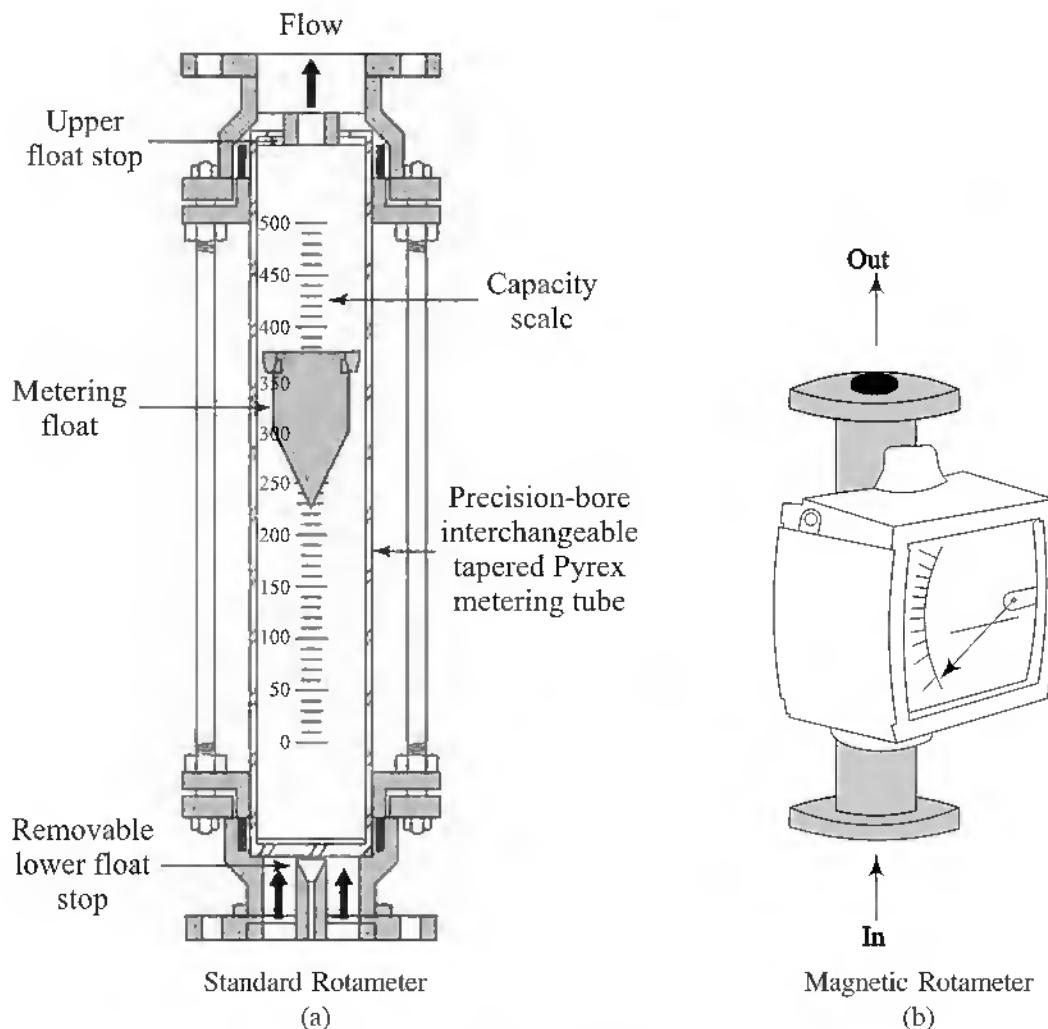


Orifice Meter with Vena Contracta Formation
(c)

Fig. 5.10 Orifice Meter

The frequency of the alternating vortex development is linearly proportional to the volumetric flow rate. Although vortex meters are as reliable and as accurate as other DP instruments, they can be used for only clean liquids and are recommended for turbulent flows.

Another popular category is of variable area flow meters which are also known as rotameters. Figure 5.11 shows two different types of rotameters. While DP type instruments can be installed in horizontal position, rotameters are installed in vertical position. Bypass rotameters are installed in horizontal position which is a combination of an orifice and a rotameter. Bypass rotameters are installed on

**Fig. 5.11 Rotameters**

larger size pipelines and occupy quite less space. Near linear scale of the meter is an advantage over logarithmic scale for the DP meters. However, rotameters with glass tubes are relatively less accurate and are mainly used for low pressure applications.

Disadvantage of low pressure applications due to glass tubes has led to the development of magnetic rotameters in which a magnetic float is used in a metallic tapered tube and its accurate position is monitored outside the tube. Float's movement is calibrated on a dial type indicator. Both DP type and magnetic rotameter can be connected to a microprocessor based control unit for totalizing and/or controlling purpose.

Positive displacement type flow meters incorporate a pair of gears or a turbine which rotate on their axes and the revolutions are measured. Since volume displaced per revolution is nearly constant, volumetric flow is calculated by multiplying two parameters. Traditional water meters are of gear type. Turbine flow meters are available for number of liquids such as ammonia, chlorine, etc. which flow under relatively high pressure. These instruments are sufficiently accurate and are used for custody transfer.

Weirs are popularly used for volumetric flow measurements in open canals and alike situations. Figure 5.12 shows different types of weirs in use.

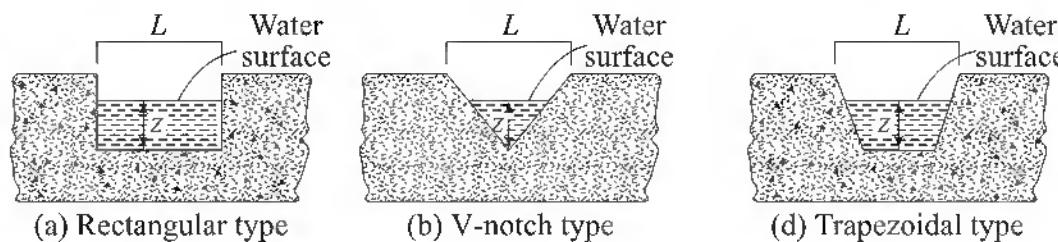


Fig. 5.12 Flow Measurement with Weirs

Another category relates to ultrasonic flow meters. Its underlying principle is measurement of the effect a flowing fluid has on an ultrasound signal either transmitted through or reflected from the fluid. These instruments are largely clamp-on designs and offer simplicity of installation. These meters suffer from a reputation for unreliability and poor long-term stability, compared with DP type meters. However, newer designs are being developed for high pressure and temperature fluids, including gases. Designs are available when a single instrument can be clamped on a range (though limited) of pipes and volumetric flows can be checked periodically. This type of meter is quite handy to a process engineer in checking plant performance from time to time.

Electromagnetic flow meters (also known as magmeters) work on Faraday's law of conductance which states that when a conductive fluid flows through a magnetic field, generated perpendicular to the flow, the voltage induced in the fluid is linearly proportional to the volumetric flow rate. Thus, this type of instrument operates only with electrically conductive liquid. Non-invasive nature of ultrasonic flow meter is also the feature of magmeter. Both permit unobstructed flow through the measuring element. Both are clamp on meters whose transducers are strapped to the pipeline. Unlike ultrasonic meters, magmeters have a reputation of accuracy.

True mass flow meter based on Coriolis principle was introduced in 1977 by Mirco Motion, Inc., USA, a division of Emerson Process Management. The Coriolis principle, discovered by Gustave Coriolis in 1935 states that fluid flowing through a tube, vibrating at its natural frequency, generates a force that slightly distorts the tube. This extremely small distortion, picked up by sensors, is directly proportional to the mass flow rate (Newton's second law of motion) and is independent of any other property of the fluid; say pressure, temperature, density or viscosity. Figure 5.13 schematically represents the mass flow meter.

In a single mass flow meter, four parameters of the fluid can be measured at a time; mass flow, volume flow temperature, and density. This instrument offers high accuracy and can be used for any fluid; clean or dirty, transparent or opaque, conductive or non-conductive. Originally developed instrument required extra space for installation but recently developed meter has a straight tube configuration. Among different types of flow meters, Coriolis device is the costliest.

In the forthcoming sections, process designs of orifice meter and rotameter are discussed. Reader is advised to refer standard handbooks on instrumentation/flow meters for design of other flow meters.

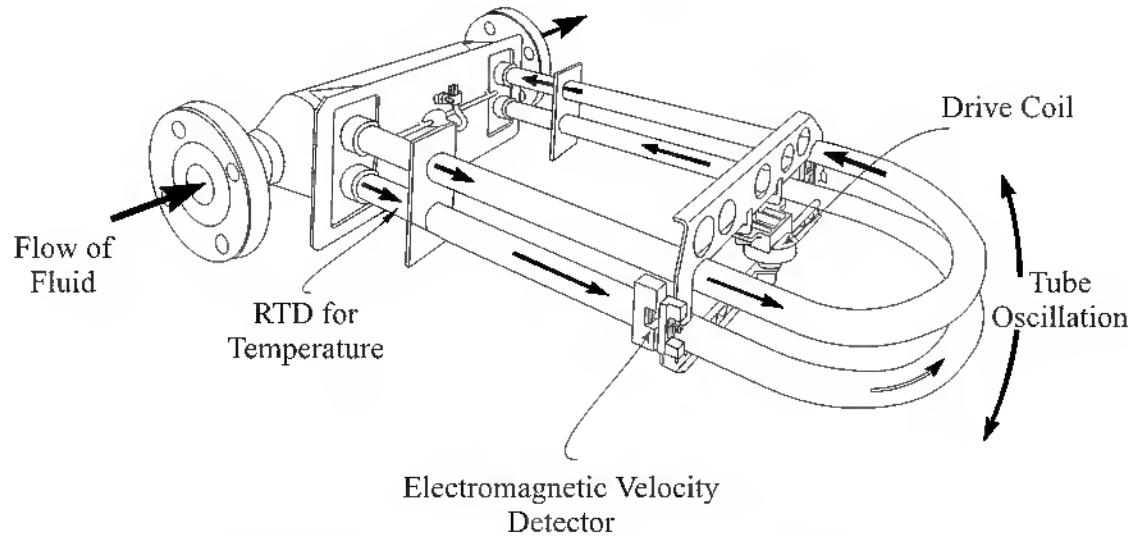


Fig. 5.13 Coriolis Mass Flow Meter (Courtesy: Micro Motion, Inc., USA)

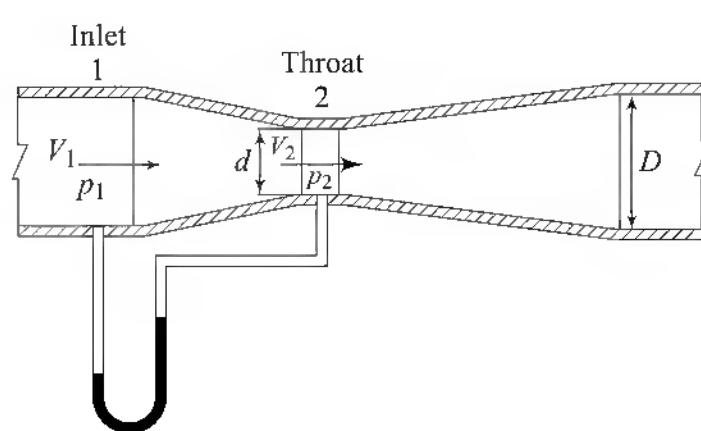
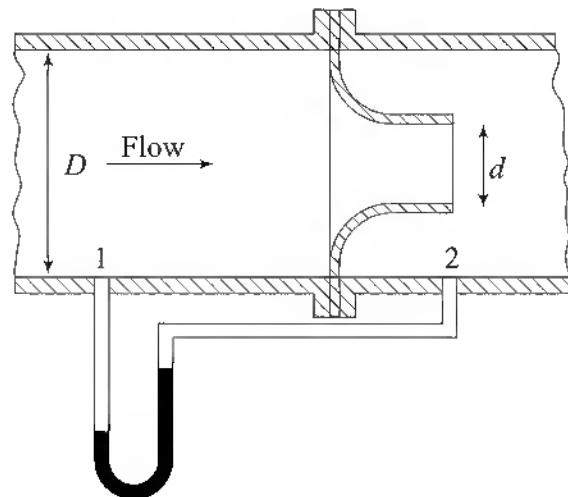


Fig. 5.14

5.7 PROCESS DESIGN OF ORIFICE METER

Orifice meter is a widely used flow meter in chemical industry, compared to venturi meter and rotameter. Advantages of orifice meter are:

- (i) Fixed cost is less
- (ii) Easy to fabricate and install
- (iii) Occupies less space as compared to the venturi meter
- (iv) Provides more flexibility. Orifice plate can be easily replaced.

Disadvantages:

- (i) Power consumption and hence operating cost of orifice meter is higher than the same of venturi meter and of rotameter.

In orifice meter, as shown in Fig. 5.10, a square edged or sharp edged orifice plate is mounted between two flanges at the flanged joint. When fluid flows through the orifice it forms free flowing jet. This free flowing jet first contracts and then expands. Minimum flow area achieved by free flowing jet is known as Vena Contracta.

Mass flow rate through orifice is given by following equation.

$$\dot{m} = C_o Y A_o \sqrt{\frac{2g_c(p_2 - p_1)\rho}{1 - \beta^4}} \quad (5.29)$$

where \dot{m} = Mass flow rate of fluid, kg/s

A_o = Area of orifice = $(\pi/4)d_o^2$, m²

p_1, p_2 = Pressure at upstream and downstream static pressure taps, respectively, Pa

ρ = Density of fluid, kg/m³

$$\beta = \frac{\text{Diameter of orifice}}{\text{Inside diameter of pipe}}$$

Y = expansion factor

= 1 for liquids

= $1 - [(1 - r)/k](0.41 + 0.35 \beta^4)$] for gases

(5.30)

where, $r = \frac{p_2}{p_1}$, ratio of downstream to upstream pressure

$$k = \frac{C_p}{C_v}, \text{ specific heat ratio}$$

C_o = Coefficient of orifice = $f(Re_o, \beta, \text{Location of taps})$

where, Re_o = Reynold's number for the fluid flowing through orifice

$$Re_o = \frac{d_o u_o \rho}{\mu} = \frac{4\dot{m}}{\pi d_o \mu} \quad (5.31)$$

where, u_o = Velocity of fluid through orifice, m/s

μ = Viscosity of fluid, kg/(m · s)

Equation of orifice meter (Eq. 5.29) is empirical in nature and it requires experimental support.

In addition to Re_o and β , C_o also depends on the location of pressure taps. Location of pressure taps on upstream side and down stream side are also standardised as discussed in Fig. 5.10 (a).

There are total five standard locations of pressure taps.

- (i) **Corner taps:** Static holes made in upstream and downstream flange. They are very close to the orifice plate. With corner taps, it is possible to drill both static holes in the orifice plate itself. Then entire orifice meter can be easily inserted in any flanged joint without drilling the holes in pipe or flanges.
- (ii) **Flange taps:** Static holes made at a distance 25.4 mm on upstream side and 25.4 mm on downstream side.
- (iii) **Radius taps:** Static holes located at a distance one pipe diameter on upstream side and 1/2 pipe diameter on downstream side. Radius taps are the best from practical stand point of view as it gives reasonably good pressure difference, compared to other taps except vena contracta taps. Higher pressure difference means more accurate measurement of flow rate.
- (iv) **Vena contracta taps:** Upstream static hole is 1/2 to 2 times pipe diameter from the plate. Downstream tap is located at the position of minimum pressure. Vena contracta taps give the maximum pressure difference for a given flow rate. But it is not suitable, if orifice size is changed from time to time.
- (v) **Pipe taps:** Static holes are located at 2.5 times pipe diameter upstream side and 8 times pipe diameter on down stream side. This means fluid is flowing normally on both sides without being affected by turbulence, created by the orifice plate.

For $Re_o > 30\,000$,

C_o = Between 0.595 to 0.62 for vena contracta taps

C_o = Between 0.595 to 0.8 for radius taps

C_o = 0.62 for corner taps

Relation between discharge coefficient C_o , β and Re_D is given by Stoltz equation (ISO: 5167).

$$C_o = 0.5959 + 0.0312 \beta^{2.1} - 0.184 \beta^8 + 0.0029 \beta^{2.5} (10^6 / Re_D)^{0.75} + 0.09 L_1 \beta^4 (1 - \beta^4)^{-1} - 0.0337 L_2 \beta^3 \quad (5.32)$$

where Re_D = Reynolds number based on internal diameter of pipe D

$$L_1 = \left(\frac{l_1}{D} \right), L_2 = \left(\frac{l_2}{d_o} \right)$$

where, d_o = diameter of orifice

$$\beta = d_o/D$$

l_1 = Distance of the upstream tapping from the upstream face of orifice plate, mm

l_2 = Distance of the downstream tapping from the downstream face of the orifice plate, mm

Example 5.21

Design an orifice meter based on the following data.

Name of fluid = water

Flow rate = 100 000 kg/h

Inside diameter of pipe = 154 mm (6 in, SCH-40 pipe)

Operating temperature = 32°C

Density of water at 32°C = 995.026 kg/m³

Viscosity of water at 32°C = 0.765 mPa · s or cP

Manometer fluid = Mercury

Density of Mercury at 32°C = 13 516.47 kg/m³

Solution:

Inside diameter of pipe D = 154 mm

Let $\beta = 0.5$, $d_o/D = 0.5$

diameter of orifice, $d_o = 77$ mm

Re_D = Reynolds number based on inside diameter of pipe

$$Re_D = \frac{4\dot{m}}{\pi D \mu} = \frac{4 \times (100\ 000/3600)}{\pi \times 0.154 \times 0.765 \times 10^{-3}}$$

$$= 300\ 210.2$$

Use square edged circular orifice plate with radius taps.

Radius taps are best from practical stand point. In radius taps downstream tap is located approximately at the mean location of vena contracta (1/2 times pipe diameter) and also upstream tap is sufficiently far so that it is not affected by distortion of flow in the vicinity of orifice.

Based on Stolz's equation Eq. (5.32),

$$C_o = 0.5959 + 0.0312\beta^{2.1} - 0.184\beta^8 + 0.0029\beta^{2.5} \left(\frac{10^6}{Re_D} \right)^{0.75}$$

$$+ 0.09L_1\beta^4(1-\beta^4)^{-1} - 0.0337L_2\beta^3 \quad (5.32)$$

$$L_1 = \frac{l_1}{D} = 1, L_2 = \frac{l_2}{d_o} = 1$$

$$C_o = 0.5959 + 7.2776 \times 10^{-3} - 7.1875 \times 10^{-4} + 1.264 \times 10^{-3} + 6 \times 10^{-3}$$

$$- 4.2125 \times 10^{-3}$$

$$= 0.6055$$

Mass flow rate of water

$$\dot{m} = C_o Y A_o \sqrt{\frac{2 g_c \Delta p \rho}{1 - \beta^4}} \quad (5.29)$$

Y = expansion factor for liquid = 1

$$\frac{100\ 000}{3600} = 0.6055 \times 1 \times \frac{\pi}{4} (0.077)^2 \times \sqrt{\frac{2 \times 1 \times \Delta p \times 995.026}{1 - 0.5^4}}$$

$$\Delta p = 45\ 722.597 \text{ Pa} \equiv 45.722 \text{ kPa} \equiv 4.661 \text{ m WC}$$

$$\Delta p = R_m \frac{g}{g_C} (\rho_m - \rho_f)$$

$$45\ 722.597 = R_m \times \frac{9.81}{1} (13\ 516.47 - 995.026)$$

$$\begin{aligned} R_m &= \text{Manometer reading} = 0.3722 \text{ m Hg} \\ &\equiv 372.2 \text{ mm Hg} \end{aligned}$$

In actual industrial practice, DP transmitter is used. Select the transmitter with 5500 mm WC range so that the normal reading is at about 85% of the range.

Similarly, one can find different values of R_m for the different variation in flow rate or in other words calibration of orifice meter can be carried out.

Example 5.22

Design an orifice meter based on the following data.

Name of fluid = Chlorine gas

Flow rate = 1500 Nm³/h

Operating Pressure = 1.2 atm a

Operating temperature = 30°C

Viscosity of Chlorine gas at 30°C = 0.0145 mPa · s or cP

Inside diameter of pipe = 154 mm (6 in. SCH-40)

Specific heat ratio for Cl₂ gas = 1.355

Solution:

Let $\beta = 0.5$

$$\frac{d_o}{D} = 0.5 \quad \text{or} \quad d_o = 77 \text{ mm}$$

$$Re_D = \frac{4\dot{m}}{\pi D \mu}$$

Density of Cl₂ gas at normal condition

$$\rho = \frac{1 \times 71}{(273 + 25)} \times \frac{273}{1 \times 22.414} = 2.902 \text{ kg/m}^3$$

$$\dot{m} = 1500 \times 2.902 = 4353 \text{ kg/h}$$

$$Re_D = \frac{4 \times (4353/3600)}{\pi \times 0.154 \times 0.0145 \times 10^{-3}}$$

$$= 689\ 457.6$$

If corner taps are made then $l_1 = l_2 = 0$

Coefficient of orifice meter can be determined by Stolz's equation.

$$\begin{aligned} C_o &= 0.5959 + 0.0312 \beta^{2.1} - 0.184 \beta^8 + 0.0029 \beta^{2.5} \left(\frac{10^6}{Re_D} \right)^{0.75} \\ &\quad + 0.09 L_1 \beta^4 (1 - \beta^4)^{-1} - 0.0337 L_2 \beta^3 \\ &= 0.5959 + 7.2776 \times 10^{-3} - 7.1875 \times 10^{-4} + 6.7755 \times 10^{-4} \\ &= 0.6031 \end{aligned} \quad (5.32)$$

Expansion factor

$$\begin{aligned} Y &= 1 - [((1-r)/k)(0.41 + 0.35\beta^4)] \\ &= 1 - [((1-r)/1.355) \times 0.4319] \end{aligned}$$

For the first trial calculation let $r = 0.8$, $Y = 0.9362$

Mass flow rate of chlorine gas

$$\dot{m} = C_o Y \frac{\pi}{4} d_o^2 \sqrt{\frac{2g_c \Delta p \rho}{1 - \beta^4}} \quad (5.29)$$

Density of chlorine gas at operating condition:

$$\rho = \frac{1.2 \times 71}{(273 + 30)} \times \frac{273}{1 \times 22.414} = 3.425 \text{ kg/m}^3$$

$$\frac{4353}{3600} = 0.6031 \times 0.9362 \times \frac{\pi}{4} (0.077)^2 \times \sqrt{\frac{2 \times 1 \times \Delta p \times 3.425}{1 - 0.5^4}}$$

$$\begin{aligned} \Delta p &= 28946.4 \text{ Pa} \\ &= 28.946 \text{ kPa} \end{aligned}$$

$$\begin{aligned} p_1 &= 1.2 \text{ atm}, p_2 = p_1 - \Delta p = 1.2 \times 101.325 - 28.946 \\ p_2 &= 92.644 \text{ kPa} \end{aligned}$$

$$r = \frac{p_2}{p_1} = \frac{92.644}{121.59} = 0.762, Y = 0.9241$$

$$\Delta p \propto \frac{1}{Y^2} \quad \text{or} \quad \Delta p_1 Y_1^2 = \Delta p_2 Y_2^2$$

$$\Delta p_2 = 28.946 \times \left(\frac{0.9362}{0.9241} \right)^2 = 29.7 \text{ kPa}$$

$$r = \frac{(121.59 - 29.7)}{121.59} = 0.7557 \approx 0.762 \text{ (close enough to the previous value of } r \text{)}$$

$$\Delta p_2 = 29.7 \text{ kPa} \equiv 3.028 \text{ m WC (final)}$$

Similarly, for the different variation in the flow rate Δp can be determined. In this case also, a DP transmitter having 3500 mm WC range can be selected. Normal reading will be at about 87% of the range.

5.8 PROCESS DESIGN OF ROTAMETER

Rotameter is used for the flow measurement of liquids and gases. It is a variable flow area meter in which pressure drop is nearly constant and the area through which the fluid flows varies with the flow rate. It consists of tapered tube with the smallest diameter at the bottom. The tube contains a freely moving float which rests on a stop at the base of the tube. When the fluid is flowing the float rises until its weight is balanced by the upthrust of the fluid, its position then indicates the rate of flow. Float is **not** floating but it is completely submerged in the fluid and its density is greater than the density of fluid. The tube is marked in divisions

and the reading of the meter is obtained from the scale reading at the reading edge of the float which is taken at the largest cross section of the float. It is always mounted in the perfect vertical position. Rotameters are designed to measure the flow rate of fluid in certain range like $5 \text{ m}^3/\text{h}$ to $50 \text{ m}^3/\text{h}$, $10 \text{ m}^3/\text{h}$ to $100 \text{ m}^3/\text{h}$, 3 Lpm to 30 Lpm , etc.

For a linearly tapered tube with a diameter at the bottom about the float diameter, flow is almost a linear function of the height or position of float. As a tube material glass is the first choice. For high temperatures or pressures or for other conditions where glass is impracticable, metal tubes are used. In this case since, in metal tube, the float is invisible, means must be provided for either indicating or transmitting the meter reading.

Mass flow rate through rotameter is given by equation

$$q_m = C_D A_2 \sqrt{\frac{2gV_f (\rho_f - \rho) \rho}{A_f [1 - (A_2/A_1)^2]}} \quad (5.33)$$

where, q_m = Mass flow rate, kg/s

C_D = Discharge co-efficient depends on the shape of the float and the Reynolds number though annulus. It can be obtained from literature such as Fig. 2.22 of Ref: 11. For the float having the shape same as shown in Fig. 5.15, value of C_D is almost constant for the value of Re above 500 and it is 0.775.

A_2 = Cross sectional area of annulus between float and tube, m^2

$A_2 = A_1 - A_f$

A_1 = Cross sectional area of tube at the float position, m^2

A_f = Maximum cross sectional area of the float in a horizontal plane, m^2

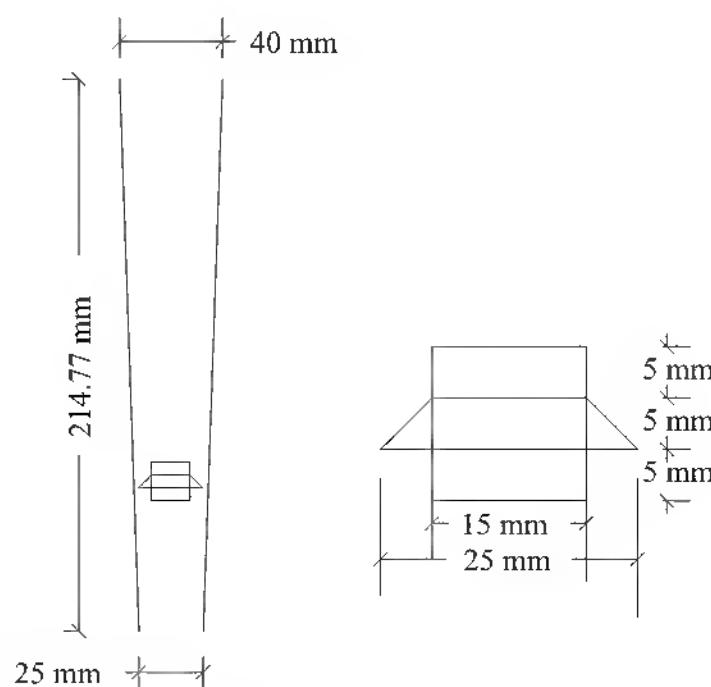


Fig. 5.15 Typical Details of Float and Tube

g = Acceleration of gravity = 9.81 m/s² at mean sea level

V_f = Volume of float, m³

ρ_f = Density of float, kg/m³

ρ = Density of fluid, kg/m³

Example 5.23

Determine the minimum and maximum flow rates of water at 40°C, which can be measured by rotameter of the given dimensions. Dimensions of tube and float of rotameter are given in Fig. 5.15.

Viscosity of water of 40°C = 0.7 cP or mPa · s

Density of water at 40°C = 992.2 kg/m³

Float material = Stainless steel

Density of stainless steel = 8000 kg/m³

Float is made from stainless steel plate of 2 mm thickness. Calibrate the rotameter or find the different values of flow rates for the different float positions.

Solution:

Volume of float:

$$V_f = \left(2 \times \frac{\pi}{4} (15)^2 \times 5 + \frac{\pi}{4} \left(\frac{25+15}{2} \right)^2 \times 5 \right) \times 10^{-9}$$

$$V_f = 3.338 \times 10^{-6} \text{ m}^3$$

Mass of float:

$$\begin{aligned} W_f &= \left(2 \times \frac{\pi}{4} (15)^2 + \pi (15)(10) + \frac{\pi}{4} (25^2 - 15^2) + \pi \left(\frac{15+25}{2} \right) \times 5 \right) \\ &\quad \times 10^{-6} \times 0.002 \times 8000 \\ &= 0.02325 \text{ kg} \end{aligned}$$

$$\text{Density of float} = \frac{W_f}{V_f} = \frac{0.02325}{3.338 \times 10^{-6}} = 6965 \text{ kg/m}^3$$

Maximum flow rate at top:

Mass flow rate through Rotameter is given by equation

$$q_m = C_D A_2 \sqrt{\frac{2 g V_f (\rho_f - \rho) \rho}{A_f \left[1 - \left(\frac{A_2}{A_1} \right)^2 \right]}} \quad (5.33)$$

For the 1st trial calculation, let discharge coefficient, $C_D = 0.75$.

A_1 = Cross sectional area of tube

$$= \frac{\pi}{4} (40)^2 = 1256.6 \text{ mm}^2 \equiv 1256.6 \times 10^{-6} \text{ m}^2$$

A_f = Maximum cross sectional area of the float in a horizontal plane, m²

$$= \frac{\pi}{4} (25)^2 = 490.87 \text{ mm}^2 \equiv 490.87 \times 10^{-6} \text{ m}^2$$

A_2 = Cross sectional area of annulus between float and tube, m^2

$$A_2 = A_l - A_f = 765.767 \times 10^{-6} \text{ m}^2, \frac{A_2}{A_l} = 0.6094$$

$$q_m = 0.75 \times 0.765767 \times 10^{-3} \sqrt{\frac{2 \times 9.81 \times 3.338 \times 10^{-6} \times 992.2 \times (6965 - 992.2)}{490.87 \times 10^{-6} [1 - 0.6094^2]}}$$

$$= 0.6441 \text{ kg/s}$$

$$\equiv 2318.78 \text{ kg/h} \equiv 2.337 \text{ m}^3/\text{h} \text{ (taking } \rho = 0.9922 \text{ kg/L for water)}$$

Mass velocity through annulus:

$$G = \frac{q_m}{A_2} = \frac{0.6441}{0.765767 \times 10^{-3}} = 841.12 \text{ kg/(m}^2 \cdot \text{s)}$$

Equivalent diameter:

$$d_e = \frac{4 \times \text{Cross Sectional area}}{\text{Wetted Parameter}}$$

$$= \frac{4 \times 765.767 \times 10^{-6}}{\pi (40 + 25) \times 10^{-3}} = 0.015 \text{ m}$$

$$Re = \frac{d_e G}{\mu} = \frac{0.015 \times 841.12}{0.7 \times 10^{-3}} = 18024$$

From Fig. 6.22 of Ref. 11

$$C_D = 0.775$$

$$q_m = \frac{0.775}{0.75} \times 0.644 = 0.6655 \text{ kg/s}$$

$$\equiv 2395.7 \text{ kg/h} \equiv 2.414 \text{ m}^3/\text{h} \text{ (corrected)}$$

First mark for indicating flow rate is at 27 mm diameter of tube.

$$A_1 = \frac{\pi}{4} (27)^2 = 572.55 \text{ mm}^2 \equiv 572.55 \times 10^{-6} \text{ m}^2$$

$$A_2 = A_1 - A_f = 81.685 \times 10^{-6} \text{ m}^2$$

$$\frac{A_2}{A_l} = 0.14267$$

Let $C_D = 0.775$ (for 1st trial)

$$q_m = 0.775 \times 81.685 \times 10^{-6} \sqrt{\frac{2 \times 9.81 \times 3.338 \times 10^{-6} \times 992.2 \times (6965 - 992.2)}{490.87 \times 10^{-6} [1 - 0.14267^2]}}$$

$$= 0.056873 \text{ kg/s} \equiv 204.7 \text{ kg/h} \equiv 0.2063 \text{ m}^3/\text{h}$$

$$G = \frac{0.056873}{81.685 \times 10^{-6}} = 696.25 \text{ kg/(m}^2 \cdot \text{s)}$$

$$d_e = \frac{4 \times 81.685 \times 10^{-6}}{\pi(27 + 25) \times 10^{-3}} = 0.002 \text{ m}$$

$$Re = \frac{d_e G}{\mu} = \frac{0.002 \times 696.25}{0.7 \times 10^{-3}} = 1989.3$$

For $Re = 1989.3$, $C_D = 0.775$ (from Fig. 6.22 of Ref. 11). Hence, no change in q_m or q_v . Based on similar calculations, Table 5.11 is prepared.

Table 5.11 Flow Rate Calculation for Rotameter

Tapered tube Diameter, mm	A_1 mm^2	A_2 mm^2	A_2/A_1	$q_v \cdot \text{m}^3/\text{h}$
25	490.87	—	—	0
27	572.55	81.685	0.142 67	0.2063
30	706.85	215.98	0.3056	0.5672
32	804.25	313.38	0.3897	0.85
34	907.92	417.05	0.4593	1.174
36	1017.876	527	0.5178	1.54
38	1134.11	643.24	0.5672	1.9528
40	1256.6	765.73	0.6094	2.4148

Let angle made by tapered tube with vertical plane is 2 degrees.

Let h = Float position in mm from the bottom most point (i.e. from the point where diameter of tube is 25 mm)

For $q_v = 2.4146 \text{ m}^3/\text{h}$, inside diameter of tapered tube = 40 mm

Value of h at 40 mm diameter

$$h = \frac{(40 - 25)/2}{\tan 2} = 214.77 \text{ mm}$$

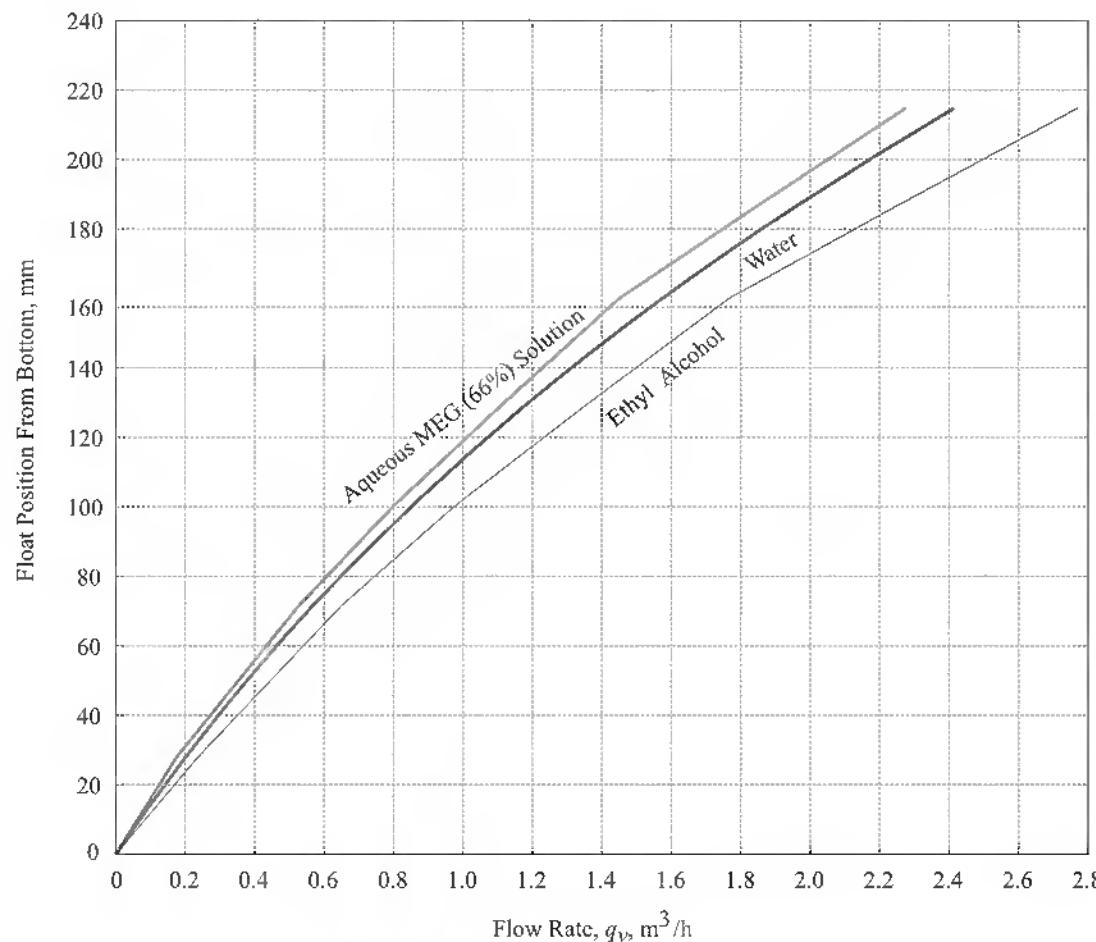
Similarly float position, h can be determined for the different flow rates.

Table 5.12 Float Position Calculations for Rotameter

Diameter of tapered tube, mm	$q_v \cdot \text{m}^3/\text{h}$	h , float position from bottom, mm
25	0	0
27	0.2063	28.64
30	0.5672	71.59
32	0.85	100.23
34	1.174	128.86
36	1.54	157.5
38	1.9528	186.135
40	2.4148	214.77

Draw the graph of h vs q_v .

Refer Fig. 5.16.

**Fig. 5.16 Calibration of Rotameter****Example 5.24**

In Example 5.23 a rotameter is calibrated to measure the flow rate of water at 40°C. If the same rotameter is used for measuring the flow rates of following fluids then what is the change in calibration?

- (a) Monoethylene glycol (MEG) brine containing 60% MEG by mass¹²
Operating temperature: -25°C
Operating pressure: 3 bar g
Density: 1098 kg/m³
Viscosity: 40 mPa · s (or cP)
- (b) Ethyl alcohol
Operating temperature: 35°C
Pressure: 3 bar g
Density: 781 kg/m³
Viscosity: 0.9 mPa · s (or cP)

Solution:

At the top most position of the float,
 $h = 214.77$ mm, diameter of tapered tube = 40 mm,

$$A_1 = 1256.6 \text{ mm}^2, \quad A_2 = 765.73 \text{ mm}^2, \quad \frac{A_2}{A_1} = 0.6094$$

For this position of float, flow rate of MEG:

$$q_m = C_D A_2 \sqrt{\frac{2gV_f(\rho_f - \rho)\rho}{A_f \left[1 - \left(\frac{A_2}{A_1} \right)^2 \right]}} \quad (5.33)$$

For the 1st trial calculations, let $C_D = 0.775$

$$q_m = 0.775 \times 765.73 \times 10^{-6} \sqrt{\frac{2 \times 9.81 \times 3.338 \times 10^{-6} \times 1098 \times (6965 - 1098)}{490.87 \times 10^{-6} [1 - 0.6094^2]}}$$

$$= 0.6939 \text{ kg/s}$$

$$\text{or } q_v = \frac{0.6939}{1098} \times 3600 = 2.275 \text{ m}^3/\text{h}$$

$$\text{Mass velocity, } G = \frac{q_m}{A_2} = \frac{0.6939}{765.73 \times 10^{-6}} = 906.194 \text{ kg/(m}^2 \cdot \text{s)}$$

$$Re = \frac{d_e G}{\mu} = \frac{0.015 \times 906.194}{40 \times 10^{-3}} = 339.82$$

Value of $C_D = 0.77$ (Fig. 6.22 of Ref.: 11)

$$q_m = \frac{0.77}{0.775} \times 0.6939 = 0.6894 \text{ kg/s}$$

$$\text{or } q_v = 2.26 \text{ m}^3/\text{h}$$

For the same position of float, flow rate of ethyl alcohol:

Let $C_D = 0.775$ (for 1st trial calculations)

$$q_m = 0.775 \times 765.73 \times 10^{-6} \sqrt{\frac{2 \times 9.81 \times 3.338 \times 10^{-6} \times 781 \times (6965 - 781)}{490.87 \times 10^{-6} [1 - 0.6094^2]}}$$

$$= 0.6 \text{ kg/s}$$

$$q_v = \frac{0.6}{781} \times 3600 = 2.77 \text{ m}^3/\text{h}$$

$$\text{Mass velocity, } G = \frac{q_m}{A_2} = \frac{0.6}{765.73 \times 10^{-6}} = 783.57 \text{ kg/(m}^2 \cdot \text{s)}$$

$$Re = \frac{d_e G}{\mu} = \frac{0.015 \times 783.57}{0.9 \times 10^{-3}} = 13059.5$$

$C_D = 0.775$ (from Fig. 6.22 of Ref.: 11)

No change in the values of q_m and q_v .

At diameter of tapered tube = 27 mm,

Float position, $h = 28.64$ mm,

$$A_1 = 572.55 \text{ mm}^2, \quad A_2 = 81.685 \text{ mm}^2, \quad A_2/A_1 = 0.14267$$

Flow rate of MEG: For the 1st trial, let $C_D = 0.77$

$$q_m = 0.77 \times 81.685 \times 10^{-6} \sqrt{\frac{2 \times 9.81 \times 3.338 \times 10^{-6} \times 1098(6965 - 1098)}{490.87 \times 10^{-6}[1 - 0.14267^2]}}$$

$$q_m = 0.059 \text{ kg/s}$$

$$q_v = \left(\frac{0.059}{1098}\right) \times 3600 = 0.19344 \text{ m}^3/\text{h}$$

$$G = \frac{q_m}{A_2} = \frac{0.059}{81.685 \times 10^{-6}} = 722.287 \text{ kg/(m}^2 \cdot \text{s)}$$

$$Re = \frac{d_e G}{\mu} = \frac{0.002 \times 722.287}{40 \times 10^{-3}} = 36.114$$

$C_D = 0.7$ (from Fig. 6.22, Ref. 11)

$$q_m = \frac{0.7}{0.77} \times 0.059 = 0.0536 \text{ kg/s}$$

$$q_v = 0.1757 \text{ m}^3/\text{h}$$

For the same position of float, flow rate of ethyl alcohol:

Let $C_D = 0.775$ (for 1st trial calculation)

$$q_m = 0.775 \times 81.685 \times 10^{-6} \sqrt{\frac{2 \times 9.81 \times 3.338 \times 10^{-6} \times 781 \times (6965 - 781)}{490.87 \times 10^{-6}(1 - 0.14267^2)}}$$

$$q_m = 0.05134 \text{ kg/s}$$

$$q_v = 0.2367 \text{ m}^3/\text{h}$$

$$G = \frac{q_m}{A_2} = \frac{0.05134}{81.685 \times 10^{-6}} = 628.5 \text{ kg/(m}^2 \cdot \text{s)}$$

$$Re = \frac{d_e G}{\mu} = \frac{0.002 \times 628.5}{0.9 \times 10^{-3}} = 1396.67$$

$C_D = 0.775$ (From Fig. 6.22 of Ref.: 11)

Hence q_m and q_v remain unchanged.

By similar calculations following table is prepared.

Table 5.13 Flow Rates of Different Fluids for the Same Position of Float

Float position h , mm	Diameter of tapered tube D , mm	q_v m ³ /h		
		Water	MEG Solution	Ethyl alcohol
0	25	0	0	0
28.64	27	0.2063	0.1757	0.2367
71.59	30	0.5672	0.5274	0.65
100.23	32	0.85	0.8	0.976
128.86	34	1.174	1.106	1.348
157.5	36	1.54	1.45	1.766
186.135	38	1.9528	1.84	2.24
214.77	40	2.4148	2.26	2.77

Following Table is made from the graph (Fig. 5.16).

Table 5.14 Calibration for Rotameter

$q_v, \text{m}^3/\text{h}$	Float Position, h mm		
	Water	MEG Solution	Ethyl Alcohol
0.1	14.31	15.94	11.828
0.2	27.8	31.02	23.66
0.4	52.67	55.8	45.224
0.6	75.06	79.22	66.28
0.8	95.21	100.00	84.77
1.0	113.81	118.94	102.08
1.2	131.0	137.37	117.47
1.4	146.93	155.47	132.73
1.6	161.89	170.05	147.63
1.8	175.96	183.46	161.88
2.0	189.19	196.67	172.9
2.2	201.81	209.83	183.93
2.4	213.91	—	194.78

Comments: It can be seen from Table 5.13 that rotameter calibration is essentially immune from the viscosity effect (say upto about 100 mPa.s or cP). However, calibration is significantly affected by the density of the fluid which changes drag force.

In actual practice, rotameters are tested for actual water capacities on a test bench to confirm the calibration.

5.9 TWO PHASE FLOW

In many applications, two-phase flow is experienced in industry. If pipe sizing is not done properly, it can result in serious problems. A few such problems are illustrated as follows:

- (a) When a saturated liquid under pressure is flashed through a control valve, two-phase flow will be realised in the downstream piping. Pressure drop in the two-phase flow could be much higher (several fold in some cases) in the pipe. This will result in much higher back pressure on the control valve, restricting its capacity. Apart from excessive pressure drop, there could be erosion in the pipeline.
- (b) When vapour and liquid flow in upward direction and the line is sized for low pressure drop, it may cause slug flow; i.e. slugs of liquid flow in vapour. This can result in pressure pulsation and severe vibration. This phenomenon is commonly observed and is known as water hammer in steam lines, particularly during cold start-up when steam condenses during travel and water slugs flow with steam. Water hammers can break open the flanges or damage the supports. Adequate draining facility is provided on long steam pipelines to avoid such water hammers.
- (c) If cooling water is flowing downward at temperature in excess of 40°C from a height of 10.5 m or more to an atmospheric pond, vibrations could result due to vapour formation at the top and two phase formation in downflow pipe. A provision of appropriate size orifice at the base of the pipe could solve the problem.

A process engineer should visualize such a problem and design the piping suitably so that both the phases flow smoothly without creating any problem.

Estimation of pressure drop in an isothermal two-phase system can be done with empirical relations, developed by Martinelli's and Dukler's correlations. Simpson⁷ has dealt at a length on both the correlations and original article by Simpson may be referred for the details.

A common problem of two-phase flow is experienced in steam condensate lines. When steam is used for heating in a heat exchanger, its condensate is discharged through a steam trap. While the heat exchanger operates under pressure, the trap discharges condensate to low pressure. In most cases, the condensate line opens into an atmospheric storage tank while in a few instances, condensate is conveyed to a deaerator, operating under positive pressure. In both cases, flashing of condensate takes place which calls for larger size pipe. If not designed properly, water hammer could be anticipated and also the back pressure on the trap could be much higher than anticipated. Steam traps are sized and rated for a specific pressure drop and condensate flow discharge rate. If the pressure drop is less than design, trap's capacity will be reduced and condensate accumulation is expected in the heat exchanger, thereby heat transfer will be affected.

Empirical correlations could be used for calculating pressure drop in steam condensate pipes. However, pipe sizing could be done with the help of Fig. 5.17 which is based on approximate pressure drop of 0.1 kPa/m (or 1 mbar/m) length of pipe. This represents only friction loss. To this head loss and equipment pressure (such as that of deaerator) are to be added to arrive at a back pressure of the trap.

Example 5.25

Saturated steam at 10 bar g is used in a heat exchanger at the rate of 2000 kg/h. Condensate is discharged through a trap and taken to a deaerator, located at 10 m above the heat exchanger. Pipe length, including equivalent length of fittings, is calculated to be 60 m. Deaerator operates at 0.4 bar g. Size the condensate pipe and calculate the back pressure on the trap.

Solution:

Assume back pressure of 2 bar on the trap.

Steam at 10 bar g has saturation temperature of 184.13°C and condensate has specific enthalpy of 781.6 kJ/kg (from Steam Tables). At 2 bar g, specific enthalpy of condensate and flash vapours will be 562.2 kJ/kg and 2725.5 kJ/kg, respectively. If \dot{m} is the flash steam rate after the trap, enthalpy balance can be written as

$$2000 \times 781.6 = \dot{m} \times 2725.5 + (2000 - \dot{m}) 562.2$$

$$\dot{m} = 202.8 \text{ kg/h}$$

This represents 10.14 % flashing. Specific volume of flash steam at 2 bar g and 133.7°C is 0.603 m³/kg.

Thus volumetric flow rate of flash steam will be 122.29 m³/h.

From Fig. 5.17, pipe size for 25 m/s flash steam velocity is read between 40 and 50 mm NB. Select 50 mm NB condensate pipe, downstream of trap which could be of 25 mm size.

Friction loss in pipe = $0.1 \times 60 = 6 \text{ kPa} \equiv 0.06 \text{ bar}$

Deaerator pressure = 0.4 bar

Elevation of deaerator = 10 m = 0.98 bar

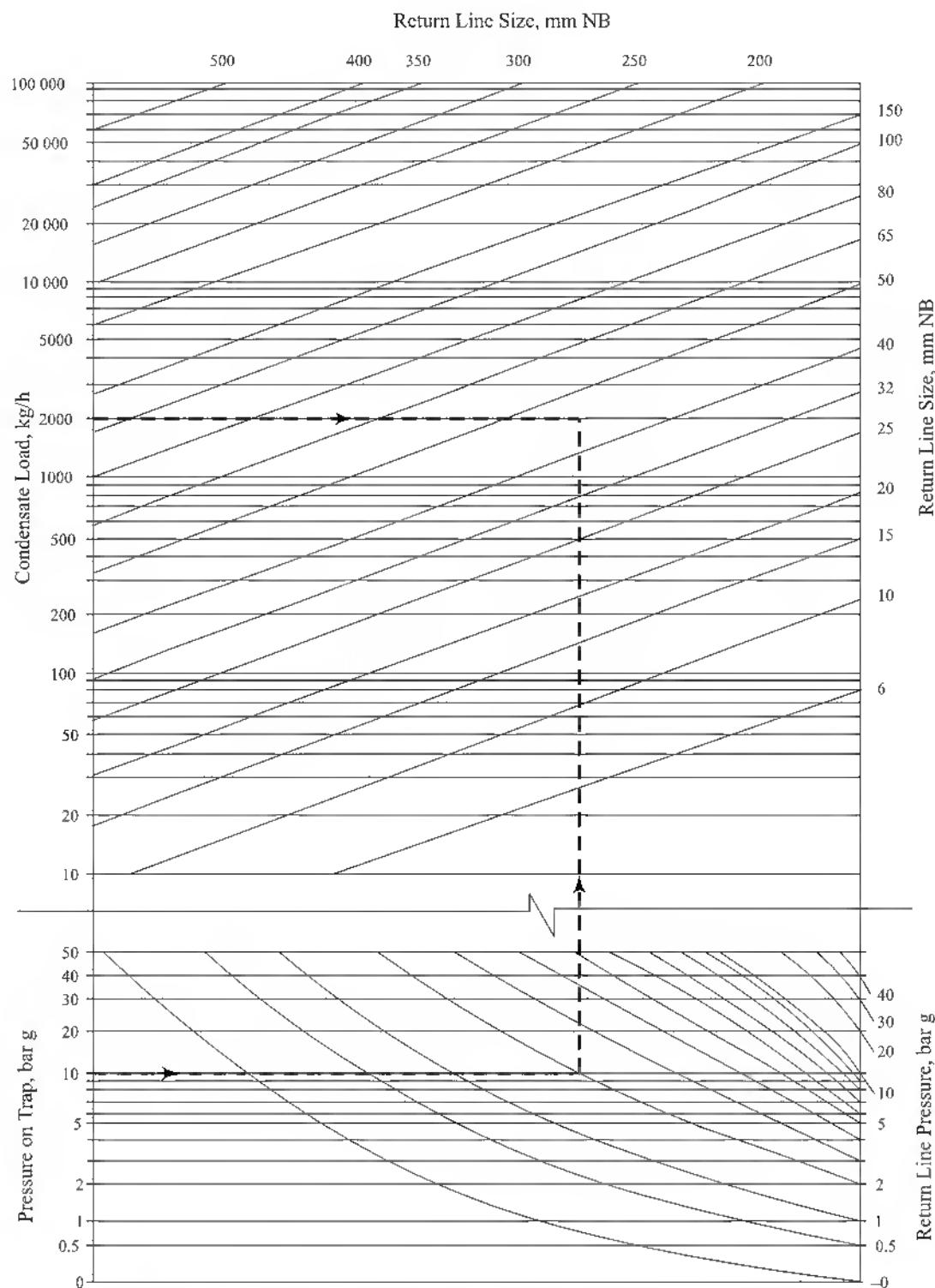


Fig. 5.17 Steam Condensate Line Sizing Chart
 (Reproduced with the Permission of Spirax-Sarco Ltd., UK)

$$\text{Total back pressure} = 0.06 + 0.4 + 0.98 = 1.44 \text{ bar}$$

Calculated back pressure is less than the assumed back pressure. Condensate pipe design should therefore be satisfactory.

Refrigerants are another class of fluids which produce flash vapours on pressure reduction of saturated liquids. Quantity of flash vapour normally varies from 15 to 40 %, depending on the refrigerant and the pressure difference across the

expansion valve. To avoid any problem of high pressure drop, the expansion valve is situated close to the evaporator.

Charts, such as Fig. 5.17, are not readily available in literature for other fluids. Hence, empirical equations for two phase flow are to be used for pressure drop estimation.

5.10 TROUBLESHOOTING OF FLUID FLOW SYSTEMS

Utilities such as cooling water, steam, refrigeration, etc. are important to process industry. Design of utility network should therefore be carried out carefully so that no user equipment starves of any utility. This can also result in energy saving.

Apart from utilities, piping of process of fluids also require careful design. Piping should neither offer excessive pressure drop nor should be overdesigned as initial cost could be a deterrent.

In many instances, problems might arise in actual operation due to various reason. Plant might be operated at a higher capacity thereby pumping capacity might become limiting, higher pressure drop might be experienced in pipe line, etc. In another example, fluid distribution in manifold piping might not be satisfactory. In yet another instance, a new boiler of higher capacity is added and steam from the new boiler is carried in the existing pipelines. In all these situations, process engineer is required to evaluate the bottlenecks and carry out troubleshooting exercise.

Take the instance of installation of new higher capacity boiler. If pressure drop calculations reveal high pressure drop in the existing pipeline, process engineer can consider laying of another parallel pipeline from the boiler to nearest steam consuming equipment. Alternately, steam at a higher pressure is conveyed from the boiler if the existing pipeline rating permits its operation at higher pressure.

Instrument air manifold becomes a bottleneck in many situations as more instruments are added over a period of operation. Also, fluctuations in supply instrument air pressure could be a problem. After careful evaluation, installation of an air receiver of adequate size near the plant may prove valuable.

For pumping system, upgradation of existing centrifugal pumps offers many options. While evaluating the options, safety, reliability, ease of maintenance and cost benefit analysis must be considered. Chaware *et al*¹³ have dealt in detail the upgrading options of a centrifugal pump. Impeller replacement, operation of two pumps in parallel, installation of a bigger pump, change of a type of a pump, etc. are the options that can be considered. Process engineer can contribute significantly in troubleshooting.

EXERCISES

5.1 Answer the following:

- (a) At 7 bar g, the leakage of steam through 3 mm, 6 mm and 12.5 mm diameter holes amounts to 34, 230 and 540 kg/h, respectively. One of the leakage figures is a typographical error. Which figure? Why?

- (b) A 2-stage reciprocating compressor is operating at 0.7 bar a inlet and 6.2 bar a outlet pressure. If the pressure drop across the intercooler is 0.14 bar, the pressure at the inlet of second stage will be approximately; (a) 3.38 bar a, (b) 2.69 bar a or (c) 2.0 bar a.
- (c) If volumetric flow rate and head of a centrifugal pump are proportional to speed raised to power 0.8 and 1.5, respectively (for a given specific speed), then power would be proportional to speed raised to power: (a) 3, (b) 2.3, (c) 2.0, (d) 1.5 or (e) 1.7.
- (d) Pressure drop in a 150 mm NB diameter pipe in a well designed distribution system is 0.07 bar due to water flowing at a certain rate. What will be the pressure drop in 80 mm NB pipe for the same length and same flow rate? (a) 4.4 bar (b) 1.62 bar (c) 1.1 bar (d) 0.55 bar (e) 0.276 bar or (e) 0.138 bar.
- (e) An orifice type flow meter, installed on a liquid stream is coupled to a differential pressure (DP) transmitter and a flow recorder. Normal reading is 5 on a 0–10 square-root scale and 25 on a 0–100 linear scale. If the range of the DP transmitter is doubled, the reading will be
 (i) 2.5 on square-root scale and 6.3 on linear scale.
 (ii) 2.25 on square-root scale and 12.5 on linear scale or
 (iii) 7.1 on square-root scale and 50 on linear scale.
- (f) For a centrifugal pump, power formula can be written as $P = 80 K (d)^3$
 where, P = power in hp
 K = dimensionless pump constant
 d = impeller diameter, in

To write this formula in metric units, P should be in kW and d should be in cm.
 What will be the constant (instead of 80)?

- 5.2 A saturated gas mixture, obtained from a coal liquefaction plant after the acid removal section, has composition of 38.2% H₂, 12.3% CO, 18% CO₂, 31.0% CH₄ and 0.5% N₂ (by mole) on dry basis. It is available at 120 kPa a and 60°C. It is cooled to 40°C in a heat exchanger and sent to suction drum of a compressor. Condensate from the drum is to be continuously pumped out with the help of a centrifugal pump. Calculate the volume fraction of the gas mixture over the condensate and the artificial vapour pressure using ideal gas law.

Table 5.15 Solubility of Gases⁹ in Water at 1 atm and 40°C

Gas	Solubility, kg/100 kg water
H ₂	0.000 1384
CO	0.002 075
CO ₂	0.0973
CH ₄	0.001 586
N ₂	0.001 391

- 5.3 Natural gas is to be delivered through 0.6 m diameter steel pipe. Straight length of pipe is 3 km. Equivalent length of fittings and valves of this pipe line is 300 m. Desired flow rate of natural gas is 180 000 Nm³/h. For the purpose, a centrifugal compressor is installed at the Gas Gathering Station (GGS). Natural gas is to be delivered at the plant at 313 K and 170 kPa a and it leaves the compressor at 313 K. What pressure must be developed at the compressor discharge in order to achieve this flow rate? Average molar mass of natural gas is 17.4 and its viscosity at 313 K is 0.01 cP.

- 5.4 It is proposed to pump 5000 kg/h of acetic acid at 129.5°C and 1.4 atm a pressure from the reboiler of a distillation column to a hold up tank without cooling. If the friction loss in the line between the reboiler and pump is 8 kPa and the density of acetic acid is 1048 kg/m³, how far above the pump must the liquid level in the reboiler be maintained to give a net positive suction head of 2.5 m? Friction loss in discharge line is 40 kPa. Maximum liquid level of acetic acid above the discharge point is 5 m. Pump efficiency is 60%. Calculate the power required to drive the pump.
- 5.5 Performance of centrifugal pump on water is known. It can deliver 100 m³/h of water at 80 m total head of water with 65% efficiency. By using Fig. 5.7, predict the performance of this pump, if it is used to deliver the oil of viscosity 176 cSt (or 800 SSU) at the pumping temperature. Determine the head, capacity and power required by this pump when it is used to deliver oil. Density of oil at pumping temperature is 800 kg/m³.
- 5.6 Design an Orifice meter based on the following data:
 Name of fluid = Natural gas
 Flow rate = 1000 Nm³/h
 Operating pressure = 1.35 atm a
 Operating temperature = 38°C
 Average molar mass of natural gas = 17.2
 Viscosity of natural gas = 0.01 cP at 38°C
 Inside diameter of pipe = 128.2 mm (5 in, SCH-40)
 Specific heat ratio for natural gas = 1.3
- 5.7 Design a venturi meter based on the data given in Example 5.21.
 Mass flow rate through venturi meter is calculated by following equation.

$$\dot{m} = \frac{C_v A_t}{\sqrt{1 - \beta^4}} \sqrt{2 g_c \Delta p \rho}$$

where, C_v = Venturi coefficient = 0.98

$$A_t = \text{Throat area of venturi} = \frac{\pi}{4} D_t^2$$

D_t = Diameter of throat, m

β = Ratio of diameter of throat to diameter of pipe

g_c = 1

Δp = Pressure drop measured by manometer, Pa

ρ = Density of flowing fluid, kg/m³

Angle of upstream cone of venturi meter is 30° and the same of downstream cone is 10°. Take $\beta = 0.5$.

Determine: (a) Throat diameter of venturi (b) Pressure drop and manometer reading and (c) Lengths of upstream and downstream cones.

- 5.8 Determine the minimum and maximum flow rates of air at 40°C and at 1.5 atm a, which can be measured by the rotameter of the dimension of tube and float of rotameter, given in Fig. 5.15, except material of float is polypropylene.
 Viscosity of air at 40°C = 1850×10^{-5} cP
 Density of polypropylene = 905 kg/m³
 Thickness of float = 2 mm
 Calibrate the rotameter or find the different values of flow rates for the different float positions.

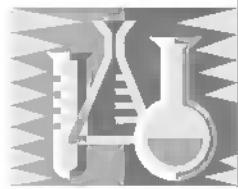
- 5.9 In the Example 5.23 a rotameter is calibrated to measure the flow rate of water at 40°C. If the same rotameter is used for measuring flow rates of glycerine at 30°C then what is the change in calibration?

Viscosity of glycerine at 30°C = 400 cP

Density of glycerine at 30°C = 1.25 kg/L

REFERENCES

1. Sinnott, R. K., *Coulson & Richardson's Chemical Engineering*, Vol. 6, Asian Book Private Ltd., Revised 2nd Ed., New Delhi, 1998.
2. Perry, R. H. and Green D, *Perry's Chemical Engineer's Handbook*, 6th Ed., McGraw-Hill Book Company, USA, 1984.
3. Techo, R., Tinker, R. R. and R. E. James, *J. Appl. Mech.*, **32**, 1965, p. 443.
4. Ludwig, E. E., *Applied Process Design for Chemical and Petrochemical Plants*, Gulf Publishing Company, Vol. 1, 3rd Ed., USA, 1999.
5. *Flow of Fluids Through Valves, Fittings and Pipe*, Published by Crane Co., New York, USA, 1977.
6. Brown G. G., *Unit Operations*, John Wiley and Sons, Inc, USA, 1950.
7. Simpson, L. L., *Chem. Engg.*, **75** (13), June 17, 1968, p. 190.
8. *Hydraulic Institute Standards*, 13th Ed., Hydraulic Institute, USA.
9. Tsai, M. J., *Chem. Engg.*, **89**(15), July 26, 1982, p. 65.
10. McCabe, W. L., Smith, J. C. and Harriott P., *Unit Operations of Chemical Engineering*, 6th Ed., McGraw-Hill Book Company, USA, 2001.
11. Coulson, J. M. and J. F. Richardson, *Chemical Engineering*, Vol. 1, Butter Worth-Heinemann, 6th Ed., UK, 1999.
12. *Carrier System Design Manual: Refrigerants, Brines, Oil* by Carrier Corporation, USA, 1969.
13. Chaware, D. K. Shukla and R. B. Swamy, *Hydrocarbon Processing*, **84**(5), 2005, p. 47.



Process Design of Heat Exchangers

6.1 SHELL AND TUBE HEAT EXCHANGER

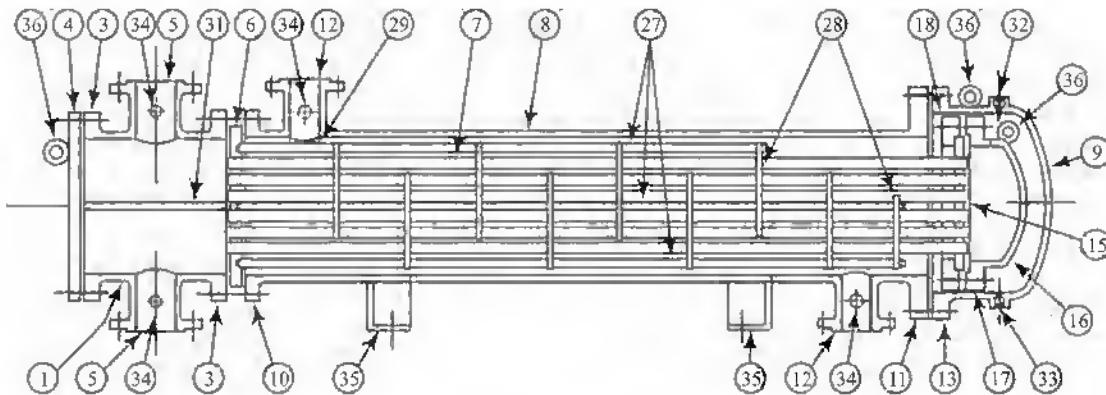
Shell and tube heat exchangers are the most widely used equipments in chemical industry. They are mostly used as heat transfer equipments but in a few cases they are also used as Reactors, Falling Film Absorbers, etc. Sizes of various parts of shell and tube heat exchangers like shell, tubes, tierods are standardized. Standards developed by Tubular Exchanger Manufacturers Association, USA (TEMA) are universally used for design of shell and tube heat exchangers. Equivalent Indian Standard is IS: 4503. TEMA or IS: 4503 specify standard sizes of shell, tubes, tierods, etc. and also maximum allowable baffle spacing, minimum tube sheet thickness, baffle thickness, numbers of tierods required, etc. For mechanical design and fabrication, in addition to TEMA standard, ASME Code Section VIII DIV. I is used. In TEMA standard, shell and tube heat exchangers are classified in three category;

- (i) Class R covers heat exchangers which are used for severe duties in petroleum and related industries,
- (ii) Class B covers the heat exchangers which are used in chemical process industries not involving severe duties, and
- (iii) Class C covers the heat exchangers which are used in commercial and in less important process applications.

Most popular and reliable softwares used for the design of shell and tube heat exchangers are of:

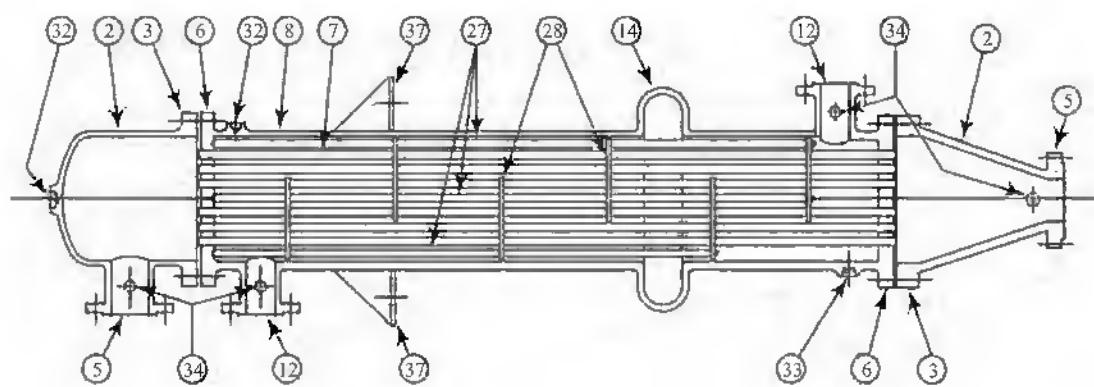
- (i) **HTRI**: Heat Transfer Research Institute, USA
- (ii) **HTFS**: Heat Transfer Fluid Flow Services, UK
- (iii) **BJAC**: USA based company
- (iv) **HEI**: Heat Exchange Institute, USA.

Design methods and equations used by these softwares are not available in open literature. For the design of shell and tube heat exchanger involving fluid without phase change, methods used by these softwares are based on Tinker's flow model.



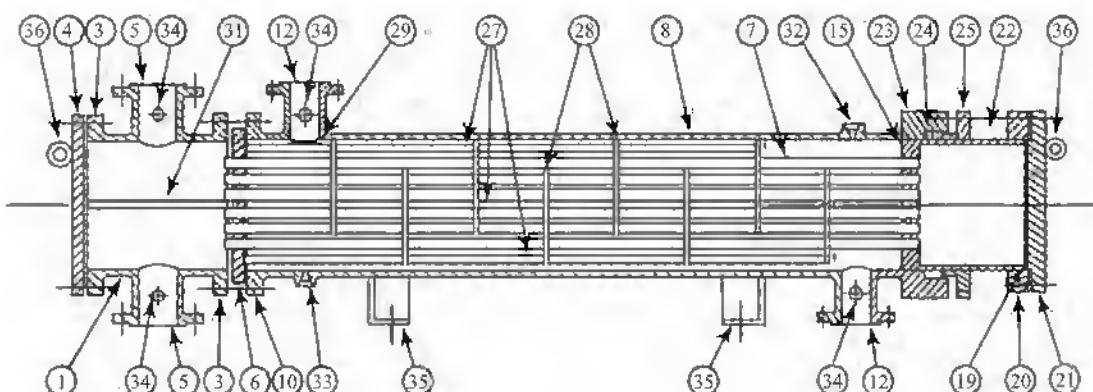
Internal Floating Head Heat Exchanger with Backing Ring, Type AES

(a)



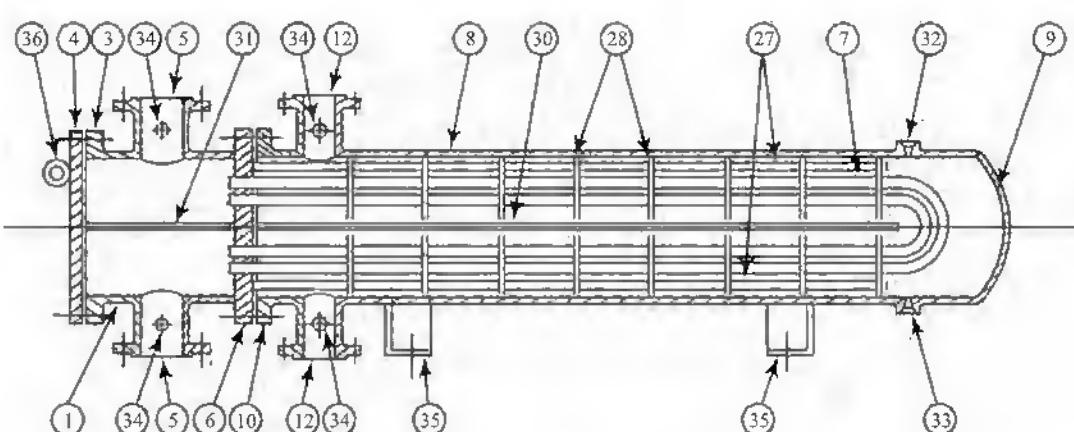
Fixed Tube Sheet Heat Exchanger, Type BEM

(b)



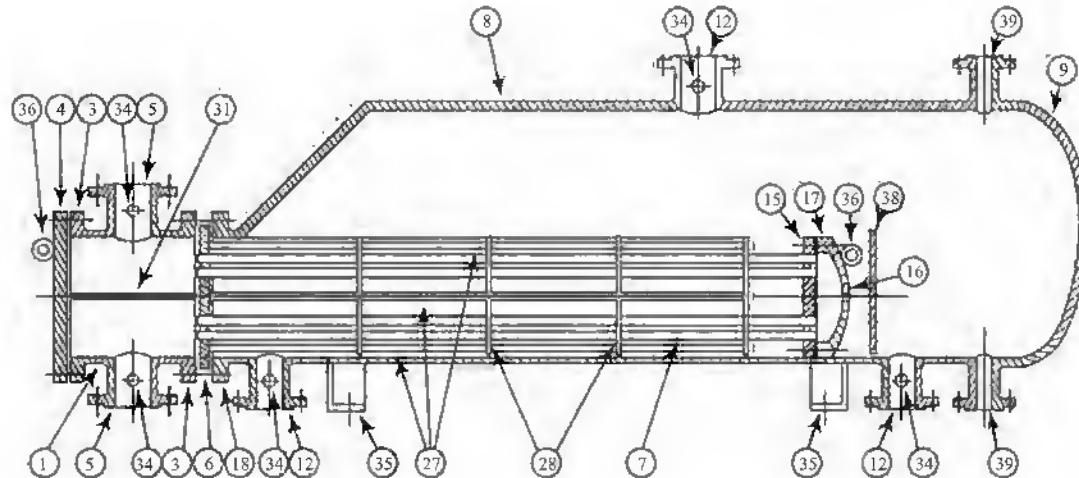
Outside-Packed Floating Head Heat Exchanger, Type AEP

(c)



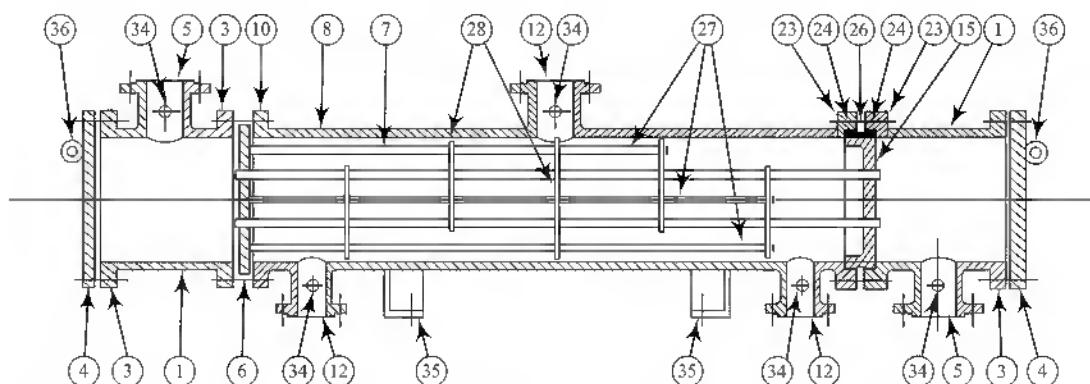
U Tube Heat Exchanger, Type CFU

(d)



Kettle Type Floating Head Reboiler, Type AKT

(e)



Exchanger with Packed Floating Tube sheet and Lantern Ring, Type AJW

(f)

Nomenclature of Parts of Various Shell and Tube Heat Exchangers

- | | | |
|---|---|--|
| 1. Stationary Head—Channel | 14. Expansion Joint | 27. Tie rods and Spacers |
| 2. Stationary Head—Bonnet | 15. Floating Tube sheet | 28. Transverse Baffles or Support Plates |
| 3. Stationary Head Flange—
Channel or Bonnet | 16. Floating Head Cover | 29. Impingement Plate |
| 4. Channel Cover | 17. Floating Head Flange | 30. Longitudinal Baffle |
| 5. Stationary Head Nozzle | 18. Floating Head Split Backing
Ring | 31. Pass Partition |
| 6. Stationary Tube sheet | 19. Split Shear Ring | 32. Vent Connection |
| 7. Tubes | 20. Slip-on Backing Flange | 33. Drain Connection |
| 8. Shell | 21. Floating Head Cover—
External | 34. Instrument Connection |
| 9. Shell Cover | 22. Floating Tube sheet Skirt | 35. Support Saddle |
| 10. Shell Flange—Stationary
Head End | 23. Packing Box Flange | 36. Lifting Lug |
| 11. Shell Flange—Rear Head End | 24. Packing | 37. Support Bracket |
| 12. Shell Nozzle | 25. Packing Gland | 38. Weir |
| 13. Shell Cover Flange | 26. Lantern Ring | 39. Liquid Level Connection |

Fig. 6.1 Different Types of Shell and Tube Heat Exchangers¹ (Fig. N-2 of TEMA 8th Edition,
Reproduced with the Permission of the Tubular Exchanger Manufacturers Association Inc.,
USA)

6.2 DESIGN OF HEAT EXCHANGERS

6.2.1 Various Parts of Shell and Tube Heat Exchangers

6.2.1.1 Shell

Shell is the costliest part of the heat exchanger. Cost of shell and tube heat exchanger sensitively changes with change in the diameter of shell. As per the TEMA standard, shell size ranges from 6 in (152 mm) to 60 in (1520 mm). Standard pipes are available up to 24 in size (600 mm NB). If shell size is greater than 24 in, it is fabricated by rolling a plate.

Shell diameter depends on tube bundle diameter. For fixed tube sheet shell and tube heat exchanger, the gap between shell and tube bundle is minimum, ranging from 10 to 20 mm. For pull through floating head heat exchanger, it is maximum, ranging from 90–100 mm.

Required shell diameter is determined by one of the following three methods:

(a) Based on the actual tube sheet layout drawing: All latest computer softwares available for the design of heat exchangers use this method to find shell inside diameter. Popular computer program, written in Autolips, is used to draw the tube sheet layout. To draw the tube sheet layout or to find the tube bundle diameter total six parameters are fixed. (i) Outside diameter (OD) of tube, (ii) Numbers of tube side passes, (iii) Tube pitch (center to center distance between adjacent tubes), (iv) Tube arrangement (triangular or square), (v) Total number of tubes and (vi) Type of shell and tube heat exchanger (fixed tube sheet, U tube or floating head).

(b) Use of standard tables: Based on actual tube sheet layout, standard tables are prepared. These tables give the maximum number of tubes that can be accommodated in various standard sizes of shell ranging from 6 in (150 mm NB) to 120 in (3000 mm NB), for the different numbers of tube side passes, different values of tube pitch and size and for the different arrangements of tubes. Table 6.1 gives the data on tube sheet layouts.

Table 6.1 Tube Sheet Tube Hole Count^{2,3}

Table 6.1 (a) $\frac{5}{8}$ in OD tubes on $\frac{13}{16}$ in square pitch

Shell ID		TEMA P or S Number of Passes				TEMA U Number of Passes		
mm	in	1	2	4	6	2	4	6
203	8	55	48	34	24	52	40	32
254	10	88	78	62	56	90	80	74
305	12	140	138	112	100	140	128	108
337	$13\frac{1}{4}$	178	172	146	136	180	164	148
387	$15\frac{1}{4}$	245	232	208	192	246	232	216
438	$17\frac{1}{4}$	320	308	274	260	330	312	292
489	$19\frac{1}{4}$	405	392	352	336	420	388	368

(Contd.)

Tube Sheet Tube Hole Count (*Contd.*)**Table 6.1 (a) (*Contd.*)**

Shell ID		TEMA P or S Number of Passes				TEMA U Number of Passes		
mm	in	1	2	4	6	2	4	6
540	21 $\frac{1}{4}$	502	484	442	424	510	488	460
591	23 $\frac{1}{4}$	610	584	536	508	626	596	562
635	25	700	676	618	600	728	692	644
686	27	843	812	742	716	856	816	780
737	29	970	942	868	840	998	956	920
787	31	1127	1096	1014	984	1148	1108	1060
838	33	1288	1250	1172	1148	1318	1268	1222
889	35	1479	1438	1330	1308	1492	1436	1388
940	37	1647	1604	1520	1480	1684	1620	1568
991	39	1840	1794	1700	1664	1882	1816	1754
1067	42	2157	2112	2004	1968	2196	2136	2068
1143	45	2511	2458	2326	2288	2530	2464	2402
1219	48	2865	2808	2686	2656	2908	2832	2764
1372	54	3656	3600	3462	3404	3712	3624	3556
1524	60	4538	4472	4310	4256	4608	4508	4426

Table 6.1 (b) $\frac{3}{4}$ in OD tubes on $\frac{15}{16}$ -in triangular pitch

Shell ID		TEMA L or M Number of passes				TEMA P or S Number of passes				TEMA U Number of passes		
mm	in	1	2	4	6	1	2	4	6	2	4	6
114	4.5	14	14	—	—	—	—	—	—	—	—	—
152	6	30	30	24	22	—	—	—	—	—	—	—
203	8	64	48	34	24	34	32	16	18	32	24	24
254	10	85	72	52	50	60	62	52	44	64	52	52
305	12	122	114	94	96	109	98	78	68	98	88	78
337	13 $\frac{1}{4}$	151	142	124	112	126	120	106	100	126	116	108
387	15 $\frac{1}{4}$	204	192	166	168	183	168	146	136	180	160	148
438	17 $\frac{1}{4}$	264	254	228	220	237	228	202	192	238	224	204
489	19 $\frac{1}{4}$	332	326	290	280	297	286	258	248	298	280	262
540	21 $\frac{1}{4}$	417	396	364	348	372	356	324	316	370	352	334
591	23 $\frac{1}{4}$	495	478	430	420	450	430	392	376	456	428	408
635	25	579	554	512	488	518	498	456	444	534	500	474
686	27	676	648	602	584	618	602	548	532	628	600	570
737	29	785	762	704	688	729	708	650	624	736	696	668
787	31	909	878	814	792	843	812	744	732	846	812	780
838	33	1035	1002	944	920	962	934	868	840	978	928	904
889	35	1164	1132	1062	1036	1090	1064	990	972	1100	1060	1008

(Contd.)

Tube Sheet Tube Hole Count (*Contd.*)**Table 6.1 (b) (*Contd.*)**

Shell ID		TEMA L or M				TEMA P or S				TEMA U		
mm	in	1	2	4	6	1	2	4	6	2	4	6
940	37	1304	1270	1200	1168	1233	1196	1132	1100	1238	1200	1152
991	39	1460	1422	1338	1320	1365	1346	1266	1244	1390	1336	1290
1067	42	1703	1664	1578	1552	1611	1580	1498	1464	1632	1568	1524
1143	45	1960	1918	1830	1800	1875	1834	1736	1708	1882	1820	1770
1219	48	2242	2196	2106	2060	2132	2100	1998	1964	2152	2092	2044
1372	54	2861	2804	2682	2660	2730	2684	2574	2536	2748	2680	2628
1524	60	3527	3476	3360	3300	3395	3346	3228	3196	3420	3340	3286
1676	66	4292	4228	4088	4044							
1829	72	5116	5044	4902	4868							
1981	78	6034	5964	5786	5740							
2134	84	7005	6934	6766	6680							
2286	90	8093	7998	7832	7708							
2438	96	9203	9114	8896	8844							
2743	108	11696	11618	11336	11268							
3048	120	14459	14378	14080	13984							

Table 6.1 (c) $\frac{3}{4}$ in OD tubes on 1 in square pitch

Shell ID		TEMA P or S				TEMA U		
mm	in	1	2	4	6	2	4	6
152	6	12	12	—	—	14	—	—
203	8	28	26	16	12	28	24	12
254	10	52	48	44	24	52	44	32
305	12	80	76	66	56	78	72	70
337	13 $\frac{1}{4}$	104	90	70	80	96	92	90
387	15 $\frac{1}{4}$	136	128	128	114	136	132	120
438	17 $\frac{1}{4}$	181	174	154	160	176	176	160
489	19 $\frac{1}{4}$	222	220	204	198	224	224	224
540	21 $\frac{1}{4}$	289	272	262	260	284	280	274
591	23 $\frac{1}{4}$	345	332	310	308	348	336	328
635	25	398	386	366	344	408	392	378
686	27	477	456	432	424	480	468	460
737	29	554	532	510	496	562	548	530
787	31	637	624	588	576	648	636	620
838	33	730	712	682	668	748	728	718
889	35	828	812	780	760	848	820	816

(Contd.)

Tube Sheet Tube Hole Count (*Contd.*)**Table 6.1 (c) (*Contd.*)**

Shell ID		TEMA P or S				TEMA U		
		Number of Passes				Number of Passes		
mm	in	1	2	4	6	2	4	6
940	37	937	918	882	872	952	932	918
991	39	1048	1028	996	972	1056	1044	1020
1067	42	1224	1200	1170	1140	1244	1224	1212
1143	45	1421	1394	1350	1336	1436	1408	1398
1219	48	1628	1598	1548	1536	1640	1628	1602
1372	54	2096	2048	2010	1992	2108	2084	2068
1524	60	2585	2552	2512	2476	2614	2584	2558

Table 6.1 (d) $\frac{3}{4}$ in OD tubes on 1 in triangular pitch

Shell ID		TEMA L or M				TEMA P or S				TEMA U		
		Number of passes				Number of passes				Number of passes		
mm	in	1	2	4	6	1	2	4	6	2	4	6
114	4.5	14	14	—	—	—	—	—	—	—	—	—
152	6	26	26	24	22	14	14	—	—	14	12	10
203	8	42	40	26	24	31	26	16	12	32	24	24
254	10	73	66	52	44	56	48	42	40	52	48	40
305	12	109	102	88	80	88	78	62	68	84	76	74
337	13 $\frac{1}{4}$	136	128	112	102	121	106	94	88	110	100	98
387	15 $\frac{1}{4}$	183	172	146	148	159	148	132	132	152	140	136
438	17 $\frac{1}{4}$	237	228	208	192	208	198	182	180	206	188	182
489	19 $\frac{1}{4}$	295	282	258	248	258	250	228	220	226	248	234
540	21 $\frac{1}{4}$	361	346	318	320	320	314	290	276	330	316	296
591	23 $\frac{1}{4}$	438	416	382	372	400	384	352	336	400	384	356
635	25	507	486	448	440	450	442	400	392	472	440	424
686	27	592	574	536	516	543	530	488	468	554	528	502
737	29	692	668	632	604	645	618	574	556	648	616	588
787	31	796	774	732	708	741	716	666	648	744	716	688
838	33	909	886	836	812	843	826	760	740	852	816	788
889	35	1023	1002	942	920	950	930	878	856	974	932	908
940	37	1155	1124	1058	1032	1070	1052	992	968	1092	1056	1008
991	39	1277	1254	1194	1164	1209	1184	1122	1096	1224	1180	1146
1067	42	1503	1466	1404	1372	1409	1378	1314	1296	1434	1388	1350
1143	45	1726	1690	1622	1588	1635	1608	1536	1504	1652	1604	1560
1219	48	1964	1936	1870	1828	1887	1842	1768	1740	1894	1844	1794
1372	54	2519	2466	2380	2352	2399	2366	2270	2244	2426	2368	2326
1524	60	3095	3058	2954	2928	2981	2940	2932	2800	3006	2944	2884
1676	66	3769	3722	3618	3576							
1829	72	4502	4448	4324	4280							
1981	78	5309	5252	5126	5068							

(Contd.)

Tube Sheet Tube Hole Count (*Contd.*)**Table 6.1 (d) (*Contd.*)**

Shell ID		TEMA L or M Number of passes				TEMA P or S Number of passes				TEMA U Number of passes		
mm	in	1	2	4	6	1	2	4	6	2	4	6
2134	84	6162	6108	5954	5900							
2286	90	7103	7040	6898	6800							
2438	96	8093	8026	7848	7796							
2743	108	10260	10206	9992	9940							
3048	120	12731	12648	12450	12336							

Table 6.1 (e) *I in OD tubes on 1 1/4 in square pitch*

Shell ID		TEMA P or S Number of Passes				TEMA U Number of Passes		
mm	in	1	2	4	6	2	4	6
203	8	17	12	8	12	14	8	6
254	10	30	30	16	18	30	24	12
305	12	52	48	42	24	44	40	32
337	13 1/4	61	56	52	50	60	48	44
387	15 1/4	85	78	62	64	80	72	74
438	17 1/4	108	108	104	96	104	100	100
489	19 1/4	144	136	130	114	132	132	120
540	21 1/4	173	166	154	156	172	168	148
591	23 1/4	217	208	194	192	212	204	198
635	25	252	240	230	212	244	240	230
686	27	296	280	270	260	290	284	274
737	29	345	336	310	314	340	336	328
787	31	402	390	366	368	400	384	372
838	33	461	452	432	420	456	444	440
889	35	520	514	494	484	518	504	502
940	37	588	572	562	548	584	576	566
991	39	661	640	624	620	664	644	640
1067	42	776	756	738	724	764	748	750
1143	45	900	882	862	844	902	880	862
1219	48	1029	1016	984	972	1028	1008	1004
1372	54	1310	1296	1268	1256	1320	1296	1284
1524	60	1641	1624	1598	1576	1634	1616	1614

(Contd.)

Tube Sheet Tube Hole Count (*Contd.*)**Table 6.1 (f)** *I* in OD tubes on *I* $\frac{1}{4}$ in triangular pitch

Shell ID		TEMA L or M Number of passes				TEMA P or S Number of passes				TEMA U Number of passes		
mm	in	1	2	4	6	1	2	4	6	2	4	6
203	8	27	26	8	12	18	14	8	12	14	12	6
254	10	42	40	34	24	33	28	16	18	28	24	24
305	12	64	66	52	44	51	48	42	44	52	40	40
337	13 $\frac{1}{4}$	81	74	62	56	73	68	52	44	64	56	52
387	15 $\frac{1}{4}$	106	106	88	92	93	90	78	76	90	80	78
438	17 $\frac{1}{4}$	147	134	124	114	126	122	112	102	122	112	102
489	19 $\frac{1}{4}$	183	176	150	152	159	152	132	136	152	140	136
540	21 $\frac{1}{4}$	226	220	204	186	202	192	182	172	196	180	176
591	23 $\frac{1}{4}$	268	262	236	228	249	238	216	212	242	224	216
635	25	316	302	274	272	291	278	250	240	286	264	246
686	27	375	360	336	324	345	330	298	288	340	320	300
737	29	430	416	390	380	400	388	356	348	400	380	352
787	31	495	482	452	448	459	450	414	400	456	436	414
838	33	579	554	520	504	526	514	584	464	526	504	486
889	35	645	622	586	576	596	584	548	536	596	572	548
940	37	729	712	662	648	672	668	626	608	668	636	614
991	39	808	792	744	732	756	736	704	692	748	728	700
1067	42	947	918	874	868	890	878	834	808	890	856	830
1143	45	1095	1068	1022	1000	1035	1008	966	948	1028	992	972
1219	48	1241	1220	1176	1148	1181	1162	1118	1092	1180	1136	1100
1372	54	1577	1572	1510	1480	1520	1492	1436	1416	1508	1468	1442
1524	60	1964	1940	1882	1832	1884	1858	1800	1764	1886	1840	1794
1676	66	2390	2362	2282	2260							
1829	72	2861	2828	2746	2708							
1981	78	3368	3324	3236	3216							
2134	84	3920	3882	3784	3736							
2286	90	4499	4456	4370	4328							
2438	96	5144	5104	4986	4936							
2743	108	6546	6494	6360	6300							
3048	120	8117	8038	7870	7812							

Table 6.1 (g) *I* $\frac{1}{4}$ in OD tubes on *I* $\frac{1}{16}$ in square pitch

Shell ID		TEMA P or S Number of Passes				TEMA U Number of Passes		
mm	in	1	2	4	6	2	4	6
203	8	12	12	4	0	4	4	6
254	10	21	12	8	12	12	8	12

(Contd.)

Tube Sheet Tube Hole Count (*Contd.*)**Table 6.1 (g) (Contd.)**

Shell ID		TEMA P or S						TEMA U					
		Number of Passes						Number of Passes					
mm	in	1	2	4	6	2	4	6	2	4	6	2	4
305	12	29	28	16	18	26	20	12					
337	13 $\frac{1}{4}$	38	34	34	24	36	28	15					
387	15 $\frac{1}{4}$	52	48	44	48	44	44	32					
438	17 $\frac{1}{4}$	70	66	56	50	60	60	56					
489	19 $\frac{1}{4}$	85	84	70	80	82	76	79					
540	21 $\frac{1}{4}$	108	108	100	96	100	100	100					
591	23 $\frac{1}{4}$	136	128	128	114	128	120	120					
635	25	154	154	142	136	154	148	130					
686	27	184	180	158	172	176	172	160					
737	29	217	212	204	198	212	204	198					
787	31	252	248	234	236	242	240	234					
838	33	289	276	270	264	280	280	274					
889	35	329	316	310	304	324	312	308					
940	37	372	368	354	340	358	352	350					
991	39	420	402	402	392	408	400	392					
1067	42	485	476	468	464	480	476	464					
1143	45	565	554	546	544	558	548	550					
1219	48	653	636	628	620	644	628	632					
1372	54	837	820	812	804	824	808	808					
1524	60	1036	1028	1012	1008	1028	1016	1008					

Table 6.1 (h) 1 $\frac{1}{4}$ in OD tubes on 1 $\frac{9}{16}$ in triangular pitch

Shell ID		TEMA L or M				TEMA P or S				TEMA U			
		Number of Passes				Number of Passes				Number of Passes			
mm	in	1	2	4	6	1	2	4	6	2	4	6	
203	8	15	10	8	12	13	10	4	0	6	4	6	
254	10	27	22	16	12	18	20	8	12	14	12	12	
305	12	38	36	26	24	33	26	26	18	28	20	18	
337	13 $\frac{1}{4}$	55	44	42	40	38	44	34	24	34	28	30	
387	15 $\frac{1}{4}$	66	64	52	50	57	58	48	44	52	48	40	
438	17 $\frac{1}{4}$	88	82	78	68	81	72	62	68	72	68	64	
489	19 $\frac{1}{4}$	117	106	98	96	100	94	86	80	90	84	78	
540	21 $\frac{1}{4}$	136	134	124	108	126	120	116	102	118	112	102	
591	23 $\frac{1}{4}$	170	164	146	148	159	146	132	132	148	132	120	
635	25	198	188	166	168	183	172	150	148	172	160	152	

(Contd.)

Tube Sheet Tube Hole Count (*Contd.*)**Table 6.1 (h) (*Contd.*)**

Shell ID		TEMA L or M				TEMA P or S				TEMA U		
		Number of Passes				Number of Passes				Number of Passes		
mm	in	1	2	4	6	1	2	4	6	2	4	6
686	27	237	228	208	192	208	206	190	180	200	188	180
737	29	268	266	242	236	249	238	224	220	242	228	216
787	31	312	304	284	276	291	282	262	256	282	264	250
838	33	357	346	322	324	333	326	298	296	326	308	292
889	35	417	396	372	364	372	368	344	336	362	344	336
940	37	446	446	422	408	425	412	394	384	416	396	384
991	39	506	490	472	464	478	468	442	432	472	444	428
1067	42	592	584	552	544	558	546	520	512	554	524	510
1143	45	680	676	646	632	646	634	606	596	636	624	592
1219	48	788	774	736	732	748	732	704	696	736	708	692
1372	54	1003	980	952	928	962	952	912	892	946	916	890
1524	60	1237	1228	1188	1152	1194	1182	1144	1116	1176	1148	1116
1676	66	1520	1496	1448	1424							
1829	72	1814	1786	1736	1724							
1981	78	2141	2116	2068	2044							
2134	84	2507	2470	2392	2372							
2286	90	2861	2840	2764	2744							
2438	96	3275	3246	3158	3156							
2743	108	4172	4136	4046	4020							
3048	120	5164	5128	5038	5000							

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(c) Use of approximate equations: Approximate equations are available in literature to find the shell diameter or to find the tube bundle diameter. One of such equations⁴ is given as follows:

$$D_b = d_o \left(\frac{N_t}{k_1} \right)^{\frac{1}{n_1}} \quad (6.1)$$

where, D_b = Tube bundle diameter, mm

d_o = Tube OD, mm

N_t = Total number of tubes

k_1 and n_1 are constants, values of which depend on ratio $\frac{P_t}{d_o}$ $\left(= \frac{\text{tube pitch}}{\text{tube OD}} \right)$, arrangement of tubes and number of tube side passes.

Use Tables 6.2 and 6.3 for finding constants of Eq. (6.1).

Table 6.2 Constants for Eq. (6.1)

A. For $P/d_o = 1.25$, Triangular Pitch

No. of tube side passes	1	2	4	6	8
k_l	0.319	0.249	0.175	0.0743	0.0365
n_l	2.142	2.207	2.285	2.499	2.675

Table 6.3 Constants for Eq. (6.1)

B. For $P_t/d_o = 1.25$, Square Pitch

No. of tube side passes	1	2	4	6	8
k_l	0.215	0.156	0.158	0.0402	0.0331
n_l	2.207	2.291	2.263	2.617	2.643

Equation (6.1) can be used for fixed tube sheet type and floating head shell and tube type heat exchangers.

But for U-tube heat exchangers, following equation can be used.

$$D_b = d_o \left(\frac{N_t'}{k_l} \right)^{\frac{1}{n_l}} \quad (6.2)$$

where, $N_t' = N_t + \frac{D_b}{P_t}$ (6.3)

N_t = Number of tube holes on tube sheet

After finding tube bundle diameter D_b , shell ID (Inside diameter) D_i can be determined by following equation.

$$D_i = D_b + C \quad (6.4)$$

where, C = Clearance between shell ID and D_b

$C = 10$ to 20 mm for fixed tube sheet and U-tube

$C = 50$ to 80 mm for split-ring floating head

$C = 90$ to 100 mm for pull through floating head

6.2.1.2 Shell Side Pass Partition Plate

Single pass shell is used in the most of the cases. Two pass shell is rarely used and is recommended where shell and tube temperature difference is unfavorable for the single pass. For such cases normally two or more heat exchangers, connected in series, are recommended. Shell side pass partition plate is not provided to improve shell side heat transfer coefficient but it is provided to avoid the unfavourable temperature difference or to avoid the cross of temperatures between hot fluid and cold fluid.

Example 6.0

Hot oil is to be cooled from 45°C to 37°C by cooling water. Cooling water will enter at 32°C and leave at 40°C . Minimum driving force required for heat transfer in shell and tube heat exchanger is 3 to 5°C . Hence, for this case 1-1 shell and tube heat exchanger

can be selected. But it will provide poor heat transfer coefficient and may require large heat transfer area. 1-2 shell and tube heat exchanger cannot be selected for this case because of the occurrence of hypothetical temperature cross between hot and cold fluids. 2-2 shell and tube heat exchanger can be selected. 2-2 heat exchanger will provide higher heat transfer coefficient than 1-1 heat exchanger.

6.2.1.3 Baffles

There are two functions of baffles.

- (i) Baffles are used in shell to direct the fluid stream across the tubes to increase the velocity of shell side flow and thereby to improve the shell side heat transfer coefficient. In other words baffles are used in shell to increase the turbulence in shell side fluid.

Note: This function is useful only if there is no phase change in shell side fluid

- (ii) Baffles indirectly support the tubes and thereby reduce the vibration in tubes. If shell side liquid velocity is higher; say more than 3 m/s, vibration analysis calculations should be carried out to check whether baffle spacing is sufficient or not. Similarly, for very high velocity of gas or vapour and also for the higher baffle spacing (higher than shell ID), vibration analysis calculation must be carried out to check the baffle spacing. Vibration analysis calculations are given in TEMA standard.

Different types of baffles are used in shell and tube heat exchangers; (i) Segmental baffle, (ii) Nest baffle, (iii) Segmental and Strip baffle (iv) Disc and Doughnut baffle, (v) Orifice baffle (vi) Dam baffle, etc. Figure 6.2 shows various types of baffles.

Most widely used type of baffle is segmental baffle. Other types of baffles like nest baffle, segmental and strip baffle and disc and doughnut baffle provide less pressure drop for the same baffle spacing but provide lower heat transfer coefficients as compared to segmental baffle. Examples: Situation when other types of baffles might be used.

- (a) For shell and tube heat exchangers shell side pressure drop controls the overall design. For example, intercoolers of compressors and heat exchangers used in very high vacuum system. In intercoolers of compressors maximum allowable shell side pressure drop may be as low as 3.45 kPa. For such cases different types of shells are also used. Split flow (G shell) or divided flow (J shell) designs provide low shell side pressure drop compared to commonly used single pass (E shell). Refer Fig. 6.3
- (b) For shell and tube heat exchanger in which boiling or condensation is carried out on shell side. In such a case baffles are required only to reduce the vibrations in tubes.

For other type of baffles (other than segmental baffle) correlations for finding heat transfer coefficient and pressure drop are not easily available in open literature.

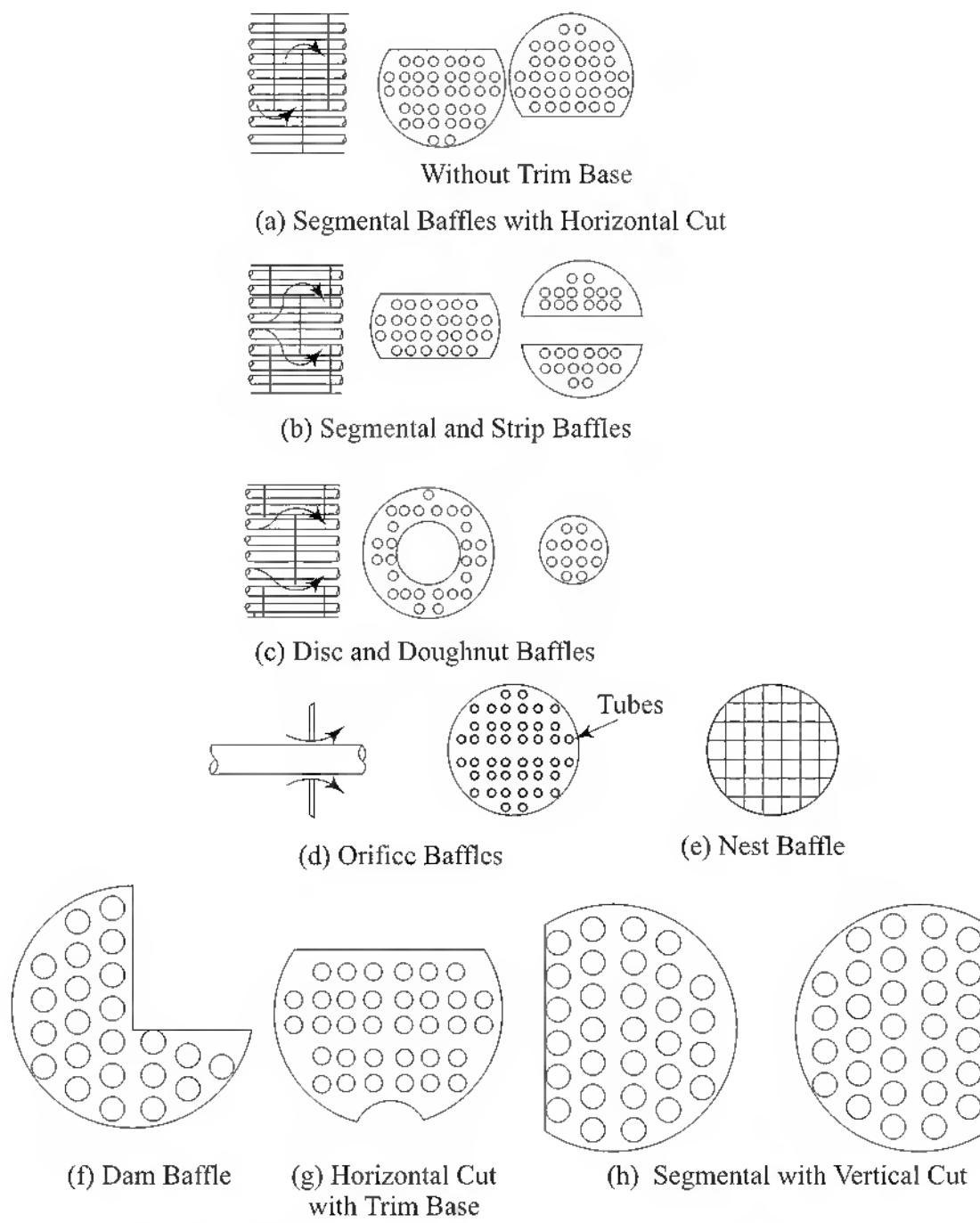


Fig. 6.2 Different Types of Baffles used in Shell and Tube Heat Exchangers²
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Segmental baffle is made by cutting the circular metal disc. Segmental baffles are specified in terms of % baffle cut.

$$\% \text{ Baffle cut} = h/d \times 100 \quad (6.5)$$

where, h = Height of segment removed

$$\begin{aligned} D &= \text{Diameter of circular metal disc from which segment is removed} \\ &= \text{ID of shell} - 2 \times \text{Clearance} \end{aligned}$$

% Baffle cut ranges from 15 to 45%. If there is no phase change on shell side fluid, then decrease in % baffle cut increases shell side heat transfer coefficient. Decrease in window area results in increase in window velocity but at the expense of pressure drop. 20 to 25% Baffle cut is found to be common in use.

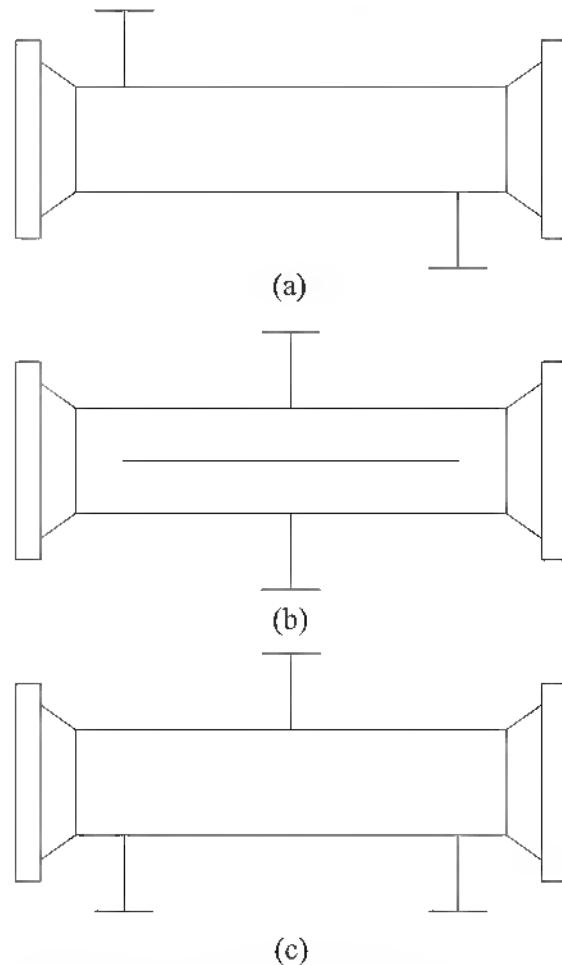


Fig. 6.3 (a) One Pass Shell (E Shell) (b) Split Flow (G Shell) (c) Divided Flow (J Shell)

Baffle outside diameter is always less than shell inside diameter. Certain clearance between baffle OD and shell ID is provided to facilitate removal and insertion of tube bundle for maintenance. Recommended clearance between shell ID and baffle OD is as follows.

Table 6.4 Clearance Between Shell and Segmental Baffle

Shell ID	Clearance (Shell ID - Baffle OD)
Standard pipe shell 6 in to 24 in (150 mm to 600 mm)	1.6 mm
Plate shell 6 in to 25 in (150 mm to 625 mm)	3.2 mm
Plate shell 27 in to 42 in (675 mm to 1050 mm)	4.8 mm

If shell is fabricated from plate then it may not be a perfect cylinder. Hence, for plate shell, more clearance is permitted. For heat exchanger without phase change, increase in clearance between shell ID and baffle OD could mean decrease in shell side heat transfer coefficient due to leakage. Fraction of shell side fluid flowing through this clearance is not utilized for heat transfer. Hence this clearance, if it is more than recommended value, is undesirable.

In kettle type reboiler, full baffle (with 0% baffle cut) is used, as in this type of heat exchanger extra space is available for the flow of shell side liquid.

In case of horizontal inclined condenser, segmental baffles with horizontal cut should not be used as they create resistance to the flow of condensate. Hence, in this case there are two options; (i) Use segmental baffles with vertical cut (Fig. 6.2 (h)) or (ii) Use segmental baffles with horizontal cut but their base must be trimmed (Fig. 6.2 (g)).

Tube holes are made in baffle. Diameter of tube hole in baffle is kept higher than tube OD. Normally this clearance is less than 0.8 mm.

Thickness of baffle depends on size of shell and spacing between baffles. For the given size of shell, thickness of baffle increases with increase in baffle spacing. It can be determined from the table given in TEMA standard. As per Kern⁵, baffle spacing ranges from 0.2 to 1 times shell ID. Lesser baffle spacing gives higher heat transfer coefficient but at the expense of higher pressure drop. Optimum baffle spacing is in between 0.3 to 0.5 times shell inside diameter.

6.2.1.4 Tube

Tube size range from 1/4 in (6.35 mm) to 2.5 in (63.5 mm) in shell and tube heat exchanger. Data for standard tubes are given in TEMA standard and Table 11.2 of Ref. 2. For the standard tubes, its size is equal to outer diameter of tube. Thickness of standard tubes are expressed in BWG (Birmingham Wire Gauge). Increase in the value of BWG means decrease in tube thickness. For no phase change heat exchangers and for condensers, 3/4 in (19.05 mm OD) tube is widely used in practice. While for reboiler 1 in (25.4 mm OD) tube size is common. Tubes are available in standard lengths like 6 ft (1.83 m), 8 ft (2.44 m), 12 ft (3.66 m), 16 ft (4.88 m) and 6 m.

6.2.1.5 Tube Side Pass Partition Plate

Tube side passes are provided to decrease the tube side flow area and to increase tube side fluid velocity thereby to improve the tube side heat transfer coefficient, at the expense of pressure drop. This is true only if there is no phase change on tube side. Hence, more number of tube side passes are recommended only if there is no change in the phase of tube side fluid.

For example, at the design stage, if the number of tube side passes are increased from one to two, then for the given volumetric flow rate, flow area becomes half and velocity becomes double. Since, tube side heat transfer coefficient, $h_i \propto u_t^{0.8}$ (where u_t is tube side fluid velocity), on increasing number of tube side passes from 1 to 2, h_i nearly becomes 1-7 times. But, $\Delta p_t \propto u_t^{2.8}$, so the pressure drop increases by 6.96 times. Increase in h_i means decrease in heat transfer area required and decrease in fixed cost. Increase in Δp_t means increase in power required for pumping the tube side fluid and increase in operating cost. Hence, ideally optimum number of tube side passes must be decided.

Tube side passes are very common and are advantageously used for improving tube side heat transfer coefficient. These passes can be achieved in many ways by locating partition plates in channel covers. Figure 6.4 gives different designs for achieving desired tube passes.

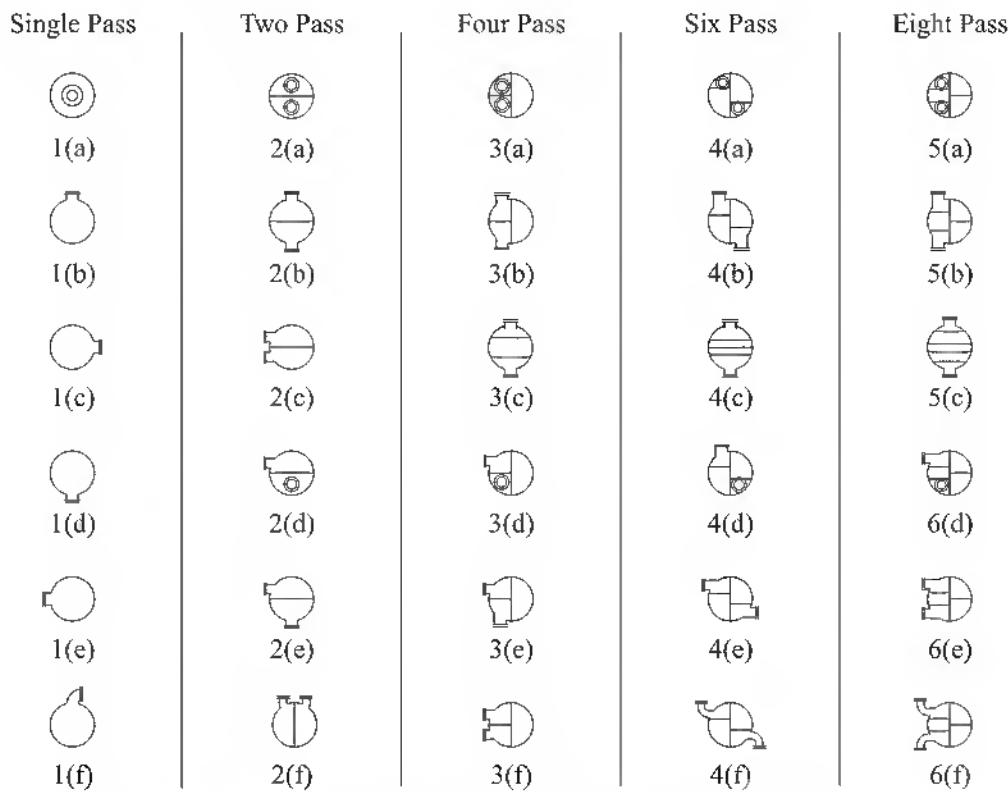


Fig. 6.4 Nozzle Orientation Designs for Tube Side Passes³

6.2.1.6 Tie Rods

Baffles are supported by tie rods. Tie rods are made from solid metal bar. Normally four or more tie rods are required to support the baffles. Diameter of tie rod is less than the diameter of tube. Diameter and number of tie rods required for given shell diameter are specified by TEMA standard and IS: 4503.

6.2.1.7 Spacers

Spacers (Fig. 6.5) are used to maintain the space between baffles. Spacers are the pieces of pipes or in the most of the cases they are the pieces of extra available tubes. Spacers are passed over the tie rods and because of them baffles do not slide over tie rods under the effect of the force of fluid. Hence, spacers fix the location of baffles and maintain the space between them. Length of spacer is equal to space between the baffles.

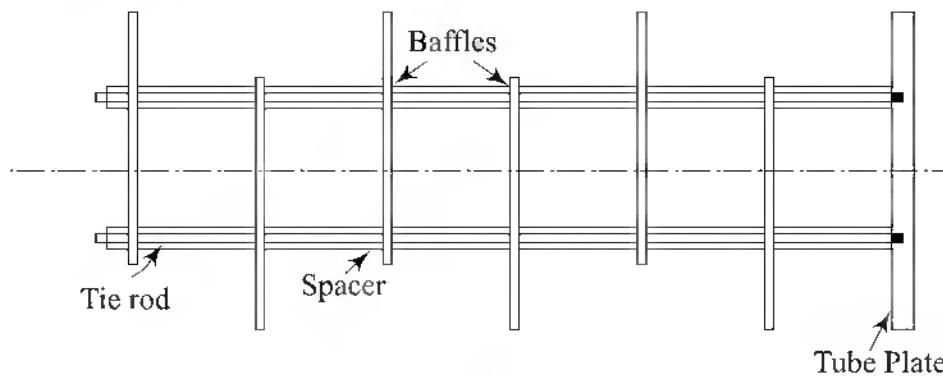


Fig. 6.5 Baffle Spacer Detail

6.2.1.8 Tube Sheet

Tubes and one end of tierods are attached to tube sheet (also called tube plate). Hence, entire load of tube bundle is transferred to one or two tube sheets. In U-tube shell and tube heat exchanger (as shown in Fig. 6.1(d), type CFU or Fig. 6.1(e), type AKT) only one tube sheet is used. While in fixed tube sheet shell and tube heat exchanger, two tube sheets are used. One surface of tube sheet is exposed to tube side fluid and other surface is exposed to shell side fluid. This point is very important in the selection of material for tube sheet and also in determining tube sheet thickness.

Tube to tube sheet joints are two types; (a) Expanded joint and (b) Welded joint as shown in Fig. 6.6.

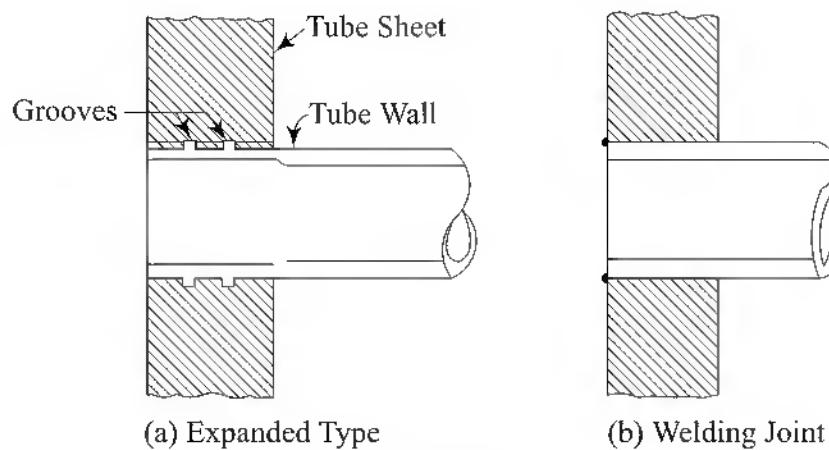


Fig. 6.6 Tube to Tube Sheet Joint

In expanded type joint, tube holes are drilled in a tube sheet with a slightly greater diameter than the tube OD. Two or more grooves are cut in the wall of each hole. The tube is placed inside the tube hole and a tube roller is inserted into the end of the tube. Roller is slightly tapered. On application of the roller, tube expands and tube material flows into grooves and forms an extremely tight seal. Welding joint is used only for the cases where leakage of fluid can be disastrous.

Detailed method for calculating tube sheet thickness is given in TEMA and in IS: 4503. Minimum required tube sheet thickness is also specified in the same. Simplified equation is also available for calculating tube sheet thickness.

$$t = FG \left(\frac{0.25 P}{f} \right)^{\frac{1}{2}} \quad (6.6)$$

where, t = Effective tube sheet thickness, mm

F = constant (1 for fixed tube sheet and 1.25 for U-tube)

P = Design pressure, kPa

f = Allowable stress of tube sheet material at design temperature, kPa

G = Mean diameter of gasket, mm

Then thickness of tube sheet T is given by equation

$$T \geq (t + \text{depth of pass partition groove} + \text{tube side corrosion allowance} + \text{shell side corrosion allowance})$$

Also $T \geq$ Minimum thickness specified in the standard

6.2.1.9 Sealing Strip

It is a shell side component. Sealing strips are attached on the inside surface of shell as shown in Fig. 6.7 throughout the length of shell.

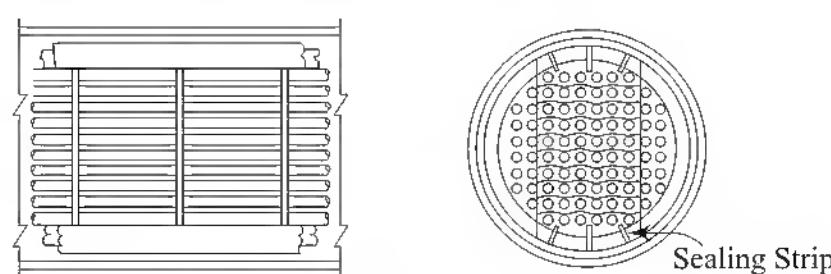


Fig. 6.7 Sealing Strips

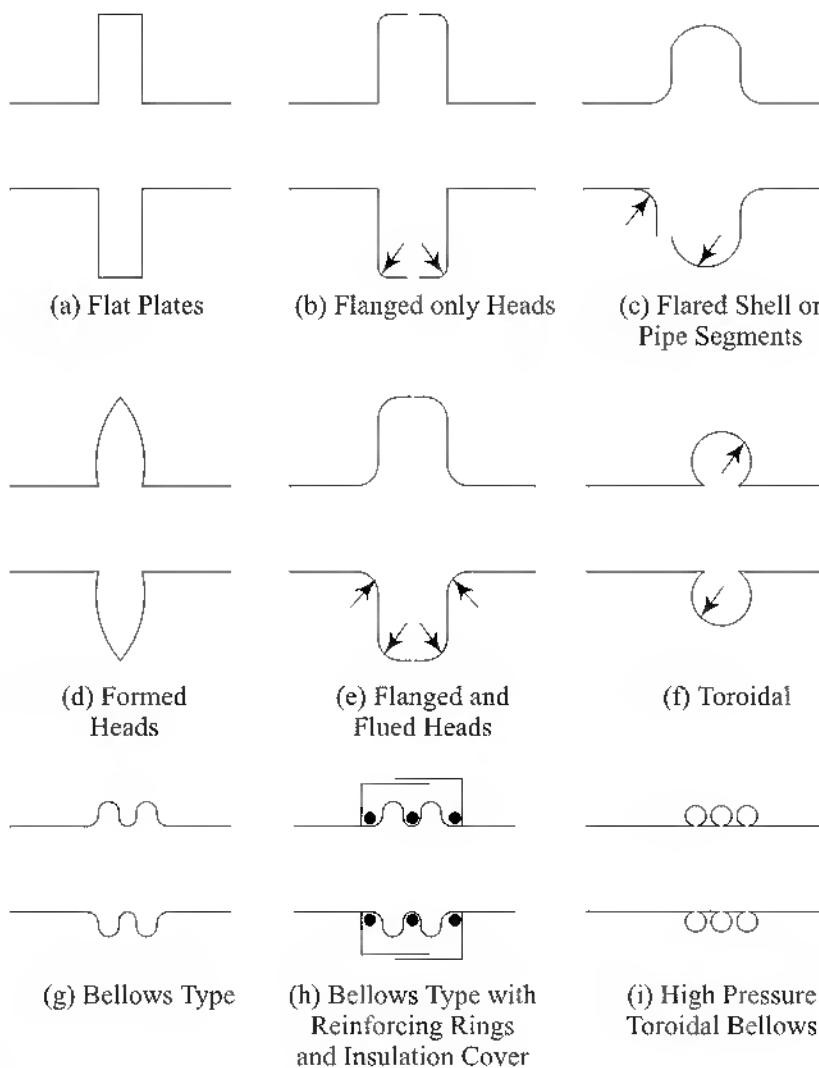
There are two functions of sealing strips.

- (i) Sealing strips reduce the amount of bypass stream of shell side fluid flowing through the clearance between shell inside diameter and tube bundle diameter and thereby improve the shell side heat transfer coefficient. (This is valid only if there is no phase change of shell side fluid).
- (ii) Sealing strips also make the removal of tube bundle from the shell easy. Hence, they are also known as sliding strips.

In Kern's method, it is assumed that entire shell side fluid is flowing across the tube bundle and between the baffles. Actually, shell side fluid is flowing in various ways. In all latest methods of process design, shell side fluid flow is divided in five streams. Among these streams, one of the streams is flowing through the clearance between shell ID and tube bundle. This clearance is significant in case of pull through floating head heat exchanger (about 90 to 100 mm). In addition, this clearance provides low pressure drop path for shell side fluid. Hence, actual fraction of shell side fluid bypassed through this clearance is considerably higher. Sealing strip blocks the gap between bundle and shell and thereby reduce the amount of this bypass stream considerably. In case of pull through floating head heat exchanger, all latest computer programmes, like HTRE, suggest the use of sealing strip. Also in such programs shell side heat transfer coefficient is a function of dimension and numbers of sealing strips provided. Kern's method is not recommended for use to predict heat transfer coefficient and pressure drop for pull through floating head heat exchanger.

6.2.1.10 Expansion Joint

Expansion joint is attached to shell wall. In this case shell is made from two pipe pieces. Two pipe pieces are joined together by an expansion joint as shown in Fig. 6.8.

**Fig. 6.8 Expansion Joints²**

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Expansion joint is used in fixed tube heat exchanger to permit the differential thermal expansion or contraction between shell and tubes which otherwise is not permitted by fixed tube sheet heat exchanger. Differential thermal expansion between shell and tube is significant if there is a large temperature difference between temperature of shell material and temperature of tube material during operation or if tube material and shell material are different. For the given case of fixed tube sheet heat exchanger, whether an expansion joint is required or not can be determined by calculations given in TEMA. If expansion joint is not provided in fixed tube sheet heat exchanger, then fixed tube sheet does not permit the unequal expansion or contraction between shell and tube and it can result in the development of thermal stress across the tube sheet. If this thermal stress is higher than permissible value, then it may develop a crack in tube sheet, and can result in leakage at tube to tube sheet joint. Other options, available to avoid the development of thermal stress in the tube sheet are use of either U tube heat exchanger or floating head heat exchanger.

6.2.2 Advantages and Disadvantages of Different Type of Shell and Tube Heat Exchangers Over Each Other

6.2.2.1 Advantages of U-tube and Floating Head Heat Exchangers (Figs 6.1(a) and 6.1(d)) Over Fixed Tube Sheet Heat Exchangers are

- (i) Maintenance and cleaning of tube bundle is easier. In U-tube and floating head heat exchanger entire tube bundle can be easily taken out for cleaning and maintenance. While the same is not possible in fixed tube sheet heat exchanger.
- (ii) U-tube and floating head heat exchangers permit the differential thermal expansion or contraction between shell and tube. Therefore, use of expansion joint is not required in U-tube and floating head heat exchangers.

6.2.2.2 Disadvantages of U-tube and Floating Head Heat Exchangers Over Fixed Tube Sheet

- (i) For a given heat duty, U-tube and floating head heat exchangers are costlier than fixed tube sheet heat exchangers because of the following reasons.
 - (a) For the same number of tubes, tube arrangement and tube pitch, shell sizes required by U-tube and floating head heat exchangers are higher than the same required by fixed tube sheet heat exchangers. In U-tube heat exchanger, minimum bend radius of U-tube is $1.5d_o$, hence in central portion of tube bundle, tubes cannot be provided. In floating head heat exchanger more clearance between shell ID and tube bundle diameter (50 to 100 mm) is required.
 - (b) In case of cooling or heating of shell side fluid (no phase change) heat transfer coefficients obtained in U-tube and floating head heat exchangers for the given heat duty are less than the same obtained in fixed tube sheet heat exchangers as bypassing area is higher in case of U-tube or floating head as compared to fixed tube sheet heat exchanger.

6.2.2.3 Advantages and Disadvantages of U-tube Heat Exchanger Over Floating Head Heat Exchanger

Advantage:

Fixed cost of U-tube heat exchanger is always less than the same of floating head heat exchanger for the given duty.

Disadvantages:

- (i) In U-tube, tube material becomes weak in bending portion, hence use of U-tube heat exchanger is not recommended for severe conditions. While for the same conditions floating head heat exchanger can be used.
- (ii) In U-tube heat exchanger number of tube side passes are fixed (two). It reduces the flexibility in design calculations.
- (iii) U-tube heat exchanger is not recommended to use for tube side dirty fluid. As it is difficult to remove dirt from U-tube. While floating head heat exchanger can be used where both shell side fluid and tube side fluid are dirty.

6.2.2.4 Floating Head Heat Exchangers are of Two Types

- (a) Split-ring floating head heat exchanger
- (b) Pull through floating head heat exchanger.

Split-ring floating head heat exchanger is widely used in the chemical process industries (Fig. 6.1 (a)). With this type split backing ring is provided. Advantages and disadvantages of this heat exchanger over pull through floating head heat exchanger are as follows.

Advantage:

Compared to pull through floating head heat exchanger, less diameter is required for shell to accommodate the same number of tubes.

Disadvantages:

More maintenance time is required. In maintenance, shell cover is removed first, then the split backing ring, then floating head cover and finally tube bundle is removed from the stationary end.

Pull through floating head heat exchanger is the costliest type of shell and tube heat exchanger because it requires largest shell diameter for the given numbers of tubes. With this heat exchanger it is possible to remove the entire tube bundle including floating head assembly from the stationary end.

6.2.3 Different Types of TEMA Designations

In TEMA standard different types of shell and tube heat exchangers are designated by three letters. First letter indicates type of front head, second letter indicates type of shell and third indicates type of rear head. Different types of TEMA designations are shown in Fig. 6.9.

For example, BEM shell and tube type heat exchanger means bonnet type (B type) front head, single pass (E type) shell and stationary head with fixed tube sheet (M type) rear head. BFL type shell and tube heat exchanger means bonnet type front head, two pass shell with longitudinal baffle (F type shell) and stationary head with fixed tube sheet and removable flat cover (L type rear head).

6.3 GENERAL DESIGN METHOD FOR SHELL AND TUBE HEAT EXCHANGER

6.3.1 Calculation of Heat Duty

- (a) For cooling or heating or for no phase change, heat duty is calculated by equation

$$\phi = \dot{m}C_p \Delta t \quad (6.7)$$

where, \dot{m} = Mass flow rate of fluid, kg/s

C_p = Specific heat of fluid, in kJ/(kg \cdot $^{\circ}$ C)

Δt = Temperature difference to be carried out, $^{\circ}$ C

ϕ = Heat duty required, kW

- (b) For condensation with subcooling

$$\phi = \dot{m}\lambda + \dot{m}C_L \Delta t \quad (6.8)$$

	Front End Stationary Head Types	Shell Types	Rear End Head Types
A	Channel and Removable Cover	E	L
B	Bonnet (Integral Cover)	F	M
C	Removable Tube Bundle Only	G	N
	Channel Integral with Tube Sheet and Removable Cover	H	P
N	Channel Integral with Tube Sheet and Removable Cover	J	S
D	Special High Pressure Closure	K	T
		X	U
			W

Front End Stationary Head Types:

- A: Channel and Removable Cover
- B: Bonnet (Integral Cover)
- C: Removable Tube Bundle Only
- N: Channel Integral with Tube Sheet and Removable Cover
- D: Special High Pressure Closure

Shell Types:

- E: One Pass Shell
- F: Two Pass Shell with Longitudinal Baffle
- G: Split Flow
- H: Double Split Flow
- J: Divided Flow
- K: Kettle Type Reboiler
- X: Cross Flow

Rear End Head Types:

- L: Fixed Tube Sheet Like "A" Stationary Head
- M: Fixed Tube Sheet Like "B" Stationary Head
- N: Fixed Tube sheet Like "C" Stationary Head
- P: Outside Packed Floating Head
- S: Floating Head with Backing Device
- T: Pull Through Floating Head
- U: U-tube Bundle
- W: Externally Sealed Floating Tube sheet

Fig. 6.9 TEMA-type Designation for Shell and Tube Heat Exchangers¹. (Fig. No. N-1.2 of TEMA 8th Edition)
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where, \dot{m} = Mass flow rate of vapour, kg/s
 C_L = Specific heat of condensate, kJ/(kg · °C)
 λ = Latent heat of vaporization, kJ/kg

(c) For reboilers

$$\phi = \dot{m}_V \lambda \times 1.05 \quad (\text{considering } 5\% \text{ heat loss}) \quad (6.9)$$

where, \dot{m}_V = Vaporization rate, kg/s

λ = Latent heat of vaporization, kJ/kg

Actually heat duty of the reboiler is determined based on energy balance around distillation column.

6.3.2 Selection of Cooling Medium or Heating Medium

Selection of cooling or heating medium mainly depends on the temperature (inlet temperature) at which cooling medium or heating medium is required. Other factors like heat transfer coefficient provided by cooling medium or heating medium, cost of the medium, etc. are also considered in the final selection. Table 6.5 and Table 6.6 can be used as a guideline for the selection of heating medium and cooling medium respectively.

Table 6.5 Commonly Used Heating Fluids

Heating medium	Recommended temperature range
Saturated steam	100 to 180° C
Oil (Thermic Fluid)	180 to 300° C
Dowtherm E	180 to 260° C
Dowtherm A	300 to 400° C
Molten salt	400 to 590° C
Na-K alloys	590 to 750° C
Flue gas or Hot air	750 to 1100° C
Electric heating	> 1100° C

Table 6.6 Commonly Used Cooling Medium

Cooling medium	Operating range	Recommended to use for temperature
1. Brines	-68 to 5°C	< 8°C
Examples:		
Ethanol water solution	-5 to 5°C	< 8°C
Methanol water solution	-33 to -1°C	< 8°C
Ethylene glycol	-34 to 5°C	< 8°C
Aqueous solution of calcium chloride	-20 to 0°C	< 8°C
Aqueous solution of sodium chloride	-9.4 to 5°C	< 8°C
Methylene chloride	-67.7 to -37.2°C	< -33°C
Trichloroethylene	-67.7 to -37.2°C	< -33°C
Trichlorofluoromethane	-67.7 to -37.2°C	< -33°C

(Contd.)

Table 6.6 (Contd.)

Cooling medium	Operating range	Recommended to use for temperature
2. Chilled water	5 to 12°C	< 35°C
3. Cooling water	32 to 60°C	35 to 100°C
4. Oil	-1 to 316°C	< 300°C
5. Air	Atmospheric temperature	> 60°C

6.3.2.1 Heating Mediums

If temperature of heating medium is required in between 100° to 180°C, saturated steam is used as heating medium. Saturated steam condenses in dropwise manner, hence it provides very high heat transfer coefficient [about 6000 W/(m² · °C)]. Superheated steam is not selected as heating medium for indirect (through the wall) heat transfer because superheating zone provides very low heat transfer coefficient.

If the temperature of heating medium required is greater than 180°C, then use of saturated steam as a heating medium is not economical. From 180°C to 300°C, hot oil (thermic fluid) is preferred as heating medium. Hot oil system is easily available at comparatively low cost. Hot oil provides low heat transfer coefficient and also properties of oil are uncertain. For the precise application at large scale Dowtherm E can be considered as a heating medium. Dowtherm E is specially processed orthodichlorobenzene. Aluminium should not be used with Dowtherm E. Dowtherm A is an organic fluid of high heat stability, an eutectic mixture containing 73.5 percent diphenyl oxide and 26.5 percent diphenyl by mass. Both, Dowtherm A and Dowtherm E were developed by Dow Chemical Company, USA. Molten salts are a molten mixtures of inorganic salts, one of which is an eutectic consisting 40% NaNO₂, 7% NaNO₃ and 53% KNO₃. 56% Na and 44% K (by mass) and 22% Na-78% K (by mass) are used as sodium potassium alloys.

6.3.2.2 Cooling Mediums

In refrigeration terminology a brine is any liquid cooled by a refrigerant and circulated as a heat transfer fluid. Operating temperature range is -68°C to 5°C. For any fluid to be cooled below 8°C (minimum possible temperature of chilled water, 5°C + minimum driving force required for heat transfer in shell and tube heat exchanger, 3°C), brines are used as cooling medium. Brine may be:

- (a) An aqueous solutions of inorganic salts, e.g. sodium chloride solution, calcium chloride solution, etc.
- (b) Aqueous solutions of organic compounds such as alcohols or glycols, e.g. ethanol, methanol, ethylene glycol, propylene glycol, etc
- (c) Chlorinated or fluorinated hydrocarbons, ex. methylene chloride, trichloroethylene, trichlorofluoromethane, halogenated hydrocarbons as refrigerants, etc.

Outlet temperature of chilled water from chilled water plant ranges from 5 to 15°C. While its return temperature is kept from 7°C to 12°C. When any fluid is to be cooled below the temperature equal to design temperature of cooling water

from cooling tower + minimum driving force required for heat transfer in shell and tube heat exchanger (3°C), chilled water is used as cooling medium.

Cooling water is the most widely used cooling medium at near ambient temperature.

Design temperature of cooling water from cooling tower = Wet bulb temperature + Approach to the wet bulb temperature (ranges from 4 to 6°C).

Cooling water can be used as cooling medium even though inlet temperature of hot fluid is more than 100°C . But in this case extra care must be taken as at any time cooling water can be converted into steam. In case of exothermic reaction, oil (thermic fluid) is used for dual purpose. Initially to achieve the reaction temperature, oil is used as heating medium. Then after as the reaction proceeds, the same oil is used as cooling medium. For this, one heater and one cooling water cooler (or economizer) are provided in oil cycle.

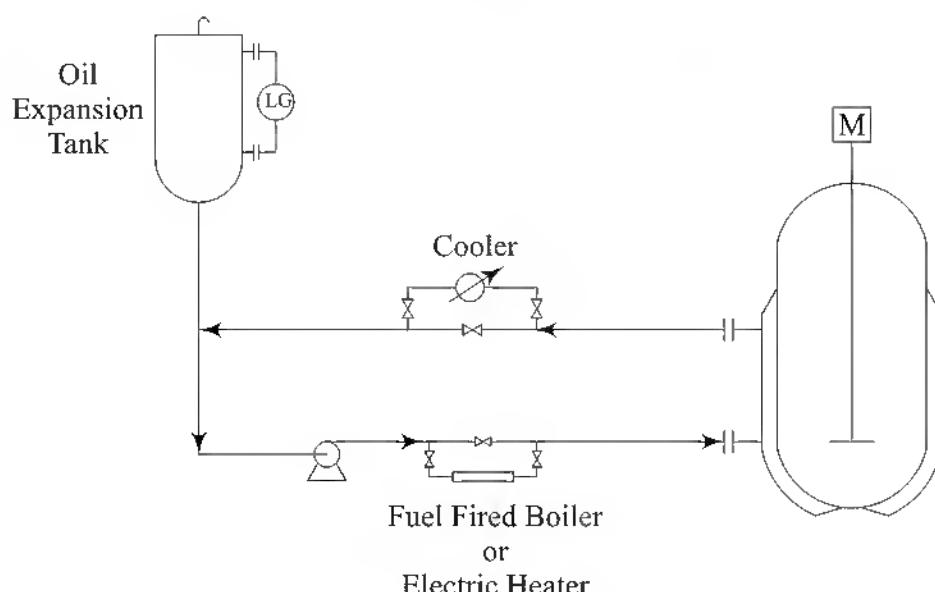


Fig. 6.10 Hot Oil (Thermic Fluid) Cycle

6.3.2.3 Air Cooling

Air cooled heat exchanger has certain advantages as well as disadvantages over cooling water cooled heat exchanger using cooling water.

Advantages:

- (i) Generally operating cost of Air Cooled Heat Exchanger (ACHE) is less than the same of Water Cooled Heat Exchanger (WCHE). Operating costs of WCHE includes cost of make-up water for the cooling tower, power required for the cooling tower fans and for the circulating pump, cooling tower maintenance, etc. While operating cost of ACHE includes cost of power consumed by fan. Day by day cost of water increases and hence the difference in operating cost between WCHE and ACHE increases.
- (ii) Inlet temperature of water to cooling tower (or outlet temperature of cooling water from heat exchanger circuit) should not be greater than 60°C otherwise it creates the scale formation and also affects the material of construction of cooling tower. While for air there is no limit for temperature.
- (iii) Fouling or scale formation does not take place with aircooling.

Disadvantages:

- (i) Fixed cost of ACHE is higher than the same of WCHE. Heat transfer coefficient of air is quite less than heat transfer coefficient provided by cooling water and hence heat transfer area required by ACHE is more. Also design inlet temperature of cooling water from cooling tower is always less than design ambient temperature. It gives lesser Mean Temperature Difference (MTD) for ACHE compared to the same for WCHE and hence it increases heat transfer area required for ACHE.
- (ii) Minimum driving force required for heat transfer, Δt_{\min} is 10° to 15°C for ACHE and 3 to 5°C for WCHE. Hence minimum temperature that can be achieved with ACHE is ambient temperature plus 10°C , while with WCHE it is design temperature of cooling water from cooling tower plus 3°C . Hence, if the fluid is to be cooled or condensed below 55°C in a tropical location, ACHE cannot be used.

Based on atmospheric conditions, cost of heat exchangers and cost of water, one can find out the temperature above which ACHE becomes cheaper than WCHE.

6.3.3 Fluid Allocation

- (a) Fluid allocation in case of phase change:
Condensing or vaporizing fluid is normally taken on shell side as in the shell liquid and vapour phases are easily separated. In case of a condenser, if condensation is carried out in tube side, then the entire liquid condensate forms a film on heat transfer surface which provides additional resistance to heat transfer. In case of reboilers normally on both sides phase change take place and hence fluid allocation depends on other factors.
- (b) Fluid allocation in case of no phase change:
If there is no phase change in shell side and tube side fluid, then fluid allocation depends on the following factors.
 - (i) **Corrosion:** Corrosive fluid is allocated tube side as the cheaper material like mild steel can be used for costly shell, baffles, etc. Also, other costlier parts like tubes and tube sheets require the use of non-corrosive or costlier material of construction in case of corrosive fluid, whether it is on tube side or in shell side. For example, in cooling of ethyl iodide, it should be taken on tube side as it requires the use of special material (Hastelloy-B).
 - (ii) **Fouling:** In fixed tube sheet heat exchanger inside surface of tubes can be easily cleaned and hence the fluid which has the greatest tendency to foul on heat transfer surface should be placed in the tubes. For example cooling water has a tendency to foul. Hence normally it is passed through tubes.
 - (iii) **Fluid Temperature:** At very high temperature as well as at very low temperature use of special alloy is required. Hence, very hot or very cold fluid is placed on tube side to avoid the use of costlier material for the shell. At moderate temperature, hotter fluid is better passed through

tubes. If it is placed on shell side more insulation is required not only to reduce the heat loss but also for safety purpose.

- (iv) **Operating Pressure:** High pressure stream should be placed on tube side as high pressure tubes are cheaper than high pressure shell. Diameter of shell is very much greater than diameter of tubes hence its thickness is more sensitively changed with change in pressure as compared to that of the tubes.
- (v) **Fluid Flow Rates and Viscosity:** Very low value of flow rate of fluid and high value of viscosity of fluid give low value of Reynolds number. Fluid which provides very low value of Reynolds number should be placed on shell side as the dependency of shell side heat transfer coefficient on Reynolds number is less as compared to the same of tube side heat transfer coefficient. Hence, for the lower value of Reynolds number shell side heat transfer coefficient is higher than tube side heat transfer coefficient. For example, in cooling of viscous oil, it is allocated shell side as it provides poor Reynolds number.

6.3.4 Establish Energy Balance or Heat Duty Balance and based on that Find the Mass Flow Rate of Heating Medium or Cooling Medium Required

For example in case of condenser

$$\phi_t = \dot{m}\lambda = \dot{m}_w C_{Lw}(t_2 - t_1) \quad (6.10)$$

where, \dot{m} = Mass flow rate of vapour condensed, kg/s

λ = Latent heat of condensation kJ/kg

\dot{m}_w = Mass flow rate of cooling medium required kg/s

C_{Lw} = Specific heat of cooling medium kJ/(kg · °C)

t_2, t_1 = Outlet inlet temperatures of cooling medium, °C

6.3.5 Mean Temperature Difference

Mean temperature difference (MTD) is calculated from equation

$$\Delta T_m = F_t \Delta T_{lm} \quad (6.11)$$

where, ΔT_{lm} = Logarithmic mean temperature difference, °C

ΔT_{lm} can be determined by following equation

$$\Delta T_{lm} = \frac{\Delta T_2 - \Delta T_1}{\ln\left(\frac{\Delta T_2}{\Delta T_1}\right)} \quad (6.12)$$

where, ΔT_2 and ΔT_1 are terminal temperature differences.

For 1-1 heat exchanger (one shell side pass and one tube side pass), $F_t = 1$

When shell side passes and/or tube side passes are more than one, F_t must be determined.

$$F_t = f(R, S) \text{ where } R \text{ and } S \text{ are temperature ratios} \quad (6.13)$$

$$R = \frac{T_1 - T_2}{t_2 - t_1} \quad (6.14)$$

$$S = \frac{t_2 - t_1}{T_1 - t_1} \quad (6.15)$$

where,
 T_1 = Hot fluid inlet temperature
 T_2 = Hot fluid outlet temperature
 t_1 = Cold fluid inlet temperature
 t_2 = Cold fluid outlet temperature

Graphs of F_t vs S for the different values of R , for the different cases are given in Ref. 1. Figure 6.11 gives values of F_t for even number of tube passes. If $F_t < 0.75$, economic design of shell and tube heat exchanger cannot be achieved. For such a case alternate type of heat exchanger design should be considered. In the cases where temperature cross is likely to occur, multiple shell pass heat exchanger or more numbers of heat exchangers in series, should be considered.

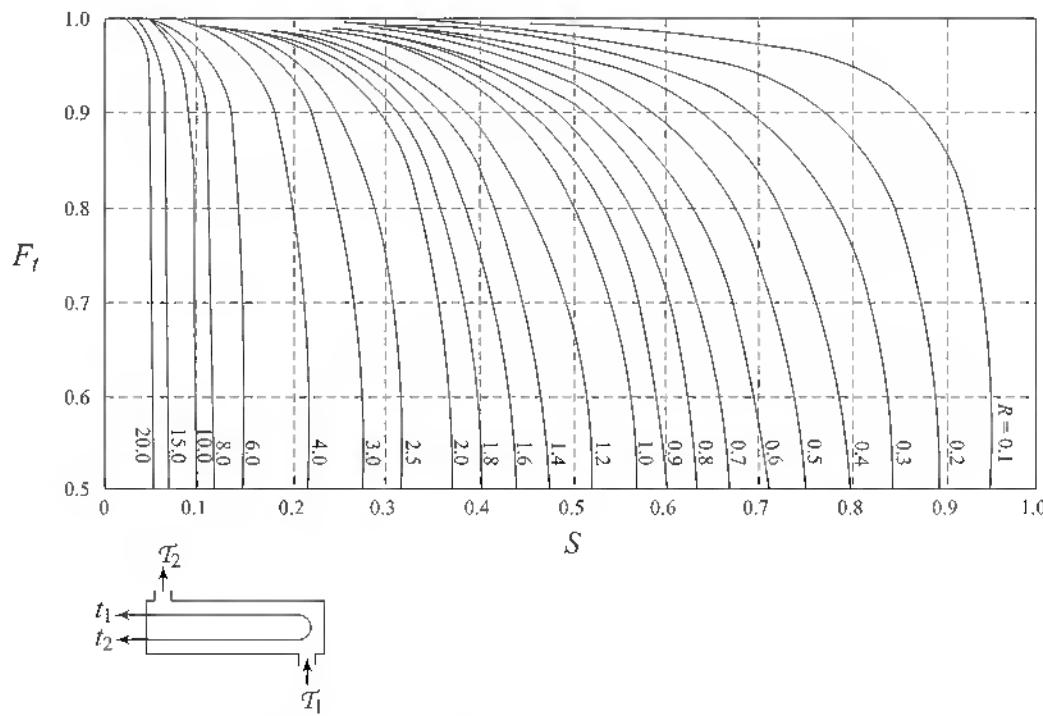


Fig. 6.11 Temperature Correction Factor: One Shell Pass; Even Number of Tube Passes¹
 (Fig. T-3.2A of TEMA, 8th Edition)
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 USA.)

6.3.6 Estimation of Overall Heat Transfer Coefficient

Assume the value of U , overall heat transfer coefficient. For this, use the standard tables, in which the range of the values of U for the different cases are given. Refer Table 6.7 for the recommended values.

Table 6.7 Typical Overcell Coefficients for Shell and Tube Heat Exchangers

Shell side	Tube side	U, W/(m ² · °C)
[A] Heat exchangers		
1. Water	Water	800–1400
2. Organic solvent	Water	300–850
3. Organic solvent	Brine	200–500
4. Light oil	Water	350–900
5. Heavy oil	Water	60–300
6. Water	Brine	600–1200
7. Gas	Brine	15–250
8. Gas	Gas	10–50
9. Gas	Water	20–300
10. Naptha	Water	300–400
11. Lube oil	Water	150–500
12. Gasoline	Water	350–550
13. Kerosene	Water	150–300
14. Water	10 to 30% Caustic soda solution	500–1250
[B] Condensers		
1. Organic vapour	Water	700–1000
2. Organic vapour + Gas	Water or Brine	100–300
3. Low boiling hydrocarbons Vapour mixture	Water	450–1100
4. Naptha vapour	Water	300–425
5. Steam	Water	2000–5000
6. Alcohol vapour	Water	500–1000
7. Saturated Organic vapour	Water or Brine	280–680
[C] Vaporizers/Reboilers		
1. Light organics	Steam	800–1200
2. Heavy organics	Steam	600–900
3. Aqueous solution	Steam	1000–1500
4. Chlorine	Steam	700–1500
5. Ammonia	Steam	700–1500
6. Water	Steam	1250–2000
7. Refrigerants	Water	425–850

Find out heat transfer area based on this selected or assumed value of U.

$$A = \frac{\phi_t}{U \times \Delta T_{lm}} \quad (6.16)$$

For the 1st trial calculation, this A is the heat transfer area provided or actual heat transfer area of heat exchanger.

$$A = A_{\text{pro}} = N_i \pi d_o L \quad (6.17)$$

where, d_o = Outside diameter of tube, m

L = Tube length, m

N_t = Total number of tubes

Values of d_o and L are decided by designer and from Eq. (6.17), value of N_t is determined. Then for the first trial, number of tube side passes and shell side passes are decided. Tube arrangement and type of heat exchanger are also decided.

6.3.7 Finding Shell Diameter

Based on all these informations find the inside diameter of shell. Select the type of baffle. First choice is 25% cut segmental baffle. Fix the value of baffle spacing. If there is cooling or heating on shell side then for the first trial baffle spacing B_s could be 0.3 to 0.5 times shell ID and if there is condensation or boiling on shell side, B_s could be equal to shell ID for the 1st trial.

6.3.8 Calculations of Tube Side Heat Transfer Coefficient

(a) For heating or cooling on tube side (no phase change), tube side heat transfer coefficient is determined by Sieder-Tate equation. If Re is less than 2000.

$$Nu = \frac{h_i d_i}{k_f} = 1.86 \left(Re \cdot Pr \cdot \frac{d_i}{L} \right)^{0.33} \left(\frac{\mu}{\mu_w} \right)^{0.14} \quad (6.18)$$

In this equation

If $Nu \leq 3.5$ then Nu is taken 3.5.

If $Re > 4000$, tube side heat transfer coefficient is determined by Dittus-Bolter equation:

$$Nu = \frac{h_i d_i}{k_f} = C Re^{0.8} Pr^{0.33} \left(\frac{\mu}{\mu_w} \right)^{0.14} \quad (6.19)$$

where, Nu = Nusselt number = $\frac{h_i d_i}{k_f}$

$$Re = \text{Reynolds number} = \frac{d_i u_t \rho}{\mu} = \frac{d_i G_t}{\mu}$$

$$Pr = \text{Prandtl number} = \frac{C_p \mu}{k}$$

h_i = Tube side heat transfer coefficient, W/(m² · °C)

d_i = Tube ID, m

L = Length of tube, m

k = Thermal conductivity of fluid, W/(m · °C)

C_p = Specific heat of fluid, kJ/(kg · °C)

μ = Viscosity of fluid at the bulk fluid temperature, (N · s)/m²

μ_w = Viscosity of fluid at tube wall temperature, (N · s)/m²

C = Constant

$C = 0.021$ for gases

= 0.023 for non-viscous liquid = 0.027 for viscous liquid

G_t = Tubeside mass velocity, kg/(m² · s)

$$G_t = \frac{\dot{m}}{a_t}$$

where, a_t = Tube side flow area = $\frac{N_t}{N_p} \times \frac{\pi}{4} d_i^2$ (6.20)

\dot{m} = Tube side mass flow rate of fluid, kg/s

N_t = Number of tubes

N_p = Number of tube side passes

u_t = Tube side fluid velocity = G_t/ρ , m/s

ρ = Density of fluid, kg/m³

To calculate tube side heat transfer coefficient, first calculate tube side flow area (a_t) then tube side mass velocity (G_t), then tube side Reynolds number (Re) and tube side Prandtl number (Pr), then depending on the value of Re , use Eq. 6.18 or Eq. 6.19 to calculate h_i .

For the transition region or for $2000 < Re < 4000$, no reliable equation is available, Hence, for this region h_i is found by using both Eqs (6.18) and (6.19) and the lesser of two values is considered.

Tube side heat transfer coefficient h_i can be calculated from the value of "heat transfer factor, J_h ", for the entire range of Reynolds number (from $Re = 10$ to 10^6). Equation relating h_i and J_h is

$$N_u = h_i \frac{d_i}{k} = J_h Re \cdot Pr^{0.33} \left(\frac{\mu}{\mu_w} \right)^{0.14} \quad (6.21)$$

Graph of J_h versus Re is given in Fig. 6.12 for the different values of L/d_i .

(b) For condensation inside the tubes.

Condensation coefficient depends on the position of condenser. For condensation, shell side condensation with horizontal position is the best which gives the maximum value of the coefficient. But if condensation is carried out in tube side then for horizontal position h_i is calculated by following two equations and higher of the two values is considered.

$$(i) \quad h_{ci} = 0.76 k_L \left(\frac{\rho_L (\rho_L - \rho_g) g}{\mu_L \tau_h} \right)^{1/3} \quad (6.22)$$

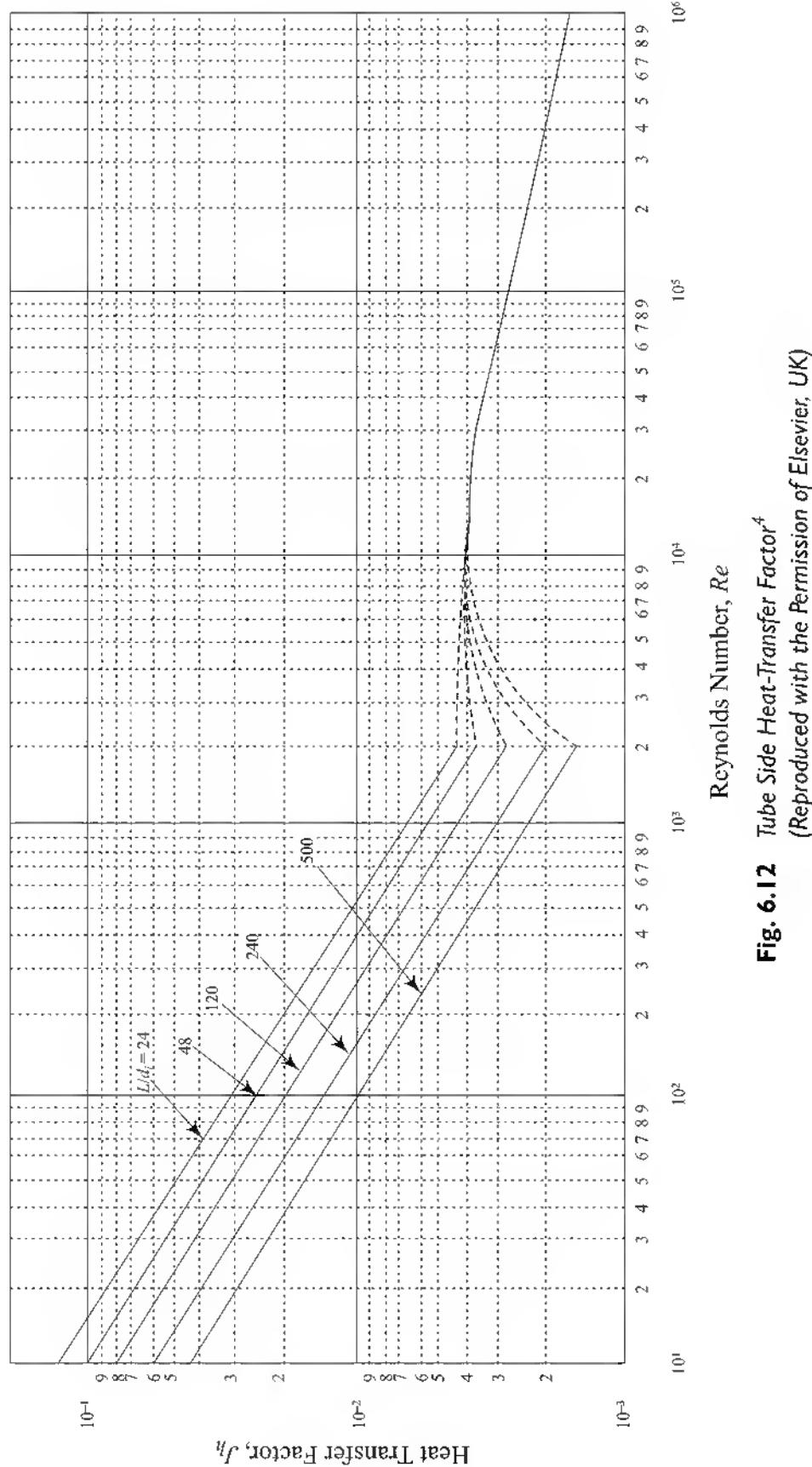


Fig. 6.12 Tube Side Heat-Transfer Factor⁴
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This equation is Nusselt equation, valid for stratified flow.

where, h_{ci} = Condensation coefficient, $\text{W}/(\text{m}^2 \cdot ^\circ\text{C})$

k_L = Thermal conductivity of liquid condensate, $\text{W}/(\text{m} \cdot ^\circ\text{C})$

ρ_L = Density of liquid condensate, kg/m^3

ρ_V = Density of vapour, kg/m^3

g = Acceleration of gravity = 9.81 m/s^2

μ_L = Viscosity of liquid condensate, $(\text{N} \cdot \text{s})/\text{m}^2$ or $\text{kg}/(\text{m} \cdot \text{s})$

τ_h = Horizontal tube loading or flow of condensate per unit length of tube, $\text{kg}/(\text{m} \cdot \text{s})$

All liquid condensate properties k_L , ρ_L and μ_L must be determined at mean temperature of condensate film.

$$\tau_h = \frac{W_C}{L \times N_t}$$

where, L = Tube length, m

W_C = Total condensate flow, kg/s

N_t = Total number of tubes

$$(ii) \quad h_{ci} = h_i' \left[\frac{1 + \sqrt{\rho_L/\rho_V}}{2} \right] \quad (6.23)$$

$$\text{where, } h_i' = 0.021 \left(\frac{k_L}{d_i} \right) Re_c^{0.8} Pr_c^{0.43}$$

This equation is known as Boyko-Kruzhilin equation, valid for annular flow.

$$Re_c = \text{Reynolds number for the condensate film} = \frac{4\tau_h}{\mu_L} \quad (6.24)$$

$$Pr_c = \text{Prandtl number of liquid condensate} = \frac{Cp_L \mu_L}{k_L} \quad (6.25)$$

where Cp_L , μ_L and k_L are properties of liquid condensate which must be determined at the mean temperature of condensate film.

$$\text{For vertical position, } h_{ci} = 0.926 k_L \left(\frac{\rho_L (\rho_L - \rho_v) g}{\mu_L \tau_v} \right)^{1/3} \quad (6.26)$$

$$\text{where, } \tau_v = \frac{W_c}{N_t \pi d_i}, \quad W_c = \text{Mass flow rate of liquid condensate kg/s}, \quad Re_c = \frac{4\tau_v}{\mu_L}$$

This equation is also called Nusselt equation, valid for $Re_c \leq 2000$. For $Re_c > 2000$, use Boyko-Kruzhilin equation, i.e. Eq. (6.23).

6.3.9 Calculation of Tube Side Pressure Drop

(a) If there is no phase change in tube side fluid, then tube side pressure drop can be calculated by following equation.

$$\Delta p_t = N_p \left(8J_f \left(\frac{L}{d_i} \right) \left(\frac{\mu}{\mu_w} \right)^{-m} + 2.5 \right) \frac{\rho u_t^2}{2} \quad (6.27)$$

$m = 0.25$ for $Re \leq 2100$ and $m = 0.14$ for $Re > 2100$

where, Δp_t = Tube side friction pressure drop, N/m^2 or Pa

N_p = Number of tube side passes

J_f = Tube side friction factor can be obtained from Fig. (6.13)

ρ = Density of tube side fluid, kg/m^3

u_t = Tube side fluid velocity, m/s

Δp_t calculated by this equation is actually permanent pressure loss. Calculated pressure drop (loss) should be less than maximum allowable pressure drop. In some applications, maximum allowable pressure drop is decided by process conditions. While in other applications, maximum allowable pressure drop is actually optimum pressure drop (loss). Heat exchanger design means the balance between two opposite factors, heat transfer coefficients related to fixed cost and pressure drop related to operating cost. Increase in heat transfer coefficient by modifying the heat exchanger design also increases pressure drop. Hence, ideally actual pressure drop should be equal to optimum pressure drop which gives the total cost of heat exchanger (i.e. fixed cost + operating cost) minimum. As a general guideline (Table 6.8) can be used to decide the value of maximum or optimum pressure drop. As per Kern, optimum pressure drop for gases is 2 psi (13.8 kPa) and for liquids it is 10 psi (69 kPa) (Ref. 5).

(b) If condensation takes place inside the tube then it is difficult to predict the pressure drop as vapour mass velocity is changing throughout the condenser. A common practice is to calculate $\Delta p'_t$ by Eq. (6.27) for inlet vapour flow rate and conditions, and multiply it by factor 0.5.

$$\Delta p_t = 0.5 \Delta p'_t \quad (6.28)$$

6.3.10 Calculation of Shell Side Heat Transfer Coefficient

(A) For heating or cooling on shell side or for no phase change on shell side, heat transfer coefficient h_o can be calculated by following procedure.

(a) Calculate shell side flow area by following equation

$$A_S = \frac{(P_t - d_o) D_S B_S}{P_t} \quad (6.29)$$

where, A_S = Shell side flow area, m^2

p_t = Tube pitch, m

d_o = Outside diameter of tube, m

B_S = Baffle spacing, m

D_S = Shell inside diameter, m

(b) Calculate shell side mass velocity G_s and linear velocity (u_s)

$$G_s = \frac{\dot{m}_s}{A_S} \text{ and } u_s = \frac{G_s}{\rho_s} \quad (6.30)$$

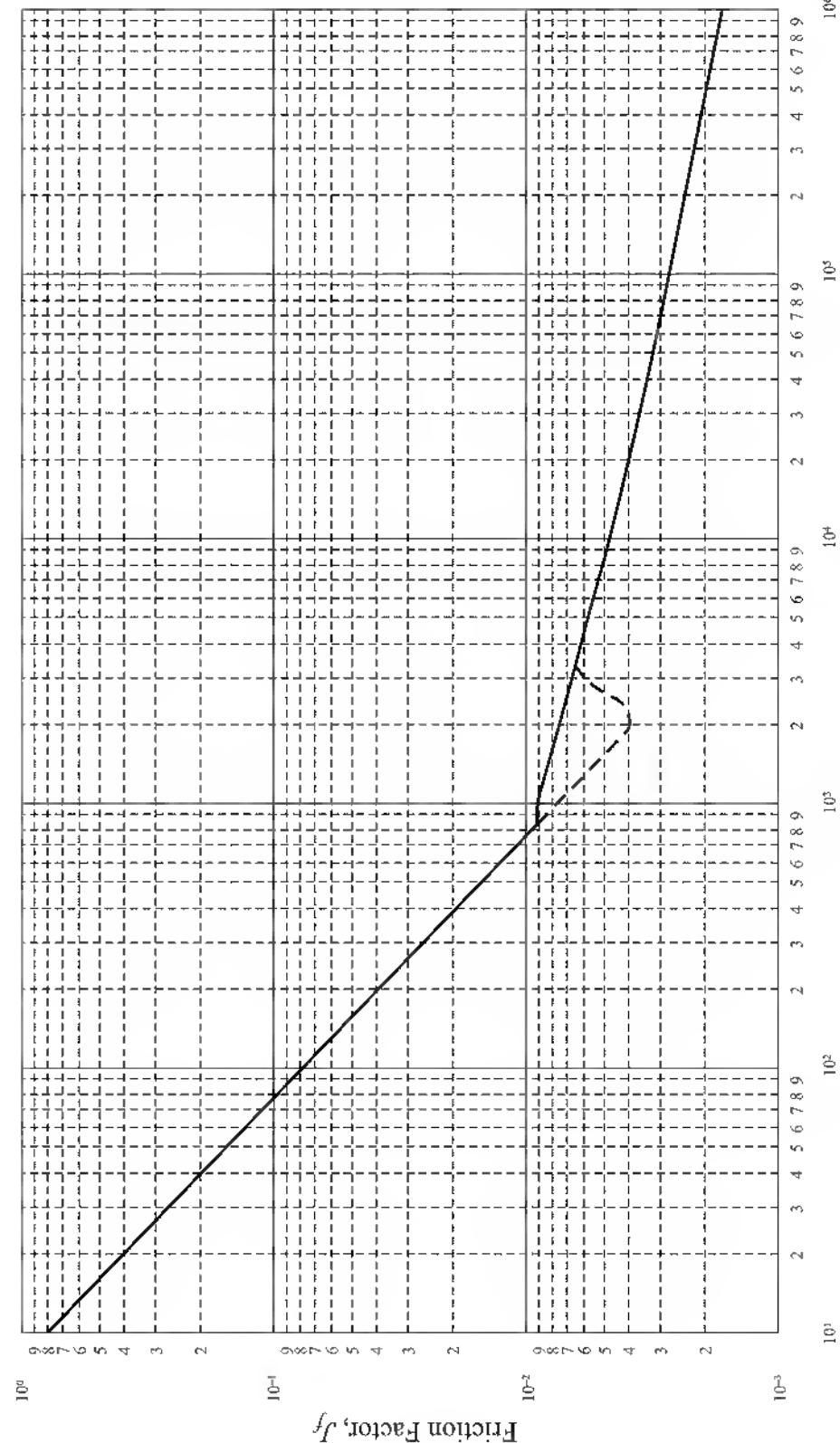


Fig. 6.13 Tube Side Friction Factors⁴
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where, G_S = Shell side mass velocity, $\text{kg}/(\text{m}^2 \cdot \text{s})$

\dot{m}_S = Shell side mass flow rate of fluid, kg/s

ρ_S = Density of shell side fluid, kg/m^3

- (c) Calculate the shell side equivalent diameter (d_e) for square pitch arrangement.

$$\begin{aligned} d_e &= \frac{4 \times \text{cross sectional area}}{\text{wetted perimeter}} \\ &= \frac{4 \times \left(P_t^2 - \frac{\pi}{4} d_o^2 \right)}{\pi d_o} \\ &= \frac{1.27}{d_o} (P_t^2 - 0.785 d_o^2) \end{aligned} \quad (6.31)$$

For triangular pitch arrangement,

$$\begin{aligned} d_e &= \frac{4 \times \left(\frac{1}{2} P_t \times P_t \sin 60 - \frac{\pi}{8} d_o^2 \right)}{(\pi d_o / 2)} \\ d_e &= \frac{1.1}{d_o} (P_t^2 - 0.907 d_o^2) \end{aligned} \quad (6.32)$$

- (d) Calculate shell side Reynolds numbers

$$Re_o = \frac{d_e G_S}{\mu} \quad (6.33)$$

where, μ = Viscosity of shell side fluid at average temperature, $\text{kg}/(\text{m} \cdot \text{s})$

Also calculate shell side Prandtl number

$$Pr = \frac{C_p \mu}{k} \quad (6.34)$$

C_p , μ and k are the properties of shell side fluid at average temperature.

- (e) Calculate the shell side heat transfer coefficient by following correlation (Ref. 5).

$$Nu = \frac{h_o d_e}{k} = 0.36 Re^{0.55} Pr^{0.33} \left(\frac{\mu}{\mu_w} \right)^{0.14} \quad (6.35)$$

This correlation is valid for the range of Reynolds number from 2000 to 1000 000. Shell side heat transfer coefficient can be found out from “shell side heat transfer factor, J_h ”.

$$Nu = \frac{h_o d_e}{k} = J_h \cdot Re \cdot Pr^{0.33} \left(\frac{\mu}{\mu_w} \right)^{0.14} \quad (6.36)$$

Graph of J_h versus Re_s given in Fig. 6.14.

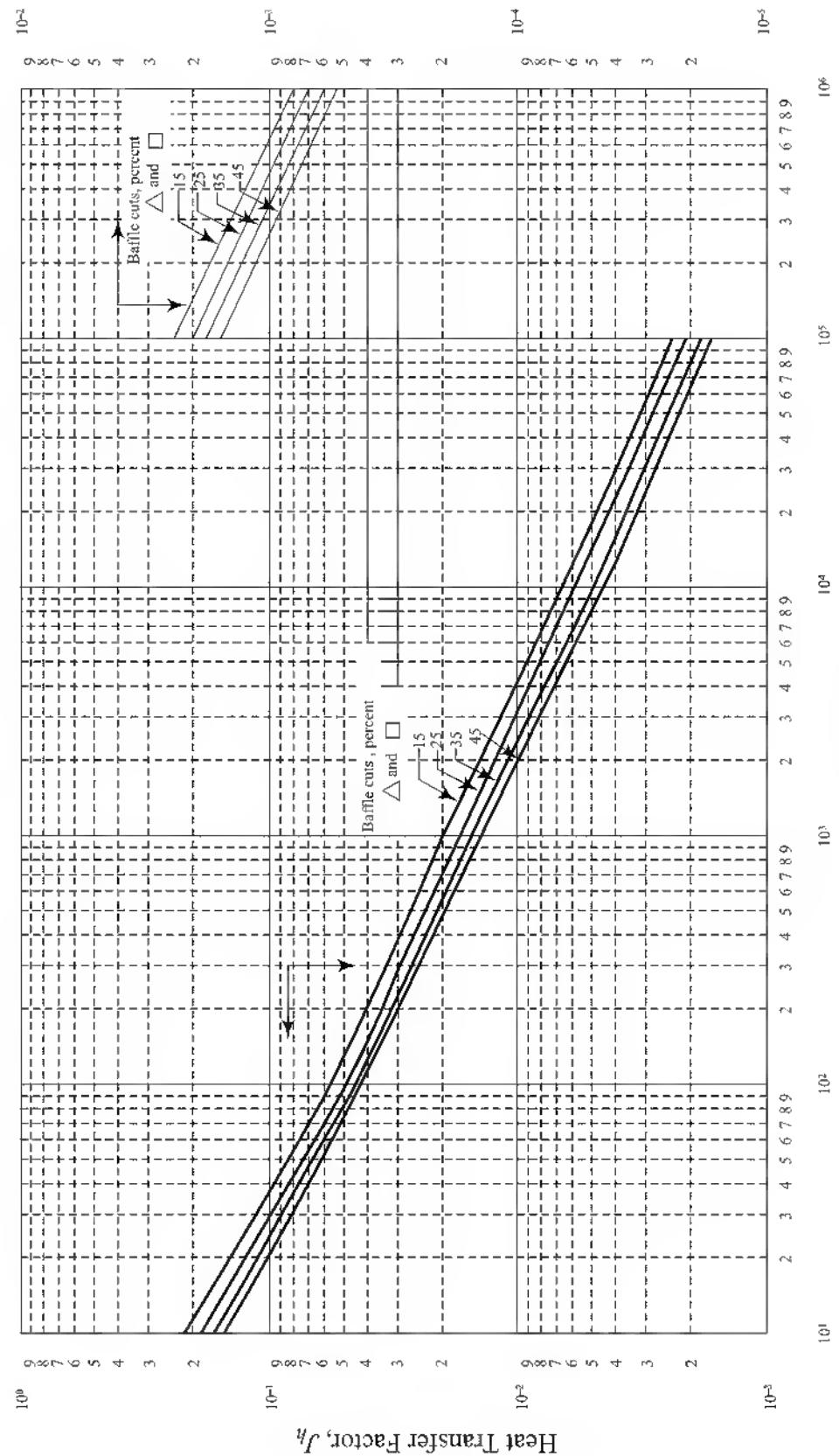


Fig. 6.14 Shell Side Heat-transfer Factors for Segmental Baffles⁴
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(B) If condensation takes place on shell side then its coefficient depends on the position of condenser.

Shell side condensation coefficient for horizontal position can be determined by following modified Nusselt equation.

$$h_{CO} = 0.95 k_L \left[\frac{\rho_L(\rho_L - \rho_v) g}{\mu_L \tau_h} \right]^{1/3} N_r^{-1/6} \quad (6.37)$$

where, h_{CO} = Shell side condensation coefficient, $\text{W}/(\text{m}^2 \cdot {}^\circ\text{C})$

k_L = Thermal conductivity of liquid condensate, $\text{W}/(\text{m} \cdot {}^\circ\text{C})$

ρ_L = Density of liquid condensate, kg/m^3

ρ_v = Vapour density, kg/m^3

g = Acceleration of gravity = 9.81 m/s^2

μ_L = Viscosity of liquid condensate, $\text{N} \cdot \text{s}/\text{m}^2$

τ_h = Horizontal tube loading or flow of condensate per unit length of tube

$$= \frac{W_C}{LN_t}$$

where, W_C = Total condensate flow, kg/s

L = Tube length, m

N_t = Number of tubes

N_r = Average number of tubes in vertical tube row

$$N_r \equiv \frac{2}{3} N'_r \quad (6.38)$$

N'_r = Number of tubes in central row

$$N'_r = \frac{D_b}{P_t} \quad (6.39)$$

D_b = Tube bundle diameter, m

P_t = Tube pitch, m

For condensation outside the vertical tube bundle, shell side condensation coefficient is given by Nusselt equation.

$$h_{CO} = 0.926 k_L \left(\frac{\rho_L(\rho_L - \rho_v) g}{\mu_L \tau_v} \right)^{1/3} \quad (6.26a)$$

$$\text{where, } \tau_v = \frac{W_C}{N_t \pi d_o} \quad (6.26b)$$

6.3.11 Calculation of Shell Side Pressure Drop

(a) For no phase change on shell side or for heating or cooling on shell side, shell side pressure drop Δp_s can be calculated by following equation.

$$\Delta p_s = 8 J_f \left(\frac{D_s}{d_e} \right) \left(\frac{L}{B_s} \right) \frac{\rho_s u_s^2}{2} \left(\frac{\mu}{\mu_w} \right)^{-0.14} \quad (6.40)$$

where, Δp_s = Shell side pressure drop, N/m² or Pa
 D_s = Shell inside diameter, m
 d_e = Shell side equivalent diameter, m
 L = Tube length, m
 B_s = Baffle spacing, m
 ρ_s = Density of shell fluid kg/m³
 u_s = Shell side linear velocity, m/s
 μ = Viscosity of fluid at the bulk fluid temperature. (N · s)/m
 μ_w = Viscosity of fluid at tube wall temperature, (N · s)/m
 J_f = Shell side friction factor for the different values of % baffle cut of segmental baffle and for the different values of Re , it can be obtained from Fig. (6.15).

Shell side pressure drop Δp_s should be less than maximum allowable pressure drop. Maximum allowable pressure drop for shell side is also decided either based on process requirement or based on economic considerations.

Table 6.8 can be used to judge optimum pressure drop.

Table 6.8 Optimum Pressure Drop (Maximum Allowable Pressure Drop) Based on Economic Considerations (Ref. 4)

Fluid	Optimum, Δp_t or Δp_s , kPa
Liquids of $\mu_L < 1$ cP	35
Liquids of $\mu_L = 1$ to 10 cP	50 to 70
Gas or vapour at 1 to 2 atm	13.8
Gas or vapour at high vacuum (up to 60 torr absolute pressure)	0.4 to 0.8 (3 to 6 torr)
Gas or vapour at high pressure > 10 atm	0.1 times operating pressure

(b) In case of shell side condensation no reliable correlation is available for predicting Δp_s . Hence, Δp_s can be approximately calculated by following equation.

$$\Delta p_s = 0.5 \Delta p'_s \quad (6.41)$$

where $\Delta p'_s$ = shell side pressure drop calculated by Eq. (6.40) based on inlet vapour flow rate and inlet conditions.

This also should be less than maximum allowable pressure drop.

6.3.12 Calculation of the Overall Heat Transfer Coefficients by Following Equation

$$U_o = \frac{1}{\frac{1}{h_o} + \frac{1}{h_{od}} + \frac{d_o \ln(d_o/d_i)}{2k_w} + \frac{d_o}{d_i} \times \frac{1}{h_{id}} + \frac{d_o}{d_i} \times \frac{1}{h_i}} \quad (6.42)$$

where, U_o = Overall heat transfer coefficient based on outside area of tubes, W/(m² · °C)

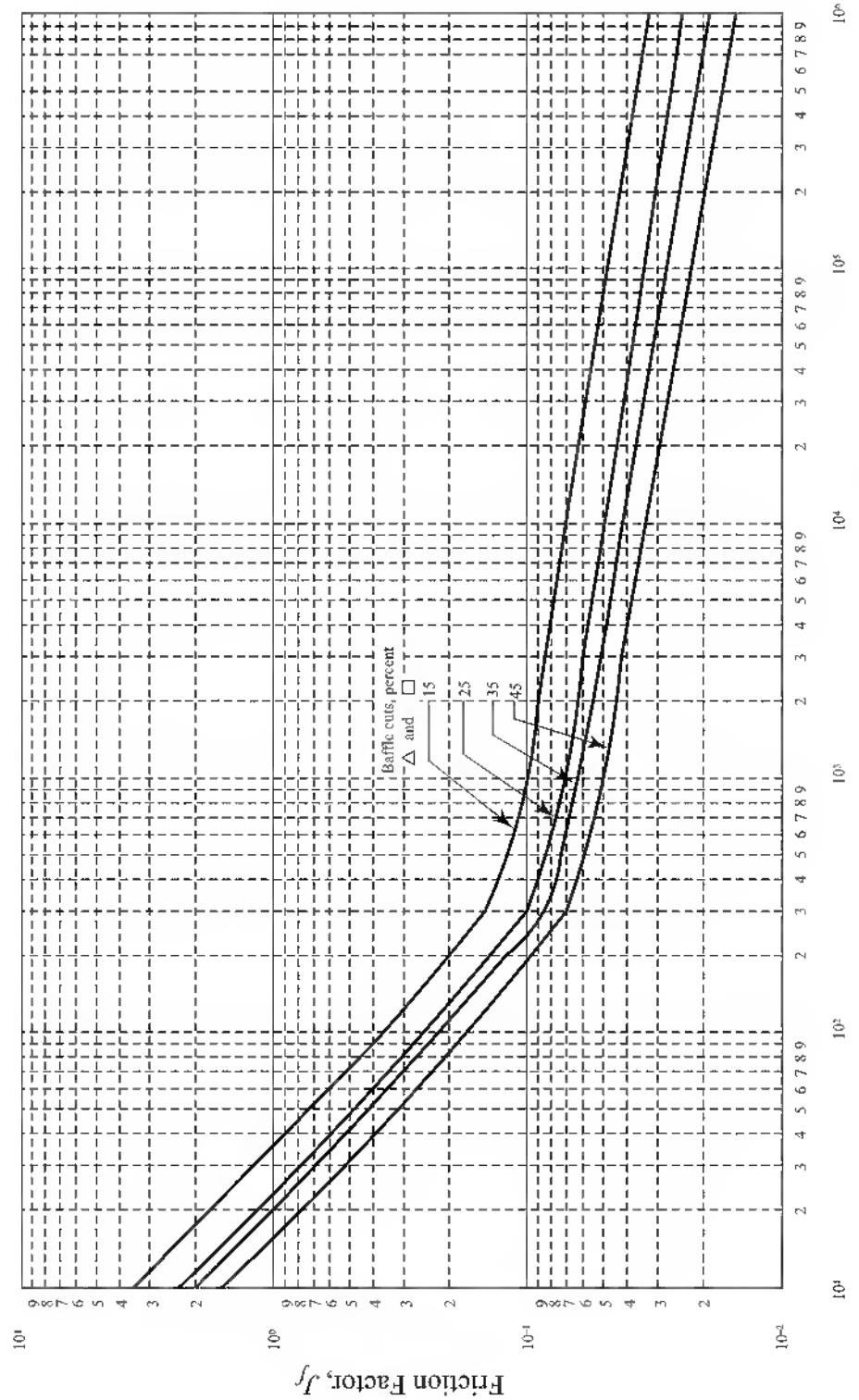


Fig. 6.15 Shell Side Friction Factors for Segmental Baffles⁴
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h_o = Shell side heat transfer coefficient, W/(m² · °C)

d_o = OD of tube, m

d_i = ID of tube, m

k_w = Thermal conductivity of tube wall material, W/(m · °C)

h_{id} = Fouling coefficient for tube side fluid, W/(m² · °C)

h_{od} = Fouling coefficient for shell side fluid, W/(m² · °C)

For the various types of fluids, recommended fouling coefficient are given in Table 6.9.

Table 6.9 Recommended Fouling Coefficients (Ref. 4, 6 & 7)

Fluid	Fouling Coefficient W/(m ² · °C)	Fluid	Fouling Coefficient W/(m ² · °C)
Cooling water	3000–6000	Aqueous Organic Solution	5000
Steam	4000–10 000	Vegetable oils	3000
Steam condensate	1500–5000	Caustic Solutions	5000
Brine	3000–5000	Gasoline	5000
Air	5000–10 000	Kerosene	3000–5000
Organic vapours	10 000	Light gas oil	3000–5000
Organic liquids	5000	Heavy gas oil	2000–3000
Heavy hydrocarbons	2000	Naphtha	5000
Aqueous salt solutions	3000–5000	Coal flue gas	1000
Light Hydrocarbon	5000		
Heavy Hydrocarbon	3000		

6.3.13 Calculating the Heat Transfer Area Required

$$A_r = \frac{\phi_t}{U_o \Delta T_m} \quad (6.43)$$

Also calculate actual heat transfer area A_o .

$$A_o = N_t \pi d_o L$$

$$\frac{A_o}{A_{\text{req}}} \in \{1.1 \text{ to } 1.2\}$$

$$\frac{\Delta p_t}{\Delta p_{t \text{ max}}} \in \{0.8 \text{ to } 1\}, \frac{\Delta p_s}{\Delta p_{s \text{ max}}} \in \{0.8 \text{ to } 1\}$$

Thus an optimum design of shell and tube heat exchanger can be obtained.

Example 6.1

A lube-oil cooler for 2 MW steam turbine is to be designed for following operating conditions. Lube-oil, conforming to ISO VG-46 grade, flows at the rate of 450 L/min and is to be cooled from 65°C to 45°C with cooling water.

Kinematic viscosity of lube oil = 45.5 cSt at 40°C.

Density of lube oil = 869 kg/m³

Specific heat of lube oil = 2.1413 kJ/(kg · °C)

Thermal conductivity of lube oil at 55°C = 0.13 W/(m · °C)

Viscosity of lube oil at 55°C = 15 cP

Cooling water inlet temperature = 35°C

Cooling water outlet temperature = 39°C

Solution:

$$\text{Heat duty, } \phi_t = \dot{m}_o C_{Lo} \Delta t$$

$$\text{Volumetric flow rate of oil} = 450 \text{ L/min} = 450 \times 60 \times 10^{-3} \text{ m}^3/\text{h} = 27 \text{ m}^3/\text{h}$$

$$\dot{m} = 27 \times 869 = 23463 \text{ kg/h}$$

$$\phi_t = 23463 \text{ kg/h} \times 2.1413(65 - 45)$$

$$\phi_t = 1004826 \text{ kJ/h} \equiv 279.118 \text{ kW}$$

Heat balance

$$\phi_t = \dot{m}_w C_{Lw} \Delta t$$

$$279.118 = \dot{m}_w \times 4.1868(39 - 35)$$

$$\dot{m}_w = 16.667 \text{ kg/s} (\equiv 60 \text{ m}^3/\text{h})$$

$$\Delta T_1 = 65 - 39 = 26^\circ\text{C}$$

$$\Delta T_2 = 45 - 35 = 10^\circ\text{C}$$

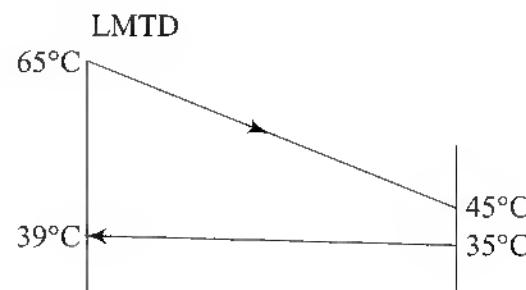


Fig. 6.16

$$\Delta T_{ln} = \frac{\Delta T_1 - \Delta T_2}{\ln\left(\frac{\Delta T_1}{\Delta T_2}\right)} = \frac{26 - 10}{\ln\left(\frac{26}{10}\right)} = 16.745^\circ\text{C} \quad (6.12)$$

For the first trial calculations, let the number of tube side passes $N_p = 4$

Allocating lube oil on tube side and cooling water on shell side.

$$R = \frac{T_1 - T_2}{t_2 - t_1}, \quad S = \frac{t_2 - t_1}{T_1 - T_2}$$

$$T_1 = 65^\circ\text{C}, T_2 = 45^\circ\text{C}, \quad t_1 = 35^\circ\text{C}, t_2 = 39^\circ\text{C}$$

$$R = \frac{65 - 45}{39 - 35} = 5, \quad S = \frac{39 - 35}{65 - 35} = 0.1333$$

From Fig. 6.11, $F_t = 0.95$

Mean temperature difference, $\Delta T_m = F_t \Delta T_{ln}$

$$\Delta T_m = 0.95 \times 16.745 = 15.908^\circ\text{C}$$

Assume overall heat transfer coefficient $U_o = 400 \text{ W}/(\text{m}^2 \cdot ^\circ\text{C})$. (Ref.: Table 6.7)

Provisional heat transfer area for the first trial calculation.

$$A = \frac{\phi_t}{U_o \Delta T_m} = \frac{279.118 \times 10^3}{400 \times 15.908}$$

$$A = 43.8644 \text{ m}^2 = N_t \pi d_o L$$

$$d_o = 5/8 \text{ in.} = 15.875 \text{ mm}, L = 6 \text{ ft} = 1.8288 \text{ m}$$

$$N_t = \frac{43.8644}{\pi \times (0.015875 \times 1.8288)} = 480.93 \approx 480$$

Select BEM type heat exchanger with tube pitch $P_t = 1.25 d_o = 19.8437 \text{ mm}$ and triangular pitch arrangement.

$$\text{Tube bundle diameter } D_b = d_o \left(\frac{N_t}{k_l} \right)^{1/n_l} \quad (6.1)$$

From Table 6.2 for $N_p = 4$, $k_1 = 0.175$, $n_1 = 2.285$

$$D_b = 15.875 \left(\frac{480}{0.175} \right)^{1/2.285} = 507.5 \text{ mm}$$

Let clearance between shell ID and $D_b = 12.5 \text{ mm} = 520 \text{ mm}$

Evaluation of h_i :

Tube side flow area:

$$a_t = \frac{480}{4} \times \frac{\pi}{4} d_i^2 \quad (6.20)$$

$d_i = 13.3858 \text{ mm}$ (for 18 BWG tube)

$$a_t = \frac{480}{4} \times \frac{\pi}{4} (0.0133858)^2 = 0.016887 \text{ m}^2$$

Tube side mass velocity $G_t = \frac{\dot{m}}{a_t}$

$$G_t = \frac{\left(23463 \times \frac{1}{3600} \right)}{0.016687} = 390.57 \text{ kg}/(\text{m}^2 \cdot \text{s})$$

Viscosity of oil:

$$\mu = 15 \text{ cP} = 15 \times 10^{-3} \text{ kg}/(\text{m} \cdot \text{s})$$

$$R_{e_t} = \frac{d_i G_t}{\mu} = \frac{0.0133858 \times 390.57}{15 \times 10^{-3}} = 348.54$$

To increase the Reynold number, let the number of tube side passes $N_p = 8$
 $K_1 = 0.0365$, $n_1 = 2.675$ from Table 6.2

$$D_b = 15.875 \left(\frac{480}{0.0365} \right)^{1/2.675} = 550 \text{ mm}$$

Shell ID = $D_b + 12 = 562 \text{ mm}$

Evaluation of h_i :

$$a_t = \frac{480}{8} \times \frac{\pi}{4} (0.0133858)^2 = 8.4436 \times 10^{-3} \text{ m}^2$$

$$G_t = 390.57 \times \frac{8}{4} = 781.14 \text{ kg}/(\text{m}^2 \cdot \text{s})$$

$$u_t = \frac{G_t}{\rho} = \frac{781.14}{869} = 0.9 \text{ m/s}$$

$$Re_s = 348.54 \times 2 = 697$$

$$Pr = \frac{C_p \mu}{K} = \frac{2.1413 \times 15 \times 10^{-3} \times 10^3}{0.13} = 247$$

Using Sieder-Tate equation ($R_{et} < 2000$)

$$\begin{aligned} Nu &= \frac{h_i d_i}{k} = 1.86(Re \cdot Pr \cdot d_i/L)^{0.33} \left(\frac{\mu}{\mu_w} \right)^{0.14} \\ &= 1.86 \left(697 \times 247 \times \frac{0.0133858}{1.8288} \right)^{0.33} \times 1 \\ &= 19.618 > 3.5 \\ h_i &= 19.62 \times \frac{k}{d_i} = \frac{19.618 \times 0.13}{0.0133858} = 190.526 \text{ W}/(\text{m}^2 \cdot ^\circ\text{C}) \quad (\text{too low}) \end{aligned} \quad (6.18)$$

To improve oil side heat transfer coefficient, allocate lube oil on shell side and water on tube side. Increase the tube length from 6 ft to 10 ft for the same value of heat transfer area provided. It will decrease the shell side flow area and will improve the shell side heat transfer coefficient

$$\begin{aligned} L &= 10 \times 0.3048 = 3.048 \text{ m} \\ A_{pro} &= 43.8644 \text{ m}^2 = N_t \pi d_o L = N_t \times \pi \times 0.015875 \times 3.048 \\ N_t &= 288.56 \cong 288 \end{aligned}$$

Let number of tube side passes $N_p = 4$

$$k_1 = 0.175, n_1 = 2.285 \quad (\text{Ref.: Table 6.2})$$

$$D_b = 15.875 \left(\frac{288}{0.175} \right)^{\frac{1}{2.285}} = 406 \text{ mm}$$

Shell ID, $D_i = 406 + 12 = 418 \text{ mm}$

Evaluation of h_i :

$$a_t = \frac{288}{4} \times \frac{\pi}{4} (0.0133858)^2 = 0.01 \text{ m}^2$$

Tube side mass velocity, $G_t = \frac{16.667}{0.01} = 1666.7 \text{ kg}/(\text{m}^2 \cdot \text{s})$

Density of water at 34°C , $\rho = 993.328 \text{ kg}/\text{m}^3$
(Table 3.28 of Ref. 2)

$$u_t = \frac{G_t}{\rho} = \frac{1666.7}{993.328} = 1.678 \text{ m/s}$$

$$Re = \frac{d_i G_t}{\mu}$$

Viscosity of water at 37°C , $\mu = 0.73 \text{ cP} = 0.73 \times 10^{-3} \text{ kg}/(\text{m} \cdot \text{s})$

$$Re = \frac{0.0133858 \times 1666.7}{0.73 \times 10^{-3}} = 30561.8 > 4000$$

Thermal conductivity of water at 37°C , $k = 0.628 \text{ W}/(\text{m} \cdot ^\circ\text{C})$

$$\text{Prandtl number of water, } Pr = \frac{C_p \mu}{K}$$

$$Pr = \frac{4.1868 \times 0.73 \times 10^{-3} \times 10^3}{0.628} = 4.867$$

Using Dittus–Bolter equation

$$Nu = \frac{h_i d_i}{k_f} = 0.023 Re^{0.8} Pr^{0.33} \left(\frac{\mu}{\mu_w} \right)^{0.14} \quad (6.19)$$

$$\begin{aligned} h_i &= 0.023 \times \frac{0.628}{0.0133858} \times (30561.8)^{0.8} \times 4.867^{0.33} \\ &= 7046.6 \text{ W/(m}^2 \cdot ^\circ\text{C}) \end{aligned}$$

Evaluation of oil side heat transfer coefficient (h_o):

Shell side flow area

$$A_s = \frac{(P_t - d_o)}{P_t} \times D_S \times B_S \quad (6.29)$$

$$P_t/d_o = 1.25, D_S = 418 \text{ mm}, B_S = \frac{D_S}{5} = \frac{418}{5} = 83.6 \text{ mm}$$

$$A_s = \frac{1.25 d_o - d_o}{(1.25 d_o)} \times 0.418 \times 0.0836 = 6.989 \times 10^{-3} \text{ m}^2$$

$$\text{Shell side mass velocity, } G_s = \frac{\dot{m}}{A_s} \quad (6.30)$$

$$G_s = \frac{23463 \times \frac{1}{3600}}{6.989 \times 10^{-3}} = 932.537 \text{ kg/m}^2 \cdot \text{s}$$

$$u_s = \frac{G_s}{\rho} = \frac{932.537}{869} = 1.073 \text{ m/s}$$

Shell side equivalent diameter for triangular pitch

$$d_e = \frac{1.1}{d_o} (P_t^2 - 0.907 d_o^2) \quad (6.32)$$

$$\begin{aligned} d_e &= \frac{1.1}{15.875} [19.8437^2 - 0.907 \times (15.875)^2] \\ &= 11.4465 \text{ m} \end{aligned}$$

$$\text{Shell side Reynolds number } Re = \frac{d_e G_s}{\mu}$$

$$Re = \frac{0.0114465 \times 932.537}{15 \times 10^{-3}} = 711.62$$

$$Pr = \frac{C_p \mu}{k} = \frac{2.1413 \times (15 \times 10^{-3}) \times 10^3}{0.13} \\ = 247$$

$Re = 711.62 \Rightarrow J_h = 2 \times 10^{-2}$ (From Fig. 6.14 for 25% baffle cut)

$$N_u = \frac{h_s d_e}{k_f} = J_h Re Pr^{1/3} \left(\frac{\mu}{\mu_w} \right)^{0.14} \quad (6.36)$$

$$h_s = 2 \times 10^{-2} \times \frac{0.13}{0.0114465} \times 711.62 \times (247)^{1/3} \times 1 \\ = 1014.18 \text{ W/(m}^2 \cdot ^\circ\text{C)}$$

Overall heat transfer coefficient

$$\frac{1}{U_o} = \frac{1}{h_o} + \frac{1}{h_{od}} + \frac{d_o \ln \left(\frac{d_o}{d_i} \right)}{2 k_w} + \frac{d_o}{d_i} \frac{1}{h_{id}} + \frac{d_o}{d_i} \times \frac{1}{h_i} \quad (6.42)$$

From Table 6.9

$$h_{od} = 3000 \text{ W/(m}^2 \cdot ^\circ\text{C}), \quad h_{id} = 5000 \text{ W/(m}^2 \cdot ^\circ\text{C)}$$

$$\frac{1}{U_o} = \frac{1}{1014.18} + \frac{1}{3000} + \frac{0.015875 \ln \left(\frac{15.875}{13.3858} \right)}{2 \times 50} + \frac{15.875}{13.3858} \times \frac{1}{5000} \\ + \frac{15.875}{13.3858} \times \frac{1}{7046.6}$$

Thermal conductivity of tube material $k_w = 50 \text{ W/(m} \cdot ^\circ\text{C)}$

Tube material: steel or cupronickel)

$$U_o = 570.8 \text{ W/(m}^2 \cdot ^\circ\text{C)}$$

$$\text{Heat transfer area required, } A_r = \frac{\phi_t}{U_o \Delta T_m} \quad (6.43)$$

$$= \frac{279.118 \times 10^3}{570.8 \times 15.908} = 30.739 \text{ m}^2$$

$$\begin{aligned} \% \text{ Excess heat transfer area} &= \left(\frac{A_{\text{pro}}}{A_r} - 1 \right) \times 100 \\ &= \left(\frac{43.8644}{30.739} - 1 \right) \times 100 = 42.7 \% \text{ (sufficiently high)} \end{aligned}$$

Shell side Pressure Drop:

$$\Delta p_s = 8J_f \left(\frac{D_s}{d_e} \right) \left(\frac{L}{B_s} \right) \frac{\rho_s u_s^2}{2} \left(\frac{\mu}{\mu_w} \right)^{-0.14} \quad (6.40)$$

$$D_s = 0.418 \text{ m}, d_e = 0.0114465 \text{ m}, L = 3.048 \text{ m}$$

$$B_s = 0.0836 \text{ m}, u_s = 1.073 \text{ m/s}, \rho_s = 869 \text{ kg/m}^3$$

J_f = shell side friction factor

For $R_{es} = 711.62$, $J_f = 7.7 \times 10^{-2}$ (From Fig. 6.15)

$$\begin{aligned} \Delta p_s &= 8 \times 7.7 \times 10^{-2} \left(\frac{0.418}{0.0114465} \right) \left(\frac{3.048}{0.0836} \right) \times \frac{869 \times 1.073^2}{2} \\ &= 410281.7 \text{ Pa} \equiv 410.282 \text{ kPa (too high)} \end{aligned}$$

To decrease the shell side pressure drop, baffles spacing can be increased from 83.6 mm to 160 mm.

Revised value of $B_s = 160 \text{ mm} = 0.16 \text{ m}$

$$\text{Shell side flow area, } A_s = 6.989 \times 10^{-3} \times \frac{0.16}{0.0836} = 0.013376 \text{ m}^2$$

$$\text{Shell side mass velocity, } G_s = 932.537 \times \frac{6.989 \times 10^{-3}}{0.013376} = 487.25 \text{ kg/(m}^2 \cdot \text{s)}$$

$$u_s = \frac{G_s}{\rho_s} = \frac{487.25}{869} = 0.56 \text{ m/s}$$

$$Re = \frac{0.0114465 \times 487.25}{15 \times 10^{-3}} = 371.82$$

$$Nu = \frac{h_s d_e}{k_f} = J_h Re Pr^{1/3} \left(\frac{\mu}{\mu_w} \right)^{0.14} \quad (6.36)$$

for $Re = 371.82 \Rightarrow J_h = 2.7 \times 10^{-2}$ (from Fig. 6.14)

$$Nu = \frac{h_s d_e}{k_f} = 2.7 \times 10^{-2} \times 371.82 \times (247)^{1/3} = 62.989$$

$$h_s = 62.989 \times \frac{0.13}{0.0114465} = 715.37 \text{ W/(m}^2 \cdot {^\circ}\text{C)}$$

$$\frac{1}{U_o} = \frac{1}{570.8} - \frac{1}{1014.18} + \frac{1}{715.37}$$

$$U_o = 462.15 \text{ W/(m}^2 \cdot {^\circ}\text{C)}$$

$$A_r = \frac{\phi_t}{U_o \Delta T_m} = \frac{279.118 \times 10^3}{462.15 \times 15.908} = 37.96 \text{ m}^2$$

$$\% \text{ Excess heat transfer area} = \left(\frac{43.8644}{37.96} - 1 \right) \times 100 = 15.55\% \text{ (adequate)}$$

Revised Shell side Pressure Drop:

For $D_s = 0.418 \text{ m}$, $B_s = 0.16 \text{ m}$, $d_e = 0.0114465 \text{ m}$, $L = 3.048 \text{ m}$, $u_s = 0.56 \text{ m/s}$, $\rho_s = 869 \text{ kg/m}^3$

$$\Delta p_s = 8J_f \left(\frac{D_s}{d_e} \right) \left(\frac{L}{B_s} \right) \frac{\rho_s u_s^2}{2} \left(\frac{\mu}{\mu_w} \right)^{-0.14} \quad (6.40)$$

For $Re = 371.82$, $J_f = 9.26 \times 10^2$ (From Fig. 6.15)

$$\Delta p_s = 8 \times 9.26 \times 10^{-2} \left(\frac{0.418}{0.0114465} \right) \left(\frac{3.048}{0.16} \right) \times \frac{869 \times 0.56^2 \times 1}{2}$$

$$\Delta p_s = 70220.7 \text{ Pa} \equiv 70.22 \text{ kPa (acceptable)}$$

Tube side Pressure Drop:

$$\Delta p_t = N_p \left(8 J_f \left(\frac{L}{d_i} \right) \left(\frac{\mu}{\mu_w} \right)^{-m} + 2.5 \right) \times \frac{\rho u_t^2}{2} \quad (6.27)$$

$N_p = 4$, $L = 3.048 \text{ m}$, $d_i = 0.0133858 \text{ m}$, $u_t = 1.678 \text{ m/s}$

$\rho = 993.328 \text{ kg/m}^3$, $\mu/\mu_w \approx 1$

$Re_t = 30561.8$, $J_f = 3.6 \times 10^{-3}$ (From Fig. 6.13)

$$\Delta p_t = 4 \left(8 \times 3.6 \times 10^{-3} \times \left(\frac{3.048}{0.0133858} \right) \times 1 + 2.5 \right) \times \frac{993.328 \times 1.678^2}{2}$$

$$\Delta p_t = 50668 \text{ Pa} \equiv 50.668 \text{ kPa}$$

Comments: In this case viscosity of lube oil is very high, hence it gives very poor Reynolds number. Fluid, which provides poor Reynolds number must be allocated shell side to get the higher heat transfer coefficient. Here also, by changing the allocation of lube oil from tube side to shell side, lube oil side coefficient increased considerably. In any (without phase change) shell and tube heat exchanger for the fixed value of heat transfer area, increase in tube length (correspondingly decrease in number of tubes and shell inside diameter) increases the overall heat transfer coefficient but at the expense of pressure drop or in other words increase in tube length for the fixed value of heat transfer area decreases the fixed cost of heat exchanger but increases its operating cost.

6.4 CRITERIA OF SELECTION BETWEEN HORIZONTAL CONDENSER AND VERTICAL CONDENSER

- (i) For only filmwise condensation, horizontal position gives higher condensation coefficient than vertical position. In filmwise condensation, thickness of condensate film over heat transfer surface decides the value of

condensation coefficient. With horizontal position and shellside condensation, the condensate travels less distance over heat transfer surface before falling down by gravity compared to vertical position. With vertical position condensate travels over the entire tube length before falling down by gravity. Hence, average condensate film thickness obtained with horizontal position is less than the same with vertical position and hence it provides higher condensation coefficient.

- (ii) In case of condensation with subcooling in a shell and tube heat exchanger, condensation coefficient is higher with horizontal position while subcooling coefficient is higher with vertical position. In case of subcooling with horizontal position, small fraction of gravitational force is acting on the pool of condensate and hence condensate is flowing with almost no turbulence. Hence, subcooling with horizontal position is natural convection heat transfer. While in subcooling with vertical position, entire gravitational force is acting on pool of condensate. Hence, it creates turbulence in pool of condensate and provides higher subcooling coefficient. Subcooling with vertical position can be considered as forced convection heat transfer.

Therefore, in the case of condensation with subcooling, shell and tube heat exchanger is designed for both positions (horizontal and vertical) and the position that gives higher value of overall coefficient is selected.

- (iii) In case of condensation with non-condensables, selection of position depends on the % of non-condensables present in inlet vapours for which guidelines are given by Frank⁵.
 - (a) If non-condensables are < 0.5 % (by mass), then the presence of non-condensables is ignored in design calculations. Heat exchanger is designed as a total condenser. For this case, horizontal position should be selected as it provides the higher condensation coefficient.
 - (b) If non condensables are > 70% (by mass), then for the entire flow rate, without phase change correlation is applied to calculate the heat transfer coefficient. However, in calculation of heat duty (ϕ_t), condensation is considered. For this case selection of the position does not depend on heat transfer coefficient, as without phase change coefficient does not depend on position of heat exchanger so in this case position is decided by other factors like available area or available height.
 - (c) Between 0.5 to 70% non-condensables, heat transfer coefficient is determined by considering both condensation as well as cooling of non-condensables and vapours. For this case horizontal position should be selected as it gives higher condensation coefficient, if condensation with cooling is carried out on shell side. If the same is carried out on tube side, then vertical position is better.

Example 6.2

10 900 kg/h of nearly pure saturated methyl ethyl ketone vapour at 13.73 kPa g is to be condensed and cooled to 60°C by cooling water which is available in plant at 32°C. Consider pressure drops of 13.7 kPa for vapour and 68.7 kPa for the water as permissible. Design the suitable shell and tube heat exchanger for the heat duty.

Solution:

Heat duty calculations

$$\phi_t = \dot{m}\lambda + \dot{m}C_L\Delta t = \phi_C + \phi_{\text{sub}}$$

where, $\phi_c = \dot{m}\lambda$ = heat duty for condensation
 $\phi_{\text{sub}} = \dot{m}C_L \Delta T$ = heat duty for subcooling
 $\dot{m} = 10\ 900 \text{ kg/h} = 3.0278 \text{ kg/s}$
 λ = Latent heat of vaporization of methyl ethyl ketone (MEK) at condensation temperature

At condensation temperature of pure MEK vapour

$$p_v = p_t = 13.73 \text{ kPa} \quad g = 115.055 \text{ kPa} \quad a = 863 \text{ torr a}$$

Antoine equation⁴ for methyl ethyl ketone

$$\ln p_v = 16.5986 - \frac{3150.42}{T - 36.65}$$

where p_v is in torr and T in K

$$p_v = p_t = 863 \text{ torr} \quad T = 356.87 \text{ K} = 83.87^\circ\text{C}$$

From Table 3-179 of Ref. 2,

$$\lambda_{\text{MEK}} \text{ at } 78.2^\circ\text{C} = 105.93 \text{ kcal/kg} = 443.5 \text{ kJ/kg}$$

Value of λ_{MEK} is required at 83.87°C .

Using Watson relation,

$$\lambda_2 = \lambda_1 \left(\frac{1 - T_{r2}}{1 - T_{r1}} \right)^{0.38}$$

T_r = reduced temperature = T/T_C

T_C of MEK = 535.6 K

$$\lambda \text{ at } 83.87^\circ\text{C} = \lambda_{78.2^\circ\text{C}} \left(\frac{1 - \frac{(83.87 + 273.15)}{535.6}}{1 - \frac{(78.2 + 273.15)}{535.6}} \right)^{0.38}$$

$$\lambda_{83.87^\circ\text{C}} = 443.5 \times 0.9882 = 438.27 \text{ kJ/kg}$$

$$\phi_c = \dot{m}\lambda = 3.0278 \times 438.27 = 1327 \text{ kW}$$

$$\phi_{\text{sub}} = \dot{m}C_L(83.87 - 60)$$

Specific heat of MEK liquid at $\frac{(83.87 + 60)}{2} = 72^\circ\text{C}$:

$$C_L = 2.298 \text{ kJ/(kg} \cdot ^\circ\text{C}) \text{ (From Table 3-184 of Ref. 2)}$$

$$\phi_{\text{sub}} = 3.0278 \times 2.298 \times (83.87 - 60) = 166 \text{ kW}$$

$$\phi_t = \phi_c + \phi_{\text{sub}} = 1327 + 166 = 1493 \text{ kW}$$

Energy Balance

$$\phi = \dot{m}_w C_{pw} (t_0 - 32)$$

Fixing the outlet temperature of cooling water as 40°C ,
mass flow rate of cooling water required

$$\dot{m}_w = \frac{\phi_t}{(C_{pw} \cdot \Delta t)} = \frac{1493}{(4.1868 \times 8)} = 44.5746 \text{ kg/s}$$

First trial calculations:

Using Table 6.7, let overall heat transfer coefficients be
for condensation $U_c = 800 \text{ W}/(\text{m}^2 \cdot ^\circ\text{C})$
for subcooling $U_{\text{sub}} = 200 \text{ W}/(\text{m}^2 \cdot ^\circ\text{C})$

Assuming that entire flow of cooling water is first utilized for subcooling and then for condensation.

$$\phi_{\text{sub}} = \dot{m}_w C_p w (t' - 32)$$

where t' = intermediate temperature of cooling water

$$166 = 44.5746 \times 4.1868 \times (t' - 32)$$

$$t' = 32.89^\circ\text{C}$$

LMTD for condensation:

$$\Delta T_1' = 83.87 - 32.89 = 50.98^\circ\text{C}$$

$$\Delta T_2' = 83.87 - 40 = 43.87^\circ\text{C}$$

$$\Delta T_{\text{ln}}' = \frac{50.98 - 43.87}{\ln\left(\frac{50.98}{43.87}\right)} = 47.336^\circ\text{C}$$

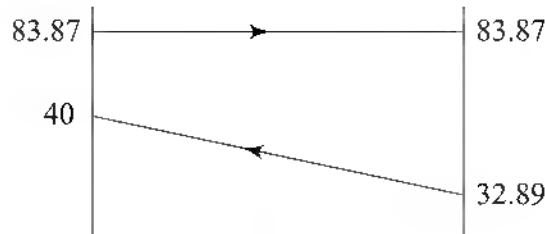


Fig. 6.17(a)

for condensation of pure component $R = 0$ and $F_t = 1$, hence LMTD = MTD

LMTD for subcooling:

$$\Delta T_1' = 83.87 - 32.89 = 50.98^\circ\text{C}$$

$$\Delta T_2' = 60 - 32 = 28^\circ\text{C}$$

$$\Delta T_{\text{ln}}' = \frac{50.98 - 28}{\ln\left(\frac{50.98}{28}\right)} = 38.349^\circ\text{C}$$

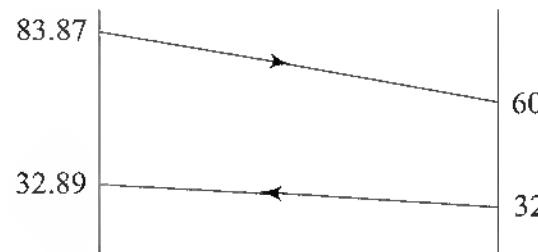


Fig. 6.17(b)

Assuming that subcooling will take place in the perfect counter current manner.

$$F_t = 1, \text{ hence LMTD} = \text{MTD}$$

Area based on assumed values of U :

For condensation:

$$A_c = \frac{\phi_c}{U_c \Delta T_{mc}} = \frac{1327 \times 1000}{800 \times 47.336} = 35.042 \text{ m}^2$$

For subcooling:

$$A_{\text{sub}} = \frac{\phi_{\text{sub}}}{U_{\text{sub}} \Delta T_{m\text{sub}}} = \frac{166 \times 1000}{200 \times 38.349} = 21.643 \text{ m}^2$$

$$\text{Total area } A = A_c + A_{\text{sub}} = 56.685 \text{ m}^2$$

$$\frac{A_{\text{sub}}}{A} = \frac{21.643}{56.685} = 0.3818 \text{ and } A_{\text{sub}} < A_c$$

Based on the selected values of overall coefficients, area required for subcooling (A_{sub}) is less than area required for condensation (A_c). Hence to get the smaller size of heat exchanger horizontal position is selected. Ideally in such a case heat exchanger should be designed for both positions and the position which requires lesser heat transfer area should be selected.

Area provided for the first trial calculation

$$A = 56.685 \text{ m}^2 = N_t \pi d_o L$$

Choose 3/4 on OD (19.05 mm OD), 6 ft (1.83 m) long tubes

$$N_t = \frac{A}{\pi d_o L} = \frac{56.685}{(\pi \times 0.01905 \times 1.83)} = 518 \text{ tubes}$$

Shell side fluid is a clean fluid, hence, select triangular pitch arrangement

$$P_t = 1.25 d_o = 23.8125 \text{ mm}$$

Number of tube side passes = 2 (for 1st trial calculations)

Tube bundle diameter:

$$D_b = d_o \left(\frac{N_t}{k_1} \right)^{\frac{1}{n_1}} \quad (6.1)$$

$$= 19.05 \left(\frac{518}{0.249} \right)^{\frac{1}{2.207}} \quad (\text{Using Table 6.2}) \\ = 607 \text{ mm}$$

Select a fixed tube sheet heat exchanger.

Let clearance between shell ID and D_b = 13 mm

Shell inside diameter = 620 mm

Calculation of tube side heat transfer coefficient:

$$a_t = \frac{N_t}{N_p} \times \frac{\pi}{4} d_i^2 = \frac{518}{2} \times \frac{\pi}{4} (0.015748)^2 = 0.05045 \text{ m}^2$$

(From Table 11.2, of Ref. 2 for 16 BWG tube $d_i = 15.748 \text{ mm}$)

Tube side mass velocity,

$$G_t = \frac{\dot{m}}{a_t} = \frac{44.5746}{0.05045} = 883.54 \text{ kg/(m}^2 \cdot \text{s)}$$

$$u_t = \frac{G_t}{\rho} \\ = \frac{883.54}{992.9} = 0.89 \text{ m/s} < 1 \text{ m/s}$$

To avoid the excessive fouling it is recommended to keep the velocity of water greater than 1 m/s.

Increase the number of tube side passes from 2 to 4.

for $N_p = 4$, $a_t = 0.02522 \text{ m}^2$

$$D_b = 19.05 \left(\frac{518}{0.175} \right)^{\frac{1}{2.285}} \quad (\text{Table 6.2}) \\ = 630 \text{ mm}$$

Let clearance between shell ID and D_b = 15 mm

Shell inside diameter = 645 mm (revised)

$$G_t = \frac{44.5746}{0.02522} = 1767.43 \text{ kg/(m}^2 \cdot \text{s)}$$

$$u_t = 1.78 \text{ m/s}$$

$$Re = \frac{d_i G_t}{\mu}$$

Viscosity of water at 36°C, $\mu = 0.72 \text{ cP} = 0.72 \times 10^{-3} \text{ kg/(m} \cdot \text{s)}$

$$Re = \frac{0.015748 \times 1767.43}{0.72 \times 10^{-3}} = 38657.6$$

Prandtl number

Thermal conductivity of water at 36°C, $k = 0.6228 \text{ W/(m} \cdot \text{C)}$

$$Pr = \frac{C_p \mu}{k} = \frac{4.1868 \times (0.72 \times 10^{-3})}{0.6228} \times \frac{10^3}{1} \\ = 4.84$$

Using Dittus-Bolter equation

$$\frac{h_i d_i}{k} = 0.023 Re^{0.8} Pr^{0.33} \left(\frac{\mu}{\mu_w} \right)^{0.14} \quad (6.19)$$

Neglecting $\left(\frac{\mu}{\mu_w} \right)^{0.14}$

$$h_i = \frac{0.023 \times 0.6228}{0.015748} \times (38657.6)^{0.8} \times (4.84)^{0.33} \\ = 7155.5 \text{ W/(m}^2 \cdot \text{°C})$$

Shell side coefficient:

(a) For condensation zone, h_{co} :

Calculation for mean temperature of condensate film:

Let t_w = Tube wall temperature, °C

$h_{co} = 1500 \text{ W/(m}^2 \cdot \text{°C})$ (assumed for 1st trial calculation)

At steady state

Heat transfer rate through condensate film = overall rate of heat transfer

$$h_{co} A_c (t_c - t_w) = U_c A_c (t_c - t_{av})$$

where, t_c = Condensation temperature of vapour on shell side, °C

t_{av} = Average tube side fluid temperature for condensation zone, °C

$$1500(83.87 - t_w) = 800 \left(83.87 - \frac{(32.89 + 40)}{2} \right)$$

$$t_w = 58.577^\circ\text{C}$$

Mean temperature of condensate film

$$= \frac{t_c + t_w}{2} = \frac{(83.87 + 58.577)}{2} = 71.22^\circ\text{C}$$

Physical properties of liquid condensate at 71.22°C

Viscosity of liquid MEK at 71.22 °C, $\mu_L = 0.32 \text{ cP} = 0.32 \times 10^{-3} \text{ kg/(m} \cdot \text{s)}$

Density of liquid MEK at 71.22 °C, $\rho_L = 805 \text{ kg/m}^3$

Thermal conductivity of liquid MEK at 71.22 °C, $k_L = 0.173 \text{ W/(m} \cdot {^\circ}\text{C)}$

Shell side condensation coefficient with horizontal position

$$h_{co} = 0.95 k_L \left(\frac{\rho_L (\rho_L - \rho_v) g}{\mu_L \tau_h} \right)^{1/3} \cdot N_r^{-1/6} \quad (6.37)$$

Acceleration of gravity $g = 9.81 \text{ m/s}^2$

Density of vapour $\rho_v = \frac{PM}{RT} = \frac{PM}{T} \times \frac{T_s}{P_s V_s}$ (s for standard conditions)

$$\rho_v = \frac{863 \times 72}{(273 + 83.87)} \times \frac{273}{760 \times 22.414}$$

$$\rho_v = 2.97 \text{ kg/m}^3$$

$$\tau_h = \frac{W_c}{LN_t} = \frac{(10900/3600)}{(1.83 \times 518)} = 3.194 \times 10^{-3} \text{ kg/(m} \cdot \text{s)}$$

Average number of tubes in vertical rows

$$N_r = 2/3 N'_r = \frac{2}{3} \left(\frac{D_b}{P_t} \right)$$

$$= \frac{2}{3} \times \frac{630}{23.8125} = 17.64 \cong 18$$

$$h_{co} = 0.95 \times 0.173 \left[\frac{805 \times (805 - 2.79) \times 9.81}{0.32 \times 10^{-3} \times 3.194 \times 10^{-3}} \right]^{1/3} \times (18)^{-1/6}$$

$$= 1864.86 \text{ W/(m}^2 \cdot {^\circ}\text{C)}$$

h_{co} is close enough to assume value of h_{co} . Hence, correction in the value of t_w is not required.

(b) For subcooling zone, h_{osub} :

With horizontal position, subcooling is taking place via natural convection only for which reliable correlations are not available. Based on Kern's recommendation (Ref. 5), h_{osub} for horizontal position.

$$h_{osub} = 50 \text{ Btu/(h} \cdot \text{ft}^2 \cdot {^\circ}\text{F)}$$

$$= 283.77 \text{ W/(m}^2 \cdot {^\circ}\text{C)}$$

Overall Heat Transfer Coefficient for Condensation:

$$U_{oc} = \frac{1}{\frac{1}{h_{oc}} + \frac{1}{h_{od}} + \frac{d_o \ln(d_o/d_i)}{2k_w} + \frac{d_o}{d_i} \left(\frac{1}{h_i} \right) + \frac{d_o}{d_i} \frac{1}{h_{id}}} \quad (6.42)$$

From Table 6.9,

For organic vapours, $h_{od} = 10000 \text{ W/(m}^2 \cdot {^\circ}\text{C)}$

For cooling water, $h_{id} = 4000 \text{ W/(m}^2 \cdot {^\circ}\text{C)}$

With methyl ethyl ketone and cooling water, stainless steel-304, is a suitable material for the tube.

Thermal conductivity of SS-304 material, $k_w = 16.3 \text{ W}/(\text{m} \cdot ^\circ\text{C})$

$$\frac{1}{U_{oc}} = \frac{1}{1864.86} + \frac{1}{10000} + \frac{0.01905 \ln\left(\frac{19.05}{15.748}\right)}{2 \times 16.3} + \frac{19.05}{15.748} \times \frac{1}{7155.5} + \frac{19.05}{15.748} \times \frac{1}{4000}$$

$$U_{oc} = 820.38 \text{ W}/(\text{m}^2 \cdot ^\circ\text{C})$$

Heat transfer area required for condensation

$$A_{cr} = \frac{\phi_{cond}}{U_{oc} \times \Delta T_{mc}} = \frac{1327 \times 1000}{820.38 \times 47.336}$$

$$A_{cr} = 34.17 \text{ m}^2$$

Overall Heat Transfer Coefficient for Subcooling:

$$U_{osub} = \frac{1}{\frac{1}{h_{osub}} + \frac{1}{h_{od}} + \frac{d_o \ln(d_o/d_i)}{2k_w} + \frac{d_o}{d_i} \frac{1}{h_{id}} + \frac{d_o}{d_i} \frac{1}{h_t}} \quad (6.42)$$

$$U_{osub} = \frac{1}{\frac{1}{283.77} + \frac{1}{10000} + \frac{0.01905 \ln(19.05/15.748)}{2 \times 16.3} + \frac{19.05}{15.748} \times \frac{1}{4000} + \frac{19.05}{15.748 \times 7155.5}}$$

$$U_{osub} = 237.72 \text{ W}/(\text{m}^2 \cdot ^\circ\text{C})$$

Heat transfer area required for subcooling

$$A_{subr} = \frac{\phi_{sub}}{U_{osub} \times \Delta T_{msub}} = \frac{166 \times 1000}{237.72 \times 38.349} = 18.21 \text{ m}^2$$

Total heat transfer area required

$$A_{tr} = A_{cr} + A_{subr} = 34.17 + 18.21 = 52.38 \text{ m}^2$$

$$\frac{A_{tpro}}{A_{treq}} = \frac{56.685}{52.38} = 1.082$$

or % Excess heat transfer area = $(1.082 - 1) \times 100 = 8.2\%$

% Excess heat transfer area should be atleast 10%. Hence, to increase the value of heat transfer area, increase the tube length. New value of tube length is

$$L \cong 1.83 \times \frac{1.15}{1.082} = 1.95 \text{ m}$$

Let new or revised tube length $L = 2 \text{ m}$

$$\text{Heat transfer area provided } A_{pro} = \frac{2}{1.83} \times 56.685 = 61.95 \text{ m}^2$$

Revised value of shell side condensation coefficient

$$h_{oc} \propto L^{1/3}$$

$$h_{oc} = \left(\frac{2}{1.83} \right)^{1/3} \times 1864.86 = 1920.9 \text{ W/(m}^2 \cdot ^\circ\text{C)}$$

New value of U_{oc}

$$\frac{1}{U_{oc}} = \frac{1}{820.38} - \frac{1}{1864.86} + \frac{1}{1920.9}$$

$$U_{oc} = 831 \text{ W/(m}^2 \cdot ^\circ\text{C)}$$

$$A_{oc} = \frac{1327 \times 1000}{831 \times 47.336} = 33.73 \text{ m}^2$$

$$A_{sub} = 18.21 \text{ m}^2$$

$$A_{req} = 51.94 \text{ m}^2$$

$$\frac{A_{tpro}}{A_{treq}} = \frac{61.95}{51.94} = 1.1927$$

% Excess heat transfer area = $(1.1927 - 1) \times 100 = 19.27\%$

If the equal percentage of excess heat transfer area (19.27%) in both zones are provided then,

Heat transfer area provided for condensation:

$$A_{cpro} = 33.73 \times 1.1927 = 40.23 \text{ m}^2$$

Heat transfer area provided for subcooling:

$$A_{subpro} = 18.21 \times 1.1927 = 21.72 \text{ m}^2$$

$$\frac{A_{sub}}{A_{tpro}} = \frac{21.72}{61.95} = 0.3506$$

Hence, 35.06% of total heat transfer area should be provided for subcooling.

Assuming that tube will be uniformly distributed in the cross section of shell.

$$\frac{A_{subpro}}{A_{tpro}} = \frac{\text{Area of segment of cross section utilized for subcooling}}{\text{Total cross sectional area of shell}}$$

$$0.3506 = \frac{x D_i^2}{\frac{\pi}{4} D_i^2}$$

$$x = \frac{\pi}{4} \times 0.3506 = 0.2754$$

From Table 1.19b of Ref. 2, for $x = 0.2754$, $h/D_i = 0.382$

Hence, 38.2% of shell inside diameter should be submerged in the pool of condensate to facilitate subcooling.

This can be achieved by providing inverted U-seal (Fig. 6.18) height of inverted U-seal from the base of shell ID

$$h = 0.382 \times 645 = 246.4 \text{ mm}$$

Shell side pressure drop Δp_s ;
Shell side flow area

$$A'_s = \frac{(P_t - d_o) B_s D_s}{P_t} \times x'$$

$$\text{where } x' = 1 - \frac{h}{d_i} = 1 - \frac{246.4}{645} = 0.618$$

Let baffle spacing $B_s = \text{Shell ID} = 645 \text{ mm}$

$$A_s = \frac{(23.8125 - 19.05)}{23.8125} \times 0.645 \times 0.645 \times 0.618 = 0.05142 \text{ m}^2$$

Shell side velocity

$$u_s = \frac{G_s}{\rho_v}$$

$$\text{Shell side mass velocity, } G_s = \frac{\dot{m}}{A_s} = \frac{3.0278}{0.05142}$$

$$G_s = 58.884 \text{ kg/(m}^2 \cdot \text{s)}$$

$$u_s = (G_s/\rho_v) = \frac{58.884}{2.79} = 21.105 \text{ m/s}$$

Equivalent diameter for triangular pitch arrangement

$$d_e = \frac{1.1}{d_o} (P_t^2 - 0.907 d_o^2)$$

$$= \frac{1.1}{19.05} (23.8125^2 - 0.907 \times 19.05^2) = 13.736 \text{ mm}$$

$$Re = \frac{0.013736 \times 58.884}{1100 \times 10^{-7} \times 10^{-1}} = 73530$$

(Viscosity of MEK vapour at $83.87^\circ\text{C} = 1100 \times 10^{-7}$ Poise)

From Fig. 6.15, for 25% cut segmental baffle

Shell side friction factor, $J_f = 0.0358$

$$\Delta p_s = 0.5 \times 8 \times J_f \left(\frac{D_s}{d_e} \right) \left(\frac{L}{B_s} \right) \frac{\rho_v u_s^2}{2} \left(\frac{\mu}{\mu_w} \right)^{-0.14} \quad (6.41)$$

$$= 0.5 \times 8 \times 0.0358 \left(\frac{645}{13.736} \right) \left(\frac{2000}{645} \right) \times \frac{2.79 \times 21.105^2}{2}$$

$$= 129.56 \text{ Pa} \equiv 12.956 \text{ kPa} < \Delta p_{smax} (13.73 \text{ kPa})$$

To decrease the shell side pressure drop, increase the % baffle cut from 25% to 35%,
then for 35% cut segmental baffle

$$J_f = 0.03$$

$$\Delta p_s = \frac{0.03}{0.0358} \times 12.956 = 10.86 \text{ kPa} < \Delta p_{smax} (13.73 \text{ kPa})$$

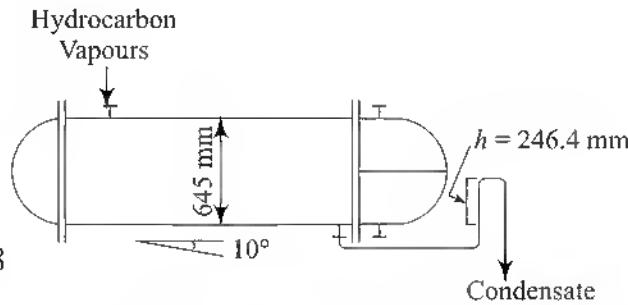


Fig. 6.18 Detail of U-Seal

Tube side pressure drop, Δp_t :

$$\Delta p_t = N_p \left(8J_f \left(\frac{L}{d_i} \right) \left(\frac{\mu}{\mu_w} \right)^{-m} + 2.5 \right) \times \frac{\rho u_t^2}{2} \quad (6.27)$$

$L = 2000$ mm, $N_p = 4$, $d_i = 15.748$ mm, $\rho = 993.684$ kg/m³, $u_t = 1.78$ m/s
 $Re = 38\,657.6$, $J_f = 3.41 \times 10^{-3}$ (from Fig. 6.13)

$$\Delta p_t = 4 \times \left(8 \times 3.41 \times 10^{-3} \times \left(\frac{2000}{15.748} \right) \times 1 + 2.5 \right) \times \frac{993.684 \times 1.78^2}{2}$$

$$\Delta p_t = 37\,558 \text{ Pa} \equiv 37.558 \text{ kPa} < \Delta p_{t\max} (68.6 \text{ kPa})$$

Resulting Heat Exchanger Specifications:

1-4, fixed tube sheet shell and tube heat exchanger (BEM type),

Shell ID = 645 mm, Baffles: 35% cut segmental,

Baffle spacing = 645 mm,

Tube OD = 19.05 mm, 16 BWG, Tube length = 2000 mm,

Δ Pitch arrangement, $P_t = 1.25 d_o$, Number of tubes, $N_t = 518$

6.5 CONDENSATION WITH NON-CONDENSABLES

For the design of this type of heat exchanger, methods available are: (i) Colburn and Hougen method and (ii) Porter and Jaffreys method. But these methods are complex requiring trial and error calculations. These methods consider both resistances; resistance provided by heat transfer and diffusional resistance provided by mass transfer.

Approximate methods are also available for the same. In approximate methods only the resistance, offered by heat transfer, is considered while mass transfer resistance is neglected. Hence in these methods it is assumed that overall rate of heat transfer in condensation with cooling of non-condensables is totally controlled by heat transfer resistance. One of the approximate methods is Gilmore's equation⁵.

$$\frac{1}{h_{cg}} = \frac{1}{h_c} + \frac{\phi_g}{\phi_t} \frac{1}{h_g} \quad (6.44)$$

where, h_{cg} = Heat transfer coefficient for condensation with cooling of non-condensables, W/(m² · °C)

h_c = Average condensate film coefficient, determined by using single component condensation correlations, at the average condensate composition and total condensate loading, W/(m² · °C)

h_g = Average gas film coefficient determined for the average vapour gas mixture flow rate, W/(m² · °C)

ϕ_g = Total sensible heat transfer from vapour-gas mixture, kW

ϕ_t = Total heat transfer, kW

ϕ_t = Latent heat of vaporization + sensible heat for cooling the gas-vapour mixture.

For non-condensables, present in vapour, guidelines given by Frank (Sec. 6.4) may be followed.

Example 6.3

Air saturated with *n*-butanol vapour at 49 kPa g pressure and at 100°C temperature is to be sent to shell and tube heat exchanger for recovering butanol. Air-butanol vapour mixture is to be cooled to 50°C by cooling water which enters at 32°C and leaves the heat exchanger at 40°C.

Air flow rate (butanol free) = 500 Nm³/h

Check that whether the shell and tube heat exchanger with following specifications is suitable for the given duty earlier.

- (i) Type of heat exchanger: BEM as per TEMA
- (ii) Material of Construction of heat exchanger: SS-304
- (iii) Shell ID = 387 mm
- (iv) Tube OD = 19.05 mm
- (v) Tube ID = 15.748 mm
- (vi) Tube length = 1500 mm
- (vii) Tube Pitch = 25.4 mm
- (viii) Type of tube arrangement = Triangular
- (ix) Number of tubes = 134
- (x) Number of tube side passes = 4
- (xi) Number of shell side passes = one
- (xii) Baffle type = 25% cut segmental
- (xiii) Baffle spacing = 120 mm

Properties of fluids and material:

- (a) Thermal conductivity of SS-304, $k_w = 16 \text{ W}/(\text{m} \cdot ^\circ\text{C})$
- (b) Antoine equation⁴ for butanol

$$\ln p_v = 17.2160 - \frac{3137.02}{T - 94.43} \quad \text{where } p_v \text{ is in torr and } T \text{ is in K.}$$

- (c) Latent heat of vaporization of butanol at 75°C, = 642 kJ/kg
- (d) Physical properties of butanol condensate at average condensate film temperature

Specific heat, $C_L = 2.8763 \text{ kJ}/(\text{kg} \cdot ^\circ\text{C})$

Viscosity, $\mu_L = 0.95 \text{ cP} = 0.95 \text{ mPa} \cdot \text{s}$

Thermal conductivity, $k_L = 0.168 \text{ W}/(\text{m} \cdot ^\circ\text{C})$

Density, $\rho_L = 810 \text{ kg/m}^3$

- (e) Physical properties of air-butanol vapour mixture of average composition at 75°C and at the operating pressure:

Specific heat, $C_p = 1.3043 \text{ kJ}/(\text{kg} \cdot ^\circ\text{C})$

Viscosity, $\mu = 0.0174 \text{ cP or mPa} \cdot \text{s}$

Thermal conductivity, $k = 0.03 \text{ W}/(\text{m} \cdot \text{K})$

Density, $\rho = 2.012 \text{ kg/m}^3$

- (f) Physical properties of cooling water

$C_{Lw} = 4.1868 \text{ kJ}/(\text{kg} \cdot ^\circ\text{C})$

$\mu_w = 0.72 \text{ cP}$

$k_w = 0.63 \text{ W}/(\text{m} \cdot \text{K})$

$\rho_w = 1000 \text{ kg/m}^3$

- (g) Fouling coefficients $h_{od} = h_{id} = 5000 \text{ W}/(\text{m}^2 \cdot ^\circ\text{C})$

Solution:

Heat duty equation for the given case

$$\phi_t = \dot{m}_a C_{pa} \Delta t + \dot{m}_v C_{pv} \Delta T + \dot{m}_{BC} \lambda = (\dot{m}_a + \dot{m}_v) C_{pav} \Delta t + \dot{m}_{BC} \lambda$$

where, ϕ_t = Heat duty, kW
 \dot{m}_a = Mass flow rate of air, kg/s
 \dot{m}_v = Average mass flow rate of butanol vapour, kg/s
 \dot{m}_{BC} = Condensation rate of butanol, kg/s
 C_{pa} = Specific heat of air, kJ/(kg · °C)
 C_{pv} = Specific heat of butanol vapour, kJ/(kg · °C)
 λ = Latent heat of vaporization of butanol, kJ/kg
 C_{pav} = Specific heat of Air-butanol vapour mixture, kJ/(kg · °C)

Density of air at normal condition

$$\rho_{\text{air}} = \frac{pM}{RT} = \frac{pM}{T} \times \frac{T_s}{p_s V_s} \quad (s \text{ for standard conditions})$$

$$\rho_{\text{air}} = \frac{1 \times 29}{(273 + 25)} \times \frac{273}{1 \times 22.414} = 1.1853 \text{ kg/m}^3$$

$$\dot{m}_a = 500 \times 1.1853 = 592.65 \text{ kg/h} \equiv 0.1646 \text{ kg/s}$$

\dot{m}_v = Average flow rate of butanol vapour

$$\dot{m}_v = \frac{\dot{m}_{vi} + \dot{m}_{vo}}{2}$$

where, \dot{m}_{vi} = Mass flow rate of butanol vapour at inlet, kg/s

\dot{m}_{vo} = Mass flow rate of Butanol vapour at outlet, kg/s

At inlet, air is saturated with butanol vapour at 100°C temperature and at 49 kPa g pressure.

Vapour pressure of butanol at 100°C = $p_v = \bar{p}_v$

where \bar{p}_v = partial pressure of butanol vapour at inlet

$$\text{At } 100^\circ\text{C } \ln p_v = 17.216 - \frac{3137.02}{T - 94.43}$$

for $T = 100 + 273 = 373 \text{ K}$, $p_v = 385.617 \text{ torr}$

Mole fraction of butanol vapour in incoming air-butanol mixture:

$$y_i = \frac{\bar{p}_v}{p_t} = \frac{385.617}{p_t}$$

$$p_t = 49 \text{ kPa g} = 49 + 101.325 = 150.325 \text{ kPa a} \equiv 1127.53 \text{ torr a}$$

$$y_i = \frac{385.617}{1127.53} = 0.342$$

$$\dot{m}_{vi} = y_i \times \left(\frac{\dot{n}_a}{1 - y_i} \right) \times \text{molar mass of butanol (C}_4\text{H}_9\text{OH)}$$

(\dot{n}_a = molar flow rate of air)

$$= 0.342 \times \left(\frac{\left(\frac{0.1646}{29} \right)}{1 - 0.342} \right) \times 74 = 0.2183 \text{ kg/s}$$

$$\dot{m}_{vo} = y_o \left(\frac{\dot{m}_a}{1 - y_o} \right) \times \text{molar mass of butanol}$$

y_o = Mole fraction of butanol vapour in air-butanol vapour mixture at outlet

$$= \frac{\bar{p}'_v}{p'_t} = \frac{p'_v}{p'_t}$$

At the outlet also air is saturated with butanol vapour.

$$\bar{p}'_v = p'_v = \text{vapour pressure of butanol at } 50^\circ\text{C temperature}$$

$$\text{For } T = 273 + 50 = 323 \text{ K, } p'_v = 32.8336 \text{ torr}$$

$$p'_t = \text{Operating pressure of air - Vapour mixture of butanol at outlet} = p_t - \Delta p_s$$

where, Δp_s = Shell side pressure drop.

$$\text{Assume shell side pressure drop } \Delta p_s = 13.8 \text{ kPa (2 psi)}$$

$$p'_t = 150.325 - 13.8 = 136.525 \text{ kPa a} \equiv 1024 \text{ torr a}$$

$$y_o = \frac{32.8336}{1024} = 0.03206$$

$$\dot{m}_{vo} = 0.03206 \left(\frac{\left(\frac{0.1646}{29} \right)}{1 - 0.03206} \right) \times 74 = 0.0139 \text{ kg/s}$$

$$\dot{m}_v = \left(\frac{0.2183 + 0.0139}{2} \right) = 0.1161 \text{ kg/s}$$

$$\dot{m}_{BC} = \text{Condensation rate of butanol} = \dot{m}_{vi} - \dot{m}_{vo}$$

$$= 0.2183 - 0.0139 = 0.2044 \text{ kg/s}$$

$$\phi_t = (0.1646 + 0.1161) \times 1.3043 (100 - 50) + 0.2044 \times 642$$

$$= 149.53 \text{ kW}$$

Heat balance:

$$\phi_t = \dot{m}_w C_{pw} \Delta t$$

$$149.53 = \dot{m}_w \times 4.1868 \times (40 - 32)$$

$$\dot{m}_w = 4.4643 \text{ kg/s} = 16.07 \text{ m}^3/\text{h}$$

$$MTD: \Delta \mathcal{T}_m = F_t \times \Delta \mathcal{T}_{ln} \quad \text{Eq. (6.11)}$$

$$\Delta \mathcal{T}_{ln}$$

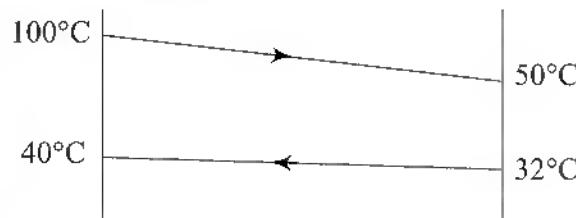


Fig. 6.19

$$\Delta \mathcal{T}_{ln} = \frac{\Delta \mathcal{T}_1 - \Delta \mathcal{T}_2}{\ln \left(\frac{\Delta \mathcal{T}_1}{\Delta \mathcal{T}_2} \right)}, \Delta \mathcal{T}_1 = 100 - 40 = 60^\circ\text{C}, \Delta \mathcal{T}_2 = 50 - 32 = 18^\circ\text{C}$$

$$\Delta \mathcal{T}_{ln} = \frac{60 - 18}{\ln \left(\frac{60}{18} \right)} = 34.88^\circ\text{C}$$

F_t = LMTD correction factor

$$R = \frac{\mathcal{T}_1 - \mathcal{T}_2}{t_2 - t_1} \quad \text{and} \quad S = \frac{t_2 - t_1}{\mathcal{T}_1 - \mathcal{T}_2}$$

$$R = \frac{100 - 50}{40 - 32} = \frac{50}{8} = 6.25, S = \frac{40 - 32}{100 - 32} = \frac{8}{68} = 0.1176$$

From Fig. 6.11 for 1–4 shell and side tube heat exchanger,

$$F_t = 0.96$$

$$\Delta T_m = 0.96 \times 34.88^\circ\text{C} = 33.485^\circ\text{C}$$

Tube side heat transfer coefficient, h_i :

$$\text{Tube side flow area, } a_t = \frac{N_t}{N_p} \times \frac{\pi}{4} d_i^2 \quad (6.20)$$

$$= \frac{134}{4} \times \frac{\pi}{4} (0.015748)^2 = 6.525 \times 10^{-3} \text{ m}^2$$

$$\begin{aligned} \text{Tube side mass velocity, } G_t &= \frac{\dot{m}_w}{a_t} = \frac{4.4643}{6.525 \times 10^{-3}} \\ &= 648.2 \text{ kg/(m}^2 \cdot \text{s)} \end{aligned}$$

$$\text{Tube side linear velocity } u_t = \frac{G_t}{p} = \frac{684.2}{1000} = 0.6842 \text{ m/s}$$

$$\begin{aligned} Re &= \frac{d_i G_t}{\mu} = \frac{0.015748 \times 684.2}{0.72 \times 10^{-3}} \\ &= 14965 \end{aligned}$$

$$\begin{aligned} Pr &= \frac{C_p \mu}{k} = \frac{4.1868 \times (0.72 \times 10^{-3}) \times 10^3}{0.63} \\ &= 4.7849 \end{aligned}$$

$$\frac{h_i d_i}{k} = 0.023 Re^{0.8} Pr^{0.33} \left(\frac{\mu}{\mu_w} \right)^{0.14} \quad (6.19)$$

$$h_i = 0.023 \times \frac{0.63}{0.015748} \times (14965)^{0.8} \times (4.7849)^{0.33} \times 1$$

$$h_i = 3374.9 \text{ W/(m}^2 \cdot {^\circ}\text{C)}$$

Shell side heat transfer coefficient, h_o :

$$\frac{1}{h_o} = \frac{1}{h_{cg}} = \frac{1}{h_c} + \frac{\phi_g}{\phi_t} \frac{1}{h_g} \quad (6.44)$$

Mean condensate film coefficient, h_c :

For horizontal position, modified Nusselt's equation is applicable

$$h_c = 0.95 k_L \left(\frac{\rho_L (\rho_L - \rho_v) g}{\mu_L \tau_h} \right)^{1/3} \times N_r^{-1/6} \quad (6.37)$$

$$\tau_h = \frac{W_c}{LN_t} = \frac{\dot{m}_{BC}}{LN_t} = \frac{0.2044}{1.5 \times 134} = 1.017 \times 10^{-3} \text{ kg/(m} \cdot \text{s)}$$

$$N_r = 2/3N'_r, N'_r = \frac{D_s}{P_t} = \frac{387}{25.4} \equiv 15$$

$$N_r = 10$$

Density of vapour, $\rho_v = \frac{pM}{RT} = \left(\frac{\left(\frac{p_t + p_t'}{2} \right) M}{RT} \right)$

$$= \frac{\left(\frac{150.325 + 136.525}{2} \right) \times 74}{(273 + 75)} \times \frac{273}{101.325 \times 22.414}$$

$$\rho_v = 3.666 \text{ kg/m}^3$$

$$h_c = 0.95 \times 0.168 \left(\frac{810(810 - 3.666) \times 9.81}{0.95 \times 10^{-3} \times 1.017 \times 10^{-3}} \right)^{1/3} \times (10)^{-1/6}$$

$$h_c = 2042.9 \text{ W/(m}^2 \cdot ^\circ\text{C})$$

Average gas film coefficient, h_g :

$$\begin{aligned} \text{Shell side flow area, } A_s &= \frac{(P_t - d_o) B_s D_s}{P_t} \\ &= \left(\frac{25.4 - 19.05}{25.4} \right) \times 0.12 \times 0.387 = 0.01161 \text{ m}^2 \end{aligned} \quad (6.29)$$

Equivalent diameter for triangular pitch arrangement

$$\begin{aligned} d_e &= \frac{1.1}{d_o} (P_t^2 - 0.907 d_o^2) = \frac{1.1}{0.01905} (0.0254^2 - 0.907 \times 0.01905^2) \\ &= 0.018247 \text{ m} \end{aligned}$$

Shell side mass velocity G_s :

$$G_s = \frac{\dot{m}_s}{A_s} = \frac{(0.1646 + 0.1161)}{0.01161} = 24.18 \text{ kg/(m}^2 \cdot \text{s)}$$

$$\begin{aligned} \text{Reynolds number, } Re &= \frac{d_e G_s}{\mu} = \frac{0.018247 \times 24.18}{0.0174 \times 10^{-3}} \\ &= 25357 \end{aligned}$$

$$\begin{aligned} Pr &= \frac{c_p \mu}{k} = \frac{1.3043 \times 0.0174 \times 10^{-3} \times 10^3}{0.03} \\ &= 0.7565 \end{aligned}$$

Gas film coefficient can be determined by following correlation.

$$h_g \frac{d_e}{k} = 0.36 Re^{0.55} Pr^{1/3} \left(\frac{\mu}{\mu_w} \right)^{0.14} \quad (6.35)$$

$$h_g = 0.36 \times \frac{0.03}{0.018247} \times (25357)^{0.55} \times (0.7565)^{1/3} \times 1 \quad \left(\frac{\mu}{\mu_w} \approx 1 \right)$$

$$= 142.59 \text{ W/(m}^2 \cdot \text{s})$$

$$\frac{1}{h_o} = \frac{1}{h_g} + \frac{\phi_g}{\phi_t} \frac{1}{h_g} = \frac{1}{2042.9} + \frac{\phi_g}{\phi_t} \times \frac{1}{142.59} \quad (6.44)$$

where, $\phi_g = (\dot{m}_a + \dot{m}_v) Cp_{av} \Delta t = (0.1646 + 0.1161) \times 1.3043 (100 - 50) = 18.306 \text{ kW}$
 $\phi_t = 149.53 \text{ kW}$

$$\frac{1}{h_o} = \frac{1}{2042.9} + \frac{18.306}{149.53} \times \frac{1}{142.59}$$

$$h_o = 741.8 \text{ W/(m}^2 \cdot {^\circ}\text{C})$$

Overall heat transfer coefficient U.

$$\frac{1}{U_o} = \frac{1}{h_o} + \frac{1}{h_{od}} + \frac{d_o \ln(d_o/d_i)}{2k_m} + \frac{d_o}{d_i} \frac{1}{h_i} + \frac{d_o}{d_i} \frac{1}{h_{id}} \quad (6.42)$$

Thermal conductivity of tube material (SS-304), $k_m = 16 \text{ W/(m} \cdot {^\circ}\text{C})$

$$\frac{1}{U_o} = \frac{1}{741.8} + \frac{1}{5000} + \frac{0.01905 \times \ln\left(\frac{19.05}{15.748}\right)}{2 \times 16} + \frac{19.05}{15.748} \times \frac{1}{3374.9}$$

$$+ \frac{19.05}{15.748} \times \frac{1}{5000}$$

$$U_o = 442.13 \text{ W/(m}^2 \cdot {^\circ}\text{C})$$

Heat transfer area required, A_{or} :

$$A_{or} = \frac{\phi_t}{U_o \Delta T_m} = \frac{149.53 \times 10^3}{442.13 \times 33.485}$$

$$= 10.1 \text{ m}^2$$

Heat transfer area provided,

$$A_{opro} = N_t \pi d_o L = 134 \times \pi \times 0.01905 \times 1.5 = 12.0293 \text{ m}^2$$

$$\% \text{ Excess heat transfer area} = \left(\frac{A_{opro}}{A_{or}} - 1 \right) \times 100 = \left(\frac{12.0293}{10.1} - 1 \right) \times 100 = 19.1\%$$

Hence heat transfer area provided by heat exchanger is sufficient for the given duty.
 Shell side pressure drop Δp_s :

$$\Delta p_s = 8J_f \left(\frac{D_s}{d_e} \right) \left(\frac{L}{B_s} \right) \frac{\rho_s u_s^2}{2} \left(\frac{\mu}{\mu_w} \right)^{-0.14} \quad (6.40)$$

$$Re = 253\ 57, J_f = 4.2 \times 10^{-2} \quad (\text{From Fig. 6.15})$$

$$L = 1500 \text{ mm}, B_s = 120 \text{ mm}, D_s = 387 \text{ mm}, d_e = 18.247 \text{ mm}$$

ρ_s = Density of shell side fluid (air – vapour mixture)

$$\rho_s = \frac{pM_{av}}{RT}$$

$$\text{Average mole fraction of butanol vapour, } y_{av} = \frac{y_i + y_o}{2}$$

$$y_{av} = \frac{0.342 + 0.032\ 06}{2} = 0.187\ 03$$

$$M_{av} = y_{av} \times \text{molar mass of butanol} + (1 - y_{av}) \times \text{molar mass of air}$$

$$M_{av} = 0.187\ 03 \times 74 + (1 - 0.187\ 03) \times 29 = 37.4163 \text{ kg/kmol}$$

$$\rho_s = \frac{pM_{av}}{RT} = \frac{\left(\frac{150.325 + 136.525}{2} \right) \times 37.4163}{(273 + 75)} \times \frac{273}{101.325 \times 22.414}$$

$$\rho_s = 1.8537 \text{ kg/m}^3$$

u_s = shell side linear velocity

$$= \frac{G_s}{\rho_s} = \frac{24.18}{1.8537} = 13.04 \text{ m/s}$$

$$\Delta p_s = 8 \times 4.2 \times 10^{-2} \left(\frac{387}{18.247} \right) \left(\frac{1500}{120} \right) \times \frac{1.8537 \times 13.04^2}{2} \times 1 \\ = 14\ 039 \text{ Pa} \equiv 14.04 \text{ kPa}$$

Calculated shell side pressure drop ($\Delta p_s = 14.04 \text{ kPa}$) is very close to optimum pressure drop ($\Delta P_{opt} = 13.8 \text{ kPa}$). Hence it is satisfactory.

Tube side pressure drop, Δp_t :

$$\Delta p_t = N_p \left(8J_f \left(\frac{L}{d_i} \right) \left(\frac{\mu}{\mu_w} \right)^{-m} + 2.5 \right) \frac{\rho u_t^2}{2} \quad (6.27)$$

$$N_p = 4, L = 1500 \text{ mm}, d_i = 15.748 \text{ mm}, 1000 \text{ kg/m}^3, u_t = 0.6842 \text{ m/s}$$

for $Re = 14\ 965$ from Fig. 6.13 $J_f = 4.3 \times 10^{-3}$

$$\Delta p_t = 4 \left(8 \times 4.3 \times 10^{-3} \left(\frac{1500}{15.748} \right) \times 1 + 2.5 \right) \times \frac{1000 \times 0.6842^2}{2}$$

$$\Delta p_t = 5408.4 \text{ Pa} \equiv 5.408 \text{ kPa} < \Delta p_{t\ max} (68 \text{ kPa})$$

Hence, tube side pressure drop is very much less than maximum or optimum pressure drop.

Considering % excess heat transfer area, Δp_s and Δp_t , given shell and tube heat exchanger is suitable for the required heat transfer duty. If the given shell and tube heat exchanger is at design stage (not fabricated), then number of tube side passes could be increased from 4 to 8 because of the following reasons:

- (i) Cooling water has a tendency to foul. To avoid the excessive fouling it is better to keep the velocity of cooling water more than 1 m/s, preferably near 2 m/s.
- (ii) Increase in tube side passes will increase the tube side heat transfer coefficient and overall coefficient. Consequently, it will decrease the required heat transfer area and hence the fixed cost. However, it will increase the operating cost for pumping the tube side fluid. Also in the given case, overall heat transfer coefficient is controlled by shell side heat transfer coefficient hence increase in number of tube side passes has little effect on the value of overall heat transfer coefficient.

6.6 MULTICOMPONENT CONDENSATION

In condensation of pure component (Example: condensation of pure ethanol vapour), condensation is taking place in isothermal manner or at constant temperature (degree of freedom = 1). While in case of multicomponent condensation, during the condensation, temperature decreases from dew point to bubble point temperature, except in the following cases.

- (a) Condensation of the vapour mixture of two immiscible components (immiscible in liquid phases) Example: Vapour mixture of toluene and water vapour.
- (b) Condensation of the azeotropic vapour mixture.
For example, condensation of vapour mixture containing 89.4% (by mole) ethanol vapour and 10.6% (by mole) steam at 1 atm pressure.

In condensation of vapour mixture of miscible components (miscible in liquid phase), condensation starts from dew point temperature and terminates at bubble point temperature. For the calculations of dew point, bubble point and compositions of vapour phase and liquid phase in between dew point and bubble point, vapour liquid equilibrium data at operating pressure for the given system is required.

For finding the vapour-liquid equilibrium data from dew point to bubble point at operating pressure, there are three options.

- (i) Find the VLE data by conducting the actual experiment. Particularly if the operating pressure is atmospheric or vacuum, then determination of VLE data via actual experiment is easier.
- (ii) Find the VLE data from literature.
- (iii) Find the VLE data by theoretical equation.

$$y_i = K_i x_i \quad (6.45)$$

where, y_i = Mole fraction of component i in vapour phase.

x_i = Mole fraction of component i in liquid phase

K_i = Equilibrium constant for component i ,

K_i is a function of operating pressure, temperature as well as of composition.

$$\text{For an ideal solution (i.e. the solution following Raoult's law), } K_i = \frac{p_{vi}}{p_t} = f(T, p) \quad (6.46)$$

where, p_{vi} = Vapour pressure of pure component i at the operating temperature

p_t = Operating pressure

For non-ideal vapour liquid equilibrium

$$K_i = \frac{p_{vi} \gamma_i}{p_t \theta_i} = f(T, p) \quad (6.47)$$

where γ_i = Activity coefficient of component i in liquid phase,

ϕ_i = Fugacity coefficient of component i in vapour phase.

If solution can be considered as ideal solution then $\gamma_i = 1$. (Example: For the liquid mixture of benzene and toluene $\gamma_{is} = 1$). If vapour mixture can be considered as ideal gas mixture then $\theta_i = 1$ (Example: Vapour mixture of benzene and hexane at 1 atm pressure)

In the design of multi-component condenser calculations, weighted temperature difference is important instead of mean temperature difference ($MTD = LMTD \times F_D$).

To calculate weighted temperature difference, entire condensing range is divided in different small intervals. The weighted temperature difference is calculated by following equation.

$$\text{Weighted } \Delta t = \frac{\sum \phi}{\sum \frac{\phi}{\Delta t_{av}}} \quad (6.48)$$

where, ϕ = Heat load for an interval

Δt_{av} = Average temperature difference for the interval

In condensing multicomponent mixture from dew point to bubble point, vapour and condensate are also cooled by sensible heat transfer. Vapour is cooled simultaneously during condensation but cooling of condensate must be facilitated by providing the inverted U seal or by using a dam baffle (Fig. 6.2(f)). Cooling of condensate formed at or near the dew point heat the condensate at or near the bubble point in condensate collecting vessel and regenerate the vapour. Hence it is necessary to cool the condensate simultaneously with vapour mixture from the dew point to bubble point in the condenser to avoid the regeneration of vapour from condensate and for the cooling of condensate certain number of tubes must be submerged in the pool of condensate which is assured by using inverted U seal or dam baffle.

Design of multicomponent condenser is illustrated by the following example.

Example 6.4

Overhead vapour flows at the rate of 500 kmol/h from a distillation column at 4 atm a. It contains saturated hydrocarbons and has the composition; 30% $n\text{-C}_3\text{H}_8$, 25% $n\text{-C}_4\text{H}_{10}$, 20% $n\text{-C}_5\text{H}_{12}$ and 25% $n\text{-C}_6\text{H}_{14}$ (mole basis). It is to be condensed in a condenser. Permissible pressure drop in the condenser is 13.8 kPa.

- (a) Determine the condensing range.
- (b) Compute the condensing curve.
- (c) Compute the weighted average temperature difference and
- (d) Design and specify a suitable condenser.

Solution:

Table 6.10 Composition of Overhead Vapour

Component	mole % ($z_i \times 100$)	kmol/h	Molar mass	kg/h	mass %
$n\text{-C}_3\text{H}_8$	30	150	44	6600	20.75
$n\text{-C}_4\text{H}_{10}$	25	125	58	7250	22.80
$n\text{-C}_5\text{H}_{12}$	20	100	72	7200	22.64
$n\text{-C}_6\text{H}_{14}$	25	125	86	10750	33.81
Total	100	500		31800	100.00

Solution:

(a) Condensing range: Condensation starts from dew point and terminates at bubble point.

Dew point:

$$\text{At dew point, } \sum \frac{y_i}{K_i} = 1 \text{ and } z_i = y_i \quad (6.49)$$

Value of equilibrium constants K_i for hydrocarbons can be obtained from Fig. 6.20.

This calculation requires trial and error.

Assume, $t = 81^\circ\text{C}$ and $p = 4 \text{ atm}$ a ,

$$K_{C_3H_8} = 5.7, \quad K_{C_4H_{10}} = 2.3, \quad K_{C_5H_{12}} = 0.89, \quad K_{C_6H_{14}} = 0.4$$

$$\sum \frac{y_i}{k_i} = \frac{0.3}{5.7} + \frac{0.25}{2.3} + \frac{0.2}{0.89} + \frac{0.25}{0.4} \cong 1.01 \cong 1$$

Dew point temperature of vapour mixture = 81°C at 4 atm a pressure

$$\text{At bubble point, } \sum K_i x_i = 1, \text{ where } x_i = z_i \quad (6.50)$$

Assume $t = 31^\circ\text{C}$

$$K_{C_3H_8} = 2.5, K_{C_4H_{10}} = 0.74, K_{C_5H_{12}} = 0.24, K_{C_6H_{14}} = 0.085$$

$$\sum K_i x_i = (0.3 \times 2.5 + 0.25 \times 0.74 + 0.20 \times 0.24 + 0.25 \times 0.085) = 1.00$$

Bubble point of vapour mixture = 31°C

Therefore, the condensation range is from 81°C to 31°C

(b) Condensing curve:

Divide the condensation range in five intervals; say 81°C to 70°C , 70°C to 60°C , 60°C to 50°C , 50°C to 40°C and 40°C to 31°C .

Equilibrium constant K_i values (From Fig. 6.20):

Table 6.11 Equilibrium Constants K_i Values (from Fig. 6.20)

Component	K_i					
	81°C	70°C	60°C	50°C	40°C	31°C
C_3H_8	5.7	4.9	4.2	3.6	3.0	2.5
C_4H_{10}	2.3	1.75	1.5	1.2	0.95	0.74
C_5H_{14}	0.89	0.7	0.56	0.42	0.32	0.24
C_6H_{14}	0.4	0.3	0.23	0.17	0.12	0.085

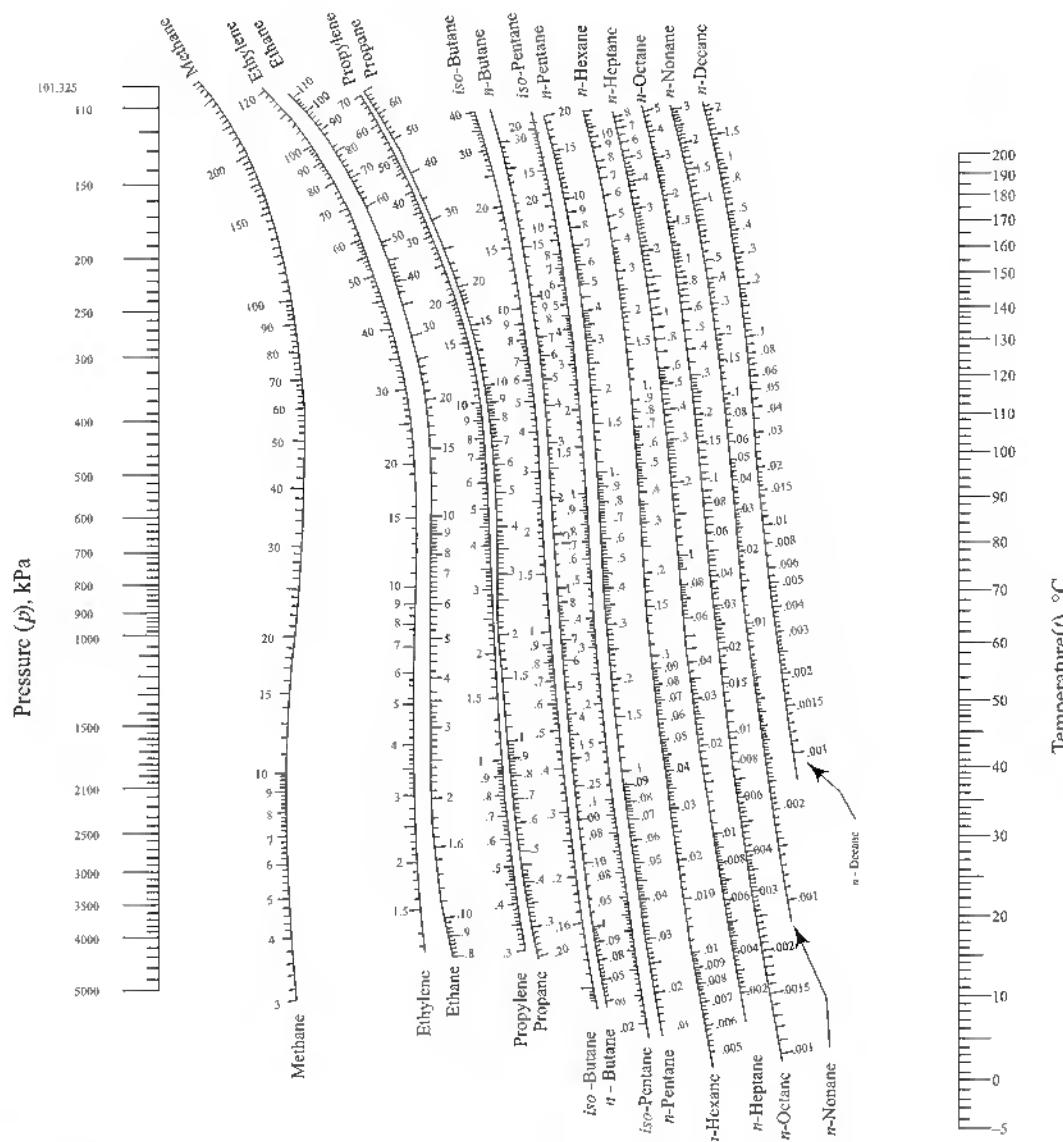


Fig. 6.20 K_i Values for Hydrocarbons at High Temperatures⁸
(Reproduced with the Permission of the American Institute of Chemical Engineers,
USA.)

Let F = Total molar flow rate of saturated vapour mixture entering to condenser
= 500 kmol/h.

First interval:

Let, L_1 = Total moles of condensate at the exist of 1st interval (at 70°C)

V_1 = Total moles of vapour at the exist of 1st interval

$$F = L_1 + V_1$$

Let C_1 = Moles of condensate formed in the interval, for first interval $C_1 = L_1$

$$\text{Component balance } F z_i = L_1 x_i + V_1 y_i \quad (6.51)$$

$$L_1 x_i = \frac{F z_i}{1 + \frac{V_1}{L_1} K_i (70^\circ\text{C})} \quad (6.52)$$

Assume the value of V_1/L_1 and find the value of $L_1 x_i$ by Eq. (6.52). Then $\sum L_1 x_i = L_1$, $F - L_1 = V_1$, $V_1/L_1 = ?$ Value of V_1/L_1 obtained at the end of calculations should be same as the assumed one. So it requires trial and error calculations.

Table 6.12 Flash Calculations for Range 81°C to 70°C

Component	Fz_i	$K_i (= 70^\circ\text{C})$	$L_1 x_i$	$C_1 x_i$	$\lambda_i \text{ kJ/kg}$	$C_{mpi} \text{ kJ/(kmol} \cdot \text{K)}$	$C_{Li} \text{ kJ/(kg} \cdot \text{K)}$
C_3H_8	150	4.90	10.917	10.917	230.27	84.253	3.12
C_4H_{10}	125	1.75	22.522	22.522	301.450	111.08	2.70
C_5H_{12}	100	0.70	35.460	35.460	303.540	137.08	2.51
C_6H_{14}	125	0.30	70.225	70.225	309.820	163.17	2.43
Total	500		139.124	139.124			

Assume $\frac{V_1}{L_1} = 2.6$

$$\frac{150}{1 + 2.6 \times 4.9} + \frac{125}{1 + 2.6 \times 1.75} + \frac{100}{1 + 2.6 \times 0.7} + \frac{125}{1 + 2.6 \times 0.3} = 139.124$$

$$L_1 = 139.124 \text{ kmol/h}$$

$$V_1 = F - L_1 = 500 - 139.124 = 360.876 \text{ kmol/h}$$

$$\frac{V_1}{L_1} = \frac{360.876}{139.124} = 2.5939$$

$$\text{difference} = 2.6 - 2.5939 = 0.006 < 0.01$$

$$\frac{V_1}{L_1} = 2.6$$

Values of λ_i at average temperature (75.5°C)

These are obtained from Fig. 3.9 of Ref. 2. Specific heat of vapour are determined by using following equation.

$$\text{C}_3\text{H}_8 : C_{mpi} = -5.338 + 310.24 \times 10^{-3} T - 164.64 \times 10^{-6} T^2 + 34.691 \times 10^{-9} T^3$$

$$\text{C}_4\text{H}_{10} : C_{mpi} = -1.779 + 386.96 \times 10^{-3} T - 193.25 \times 10^{-6} T^2 + 34.833 \times 10^{-9} T^3$$

$$\text{C}_5\text{H}_{12} : C_{mpi} = -3.411 + 485.01 \times 10^{-3} T - 251.94 \times 10^{-6} T^2 + 48.677 \times 10^{-9} T^3$$

$$\text{C}_6\text{H}_{14} : C_{mpi} = -4.738 + 582.41 \times 10^{-3} T - 310.64 \times 10^{-6} T^2 + 62.923 \times 10^{-9} T^3$$

where C_{mpi} are in $\text{kJ/(kmol} \cdot \text{K)}$ and T is in K (Table 5.1 of Ref. 9)

Specific heat of liquids are obtained from Fig. 4 of Ref. 5 at average temperature (75.5°C).

Values of C_{mp} and C_L are at 1 bar a in ideal state. However, these are not significantly affected under pressure upto 10 bar.

For the interval 81°C to 70°C

$$\begin{aligned} \phi_1 &= \sum C_1 x_i \lambda_i + \sum \left(\frac{Fz_i + V_1 y_i}{2} \right) C_{mpi} \Delta t + \sum \left(\frac{0 + L_1 x_i}{2} \right) C_{Li} \Delta t \\ \phi_1 &= (10.917 \times 230.27 \times 44 + 22.522 \times 301.45 \times 58 + 35.46 \times 303.54 \times 72 \\ &\quad + 70.225 \times 309.82 \times 86) + \left(\frac{150 + (150 - 10.917)}{2} \right) \times 84.253 \times 11 \end{aligned}$$

$$\begin{aligned}
& + \left(\frac{125 + (125 - 22.522)}{2} \right) \times 111.08 \times 11 \\
& + \left(\frac{100 + (100 - 35.46)}{2} \right) \times 137.08 \times 11 \\
& + \left(\frac{125 + (125 - 70.225)}{2} \right) \times 163.17 \times 11 + \frac{10.917}{2} (3.12 \times 44) \times 11 \\
& + \frac{22.522}{2} \times (2.7 \times 58) \times 11 + \frac{35.46}{2} (2.51 \times 72) \times 11 \\
& + \frac{70.225}{2} \times (2.43 \times 86) \times 11
\end{aligned}$$

$$\phi_1 = 3150\ 472.1 + 558\ 323.68 + 143\ 602.7$$

$$\phi_1 = 3852\ 398.5 \text{ kJ/h} \equiv 1070.1 \text{ kW}$$

Second interval:

Table 6.13 Flash Calculations for Range 70°C to 60°C

Component	$V_1 y_i (= Fz_i - L_1 x_i)$	$K_i (= 60^\circ\text{C})$	$L_2 x_i$	$V_2 y_i (= Fz_i - L_2 x_i)$	$C_2 x'_i$
C_3H_8	139.083	4.2	24.834	125.166	13.917
C_4H_{10}	102.478	1.5	44.643	80.357	22.121
C_5H_{12}	64.540	0.56	59.810	40.190	24.350
C_6H_{14}	54.775	0.23	97.962	27.038	27.737
Total	360.876		227.25	272.75	88.125

For the second interval let L_2 is total moles of condensate leaving at 60°C and C_2 is moles of condensate formed in second interval.

$$L_2 = \sum L_2 x_i = \sum \left(\frac{Fz_i}{1 + \frac{V_2}{L_2} K_i (= 60^\circ\text{C})} \right)$$

where, V_2 = Total moles of vapour at 60°C, leaving the second interval

$$L_2 = \sum L_2 x_i = \frac{150}{1 + \frac{V_2}{L_2} \times 4.2} + \frac{125}{1 + \frac{V_2}{L_2} \times 1.5} + \frac{100}{1 + \frac{V_2}{L_2} \times 0.56} + \frac{125}{1 + \frac{V_2}{L_2} \times 0.23}$$

$$\text{Assume } \frac{V_2}{L_2} = 1.2$$

$$L_2 = \sum L_2 x_i = 24.834 + 44.643 + 59.81 + 97.962 = 227.25 \text{ kmol/h}$$

$$F = V_2 + L_2$$

$$V_2 = F - L_2 = 500 - 227.25 = 272.75 \text{ kmol/h}$$

$$\frac{V_2}{L_2} = \frac{272.75}{227.25} = 1.2 \quad (\text{check})$$

$$V_2 y_i = F z_i - L_2 x_i, C_2 x'_i = V_1 y_i - V_2 y_i$$

Table 6.14 Properties at 65°C

Component	λ_i , kJ/mol	C_{mpi} , kJ/(kmol · K)	C_{Li} , kJ/(kmol · K)
C_3H_8	11 605.81	82.053	132.64
C_4H_{10}	18 212.58	108.28	150.56
C_5H_{12}	22 608.72	133.62	177.86
C_6H_{14}	28 445.12	159.06	205.24

For the interval 70 to 60°C

$$\begin{aligned}\phi_2 &= \sum C_2 x'_i \lambda_i + \sum \left(\frac{V_1 y_i + V_2 y_i}{2} \right) C_{mpi} \Delta t + \sum \left(\frac{L_1 x_i + L_2 x_i}{2} \right) C_{Li} \Delta t \\ \phi_2 &= (13.917 \times 11 605.81 + 22.121 \times 18 212.58 + 24.35 \times 22 608.72 \\ &\quad + 27.737 \times 28 445.12) \\ &\quad + \left(\frac{139.083 + 125.166}{2} \right) \times 82.053 \times 10 + \left(\frac{102.478 + 80.357}{2} \right) \times 108.28 \times 10 \\ &\quad + \left(\frac{64.54 + 40.19}{2} \right) \times 133.62 \times 10 + \left(\frac{54.775 + 27.038}{2} \right) \times 159.06 \times 10 \\ &\quad + \left(\frac{10.917 + 24.834}{2} \right) \times 132.64 \times 10 + \left(\frac{22.522 + 44.643}{2} \right) \times 150.56 \times 10 \\ &\quad + \left(\frac{35.46 + 59.81}{2} \right) \times 177.86 \times 10 + \left(\frac{70.225 + 97.962}{2} \right) \times 205.24 \times 10 \\ \phi_2 &= 1903 903.2 + 342 435 + 331 589 \\ \phi_2 &= 2577 927.2 \text{ kJ/h} \equiv 716.1 \text{ kW}\end{aligned}$$

Table 6.15 Flash Calculations for Range 60°C to 50°C

Component	$V_2 y_i$	$K_i (= 50^\circ\text{C})$	$L_2 x_i$	$L_3 x_i$	$V_3 y_i$	$C_3 x'_i$
C_3H_8	125.166	3.60	24.834	48.5751	101.425	23.741
C_4H_{10}	80.357	1.20	44.643	73.7028	51.297	29.060
C_5H_{12}	40.190	0.42	59.810	80.4117	19.588	20.600
C_6H_{14}	27.038	0.17	97.962	113.7812	11.220	15.820
Total	272.750		227.250	316.4708	183.530	89.220

Value of $L_3 x_i$, $V_3 y_i$ and $C_3 x'_i$ of above table are obtained via following calculation.

L_3 is total mole of condensate at 50°C.

$$L_3 = \sum L_3 x_i = \sum \left(\frac{F_{Z_i}}{1 + \frac{V_3}{L_3} k_{i(=50^\circ\text{C})}} \right)$$

$$L_3 = \sum L_3 x_i = \frac{150}{1 + \frac{V_3}{L_3} \times 3.6} + \frac{125}{1 + \frac{V_3}{L_3} \times 1.2} + \frac{100}{1 + \frac{V_3}{L_3} \times 0.42} + \frac{125}{1 + \frac{V_3}{L_3} \times 0.17}$$

Trial: Assume $\frac{V_3}{L_3} = 0.58$

$$L_3 = \sum L_3 x_i = \frac{150}{1 + 0.58 \times 3.6} + \frac{125}{1 + 0.58 \times 1.2} + \frac{100}{1 + 0.58 \times 0.42} + \frac{125}{1 + 0.58 \times 0.17}$$

$$L_3 = \sum L_3 x_i = 48.5751 + 73.7028 + 80.4117 + 113.7812 = 316.4708 \text{ kmol/h}$$

$$V_3 = 500 - 316.4708 = 183.5292 \text{ kmol/h}$$

$$\frac{V_3}{L_3} = 0.5799 \approx 0.58 \text{ (Check)}$$

$$C_3 x'_i = (L_3 x_i)_3 - (L_2 x_i)_2 = (V_2 y_i)_2 - (V_3 y_i)_3$$

Table 6.16 Properties at 55°C

Component	λ_i , kJ/kmol	C_{mpi} , kJ/(kmol · K)	C_{Li} , kJ/(kmol · K)
C_3H_8	12 895.3	79.932	128.95
C_4H_{10}	19 183.9	105.58	148.13
C_5H_{12}	23 513.1	150.29	173.33
C_6H_{14}	28 805.2	155.09	201.64

$$\phi_3 = \sum C_3 x'_i \lambda_i + \sum \left(\frac{V_2 y_i + V_3 y_i}{2} \right) C_{mpi} \Delta t + \sum \left(\frac{L_2 x_i + L_3 x_i}{2} \right) C_{Li} \Delta t$$

$$\phi_3 = (23.741 \times 12 895.3 + 29.06 \times 19 183.9 + 20.6 \times 23 513.1 + 15.82 \times 28 805.2)$$

$$+ \left(\frac{125.166 + 101.425}{2} \right) \times 79.932 \times 10 + \left(\frac{80.357 + 51.297}{2} \right) \times 105.58 \times 10$$

$$+ \left(\frac{40.19 + 19.588}{2} \right) \times 150.29 \times 10 + \left(\frac{27.038 + 11.22}{2} \right) \times 155.09 \times 10$$

$$+ \left(\frac{24.834 + 48.575}{2} \right) \times 128.95 \times 10 + \left(\frac{44.643 + 73.7028}{2} \right) \times 148.13 \times 10$$

$$+ \left(\frac{59.81 + 80.4117}{2} \right) \times 173.33 \times 10 + \left(\frac{97.962 + 113.7812}{2} \right) \times 201.64 \times 10$$

$$\phi_3 = 1803 699.6 + 234 646.85 + 469 986 = 2508 332.5 \text{ kJ/h} \equiv 696.76 \text{ kW}$$

Fourth interval:

Table 6.17 Flash Calculation for Range 50°C to 40°C

Component	V ₃ y _i	K _{i(=40°C)}	L ₃ x _i	L ₄ x _i	V ₄ y _i	C ₄ x' _i
C ₃ H ₈	101.425	3.00	48.5751	85.714	64.286	37.14
C ₄ H ₁₀	51.297	0.95	73.7028	101.01	23.990	27.31
C ₅ H ₁₂	19.588	0.32	80.4117	92.593	7.407	12.18
C ₆ H ₁₄	11.220	0.12	113.7812	121.36	3.640	7.58
Total	183.53		316.4708	400.677	99.323	84.21

Value of L₄x_i, V₄y_i and C₄x'_i of above table are obtained by following calculations.

$$L_4 = \sum L_4 x_i = \sum \left(\frac{F z_i}{1 + \frac{V_4}{L_4} K_{i(=40^\circ\text{C})}} \right)$$

$$\therefore L_4 = \sum L_4 x_i = \frac{150}{1 + \frac{V_4}{L_4} \times 3} + \frac{125}{1 + \frac{V_4}{L_4} \times 0.95} + \frac{100}{1 + \frac{V_4}{L_4} \times 0.32} + \frac{125}{1 + \frac{V_4}{L_4} \times 0.12}$$

Trial: Assume $\frac{V_4}{L_4} = 0.25$

$$L_4 = 85.714 + 101.01 + 92.593 + 121.36 = 400.677 \text{ kmol/h}$$

$$V_4 = 99.323, \frac{V_4}{L_4} = 0.248 \approx 0.25$$

$$V_4 y_i = F z_i - L_4 x_i, C_4 x'_i = (L_4 x_i)_4 - (L_3 x_i)_3$$

$$\phi_4 = \sum C_4 x'_i \lambda_i + \sum \left(\frac{V_3 y_i + V_4 y_i}{2} \right) C_{mp,i} \Delta t + \sum \left(\frac{L_3 x_i + L_4 x_i}{2} \right) C_{Li} \Delta t$$

Table 6.18 Properties at 45°C

Component	λ_p kJ/kmol	$C_{mp,i}$ kJ/(kmol · K)	C_{Li} kJ/(kmol · K)
C ₃ H ₈	13 448.0	77.785	125.27
C ₄ H ₁₀	19 912.4	102.850	143.27
C ₅ H ₁₂	24 116.0	126.910	168.81
C ₆ H ₁₄	29 525.3	151.440	198.04

$$\phi_4 = \sum C_4 x'_i \lambda_i + \sum \left(\frac{V_3 y_i + V_4 y_i}{2} \right) C_{mp,i} \Delta t + \sum \left(\frac{L_3 x_i + L_4 x_i}{2} \right) C_{Li} \Delta t$$

$$\phi_4 = (37.14 \times 13 448 + 27.31 \times 19 912.4 + 12.18 \times 24 116 + 7.58 \times 29 525.3)$$

$$+ \left(\frac{101.425 + 64.286}{2} \right) \times 77.785 \times 10$$

$$\begin{aligned}
& + \left(\frac{51.297 + 23.99}{2} \right) \times 102.85 \times 10 + \left(\frac{19.588 + 7.407}{2} \right) \times 126.91 \times 10 \\
& + \left(\frac{11.22 + 3.64}{2} \right) \times 151.44 \times 10 + \left(\frac{48.5751 + 85.714}{2} \right) \times 125.27 \times 10 \\
& + \left(\frac{73.7028 + 101.01}{2} \right) \times 143.27 \times 10 + \left(\frac{80.4117 + 92.593}{2} \right) \times 168.81 \times 10 \\
& + \left(\frac{113.7812 + 121.36}{2} \right) \times 198.04 \times 10
\end{aligned}$$

$$\phi_4 = (156\ 0801 + 131\ 547.2 + 588\ 128.9) = 2280\ 477.1 \text{ kJ/h} \equiv 633.46 \text{ kW}$$

Fifth (final) Interval:

Table 6.19 Flash Calculations for Range 40°C to 31°C

Component	$V_4 y_i$	$V_5 y_i$	$L_4 x_i$	$C_5 x_i$	$L_5 x_i = F z_i$
C_3H_8	64.286	0	85.714	64.286	150
C_4H_{10}	23.990	0	101.010	23.990	125
C_5H_{12}	7.407	0	92.593	7.407	100
C_6H_{14}	3.640	0	121.360	3.640	125
Total	99.323		400.677	99.323	500

Table 6.20 Properties at 35.5°C

Component	λ_p , kJ/kmol	C_{mpt} , kJ/(kmol · K)	C_{Li} , kJ/(kmol · K)
C_3H_8	14 553.3	75.72	123.43
C_4H_{10}	20 155.3	100.23	140.84
C_5H_{12}	24 718.9	123.67	165.8
C_6H_{14}	30 245.4	147.22	194.4

$$\phi_5 = \sum C_5 x'_i \lambda_i + \sum \left(\frac{V_4 y_i + 0}{2} \right) C_{mpt} \Delta t + \sum \left(\frac{L_4 x_i + L_5 x_i}{2} \right) C_{Li} \Delta t$$

$$\phi_5 = (64.286 \times 14\ 553.3 + 23.99 \times 20\ 155.3 + 7.407 \times 24\ 718.9 + 3.64 \times 30\ 245.4$$

$$\begin{aligned}
& + \left(\frac{64.286}{2} \times 75.72 \times 11 + \frac{23.99}{2} \times 100.23 \times 11 + \frac{7.407}{2} \times 123.67 \times 11 \right. \\
& \quad \left. + \frac{3.64}{2} \times 147.22 \times 11 \right)
\end{aligned}$$

$$+ \left(\frac{85.714 + 150}{2} \right) \times 123.43 \times 11 + \left(\frac{101.01 + 125}{2} \right) \times 140.84 \times 11$$

$$+ \left(\frac{92.593 + 100}{2} \right) \times 165.8 \times 11 + \left(\frac{121.36 + 125}{2} \right) \times 194.4 \times 11$$

$$\phi_5 = 17\ 12285.2 + 47\ 806.1 + 77\ 4123.5 = 25\ 34214.8 \text{ kJ/h} \equiv 703.95 \text{ kW}$$

$$\begin{aligned}\text{Total heat duty } \phi_t &= \phi_1 + \phi_2 + \phi_3 + \phi_4 + \phi_5 \\ &= 1070.1 + 716.1 + 696.76 + 633.46 + 703.95 = 3820.37 \text{ kW}\end{aligned}$$

Assume chilled water as a cooling medium.

Let inlet temperature of chilled water, $t_i = 6^\circ\text{C}$

Outlet temperature of chilled water, $t_o = 9^\circ\text{C}$

Energy balance:

$$\phi_t = \sum_1^5 \phi_i = 3820.37 \text{ kW} = \dot{m}_w \times 4.1868 \times (9 - 6)$$

Mass flow rate of chilled water required

$$\dot{m}_w = \frac{3820.37}{4.1868 \times 3} = 304.16 \text{ kg/s}$$

$$\dot{m}_w \equiv 1094\ 976 \text{ kg/h} \equiv 1095 \text{ m}^3/\text{h}$$

$$\Delta t_{avi} (\text{LMTD}) \text{ for 1st interval} = \frac{(81 - 9) - (70 - 8.159)}{\ln\left(\frac{81 - 9}{70 - 8.159}\right)} = 66.79^\circ\text{C}$$

Table 6.21 Summary of Heat Balance Calculations

Interval (Based on vapour temperature)	Temperatures of chilled water	Heat duty ϕ , kW	Δt_{avi} , (LMTD)
81°C to 70°C	8.16°C to 9°C	1070.1	66.79°C
70°C to 60°C	7.6°C to 8.16°C	716.1	56.99°C
60°C to 50°C	7.05°C to 7.6°C	696.76	47.52°C
50°C to 40°C	6.553°C to 7.05°C	633.46	38°C
40°C to 31°C	6°C to 6.553°C	703.95	29.02°C
Total		3820.37	

Figure 6.21 gives the results in diagrammatic form.

$$\text{Weighted } \Delta t = \frac{\sum \phi_i}{\sum \left(\frac{\phi_i}{\Delta t_{avi}} \right)} \quad (6.48)$$

$$\text{Weighted } \Delta t = \frac{3820.37}{\left(\frac{1070.1}{66.79} + \frac{716.1}{56.99} + \frac{696.76}{47.52} + \frac{633.46}{38} + \frac{703.95}{29.02} \right)}$$

$$\text{Weighted } \Delta t = 45.385^\circ\text{C}$$

$$\text{Heat duty } \phi_t = 3820.37 \text{ kW}$$

Heat duty for subcooling

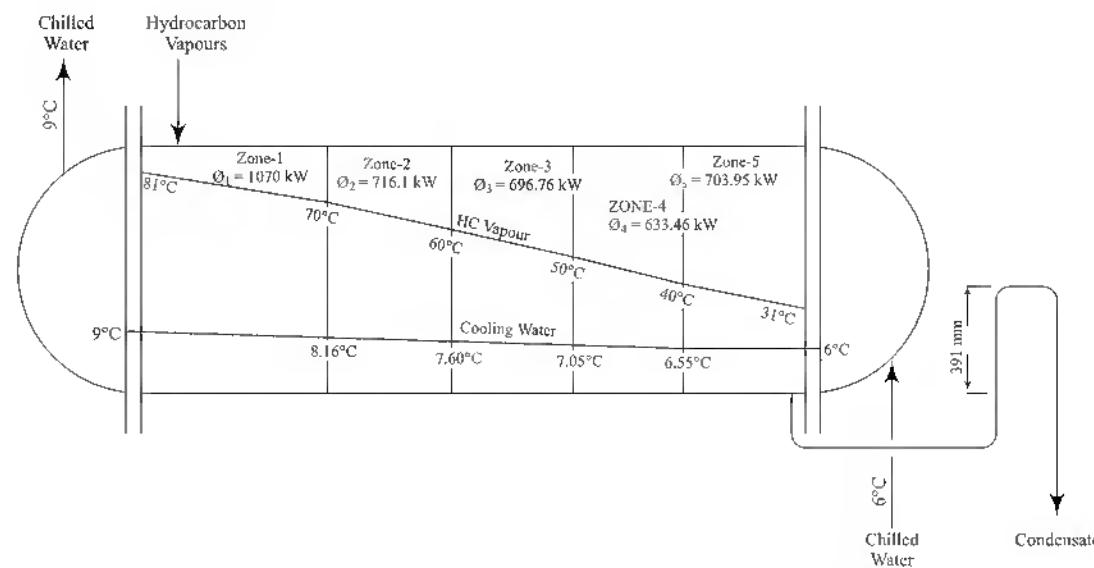


Fig. 6.21 Condenser for Condensation of Hydrocarbon Vapours

$$\begin{aligned}\phi_{\text{sub}} &= 143\ 602.7 + 331\ 589 + 469\ 986 + 588\ 128.9 + 774\ 123.5 \\ &= 2307\ 430.1 \text{ kJ/h} \equiv 640.95 \text{ kW}\end{aligned}$$

Heat duty for condensation

$$\phi_c = \phi_t - \phi_{\text{sub}} = 3820.37 - 640.95 = 3179.42 \text{ kW}$$

This value includes heat load required for cooling of vapour. Heat transfer coefficient for the cooling of vapour in presence of condensation is quite high. Also, cooling of vapour and condensation of vapour both are taking place simultaneously. Hence, as per the Kern's suggestion, separate calculation for determining heat transfer coefficient for cooling of vapour is not required.

Assume values of overall coefficients for the first trial calculation.

Overall coefficient for condensation $U_{oc} = 800 \text{ W}/(\text{m}^2 \cdot ^\circ\text{C})$

Overall coefficient for subcooling $U_{osub} = 200 \text{ W}/(\text{m}^2 \cdot ^\circ\text{C})$

Provisional heat transfer area:

$$\text{For condensation, } A_{cpro} = \frac{\phi_c}{U_{oc} \times \text{weighted } \Delta t}$$

$$A_{cpro} = \frac{3179.42 \times 10^3}{800 \times 45.385} = 87.568 \text{ m}^2$$

$$\text{For subcooling, } A_{sub, pro} = \frac{\phi_{\text{sub}}}{U_{osub} \times \text{weighted } \Delta t} = \frac{640.95 \times 10^3}{200 \times 45.386} = 70.61 \text{ m}^2$$

$$\text{Total heat transfer area } A_{pro} = 87.568 + 70.61 = 158.18 \text{ m}^2$$

$$\text{Let tube length } L = 8 \text{ ft} = 2.4384 \text{ m}$$

$$\text{Tube outside diameter } d_o = 3/4 \text{ in} = 0.01905 \text{ m}$$

$$\text{Number of tubes, } N_t = \frac{A_{pro}}{\pi d_o L} = \frac{158.18}{\pi \times 2.4384 \times 0.01905}$$

$$N_t = 1084$$

Let tube pitch $P_t = 1.25 d_o = 0.02381 \text{ m}$, triangular pitch arrangement, number of tube side passes = 4 (for first trial calculations)

Tube bundle diameter

$$D_b = d_o \left(\frac{N_t}{K_1} \right)^{\frac{1}{n_1}} \quad (6.1)$$

for $N_p = 4, K_1 = 0.175, n_1 = 2.285$ (Table 6.2)

$$D_b = 19.05 \left(\frac{1084}{0.175} \right)^{\frac{1}{2.285}} = 870 \text{ mm}$$

Let the clearance between shell inside diameter and tube bundle diameter = 16 mm.

Shell ID $D_s = 870 + 16 = 886 \text{ mm}$

Tube side heat transfer coefficient h_t :

Allocate chilled water on tube side

$$\text{Tube side flow area, } a_t = \frac{N_t}{N_p} \times \frac{\pi}{4} d_i^2 \quad (6.20)$$

Tube inside diameter, $d_i = 15.748 \text{ mm}$ (For 16 BWG tube)

$$a_t = \frac{1084}{4} \times \frac{\pi}{4} (0.015748)^2 = 0.052785 \text{ m}^2$$

$$\text{Tube side mass velocity } G_t = \frac{\dot{m}}{a_t} = \frac{304.16}{0.052785} = 5762.24 \text{ kg/(m}^2 \cdot \text{s)}$$

Density of water at 7.5°C, $\rho = 999.877 \text{ kg/m}^3$

(From Table 3.28 of Ref. 2)

$$\text{Tube side velocity, } u_t = \frac{G_t}{\rho} = \frac{5762.24}{999.877} = 5.76 \text{ m/s (very high)}$$

Velocity of water in tubes should preferably be in between 1 to 3 m/s.

Let number of tube side passes = 1

for $N_p = 1, K_1 = 0.319, n_1 = 2.142$ (From Table 6.2)

$$D_b = 19.05 \left(\frac{1084}{0.319} \right)^{\frac{1}{2.142}} = 848 \text{ mm}$$

Shell ID, $D_s = 848 + 16 = 864 \text{ mm}$ (Revised)

$$\text{Tube side flow area, } a_t = \frac{1084}{1} \times \frac{\pi}{4} (0.015748)^2 = 0.2111 \text{ m}^2$$

$$G_t = \frac{304.16}{0.2111} = 1440.83 \text{ kg/m}^2 \cdot \text{s}$$

$$u_t = \frac{G_t}{\rho} = \frac{1440.83}{999.877} = 1.44 \text{ m/s which is within acceptable range}$$

$$\text{Tube side Reynold's number, } Re_t = \frac{d_i G_t}{\mu}$$

Viscosity of water at 7.5°C, $\mu = 1.3 \times 10^{-3} \text{ kg/(m} \cdot \text{s)}$

$$Re_t = \frac{0.015748 \times 1440.83}{1.3 \times 10^{-3}} = 17454$$

$$\text{Prandtl number, } Pr = \frac{C_p \mu}{k}$$

Thermal conductivity of water at 7.5°C, $k = 0.596\ 85\ \text{W}/(\text{m} \cdot ^\circ\text{C})$

$$Pr = \frac{4.1868 \times 1.3 \times 10^{-3} \times 10^3}{0.596\ 85} = 9.12$$

Dittus-Bolter equation

$$\frac{h_i d_i}{k} = 0.023 Re^{0.8} Pr^{0.33} \left(\frac{\mu}{\mu_w} \right)^{0.14} \quad (6.19)$$

$$h_i = 0.023 \times \frac{0.596\ 85}{0.0157\ 48} \times (17\ 454)^{0.8} \times (9.12)^{0.33} \times 1$$

$$h_i = 4473.85\ \text{W}/(\text{m}^2 \cdot ^\circ\text{C})$$

h_o : Heat transfer coefficients for both condensation and subcooling must be determined separately.

For condensation \bar{h}_{oc}

For the first trial calculation let $\bar{h}_{oc} = 1500\ \text{W}/(\text{m}^2 \cdot ^\circ\text{C})$

$$\bar{h}_{oc} (\mathcal{T}_s - \mathcal{T}_w) = U_{oc} (\mathcal{T}_s - \mathcal{T}_t)$$

where $U_{oc} = 800\ \text{W}/(\text{m}^2 \cdot ^\circ\text{C})$

$$\mathcal{T}_s = \text{shell side average temperature} = \frac{81 + 31}{2} = 56^\circ\text{C}$$

\mathcal{T}_w = Tube wall temperature, °C

$$\mathcal{T}_t = \text{Tube side average temperature} = \frac{6 + 9}{2} = 7.5^\circ\text{C}$$

$$1500(56 - T_w) = 800(56 - 7.5)$$

$$T_w = 30.13^\circ\text{C}$$

$$\text{Mean temperature of condensate film, } \mathcal{T}_f = \frac{\mathcal{T}_s + \mathcal{T}_w}{2} = \frac{56 + 30.13}{2}$$

$$\mathcal{T}_f = 43^\circ\text{C}$$

Physical properties of liquid condensate should be determined for the average condensate composition. Here, properties are determined based on the composition of condensate in the intermediate (third) interval.

Table 6.22 Properties of Liquid Condensate in 3rd Interval

Component	$C_3x'_i$	Mole fraction	Mass fraction	$\rho_L, \text{kg}/\text{m}^3$	μ, cP	$k, \text{W}/(\text{m} \cdot ^\circ\text{C})$
C_3H_8	23.741	0.2660	0.1874	582	0.10	0.1211
C_4H_{10}	29.060	0.3257	0.3024	579	0.15	0.1263
C_5H_{12}	20.600	0.2309	0.2661	626	0.20	0.1305
C_6H_{14}	15.820	0.1773	0.2441	659	0.27	0.1367
	89.221	1.0000	1.0000			

(For viscosity, Fig. 14 of Ref. 5)

$$\rho_L = \sum \left(1 / \frac{W_i}{\rho_{L_i}} \right) = \frac{1}{\frac{0.1874}{582} + \frac{0.3024}{579} + \frac{0.2661}{626} + \frac{0.2441}{659}} = 609.84 \text{ kg/m}^3$$

Viscosity of liquid mixture by Irving's equation (Equation 3.107 of Ref. 2)

$$\ln \mu_{L_{\text{mix}}} = \sum w_i \ln \mu_i = [0.1874 \ln (0.1) + 0.3024 \ln (0.15) + 0.2661 \ln (0.2) \\ + 0.2441 \ln (0.27)]$$

$$\mu_{L_{\text{mix}}} = 0.1732 \text{ cP} \equiv 0.1732 \times 10^{-3} \text{ kg/(m} \cdot \text{s)}$$

$$k_{L_{\text{mix}}} = \sum k_i x_i = 0.266 \times 0.1211 + 0.3257 \times 0.1263 + 0.2309 \times 0.1305 \\ + 0.1773 \times 0.1367$$

$$k_{L_{\text{mix}}} = 0.1277 \text{ W/(m} \cdot \text{K)}$$

Average molar mass of vapour mixture,

$$M_{av} = \sum M_i y_i = 0.3 \times 44 + 0.25 \times 58 + 0.2 \times 72 + 0.25 \times 86 = 63.6 \text{ kg/kmol}$$

$$\text{Density of vapour mixture, } \rho_v = \frac{p M_{av}}{RT} \\ = \frac{4 \times 63.6}{(273 + 56)} \times \frac{273}{1 \times 22.414} = 9.418 \text{ kg/m}^3$$

Position of condenser: Horizontal

Shell side condensation coefficient

$$\bar{h}_{oc} = 0.95 k_L \left[\frac{\rho_L (\rho_L - \rho_v) g}{\mu_L \tau_h} \right]^{1/3} \cdot N_r^{-1/6} \quad (6.37)$$

$$\tau_h = \frac{W_c}{LN_t}, N_r = 2/3, N'_r = \frac{2}{3} \left(\frac{D_b}{p_t} \right) = \frac{2}{3} \times \left(\frac{848}{23.81} \right) \equiv 24$$

$$W_c = (23.741 \times 44 + 29.06 \times 58 + 20.6 \times 72 + 15.82 \times 86) / 3600$$

$$W_c = 1.548 \text{ kg/s}$$

$$\tau_h = \frac{1.548}{(1084 \times 2.4384)} = 5.856 \times 10^{-4} \text{ kg/(m} \cdot \text{s)}$$

$$\bar{h}_{oc} = 0.95 \times 0.1277 \left[\frac{609.84 (609.84 - 9.418) \times 9.81}{0.1732 \times 10^{-3} \times 5.856 \times 10^{-4}} \right]^{1/3} \times (24)^{-1/6} \\ = 0.95 \times 0.1277 \times 32839.57 \times 0.5888 = 2345.7 \text{ W/(m}^2 \cdot {^\circ}\text{C})$$

For the second trial calculation if it is assumed $\bar{h}_{oc} = 2345.7 \text{ W/(m}^2 \cdot {^\circ}\text{C})$ (resulting value of first trial calculation) condensate film temperature, $T_f = 47.73^\circ\text{C}$ (revised). Hence, minor change in the value of film temperature is noted. Also, precise temperature wise physical properties of these components (particularly density and thermal conductivity) are not available. So, second trial calculation will not make any difference in the final value.

$$\bar{h}_{oc} = 2345.7 \text{ W/(m}^2 \cdot {^\circ}\text{C})$$

As stated earlier with horizontal position, subcooling of condensate is taking place by natural convection only. No reliable correlation is available for natural convection. Hence, based on Kern's recommendation, assume

$$h_{osub} = 50 \text{ Btu}/(\text{h} \cdot \text{ft}^2 \cdot {}^\circ\text{F}) = 283.77 \text{ W}/(\text{m}^2 \cdot {}^\circ\text{C})$$

Overall Heat Transfer Coefficient for Condensation

$$\frac{1}{U_{oc}} = \frac{1}{\bar{h}_{oc}} + \frac{1}{h_{od}} + \frac{d_o \ln(d_o/d_i)}{2k_w} + \frac{d_o}{d_i} \frac{1}{h_i} + \frac{d_o}{d_i} \frac{1}{h_{id}} \quad (6.42)$$

For light hydrocarbon mixture $h_{od} = 5000 \text{ W}/(\text{m}^2 \cdot {}^\circ\text{C})$ (From Table 6.9)

For cooling water $h_{id} = 4000 \text{ W}/(\text{m}^2 \cdot {}^\circ\text{C})$

Thermal conductivity of tube material

$$k_w = 50 \text{ W}/(\text{m} \cdot {}^\circ\text{C}) \text{ (Steel or cupronickel tube)}$$

$$\begin{aligned} \frac{1}{U_{oc}} &= \frac{1}{2345.7} + \frac{1}{5000} + \frac{0.01905 \ln\left(\frac{19.05}{15.748}\right)}{2 \times 50} + \frac{19.05}{15.748} \times \frac{1}{4473.85} \\ &\quad + \frac{19.05}{15.748} \times \frac{1}{4000} \\ U_{oc} &= 809.47 \text{ W}/(\text{m}^2 \cdot {}^\circ\text{C}) \end{aligned}$$

Heat transfer area required for condensation

$$A_{creq} = \frac{\phi_c}{U_{oc} \times \text{weighted } \Delta t} = \frac{3179.42 \times 10^3}{809.47 \times 45.385}$$

$$A_{creq} = 86.54 \text{ m}^2$$

Overall heat transfer coefficient for subcooling

$$\frac{1}{U_{osub}} = \frac{1}{h_{osub}} + \frac{1}{h_{od}} + \frac{d_o \ln(d_o/d_i)}{2k_w} + \frac{d_o}{d_i} \frac{1}{h_i} + \frac{d_o}{d_i} \frac{1}{h_{id}} \quad (6.42)$$

$$\begin{aligned} \frac{1}{U_{osub}} &= \frac{1}{283.77} + \frac{1}{5000} + \frac{0.01905 \ln\left(\frac{19.05}{15.748}\right)}{2 \times 50} + \frac{19.05}{15.748} \times \frac{1}{4473.85} \\ &\quad + \frac{19.05}{15.748} \times \frac{1}{4000} \\ U_{osub} &= 230.78 \text{ W}/(\text{m}^2 \cdot {}^\circ\text{C}) \end{aligned}$$

$$A_{sub} = \frac{\phi_{sub}}{U_{osub} \times \text{weighted } \Delta t} = \frac{640.95 \times 10^3}{230.78 \times 45.385}$$

$$A_{subreq} = 61.19 \text{ m}^2$$

Total heat transfer area required,

$$A_{req} = 86.54 + 61.19 = 147.73 \text{ m}^2$$

Total heat transfer area required,

$$A_{pro} = 158.16 \text{ m}^2$$

$$\% \text{ Excess heat transfer area} = \left(\frac{158.16}{147.73} - 1 \right) \times 100 = 7.06 \%$$

It is not above 10%.

If number of tubes is increased to 1200,

$$D_b = 889, \text{ shell ID} = 905 \text{ mm}, N_t = 1200$$

$$a_t = \frac{1200}{1084} \times 0.2111 = 0.2337 \text{ m}^2$$

$$G_t = \frac{304.16}{0.2337} = 1301.5 \text{ kg/(m}^2 \cdot \text{s)}$$

$$Re = \frac{1301.5}{1440.83} \times 17454 = 15766.18$$

$$u_t = G_t/\rho = \frac{1301.5}{999.877} = 1.3016 \text{ m/s}$$

$$h_i = 4473.85 \times \left(\frac{15766.18}{17454} \right)^{0.8}$$

$$h_i = 4124.26 \text{ W/(m}^2 \cdot ^\circ\text{C)}$$

$$\bar{h}_{oc} \propto N_t^{1/3} \cdot N_r^{-1/6}$$

$$N_r = (2/3) N'_r = (2/3) \frac{D_b}{P_t} = \frac{2}{3} \left(\frac{889}{23.81} \right) = 24.9 \equiv 25$$

$$\bar{h}_{oc} = 2345.7 \times \left(\frac{1200}{1084} \right)^{1/3} \times \left(\frac{25}{24} \right)^{-1/6} = 2410 \text{ W/(m}^2 \cdot ^\circ\text{C)}$$

$$U_{oc} = 801.97 \text{ W/(m}^2 \cdot ^\circ\text{C)}$$

Heat transfer area required for condensation

$$A_{creq} = \frac{\phi_c}{U_{oc} \times \text{weighted } \Delta t} = \frac{3179.42 \times 10^3}{801.97 \times 45.385}$$

$$A_{creq} = 87.35 \text{ m}^2$$

$$U_{osub} = 229.57 \text{ W/(m}^2 \cdot ^\circ\text{C}) \quad (\text{based on new value of } h_i)$$

$$A_{subreq} = \frac{640.95 \times 10^3}{229.57 \times 45.385} = 61.52 \text{ m}^2$$

Total area required, $A_{treq} = 148.87 \text{ m}^2$

$$A_{pro} = N_t \pi d_o L = 1200 \times \pi \times 0.01905 \times 2.4384 \text{ m} = 175.12 \text{ m}^2$$

$$\% \text{ Excess heat transfer area} = \left(\frac{175.12}{148.87} - 1 \right) \times 100 = 17.63\%$$

It is adequate.

If equal % excess heat transfer area for condensation and subcooling are provided then,

Area provided for condensation $A_{cpro} = 1.1763 \times A_{creq}$

$$A_{cpro} = 102.75 \text{ m}^2$$

$$A_{sub\ pro} = 175.12 - 102.75 = 72.37 \text{ m}^2$$

$$\frac{A_{sub\ pro}}{A_{t\ pro}} = \frac{72.37}{175.12} = 0.4133$$

Thus, 41.33% of total heat transfer area is available for subcooling and the same area should be submerged in the pool of condensate. Assuming that tubes will be uniformly distributed in the crosssection of shell.

$$\frac{A_{sub\ pro}}{A_{t\ pro}} = 0.4133 = \frac{\text{Area of segment of cross-section utilized for subcooling}}{\text{Total cross-section area of shell}}$$

$$\frac{x D_i^2}{\frac{\pi}{4} D_i^2} = 0.4133 \text{ or } x = 0.3246$$

(From Table 1.19 b of Ref. 2)

$$\text{for } x = 0.3246, \frac{h}{D_i} = 0.432$$

To facilitate the subcooling of condensate or to submerge 41.33% of total heat transfer area in the pool of condensate, height of inverted U-seal required from the bottom most point of shell inside diameter.

$$h = 0.432 \times 905 = 390.96 \text{ mm} = 391 \text{ mm} \text{ (as shown in Fig. 6.21)}$$

6.7 PROCESS DESIGN OF REBOILERS AND VAPORIZERS

Design correlations for reboilers and vaporizers are same. Reboilers are used with distillation columns. In a reboiler part of liquid fed is vaporized while in a vaporizer the entired feed of liquid is vaporized.

Following types of Reboilers are used in chemical industries.

- (a) Kettle type Reboiler
- (b) Bundle-in-column Reboiler
- (c) Horizontal Thermosyphon Reboiler
- (d) Vertical Thermosyphon Reboiler
- (e) Forced Circulation Reboiler.

Process designs of Kettle type Reboiler and Vertical Thermosyphon Reboiler are given in subsequent sections.

6.7.1 Process Design of Kettle Type Reboiler

Kettle type reboiler (see Fig. 6.1(e)) is normally used with a distillation column and also as vaporizer. Vapour leaving the kettle type reboiler is always in equilibrium with the liquid. In this type of reboiler heating surface is immersed in the pool of liquid. Hence, perfect pool boiling is taking place. In pool boiling, maximum value of heat transfer coefficient is achieved in nucleate boiling region. To achieve the nucleate boiling region, ΔT (Temperature difference between

temperature of heating surface and boiling temperature of liquid) should be less than and close to critical temperature drop. Critical temperature drop for water is 20 to 30°C and for light organics is 20 to 50°C. In kettle type reboiler boiling fluid side pressure drop is negligible, hence it is preferred with high vacuum distillation column.

6.7.1.1 Process Design Steps

- Calculation of heat duty.

In actual design calculation, heat duty of reboiler is calculated based on energy balance equation around distillation column.

$$\phi_B = H_D D + H_W W - H_F F + \phi_C + \phi_L \quad (6.53)$$

where, ϕ_B = Heat duty of reboiler, kW

H_D = Enthalpy of distillate, kJ/kmol

D = Molar flow rate of distillate, kmol/s

H_w = Enthalpy of residue, kJ/kmol

W = Molar flow rate of residue, kmol/s

H_F = Enthalpy of feed, kJ/kmol

F = Molar flow rate of feed, kmol/s

ϕ_C = Heat duty of condenser, kW

ϕ_L = Heat loss of distillation system, kW

If the vaporization rate of reboiler is given then heat duty can be determined by following equation.

$$\phi_B = \dot{m}_v \lambda \times 1.05 \quad (6.54)$$

\dot{m}_v = Vaporization rate, kg/s

(considering 5% heat loss to surrounding)

- Fix the value of mean temperature difference ΔT_m . It should be less than and close to critical temperature drop. Average temperature of heating medium $t_h = \Delta T_m + t_B$
- Select the suitable heating medium.
- Based on energy balance, find the mass flow rate of heating medium. If the temperature of heating medium required is less than 180°C, then saturated steam can be selected as heating medium. In this case,

$$q_B = \dot{m}_s \lambda_s \quad (6.55)$$

where, λ_s = Latent heat of vaporization of steam, kJ/kg (to be obtained from Steam Tables)

\dot{m}_s = Mass flow rate of steam required, kg/s

- To get the first estimate of heat transfer area, assume the value of overall heat transfer coefficient (Table 6.7). Find the heat transfer area based on the assumed value of U_o .

$$A = \frac{\phi_t}{U_o \Delta T_m} = A_{\text{provided}} = N_t \pi d_o L \quad (6.43)$$

- Fix the value of tube outside diameter d_o and tube length. Find the number of tubes N_t . Decide the tube arrangement. In reboiler, normally 1 in

(25.4 mm OD) tubes are used. Square pitch arrangement with U tube-bundle is preferred. In kettle type reboiler, higher value of tube pitch ($P_t = 1.5 d_o$ to $2d_o$) is preferred to avoid the “vapour blanketing” inside the pool of liquid and over the heat transfer surface. Close distance between tubes provides more resistance to the flow of vapour bubbles. Close distance between the tubes with higher vaporization rate per unit area may result in vapour blanketing which reduces the overall heat transfer coefficient and hence reduces heat duty of reboiler considerably. With kettle type reboiler full baffles (% Baffle cut = 0) are used as extra space is available for the flow of liquid. Nucleate boiling coefficient or any phase change coefficient does not depend on type of baffle or baffle spacing. Here, baffles are provided only to reduce the vibration in tubes. Here, baffle spacing can be kept equal to tube bundle diameter or even higher than that but within permissible limit, specified in TEMA standard.

- (g) Calculate shell side boiling coefficient by Mostinski equation.

$$h_o = h_{nB} = 0.104 p_c^{0.69} \left(\frac{\phi_B}{A} \right)^{0.7} \left[1.8 \left(\frac{p}{p_c} \right)^{0.17} + 4 \left(\frac{p}{p_c} \right)^{1.2} + 10 \left(\frac{p}{p_c} \right)^{10} \right] \quad (6.56)$$

where, $h_o = h_{nB}$ = Shell side nucleate boiling heat transfer coefficient in $\text{W}/(\text{m}^2 \cdot ^\circ\text{C})$

$\frac{\phi_B}{A}$ = Heat flux of reboiler, W/m^2

p_c = Critical pressure of component or pseudo-critical pressure of the mixture of components vaporized, bar

p = Operating pressure in reboiler, bar

This equation is reliable for single component boiling. For, a mixture, actual coefficient is lesser than the same predicted by this equation.

- (h) Calculate tube side heat transfer coefficient, h_i :

If saturated steam is used as heating medium, then calculation for h_i is not required. Value of $6000 \text{ W}/(\text{m}^2 \cdot ^\circ\text{C})$ can be safely used. This value also includes fouling resistance.

$$\text{or } \frac{1}{h'_i} = \frac{1}{h_i} + \frac{1}{h_{id}} , \quad h'_i = 6000 \text{ W}/(\text{m}^2 \cdot ^\circ\text{C})$$

If hot oil is used as heating medium, then h_i is calculated by forced convection correlation. Eq. (6.18) or (6.19) depending on the value of Reynolds number.

- (i) Calculate the overall heat transfer coefficient U_o , by Eq. (6.42).
- (j) Calculate the heat transfer area required using Eq. (6.43).
- (k) Calculate % excess heat transfer area. It should be in between 10 to 20%. If not, then change the number of tubes or tube length. Repeat the calculations until % excess area is in the desired range.

- (l) Based on the final or last value of heat transfer area provided, calculate the actual heat flux.

$$\text{Actual heat flux} = \frac{\phi_B}{A_{\text{provided}}}$$

- (m) Actual heat flux $\leq 0.7 (\phi/A)_c$

where $(\phi/A)_c$ is critical heat flux. Critical heat flux is the heat flux corresponding to critical temperature drop. This condition must be satisfied. Critical heat flux can be estimated by modified Zuber's equation⁴ which can be written as

$$\left(\frac{\phi}{A}\right)_C = K_b \left(\frac{P_t}{d_o}\right) \left(\frac{\lambda_v}{\sqrt{N_t}}\right) (\sigma g (\rho_L - \rho_v) \rho_v^2)^{0.25} \quad (6.57)$$

where,

$$\left(\frac{\phi}{A}\right)_C = \text{Critical heat flux, W/m}^2$$

K_b = Constant

K_b = 0.44 for square pitch arrangement and 0.41 for triangular pitch arrangement

P_t = Tube pitch, mm

d_o = Tube outside diameter, mm

N_t = Total number of tubes in bundle

λ = Latent heat of vaporization, J/kg

ρ_v = Vapour density, kg/m³

σ = Liquid surface tension, N/m

ρ_L = Liquid density kg/m³

g = Acceleration due to gravity = 9.81 m/s²

- (n) Find the shell inside diameter. Shell inside diameter of kettle type reboiler should be greater of following two values.

- (i) Shell ID = Liquid level + 0.15 to 0.25 m

Liquid level in kettle type reboiler = Tube bundle diameter + 50 to 100 mm

To find the tube bundle diameter for U tube bundle, Eq. (6.2) and for tube bundle with floating head, Eq. (6.1), can be used.

- (ii) Minimum Shell ID can be obtained from following table.

Table 6.23 Minimum Shell Diameter of Kettle Type Reboiler

Heat flux, W/m ²	Shell ID/Tube bundle diameter
< 25 000	1.2 to 1.5
25 000 to 40 000	1.4 to 1.8
> 40 000	1.7 to 2.0

- (o) Check the liquid entrainment. To avoid the excessive entrainment, vapour velocity at liquid surface should be less than v_{\max} .

$$v_{\max} = 0.2 \left(\frac{\rho_L - \rho_v}{\rho_v} \right)^{1/2} \quad (6.58)$$

where, v_{\max} = Maximum permissible vapour velocity at liquid surface, m/s

ρ_L = Density of liquid phase kg/m³

ρ_v = Density of vapour phase kg/m³

Actual vapour velocity at liquid surface.

$$v = \frac{m_v / \rho_v}{\text{Liquid surface area}} \quad (6.59)$$

$$\text{Liquid surface area} \equiv L \times x \quad (6.60)$$

where, L = Tube length

x = width of liquid level

6.7.1.2 Boiling of Liquid Mixture

For the close boiling liquid mixture (boiling range is less than 5°C), Eq. (6.56) can be used to find the nucleate boiling coefficient. If for the given liquid mixture boiling range is wide then, use of Eq. (6.56) is not recommended. For such a mixture, Palen and Small equation can be used.

$$(h_{nB})_{\text{mixture}} = f_m \times (h_{nB})_{\text{single component}} \quad (6.61)$$

$$\text{where, } f_m = \exp [-0.0083 (\mathcal{T}_{bo} - \mathcal{T}_{bi})] \quad (6.62)$$

\mathcal{T}_{bo} = Temperature of vapour mixture leaving the reboiler, °C

\mathcal{T}_{bi} = Temperature of liquid entering the reboiler, °C

Kettle type reboiler can be considered as one equilibrium stage. Hence, the vapour leaving the reboiler is in equilibrium with the residue. Dew point temperature of vapour leaving the reboiler (\mathcal{T}_{bo}) is equal to bubble point temperature of residue. To find T_{BP} (Bubble point temperature of liquid entering to the reboiler), composition of liquid entering to reboiler must be determined. Material balance around reboiler gives composition of liquid (x_{1i}) entering to reboiler.

$$\bar{L} = \bar{V} + W \quad (6.63)$$

$$\bar{L}x_{1i} = \bar{V}y_i + Wx_i \quad (6.64)$$

where, W = Molar flow rate of Residue, kmol/s

x_i = Mole fraction of component i in residue

$y_i = K_i x_i$ = Mole fraction of component i in vapour leaving the reboiler

x_{1i} = Mole fraction of component i in liquid entering to reboiler.

\bar{V} = Molar flow rate of vapour leaves the reboiler, kmol/s

\bar{L} = Molar flow rate of liquid enters to reboiler, kmol/s

Bubble point of liquid entering to reboiler, gives the value of T_{BP} .

Example 6.5

A packed distillation tower with overhead condenser and kettle type reboiler is used for aromatics separation. Design conditions of the column are as follows.

Table 6.24 Composition of Distillation Column Streams

Component	Composition, mole %		
	Feed	Distillate	Bottoms
Benzene	2.2	22.8	0
Toulene	7.4	72.2	0.5
Ethyl benzene	43.4	5.0	47.5
Styrene	47.0	0	52.0

Feed flow rate to distillation tower is 100 kmol/h.

A reflux ratio of 6.0 is selected for the design. Operating pressure at the base of column is fixed at 21.3 kPa a (160 torr). All the streams may be taken as saturated liquids. Also assume that the mixtures are ideal mixtures. Saturated steam at 0.2 MPa a pressure is used as heating medium in kettle type reboiler.

Design the kettle type reboiler for this distillation column.

Solution:

Heat duty of kettle type reboiler

$$\phi_B = \phi_C + H_D D + H_W W - H_F F + \phi_L \quad (6.53)$$

To calculate the heat duty of reboiler ϕ_B by using above equation flow rates (F , D and W), latent heats at bubble point temperatures, specific heats of liquid streams, bubble point temperatures of streams, dew point temperature of overhead vapour, etc. must be determined.

$$F = \text{Feed flow rate} = 100 \text{ kmol/h}$$

$$F = D + W$$

where D and W are molar flow rates of distillate and residue (bottom product).

$$D + W = 100$$

Benzene balance

$$0.228 D = 2.2$$

$$\text{or } D = 9.65 \text{ kmol/h}$$

$$W = 100 - 9.65 = 90.35 \text{ kmol/h}$$

$$\phi_C = \text{Heat duty of condenser} = (R + 1) D \lambda$$

$$R = 6, D = 9.65 \text{ kmol/h}$$

Latent heat of vaporization must be determined at the average temperature of the dew point and bubble point of distillate product. Assuming negligible pressure drop in the tower, operating pressure is constant throughout the distillation column and is equal to operating pressure at the base of column for heat duty calculation, i.e. 21.3 kPa a.

Assumption of constant pressure throughout the column is not true in real case but for simplicity this assumption is made. In actual case, top pressure is to be found by distillation calculations and dew point at the real pressure at the top is to be calculated.

Bubble point of Distillate

For ideal vapour liquid equilibrium,

$$\text{at bubble point } \sum x_i p_i^{\text{sat}} = p_t \text{ and } z_i = x_i$$

Vapour Pressure Data:

Table 6.25 Antoine Constants⁹

Component	A	B	C
Benzene	6.0306	1211.0	-52.35
Toluene	6.0795	1344.8	-53.65
Ethyl benzene	6.0821	1424.3	-59.95
Styrene	6.1911	1507.4	-58.15

$$\text{Antoine equation } \log_{10} p_v = A - \frac{B}{T + C}$$

where p_v is in kPa and T is in K.

At bubble point of distillate,

$$0.228 p_{vB} + 0.722 p_{vT} + 0.05 p_{vEB} = p_t$$

Trial $T = 328.3$ K

At $T = 328.3$ K $p_{vB} = 43.865$ kPa, $p_{vT} = 15.244$ kPa and $p_{vEB} = 5.95$ kPa

$$0.228 \times 43.865 + 0.722 \times 15.244 + 0.05 \times 5.95 = 21.3 \text{ kPa} = p_t \quad (\text{Check!})$$

Bubble point of distillate $T_{BP} = 328.3$ K (55.3°C)

At dew point of distillate

$$\sum \frac{y_i p_t}{p_i^{\text{sat}}} = 1 \quad \text{and} \quad z_i = y_i$$

Try $T = 334.7$ K

$p_{vB} = 55.1565$ kPa, $p_{vT} = 19.7054$ kPa, $p_{vEB} = 7.909$ kPa

$$\left(\frac{0.228}{55.1565} + \frac{0.722}{19.7054} + \frac{0.05}{7.909} \right) \times 21.3 = 1.003 \quad (\text{Check!})$$

Dew point temperature of distillate, $T_{DP} = 334.7$ K (61.7°C)

Latent heat of vaporization must be determined at average temperature.

$$T_{av} = \frac{328.3 + 334.7}{2} = 331.5 \text{ K}$$

Table 6.26 Properties of Components of Distillate

Component	y_i	Critical temperature T_C °K	T_1 , K	λ at T_1 , kJ/kmol
Benzene	0.228	562.16	353.3	30 761
Toluene	0.722	591.79	383.7	33 179
Ethyl benzene	0.05	617.2	409.3	35 564

$$\frac{\lambda_2}{\lambda_1} = \left[\frac{T_C - T}{T_C - T_1} \right]^{0.38} \quad (\text{Watson equation})$$

For Benzene

$$\frac{\lambda_B}{30761} = \left[\frac{562.16 - 331.5}{562.16 - 353.3} \right]^{0.38}$$

$$\lambda_B = 31\ 943.68 \text{ kJ/kmol at } 331.5 \text{ K}$$

Similarly,

$$\begin{aligned}\lambda_T &= 36\ 124.5 \text{ kJ/kmol at } 331.5 \text{ K} \\ \lambda_{EB} &= 40\ 130.243 \text{ kJ/kmol at } 331.5 \text{ K} \\ \lambda_{av} &= \sum \lambda_{Vi} y_i \\ &= 0.228 \times 31\ 943.68 + 0.722 \times 36\ 124.5 + 0.05 \times 40\ 130.243 \\ &= 35\ 371.56 \text{ kJ/kmol}\end{aligned}$$

Heat duty of condensation

$$\begin{aligned}\phi_c &= (R+1)D \lambda_{av} \\ \phi_c &= (6+1) \times 9.65 \times 35\ 371.56 = 238\ 9348.9 \text{ kJ/h} \equiv 663.7 \text{ kW}\end{aligned}$$

Let 331.5 K be the reference temperature for enthalpy calculations, i.e. $T_o = 331.5 \text{ K}$

$$H_D = 0, H_D D = 0$$

$H_W W$

Residue is bottom product of Distillation and is a saturated liquid or at its bubble point. To find the enthalpy of residue, first the bubble point of residue must be determined.

At bubble point of liquid $\sum x_i p_i^{\text{sat}} = p_t$ and $z_i = x_i$

$$\text{Trial: At } T = 363.55 \text{ K or } \sum \frac{x_i p_i^{\text{sat}}}{p_t} = \sum y_i = 1$$

$$\begin{aligned}\text{Vapour pressures, } p_{vT} &= 54.959 \text{ kPa}, p_{vEB} = 24.588 \text{ kPa} \\ p_{vS} &= 18 \text{ kPa}, p_t = 21.3 \text{ kPa}\end{aligned}$$

Table 6.27 Bubble Point Calculations for Residue

Component	x_i	p_{vi}	$y_i = \frac{x_i p_{vi}}{p_t}$
Toluene	0.005	54.959	0.013
Ethyl benzene	0.475	24.588	0.548
Styrene	0.52	18	0.439
$\Sigma y_i = 1$ (check)			

Bubble point of residue $T_{BP} = 363.55 \text{ K}$ (90.55°C)

Empirical heat capacity equation for liquids

$$C_{ml} = a + bT + cT^2 + dT^3$$

where C_{ml} is in $\text{kJ / (kmol} \cdot \text{K)}$ and T is in K

Table 6.28 Heat Capacity Equation Constants

Component	a	$b \times 10^3$	$c \times 10^6$	$d \times 10^9$
Toluene	1.8083	812.223	-1512.67	1630.01
Ethyl benzene	4.3143	900.174	-1450.05	1433.6
Styrene	-38.019	1197.21	-2195.65	1933.12
Benzene	-7.2733	770.541	-1648.18	1897.94

Table 6.29 Heat Capacity Equation Constants for Residue

Component	x_i	$a_i \times x_i$	$b_i \times x_i \times 10^3$	$c_i \times x_i \times 10^6$	$d_i \times x_i \times 10^9$
Toluene	0.005	0.009	4.061	-7.563	8.15
Ethyl benzene	0.475	2.049	427.583	-688.774	680.96
Styrene	0.52	-19.77	622.549	-1141.738	1005.222
Total	1.00	-17.712	1054.193	-1838.075	1694.332

Enthalpy of residue,

$$H_w = \sum a_i x_i \times (T - T_o) + \sum b_i x_i \times \left(\frac{T^2 - T_o^2}{2} \right) + \sum c_i x_i \times \left(\frac{T^3 - T_o^3}{3} \right) + \sum d_i x_i \times \left(\frac{T^4 - T_o^4}{4} \right)$$

where, T_o = reference temperature = 331.5 K

$$H_w = -17.712(363.55 - 331.5) + 1054.193 \times 10^{-3} \left(\frac{363.55^2 - 331.5^2}{2} \right) - 1838.075 \times 10^{-6}$$

$$\times \left(\frac{363.55^3 - 331.5^3}{3} \right) + 1694.332 \times 10^{-9} \times \left(\frac{363.55^4 - 331.5^4}{4} \right)$$

$$H_w = 6338.32 \text{ kJ/kmol}$$

$$H_w W = 6338.32 \times 90.35 = 572\ 667.2 \text{ kJ/h} \equiv 159.074 \text{ kW}$$

H_F :

Table 6.30 Heat Capacity Equation Constants for Feed

Component	x_i	$a_i \times x_i$	$b_i \times x_i \times 10^3$	$c_i \times x_i \times 10^6$	$d_i x_i \times 10^9$
Benzene	0.022	-0.16	16.952	-36.26	41.755
Toluene	0.074	0.134	60.105	-111.93	120.621
Ethyl benzene	0.434	1.872	390.676	-629.322	622.182
Styrene	0.47	-17.869	562.689	-1031.956	908.566
Total	1.00	-16.023	1030.422	-1809.476	1693.124

$$H_F = -16.023(T_b - T_o) + 1030.42 \times 10^{-3} \left(\frac{T_b^2 - T_o^2}{2} \right)$$

$$- 1809.476 \times 10^{-6} \left(\frac{T_b^3 - T_o^3}{3} \right) + 1693.124 \times 10^{-9} \left(\frac{T_b^4 - T_o^4}{4} \right)$$

where, T_{BP} = bubble point of feed, T_o = reference temperature

$$\text{At bubble point } \sum \frac{x_i p_{vi}}{p_t} = \sum y_i = 1$$

Trial: At $T = 357.75$ K, $p_t = 21.3$ kPa

Table 6.31 Bubble Point Calculation

Component	x_i	p_{vi} , kPa	$y_i = \frac{x_i p_{vi}}{p_t}$
Benzene	0.022	116.227	0.12
Toluene	0.074	45.422	0.158
Ethyl benzene	0.434	19.923	0.406
Styrene	0.47	14.445	0.319
	1.00		1.003 (check)

Bubble point of feed, $T_{BP} = 357.75$ K (84.75 °C)

$$H_F = -16.023(357.75 - 331.5) + 1030.422 \times 10^{-3} \left(\frac{357.75^2 - 331.5^2}{2} \right) - 1809.476$$

$$\times 10^{-6} \left(\frac{357.75^3 - 331.5^3}{3} \right) + 1693.124 \times 10^{-9} \left(\frac{357.75^4 - 331.5^4}{4} \right) = 5079 \text{ kJ/kmol}$$

$$H_F F = 507\ 900 \text{ kJ/h} \equiv 141.08 \text{ kW}$$

Heat duty of kettle type reboiler

$$\phi_B = \phi_C + H_D D + H_W W - H_F F + \phi_L \quad (6.53)$$

$$\phi_B = 663.7 + 0 + 159.074 - 141.08 + \phi_L$$

$$\phi_B = 681.7 + \phi_L = 681.7 \times 1.05 = 716 \text{ kW} \text{ (considering 5 % heat loss)}$$

In continuous operation at steady state, boiling temperature of liquid in kettle type reboiler = Bubble point temperature of residue = Dew point temperature of vapour leaving the reboiler = Steady state equilibrium temperature of kettle type reboiler = T_{BP} .

$$T_{BP} = 363.55 \text{ K} \quad (90.55^\circ\text{C})$$

Critical temperature drop of light organics is ranging from 20°C to 50°C . For the precise calculations, value of critical temperature drop and critical heat flux at the actual operating condition must be determined by experiment.

Temperature of heating medium $\mathcal{T}_h = T_{BP} + \Delta\mathcal{T}_m$

$\Delta\mathcal{T}_m$ should be close to and less than $\Delta\mathcal{T}_c$.

Let $\Delta\mathcal{T}_m \equiv 30^\circ\text{C}$

\mathcal{T}_h = Temperature of saturated steam = 393.38 K = 120.23°C

$$\Delta\mathcal{T}_m = 393.38 - 363.55 = 29.83^\circ\text{C}$$

Saturated steam pressure = 0.2 MPa

Latent heat, $\lambda_s = 2201.6$ kJ/kg (Ref: Steam Tables)

Steam required

$$\dot{m}_s = \frac{\phi_B}{\lambda_s} = \frac{716}{2201.6} = 0.325\ 218 \text{ kg/s} \equiv 1170 \text{ kg/h}$$

For the first trial calculation, assume $U_o = 1000$ W/(m² · °C)

Heat transfer area based on assumed value of U_o :

$$A_o = \frac{\phi_B}{U_o \Delta T_m} = \frac{716 \times 10^3}{1000 \times 29.836} = 24 \text{ m}^2 = A_{o \text{ provided}}$$

$$A_{o \text{ provided}} = 24 \text{ m}^2 = N_t \pi d_o L$$

Nominal U tube length = 3.6576 m (12 ft)

$$d_o = 25.4 \text{ mm}, d_i = 21.1836 \text{ mm (14 BWG)}$$

$$A_{o \text{ provided}} = 24 \text{ m}^2 = N_t \pi \times (0.0254) \times 3.6576$$

Number of tubes = 82 (U tubes)

Number of tube holes in tube sheet = $82 \times 2 = 164$

Use square pitch arrangement.

Tube pitch $P_t = 1.5 d_o = 38.1 \text{ mm}$

Minimum U bend radius = $1.5 d_o = 38.1 \text{ mm}$

$h_o = h_{nBo}$ = Nucleate boiling heat transfer coefficient for mixture = $f_m \cdot h_{nB}$ (6.61)
where, h_{nB} can be determined by Mostinski equation²

$$h_{nB} = 0.104 p_c^{0.69} \left(\frac{\phi_B}{A} \right)^{0.7} \left[1.8 \left(\frac{p}{p_c} \right)^{0.17} + 4 \left(\frac{p}{p_c} \right)^{1.2} + 10 \left(\frac{p}{p_c} \right)^{10} \right] \quad (6.56)$$

$$\frac{\phi_B}{A} = \frac{716 \times 10^3}{24} = 29833.33 \text{ W/m}^2$$

$$P = 21.3 \text{ kPa} = 0.213 \text{ bar}$$

p_c = Critical pressure of boiling liquid mixture, bar

No reliable method is available for estimating critical pressure of a mixture.

$$p_{cn} \cong \sum y_i p_{ci}$$

f_m = correction factor

Table 6.32 Composition in Reboiler

Component	Residue composition		Composition of vapour		Composition of liquid entering reboiler	
	x_i	$Wx_i, \text{ kmol/h}$	$y_i = p_{vi}x_i/p_t$	$\bar{V}y_i, \text{ kmol/h}$	$\bar{L}x_i, \text{ kmol/h}$	x_{1i}
Toluene	0.005	0.45175	0.013	0.856	1.30775	0.0084
Ethyl benzene	0.475	42.9163	0.548	36.07	78.9863	0.5058
Styrene	0.52	46.982	0.439	28.895	75.877	0.4859
Total	1.00	90.35	1.00	65.82	156.17	1.000

Total material balance around reboiler

$$\bar{L} = \bar{V} + W$$

and component balance: $\bar{L}x_{1i} = Wx_i + \bar{V}y_i$

where

\bar{V} = Molar flow rate of vapour leaving the reboiler, kmol/h

\bar{L} = Molar flow rate of liquid leaving the bottom most equilibrium stage, kmol/h

x_{1i} = Mole fraction of component i in \bar{L}

Correction factor

$$f_m = \exp[-0.0083(\mathcal{T}_{bo} - \mathcal{T}_{bi})] \quad (6.62)$$

where, \mathcal{T}_{bo} = Temperature of vapour mixture leaving the reboiler, °C

\mathcal{T}_{DP} = Dew point temperature of vapour leaving the reboiler, °C

or \mathcal{T}_{BP} = Bubble point temperature of residue, °C

\mathcal{T}_{bi} = Temperature of liquid entering to the reboiler = Bubble point temperature of liquid entering the reboiler.

$\mathcal{T}_{bo} = 90.4^\circ\text{C}$ (363.55 K)

At \mathcal{T}_{bi}

$$\sum \frac{x_{1i} P_i^{\text{sat}}}{P_t} = 1$$

To find \bar{L} and x_{1i} by Equations 6.63 and 6.64, value of \bar{V} is required

$$\bar{V} = \frac{\phi_B}{\lambda_{av}}$$

Table 6.33 Properties of Residue Components

Component	x_i	Critical Temperature T_c, K	T_l, K	$\lambda_i, \text{kJ/kmol}$
Toluene	0.005	591.79	383.7	33 179
Ethyl benzene	0.475	617.2	409.3	35 564
Styrene	0.52	647	418.2	36 819
Total	1.00			

$$\frac{\lambda}{\lambda_1} = \left[\frac{T_c - T}{T_c - T_1} \right]^{0.38}$$

To be precise, λ must be determined at average temperature of T_{bo} and T_{bi} and also for average composition of liquid. Here, λ_{av} is determined at T_{bo} and for the residue composition as there is a negligible change in temperature and composition.

At $T = T_{bo} = 363.55 \text{ K}$

$$\lambda_T = 34 365 \text{ kJ/kmol}, \lambda_{EB} = 38 356 \text{ kJ/kmol}, \lambda_S = 39 941 \text{ kJ/kmol}$$

$$\lambda_{av} \cong 0.005 \times 34 365 + 0.475 \times 38 356 + 0.52 \times 39 941$$

$$\lambda_{av} = 39 160 \text{ kJ/kmol}$$

$$\bar{V} = \frac{716}{39 160} \times \frac{3600}{1} = 65.82 \text{ kmol/h}$$

$$\bar{L} = \bar{V} + W = 65.82 + 90.35 = 156.17 \text{ kmol/h.}$$

Values of x_{1i} are calculated by using Eqs (6.63) and (6.64) and are given in Table 6.33.

At $T_{bi} = 363.1 \text{ K}$ (Trial)
 $p_{vT} = 54.1658 \text{ kPa}$, $p_{vEB} = 24.1972 \text{ kPa}$, $p_{vS} = 17.7 \text{ kPa}$
 $p_t = 21.3 \text{ kPa}$

$$\sum \frac{x_{li} p_{vt}}{p_t} = \frac{0.0084 \times 54.1658}{21.3} + \frac{0.5058 \times 24.1972}{21.3} + \frac{0.4859 \times 17.7}{21.3}$$

$$\frac{x_{li} p_{vt}}{p_t} = 1 \quad (\text{check})$$

$$T_{bi} = 363.1 \text{ K}$$

Correction factor $f_m = \exp[-0.0083(363.55 - 363.1)]$

$$f_m = 0.99627$$

$$h_o = h_{nB\text{mix}} = f_m \cdot h_{nB} = 0.99627 \times h_{nB}$$

$$p_{cm} \cong \sum p_{ci} y_i$$

Table 6.34 Critical Pressure Data

Component	p_c , bar	y_i
Toluene	41.1	0.013
Ethyl benzene	36.1	0.548
Styrene	39.9	0.439
Total		1.00

$$p_{cm} = \sum p_{ci} y_i = 0.013 \times 41.1 + 0.548 \times 36.1 + 0.439 \times 39.9$$

$$p_{cm} = 37.83 \text{ bar}, \frac{p}{p_{cm}} = \frac{0.213}{37.83} = 0.00563 = p_{rm}$$

$$h_{nB} = 0.104 \times (37.83)^{0.69} \times (29833.33)^{0.7} [1.8 p_{rm}^{0.17} + 4 p_{rm}^{1.2} + 10 p_{rm}^{10}]$$

$$h_{nB} = 1304.72 \text{ W}/(\text{m}^2 \cdot ^\circ\text{C})$$

$$h_o = h_{nB\text{mix}} = 0.99627 \times 1304.72 = 1299.85 \text{ W}/(\text{m}^2 \cdot ^\circ\text{C})$$

For the condensation of saturated steam

$$h'_i = 6000 \text{ W}/(\text{m}^2 \cdot ^\circ\text{C})$$

This value also includes fouling resistance.

Overall heat transfer coefficient U_o

$$\frac{1}{U_o} = \frac{1}{h_o} + \frac{1}{h_{od}} + \frac{d_o \ln(d_o/d_i)}{2k_w} + \frac{d_o}{d_i} \times \frac{1}{h'_i} \quad (6.42)$$

Shell side fouling coefficient, $h_{od} = 5000 \text{ W}/(\text{m}^2 \cdot ^\circ\text{C})$

Thermal conductivity of steel tube material, $k_w = 50 \text{ W}/(\text{m} \cdot ^\circ\text{C})$

$$\frac{1}{U_o} = \frac{1}{1299.85} + \frac{1}{5000} + \frac{0.0254 \ln\left(\frac{25.4}{21.1836}\right)}{2 \times 50} + \frac{25.4}{21.1836} \times \frac{1}{6000}$$

$$U_o = 822.86 \text{ W}/(\text{m}^2 \cdot ^\circ\text{C})$$

Heat transfer area required

$$A_{o\text{req}} = \frac{\phi_B}{U_o \Delta T_m} = \frac{716 \times 10^3}{822.86 \times 29.83} = 29.17 \text{ m}^2$$

Heat transfer area provided, $A_{opro} = 24 \text{ m}^2$

$$A_{o\text{req}} > A_{opro}$$

Increase in heat transfer area will decrease the value of h_{nB} and the value of U_o . Value of overall coefficient removed ($U_o = 822.86 \text{ W}/(\text{m}^2 \cdot ^\circ\text{C})$) is within normal range (See Table 6.7). Further decrease in the value of U_o is not required. To decrease the value of $A_{o\text{req}}$ increase the pressure and temperature of saturated steam. Let saturation temperature = 404.35 K.

Pressure = 0.28 MPa

$$\lambda_s = 2170.1 \text{ kJ/kg}$$

$$\text{Steam required } \dot{m}_s = \frac{\phi_B}{\lambda_s}$$

$$\dot{m}_s = \frac{716}{2170.1} = 0.33 \text{ kg/s} \equiv 1188 \text{ kg/h}$$

$$\Delta T_m = 404.35 - 363.55 = 40.8^\circ\text{C}$$

$$A_{o\text{req}} = \frac{\phi_B}{U_o \Delta T_m} = \frac{716 \times 10^3}{822.86 \times 40.8} = 21.327 \text{ m}^2$$

$$\% \text{ Excess heat transfer area} = \left(\frac{24}{21.327} - 1 \right) \times 100$$

% Excess heat transfer area = 12.533%

$$\text{Actual heat flux, } \frac{\phi_B}{A_{opro}} = 29833.33 \text{ W/m}^2 \leq 0.7 \left(\frac{\phi}{A} \right)_c$$

This condition must be satisfied. To verify this condition critical heat flux is determined by modified Zuber's equation.

$$\left(\frac{\phi}{A} \right)_c = K_b \left(\frac{p_t}{d_o} \right) \left(\frac{\lambda}{\sqrt{N_t}} \right) (\sigma g (\rho_L - \rho_v) \rho_v^2)^{0.25} \quad (6.57)$$

$$K_b = 0.44, p_t = 38.1 \text{ mm}, d_o = 25.4 \text{ mm}$$

$$N_t = 82 \times 2 = 164, g = 9.81 \text{ m/s}^2$$

λ = Latent heat of vaporization, J/kg

$$\lambda_s = 39160 \text{ kJ/kmol}$$

Average molar mass of residue

$$M_{av} = \sum M_i x_i = 0.005 \times M_T + 0.475 \times M_{EB} + 0.52 \times M_S$$

Molar mass of toluene $M_T = 92.141$

Molar mass of ethyl benzene $M_{EB} = 106.168$

Molar mass of styrene $M_S = 104.152$

$$M_{av} = 0.005 \times 92.141 + 0.475 \times 106.168 + 0.52 \times 104.152 = 105.05 \text{ kg/kmol}$$

$$\lambda = \frac{39160}{105.05} \times 10^3$$

$$\lambda = 372\ 774.9 \text{ J/kg}$$

$$\rho_v = \frac{pM_{av}}{RT} = \frac{pM_{av}}{T} \times \frac{T_s}{p_s V_s} \quad (s = \text{standard conditions})$$

Average molar mass of vapour

$$M_{av} = \sum M_i y_i = 0.013 \times M_T + 0.548 \times M_{EB} + 0.439 \times M_S$$

$$M_{av} = 0.013 \times 92.141 + 0.548 \times 106.168 + 0.439 \times 104.152 = 105.1 \text{ kg/kmol}$$

$$\rho_v = \frac{21.3 \times 105.1}{363.55} \times \frac{273}{101.325 \times 22.414}$$

$$\rho_v = 0.74 \text{ kg/m}^3$$

$$\rho_L = \text{Density of liquid mixture} = \frac{1}{\sum \frac{w_i}{\rho_{Li}}}$$

For Residue

Table 6.35 Properties of Residue Components

Component	x_i	w_i (mass fraction)	ρ_{Li} , Liquid density, kg/m^3
		$w_i = \frac{x_i M_i}{\sum x_i M_i}$	
Toluene	0.005	0.0044	867
Ethyl benzene	0.475	0.48	867
Styrene	0.52	0.5156	906
Total	1.00	1.00	

$$\rho_L = \frac{1}{\frac{0.0044}{867} + \frac{0.48}{867} + \frac{0.5156}{906}}$$

$$\rho_L = 886.68 \text{ kg/m}^3$$

$$\sigma = \text{surface tension of liquid mixture} = \sigma_m$$

Surface tension of liquid mixture can be estimated by following equation.

$$\sigma_m^{1/4} = \sum [P_i] (\rho_{Lm} x_i - \rho_{Vm} y_i) \quad (\text{Eq. 3.152 of Ref. 2})$$

where, σ_m = Surface tension of liquid mixture, dyn/cm

$[P_i]$ = Parachor of component i can be obtained from Table 3.343 of Ref. 2

ρ_{Lm} = Liquid mixture density, mol /cm³

ρ_{Vm} = Vapour mixture density, mol/cm³

$[P_i]$ for toluene = 189.6 (For C₆H₅ – group) + 55.5 (For—CH₃ group).

$$[P_{\text{Toluene}}] = 245.1$$

$$[\mathcal{P}_{EB}] = 189.6 + 55.5 + 40 \text{ (For } -\text{CH}_2\text{— group)} \\ = 285.1$$

$$[\mathcal{P}_{\text{styrene}}] = 189.6 + (15.5 + 9) + 40 = 254.1$$

$$\rho_{Lm} = 886.68 \text{ kg/m}^3 \equiv \frac{886.68}{105.05} \equiv 8.44 \frac{\text{kmol}}{\text{m}^3}$$

$$= 8.44 \times \frac{10^3}{1} \times \frac{1}{10^6} \\ = 8.44 \times 10^{-3} \text{ mol/cm}^3$$

$$\rho_{Vm} = 0.74 \text{ kg/m}^3 = \frac{0.74}{105.1} \times 10^{-3} = 7.041 \times 10^{-6} \text{ mol/cm}^3$$

$$\sigma^{1/4} = \sigma_m^{1/4} = 245.1(8.44 \times 10^{-3} \times 0.005 - 7.041 \times 10^{-6} \times 0.013) \\ + 285.1(8.44 \times 10^{-3} \times 0.475 - 7.041 \times 10^{-6} \times 0.548) \\ + 254.1(8.44 \times 10^{-3} \times 0.52 - 7.041 \times 10^{-6} \times 0.439) = 2.2666$$

$$\sigma = 26.3936 \text{ dyn/cm}$$

$$\sigma = 26.3936 \times 10^2 \times \frac{1}{10^5}$$

$$\sigma = 26.3936 \times 10^{-3} \text{ N/m}$$

$$\left(\frac{\phi}{A}\right)_c = 0.44 \times \left(\frac{38.1}{25.4}\right) \times \frac{372774.9}{\sqrt{164}} \times (26.3936 \times 10^{-3} \times 9.81 \times (886.68 - 0.74) \\ \times 0.74^2)^{0.25}$$

$$\left(\frac{\phi}{A}\right)_c = 64317 \text{ W/m}^2$$

$$0.7 \left(\frac{\phi}{A}\right)_c = 45022 \text{ W/m}^2$$

$$\text{Actual heat flux, } \frac{\phi}{A} = 29833.33 \text{ W/m}^2 < 0.7 \left(\frac{\phi}{A}\right)_c$$

To find the tube bundle diameter for square pitch arrangement with U tube bundle, following equation can be written.

$$\frac{\pi}{4} D_b^2 = N_t P_t^2 + \text{Area of central portion}$$

$$\frac{\pi}{4} D_b^2 \equiv N_t P_t^2 + D_b \times P_t \quad (\text{for } P_t = 38.1 \text{ mm (1.5 in)} = \text{minimum U tube radius})$$

$$\frac{\pi}{4} D_b^2 - D_b P_t - N_t P_t^2 \equiv 0$$

$$D_b = \frac{P_t \pm \sqrt{P_t^2 + 4(\pi/4)N_t P_t^2}}{2\left(\frac{\pi}{4}\right)}$$

$$D_b = \frac{P_t + \sqrt{P_t^2 + \pi N_t P_t^2}}{(\pi/2)}$$

Minor correction in this equation:

$$D_b = \frac{P_t + \sqrt{P_t^2 + \pi N_t P_t^2}}{\pi/2} + d_o$$

$$D_b = \frac{38.1 + \sqrt{38.1^2 + \pi \times 164 \times 38.1^2}}{\pi/2} + 25.4 \text{ (82 U tubes} = 2 \times 82 = 164 \text{ tubes)}$$

$$D_b = 600.74 \approx 600 \text{ mm}$$

Ideally tube bundle diameter must be determined based on actual tube sheet layout as shown in Fig. 6.22.

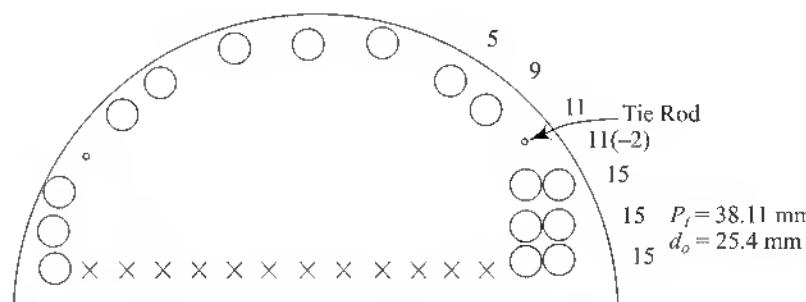


Fig. 6.22 Tube Sheet Layout Drawing for $D_b = 600$ mm, $N_t = 162$

Let the height of weir which maintains the liquid level in kettle type reboiler be 700 mm.
Shell ID, D_s :

$$D_s = 700 + 250 = 950 \text{ mm}$$

(where 250 mm is the vertical distance between shell ID and liquid level.)

$$\text{or } D_s = 1.6 D_b \text{ (From Table 6.23)}$$

$$\text{Hence, } D_s = 960 \text{ mm}$$

Based on Tube sheet layout drawing for $D_b = 600$ mm, $N_t = 162$

Let shell ID, $D_s = 960$ mm (greater of two values)

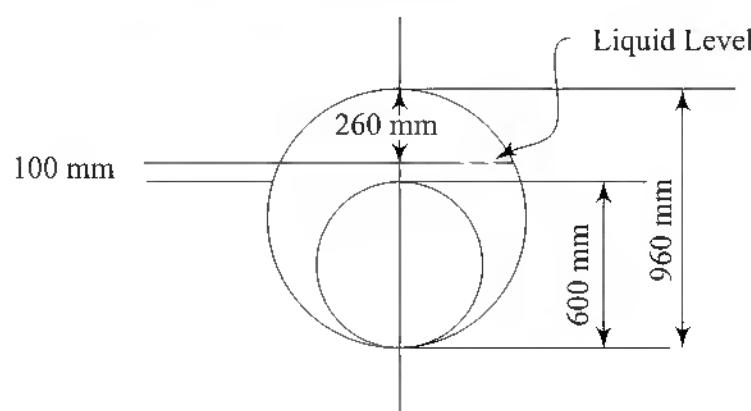


Fig. 6.23 Checking of Liquid Entrainment

Checking of liquid entrainment

To avoid the excessive liquid entrainment

$$v < v_{\max}$$

where, v = Actual vapour velocity at liquid surface, m/s

v_{\max} = Maximum permissible vapour velocity at liquid surface, m/s

$$v = \frac{(\dot{m}_v / \rho_v)}{L'x}$$

where, $L'x$ = Liquid surface area

$$L' = \text{Tube length} = \frac{3.6576}{2} \quad (\text{U tube})$$

$$L' = 1.8288 \text{ m}$$

From geometry, width of liquid surface,

$$\text{Liquid level} - \frac{960}{2} = 220$$

$$\left(\frac{x}{2}\right)^2 = \left(\frac{960}{2}\right)^2 - 220^2$$

$$x = 853.23 \text{ mm}$$

$$\begin{aligned} \text{Liquid surface area, } L'x &= 1.8288 \times 0.853 \\ &= 1.5604 \text{ m}^2 \end{aligned}$$

$$v = \frac{(\bar{V} \times \text{Average molar mass of vapour}) / \rho_v}{1.5604}$$

$$v = \frac{(65.82 \times 105.1) / 0.74}{1.5604} \times \frac{1}{3600}$$

$$v = 1.664 \text{ m/s}$$

$$v_{\max} = 0.2 \left(\frac{\rho_L - \rho_v}{\rho_v} \right)^{1/2} \quad (6.54)$$

$$v_{\max} = 0.2 \left(\frac{886.68 - 0.74}{0.74} \right)^{1/2} = 6.92 \text{ m/s}$$

$$v < v_{\max} \Rightarrow \text{There is no excessive liquid entrainment.}$$

Shell side or boiling side pressure drop Δp_s in case of kettle type reboiler is negligible.

Tube Side Pressure Drop Δp_t

On tube side, condensation of saturated steam is taking place.

$$\Delta P_t = 0.5 \Delta P_t' = 0.5 N_p \left(8 J_f \frac{L}{d_i} \left(\frac{\mu}{\mu_w} \right)^{-m} + 2.5 \right) \frac{\rho u_t^2}{2} \quad (6.28)$$

Here $N_p = 2$, $L = \frac{3.6576}{2} = 1.8288 \text{ m}$
 $d_i = 0.021\ 1836 \text{ m}$
 $\rho = \text{Density of saturated steam at } 0.28 \text{ MPa}$

$$= \frac{1}{\text{Specific volume of vapour}} = \frac{1}{0.646\ 04} = 1.548 \text{ kg/m}^3$$

Tube side mass velocity, $G_t = \frac{\dot{m}_s}{\left(\frac{N_t}{N_p}\right) \times \frac{\pi}{4} d_i^2}$

$$G_t = \frac{0.33}{\left(\frac{162}{2}\right) \times \frac{\pi}{4} (0.021\ 1836)^2} = 11.56 \text{ kg/(m}^2 \cdot \text{s)}$$

Tube side Reynolds number

$$Re = \frac{d_i G_t}{\mu} = \frac{0.021\ 1836 \times 11.56}{1350 \times 10^{-8}}$$

Viscosity of saturated steam at 404.35 K

$$\mu_v = 1350 \times 10^{-7} \text{ poise} = 1350 \times 10^{-8} \text{ kg/(m} \cdot \text{s)}$$

$$Re = 18\ 139.44$$

$$J_f = 0.0041 \text{ (From Fig. 6.13)}$$

$$\Delta p_t = 0.5 \times 2 \left(8 \times 0.0041 \left(\frac{1.8288}{0.021\ 1836} \right) \times 1 + 2.5 \right) \frac{1.548 \times 7.468^2}{2},$$

$$\left(u_t = \frac{G_t}{\rho} = \frac{11.56}{1.548} = 7.468 \text{ m/s} \right)$$

$$\Delta p_t = 230.15 \text{ Pa} \equiv 0.23 \text{ kPa (acceptable)}$$

6.7.2 Design of Vertical Thermosyphon Reboiler

As a vertical thermosyphon reboiler, BEM type fixed tube sheet heat exchanger is normally used. It is installed in the perfect vertical position. Figure 6.24 shows standard location of vertical thermosyphon reboiler in which normal level of liquid in distillation column is same as level of top tube sheet of heat exchanger. Inside the tubes liquid is partially vaporized by using the heating medium like saturated steam or hot oil on shell side. It decreases the density of fluid (vapour - liquid mixture) in "hot" leg compared to the density of saturated liquid in relatively "cold" leg and starts natural fluid circulation in the anticlockwise direction as shown in Fig. 6.24 by arrows. In this type of reboiler, syphoning effect is created by heating (thermally), hence it is called thermosyphon reboiler.

HTRI and HTFS have developed their own methods for the process design of thermosyphon reboiler which are not available in open literature. The oldest and

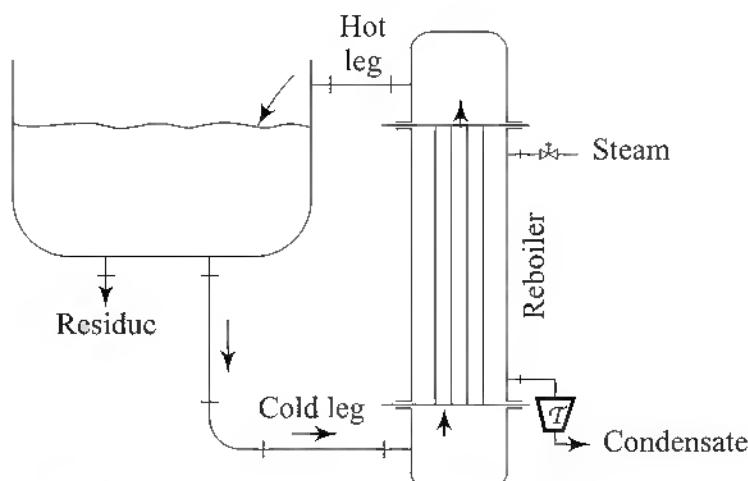


Fig. 6.24 Standard Arrangement of Vertical Thermosyphon Reboiler

the simplest method for the process design of vertical thermosyphon reboiler is Kern's method, which is outlined as follows.

- (i) Calculate the heat duty of reboiler based on energy balance equation for distillation column.

$$\phi_B = \phi_C + H_D D + H_W W - H_F F + \phi_L \quad (6.53)$$

If vaporization rate is given then heat duty can be determined by Eq. (6.54).

$$\phi_B = m_v \lambda \times 1.05 \quad (6.54)$$

- (ii) Fix the value of mean temperature difference, ΔT_m . It should be less than and close to critical temperature drop. Then, average temperature of heating medium $T_h = \Delta T_m + T_b$.
- (iii) Select the suitable heating medium. Based on the energy balance, find the mass flow rate of heating medium required.
- (iv) Assume the value of overall heat transfer coefficient, $U_o = 1000 \text{ W}/(\text{m}^2 \cdot ^\circ\text{C})$. Find the heat transfer area based on assumed value of U_o .

$$A_o = \frac{\phi_B}{U_o \Delta T_m} = A_{pro} = N_t \pi d_o L \quad (6.43)$$

Find or decide the required dimension of the heat exchanger.

- (v) Assume the recirculation ratio equal to 4 for the first trial.

$$\text{Recirculation ratio} = \frac{\text{Mass flow rate of liquid recirculated}}{\text{Vaporization rate}}$$

$$\text{or Recirculation ratio} = \frac{\dot{m}_{Lo}}{\dot{m}_{vo}} \quad (6.65)$$

where, \dot{m}_{Lo} = Mass flow rate of liquid at outlet of reboiler

\dot{m}_{vo} = Mass flow rate of vapour at outlet of reboiler.

- (vi) Do the pressure balance. At steady state,
Available differential head = Pressure loss in the circuit
or $\Delta p_{av} = \text{Pressure loss in the base of distillation column}$
+ Pressure loss in associated piping and nozzles
+ Pressure loss in reboiler (i.e. tube side pressure drop)

Velocity of liquid through the shell of distillation column is very low compared to the velocity of liquid through pipes. Hence, pressure loss in the base of distillation column may be neglected. If piping arrangement is smooth and higher sizing for the pipes is provided, then pressure loss in piping may also be neglected. Hence, $\Delta p_{av} \approx \Delta p_t$ at steady state. Δp_t = Tube side pressure drop (loss) of reboiler. Available differential head, Δp_{av} is calculated by following equation.

$$\Delta p_{av} = L (\rho_L - \rho_{av}) g \quad (6.66)$$

where, Δp_{av} = Available differential head. Pa

L = Length of tube, m

ρ_L = Density of liquid in cold leg, kg/m³

ρ_{av} = Average density of liquid vapour mixture in hot leg, kg/m³

g = Acceleration of gravity = 9.81 m/s²

$$\rho_{av} = \frac{\ln(V_o/V_i)}{V_o - V_i} \quad (6.67)$$

where, V_i = Specific volume of saturated liquid at the inlet of reboiler, m³/kg

$$V_i = 1/\rho_L$$

V_o = Specific volume of liquid vapour mixture at the outlet of reboiler, m³/kg

$$V_o = \frac{(\dot{m}_{vo}/\rho_v) + (\dot{m}_{Lo}/\rho_L)}{(\dot{m}_{vo} + \dot{m}_{Lo})} \quad (6.68)$$

where, $\dot{m}_{vo}, \dot{m}_{Lo}$ = Mass flow rates of vapour phase and liquid phase at the outlet of reboiler, kg/s

In Kern's method, it is suggested to find Δp_t by same equation that is used for no phase change in the tube side fluid [Eq. (6.27)]. In this equation, $\rho = \rho_{av}$ = average density of tube side fluid.

- (vii) Compare the value of available differential head Δp_{av} and pressure drop in the system or tube side pressure drop, (Δp_t), obtained based on assumed value of recirculation ratio. There are total five possibilities.

(a) $\Delta p_{av} \approx \Delta p_t$

(b) $\Delta p_{av} > \Delta p_t$

(c) $\Delta p_{av} > > \Delta p_t$

(d) $\Delta p_{av} < \Delta p_t$

(e) $\Delta p_{av} < < \Delta p_t$

(a) $\Delta p_{av} \approx \Delta p_t$ implies that the assumed value of recirculation ratio (4 for 1st trial calculation) is equal to the actual value of recirculation ratio. Proceed for the next step of calculation.

(b) $\Delta p_{av} > \Delta p_t$ implies that the assumed value of recirculation ratio is less than the actual recirculation ratio. Hence, assume the higher value of recirculation ratio, repeat the calculations and again compare the new values of Δp_{av} and Δp_t . By trial and error calculations, finally one can find the recirculation ratio for which $\Delta p_{av} \approx \Delta p_t$. Recirculation ratio can have a fractional value.

- (c) $\Delta p_{av} > \Delta p_t$ or $\Delta p_{av}/\Delta p_t > 50$ implies that the assumed value of recirculation ratio is very much less than actual recirculation ratio. In this case also, one can find the value of recirculation ratio for which $\Delta p_{av} \cong \Delta p_t$, by trial and error calculations but very high recirculation ratio and hence very high tube side pressure drop may be obtained which cannot be permitted. Very high tube side pressure drop will result in significant boiling point elevation inside the tubes of the reboiler and decrease the mean temperature difference. Very high tube side velocity gives erosion and vibrations in tubes. Hence, in such a case one of the following options can be considered.
- (i) Provide the control valve with flow meter in the inlet line to the reboiler. Then, pressure balance is $\Delta p_{av} = \Delta p_t + \text{Pressure loss in flow meter} + \text{Pressure drop offered by control valve}$. Pressure drop of control valve can be adjusted to the desired value. This option also provides flexibility in the actual operation. In actual plant operation, recirculation ratio or circulation rate can be corrected depending on the actual performance. This facility is not available with other options.
- (ii) Elevate the level of top tube sheet of reboiler above the liquid level at the base of distillation column as shown in Fig. 6.25. In this option, $\Delta p_{av} = L'(\rho_L - \rho_{av})g$. Hence, this modification decreases the value of available differential head Δp_{av} and may equate it with Δp_t at the lower value or reasonable value of recirculation ratio.
- (d) If $\Delta p_{av} < \Delta p_t$, then one of the following option can be considered.
- (i) Increase the number of tubes and decrease the tube length. This modification will decrease the values of both Δp_{av} and Δp_t . But, the percentage decrease in the value of Δp_t will be more compared to the same in the value of Δp_{av} . Hence, it may be possible to equate Δp_{av} and Δp_t for the lower value of recirculation rate.
- (ii) $\Delta p_{av} < \Delta p_t$, implies that the actual recirculation ratio is less than assumed or minimum recommended value of recirculation ratio. In this case actual recirculation ratio can be increased by elevating liquid level at the base of the distillation column above the top tube sheet of reboiler as shown in Fig. 6.26.

In this case equation of Δp_{av} is changed.

$$\Delta p_{av} = \rho gh + L(\rho_L - \rho_{av})g$$

But this modification may increase the height of skirt support of distillation column. If increase in the height of skirt support is not possible, then one can go for underground construction. If none of these options

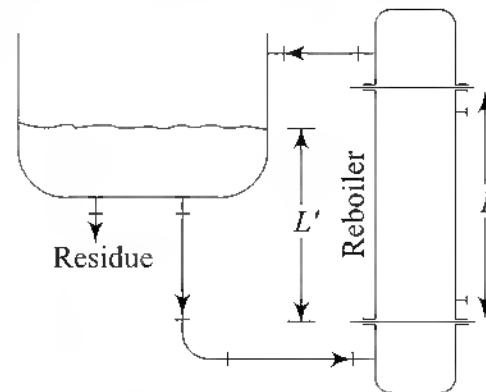


Fig. 6.25 Increase of Elevation of Reboiler

is permitted, then use pump in the inlet line to the reboiler to increase the circulation rate. Then this reboiler is called a forced circulation reboiler.

- (e) $\Delta p_{av} \ll \Delta p_t$ implies that the actual recirculation ratio is very low or very much less than minimum recommended value, i.e. 4. This is possible when the viscosity of bottom product of distillation column is very high. In this case use a pump in the inlet line to the reboiler to get the desired value of circulation rate and desirable value of tube side heat transfer coefficient. But then this reboiler becomes a forced circulation reboiler.

- (viii) After finding the recirculation ratio for which available differential head becomes nearly equal to pressure loss in the system, calculate the tube side heat transfer coefficient. In the latest method tube length is divided in different sections. For the different sections, different regimes of two phase flow are considered. Hence for the different sections different values of heat transfer coefficients are calculated by different correlations (Ref. 10, 11 & 12). But in the simplified Kern's method, it is suggested to use the same forced convection correlation which are actually derived for the sensible heat transfer (cooling or heating) of tube side fluid. Hence, in Kern's method, h_i is determined by Eq. (6.18) or (6.19). In addition to that Kern has specified the maximum limit for the value of h_i as under.

If $h_i \geq 300 \text{ Btu}/(\text{h} \cdot \text{ft}^2 \cdot ^\circ\text{F}) [= 1700 \text{ W}/(\text{m}^2 \cdot ^\circ\text{C})]$
take $h_i = 1700 \text{ W}/(\text{m}^2 \cdot ^\circ\text{C})$

- (ix) Calculate the shell side or heating medium side heat transfer coefficient. If shell side heating medium is saturated steam then take

$$h'_o = 6000 \text{ W}/(\text{m}^2 \cdot ^\circ\text{C})$$

This figure also includes fouling resistance. If hot oil is used as heating medium, then use the Eq. (6.35) or Eq. (6.36) for the calculation of h_o .

- (x) Determine the overall heat transfer coefficient by Eq. (6.42).
- (xi) Calculate the required heat transfer area.

$$A_{\text{req}} = \frac{\phi_b}{U_o \Delta T_m} \quad (6.43)$$

Heat transfer area provided $A_{\text{opro.}} = N_t \pi d_o L$
Find the % Excess heat transfer area.

$$\% \text{ Excess heat transfer area} = \left(\frac{A_{\text{opro.}}}{A_{\text{req}}} - 1 \right) \times 100$$

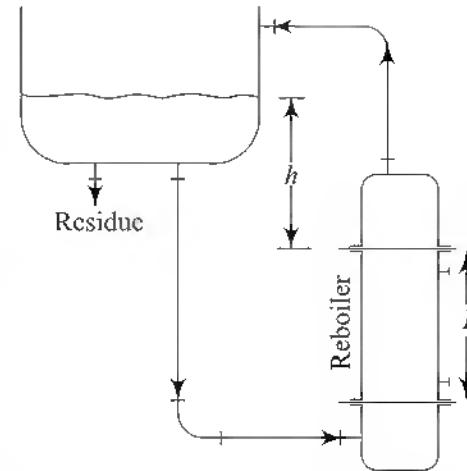


Fig. 6.26 Decrease of Elevation of Reboiler

It should be in between 10 to 20%. If it is not, then change the value of A_{opro} , either by increasing the tube length or by increasing number of tubes. An increase in tube length will keep the tube side heat transfer coefficient unchanged but it will increase the value of Δp_t . Increase in number of tubes will decrease the value of h_i and also of h_o , if hot oil is used as heating medium but it will decrease the value of Δp_t . In any case, both pressure balance calculations and heat transfer coefficient calculations must be repeated.

- (xii) Find the shell side pressure drop Δp_s . It should be less than the maximum allowable value.

Example 6.6

Design a vertical thermosyphon reboiler which must provide 10 000 kg/h of *n*-pentane vapour to a distillation tower at 10 bar a pressure.

Solution:

Determination of boiling point of pentane:

Antoine equation⁴ for *n*-pentane

$$\ln p_v = A - \frac{B}{(T + C)}, \text{ where, } p_v \text{ in torr, } T \text{ in K}$$

$$\ln p_v = 15.8333 - \frac{2477.07}{T - 39.94}$$

$$p_v = p_t = 10 \text{ bar} = 7500.6 \text{ torr}$$

$$T = 398.38 \text{ K} \equiv 125.4^\circ\text{C}$$

Heat duty, $\phi = \dot{m}\lambda \times 1.05$ (5% heat loss)

where, λ = Latent heat of vaporization at the boiling point

Latent heat of vaporization of pentane at 125.4°C:

At 25°C, $\lambda_{v1} = 366.512 \text{ kJ/kg}$

Critical temperature of pentane, $t_c = 197^\circ\text{C}$, (Ch-3, of Ref. 2)

From Watson's equation,

$$\begin{aligned} \lambda_2 &= \lambda_1 \left(\frac{1 - T_{t_2}}{1 - T_{t_1}} \right)^{0.38} \\ &= 366.512 \left(\frac{1 - \left(\frac{125.4 + 273}{197 + 273} \right)}{1 - \left(\frac{25 + 273}{197 + 273} \right)} \right)^{0.38} = 366.512 \times 0.7167 \\ &= 262.68 \text{ kJ/kg} \end{aligned}$$

Heat duty, $\phi = 1.05 \times 10000 \times 262.68$
 $\phi = 2758140 \text{ kJ/h} \equiv 766.15 \text{ kW}$

Let $\Delta T_m = 39.6^\circ\text{C}$. Assume that this value of ΔT_m is less than and close to the critical temperature drop.

Average temperature of heating medium

$$\bar{T}_h = T_b + \Delta T = 125.4 + 39.6 = 165^\circ\text{C}$$

From Steam Tables, for the steam temperature $t_s = 165^\circ\text{C}$, saturation steam pressure = 700 kPa and latent heat of condensation of steam

$$\lambda_s = 2064.9 \text{ kJ/kg}$$

$$\phi_t = \dot{m}_s \lambda_s$$

$$\dot{m}_s = \frac{766.15}{2064.9} = 0.371 \text{ kg/s} = 1335.6 \text{ kg/h}$$

For the first trial calculations, assuming overall heat transfer coefficient $U_o = 1000 \text{ W/(m}^2 \cdot ^\circ\text{C)}$

$$A_{\text{pro}} = \frac{\phi_t}{U_o \Delta T_m} = \frac{766.15 \times 10^3}{1000 \times 39.6}$$

$$A_{\text{pro}} = 19.3472 \text{ m}^2 = N_t \pi d_o L$$

Let length of tube $L = 6 \text{ ft} = 1.8288 \text{ m}$

$$d_o = 25.4 \text{ mm}$$

$$19.3472 = N_t \times \pi \times (0.0254) \times 1.8288$$

$$N_t = 132.57 \approx 133 \text{ tubes}$$

For 25.4 mm OD tube, 31.75 mm tube pitch ($P/d_o = 1.25$) triangular pitch arrangement and 1-1 shell and tube heat exchanger,

Shell ID, $D_i = 438 \text{ mm}$ (From Table 6.1 (f))

Assume recirculation ratio = 4

Available differential head

$$\Delta P_{\text{av}} = L(\rho_L - \rho_{\text{av}})g \quad (6.66)$$

ρ_L = Density of liquid pentane at $125.4^\circ\text{C} = 626 \text{ kg/m}^3$

Density of pentane vapour at 10 bar a pressure and at 125.4°C

$$\rho_v = \frac{pM}{RT} = \frac{10 \times 72}{(273 + 125.4)} \times \frac{273}{1.01325 \times 22.414}$$

$$\rho_v = 21.724 \text{ kg/m}^3$$

Average density of liquid-vapour mixture

$$\rho_{\text{av}} = \frac{\ln(V_o/V_i)}{V_o - V_i} \quad (6.67)$$

V_i = Specific volume of liquid at inlet

$$V_i = \frac{1}{\rho_L} = 1.59744 \times 10^{-3} \text{ m}^3/\text{kg}$$

For a recirculation ratio = 4

Flow rate of liquid at the outlet of reboiler

$$\dot{m}_{Lo} = 4 \times 10000 \text{ kg/h} = 40000 \text{ kg/h}$$

Flow rate of vapour at the outlet of reboiler $\dot{m}_{vo} = 10000 \text{ kg/h}$

Specific volume of vapour at outlet

$$V_o = \frac{\dot{m}_{vo}/\rho_v + \dot{m}_{Lo}/\rho_L}{\dot{m}_{vo} + \dot{m}_{Lo}} = \frac{\frac{10\ 000}{21.724} + \frac{40\ 000}{626}}{50\ 000}$$

$$V_o = 0.010\ 4843 \text{ m}^3/\text{kg}$$

$$\rho_{av} = \frac{\ln\left(\frac{0.010\ 4843}{1.597\ 44 \times 10^{-3}}\right)}{0.010\ 4843 - 1.597\ 44 \times 10^{-3}} = 211.715 \text{ kg/m}^3$$

$$\Delta P_{av} = 1.8288 \times (626 - 211.715) \times 9.81 = 7432.5 \text{ Pa}$$

Assume that higher sizes of the piping will be used in between base of distillation column and thermosyphon reboiler.

Pressure loss in the system \equiv Tube side pressure drop, Δp_t
Tube side pressure drop,

$$\Delta p_t = N_p \left(8 J_f \left(\frac{L}{d_i} \right) \left(\frac{\mu}{\mu_w} \right)^{-m} + 2.5 \right) \frac{\rho_{av} u_t^2}{2} \quad (6.27)$$

$$N_p = 1, \frac{\mu}{\mu_w} \approx 1, \rho_{av} = 211.715 \text{ kg/m}^3$$

Tube inside diameter

$$d_i = 22.098 \text{ mm (16 BWG tube)}$$

Tube inside flow area

$$a_t = \frac{N_t}{N_p} \times \frac{\pi}{4} d_i^2 = \frac{133}{1} \times \frac{\pi}{4} (0.022\ 098)^2 = 0.051 \text{ m}^2$$

Tube side mass velocity

$$G_t = \frac{\dot{m}}{a_t} = \frac{\dot{m}_{vo} + \dot{m}_{Lo}}{a_t} = \frac{50\ 000 \times \frac{1}{3600}}{0.051}$$

$$= 272.33 \text{ kg/(m}^2 \cdot \text{s)}$$

$$\text{Tube side velocity, } u_t = \frac{G_t}{\rho_{av}} = \frac{272.33}{211.715}$$

$$= 1.2863 \text{ m/s}$$

$$Re = \frac{d_i G_t}{\mu_L} = \frac{0.022\ 098 \times 272.33}{0.12 \times 10^{-3}} = 50\ 149.6$$

(Kern suggests to use liquid viscosity, instead of viscosity of liquid-vapour mixture)

$$J_f = 3.2 \times 10^{-3} \text{ (From Fig. 6.13)}$$

$$\Delta p_t = \left(8 \times 3.2 \times 10^{-3} \times \left(\frac{1.8288}{0.022\ 098} \right) + 2.5 \right) \times \frac{211.715 \times 1.2863^2}{2}$$

$$\Delta p_t = 808.94 \text{ Pa}$$

$$\Delta p_a > \Delta p_t$$

Assume the higher value of recirculation ratio (greater than 4) and by trial and error calculations find the value of recirculation ratio for which $\Delta p_{av} \cong \Delta p_t$

Trial I: Assume circulation ratio = 15.5

$$\dot{m}_{Lo} = 15.5 \times 10\ 000 = 155\ 000 \text{ kg/h}, \dot{m}_{vo} = 10\ 000 \text{ kg/h}$$

$$V_o = \frac{\left(\frac{10\ 000}{21.724}\right) + \left(\frac{155\ 000}{626}\right)}{165\ 000} = 4.2904 \times 10^{-3} \text{ m}^3/\text{kg}$$

$$V_t = 1.597\ 44 \times 10^{-3} \text{ m}^3/\text{kg}$$

$$\rho_{av} = \frac{\ln((4.2904 \times 10^{-3})/(1.597\ 44 \times 10^{-3}))}{(4.2904 \times 10^{-3} - 1.597\ 44 \times 10^{-3})} = 366.874 \text{ kg/m}^3$$

$$\Delta p_{av} = 1.8288 \times (626 - 366.874) \times 9.81 = 4648.9 \text{ Pa}$$

$$\text{Tube side mass velocity, } G_t = \frac{\left(\frac{165\ 000}{3600}\right)}{0.051} = 898.693 \text{ kg}/(\text{m}^2 \cdot \text{s})$$

Tube side Reynolds number

$$Re = \frac{d_i G_t}{\mu_L} = \frac{0.022\ 098 \times 898.693}{0.12 \times 10^{-3}} = 165\ 494$$

Tube side fraction factor, $J_f = 2.64 \times 10^{-3}$ (From Fig. 6.13)

$$\text{Tube side velocity } u_t = \frac{G_t}{\rho_{av}} = \frac{898.693}{366.874} = 2.45 \text{ m/s}$$

Tube side pressure drop

$$\begin{aligned} \Delta p_t &= Np \left(8 J_f \left(\frac{L}{d_i} \right) \left(\frac{\mu}{\mu_w} \right)^{-m} + 2.5 \right) \frac{\rho_{av} u_t^2}{2} \\ &= 1 \left(8 \times 2.64 \times 10^{-3} \times \left(\frac{1.8288}{0.022\ 098} \right) \times 1 + 2.5 \right) \times \frac{366.874 \times 2.45^2}{2} \\ &= 4677.2 \text{ Pa} \end{aligned} \quad (6.27)$$

$$\Delta p_{av} \cong \Delta p_t$$

Hence recirculation ratio of 15.5 is acceptable.

Heat transfer coefficients:

(a) Tube side heat transfer coefficient

$$Re = 165\ 494$$

$$Pr = \frac{C_L \mu_L}{k_L} \quad (\text{Kern suggests to use liquid properties})$$

Properties of liquid pentane at 125.4°C:

Specific heat $C_L = 2.2483 \text{ kJ}/(\text{kg} \cdot ^\circ\text{C})$

Viscosity $\mu_L = 0.12 \text{ cP}$

Thermal conductivity, $k_L = 0.124\ 56\ \text{W}/(\text{m} \cdot ^\circ\text{C})$

$$\begin{aligned} Pr &= \frac{C_L \mu_L}{k_L} = \frac{2.2483 \times 0.12 \times 10^{-3} \times 10^3}{0.124\ 56} \\ &= 2.166 \end{aligned}$$

$$\frac{h_i d_i}{k} = 0.023 \ Re^{0.8} \ Pr^{0.33} \left(\frac{\mu}{\mu_w} \right)^{0.14} \quad (6.19)$$

$$h_i = 0.023 \times \frac{0.124\ 56}{0.022\ 098} \times (165\ 494)^{0.8} \times (2.166)^{0.33} \times 1$$

$$h_i = 2503.5\ \text{W}/(\text{m}^2 \cdot ^\circ\text{C}) \geq 1702.6\ \text{W}/(\text{m}^2 \cdot ^\circ\text{C}) \text{ Take } h_i = 1702.6\ \text{W}/(\text{m}^2 \cdot ^\circ\text{C})$$

Shell side heat transfer coefficient $h_o' = 6000\ \text{W}/(\text{m}^2 \cdot ^\circ\text{C})$

(This value includes fouling resistance)

$$\frac{1}{U_o} = \frac{1}{h_o'} + \frac{d_o \ln\left(\frac{d_o}{d_i}\right)}{2 k_w} + \frac{d_o}{d_i} \times \frac{1}{h_i} + \frac{d_o}{d_i} \times \frac{1}{h_{id}} \quad (6.42)$$

Thermal conductivity of tube material $k_w = 45\ \text{W}/(\text{m} \cdot \text{K})$ (mild steel tubes).

Tube side fouling coefficient $h_{id} = 5000\ \text{W}/(\text{m}^2 \cdot ^\circ\text{C})$

$$\frac{1}{U_o} = \frac{1}{6000} + \frac{0.0254 \ln\left(\frac{25.4}{22.098}\right)}{2 \times 45} + \frac{25.4}{22.098} \times \frac{1}{1702.6} + \frac{25.4}{22.098} \times \frac{1}{5000}$$

$$U_o = 900.13\ \text{W}/(\text{m}^2 \cdot ^\circ\text{C})$$

Heat transfer area required

$$A_{req} = \frac{\phi}{U_o \Delta T_m} = \frac{766.15 \times 1000}{900.13 \times 39.6} = 21.494\ \text{m}^2$$

$$\frac{A_{pro}}{A_{req}} = \frac{19.3472}{21.494} = 0.9 < 1.1$$

Assume new ratio of 1.2.

Revising tube length $L_{rev} = 6 \times \frac{1.2}{0.9} = 8\ \text{ft}$

$$L_{rev} = 8\ \text{ft} = 2.4384\ \text{m}$$

$$A_{pro} = N_t \pi d_o L = 133 \times \pi \times 0.0254 \times 2.4384 = 25.8786\ \text{m}^2$$

For the new tube length and with recirculation ratio = 15.5

$$\Delta p_{av} = 4648.9 \times \frac{2.4384}{1.8288} = 6198.53\ \text{Pa}$$

Tube side pressure drop,

$$\Delta p_t = 5182.63\ \text{Pa}$$

$\Delta p_{av} > \Delta p_t$, hence assume the higher value of recirculation ratio.

Trial: For the recirculation ratio = 17

$$\dot{m}_{Lo} = 17 \times 10\ 000 = 170\ 000 \text{ kg/h}, \dot{m}_{vo} = 10\ 000 \text{ kg/h}$$

Specific volume of liquid-vapour mixture at the outlet of reboiler

$$V_o = \frac{\frac{170\ 000}{626} + \frac{10\ 000}{21.724}}{180\ 000} = 4.066 \times 10^{-3} \text{ m}^3/\text{kg}$$

$$\ln\left(\frac{4.066 \times 10^{-3}}{1.597\ 44 \times 10^{-3}}\right)$$

$$\text{Average density, } \rho_{av} = \frac{(4.066 \times 10^{-3} - 1.597\ 44 \times 10^{-3})}{(4.066 \times 10^{-3} + 1.597\ 44 \times 10^{-3})} = 378.46 \text{ kg/m}^3$$

$$\Delta p_{av} = 2.4384 \times (626 - 378.46) \times 9.81 = 5921.33 \text{ Pa}$$

Tube side pressure drop:

Tube side mass velocity

$$G_t = \frac{180\ 000/3600}{0.051} = 980.392 \text{ kg/(m}^2 \cdot \text{s)}$$

$$Re = \frac{d_i G_t}{\mu} = \frac{0.022\ 098 \times 980.392}{0.12 \times 10^{-3}}$$

$$= 180\ 539.2$$

$$J_f = 2.4 \times 10^{-3}$$

$$u_t = \frac{G_t}{\rho_{av}} = 2.59 \text{ m/s}$$

$$\Delta p_t = 1 \times \left(8 \times 2.4 \times 10^{-3} \times \left(\frac{2.4384}{0.022\ 098} \right) + 2.5 \right) \times \frac{378.46 \times 2.59^2}{2} = 5862.75 \text{ Pa}$$

$$\Delta p_{av} \equiv \Delta p_t$$

For the recirculation ratio = 17

$$Re_i = 180\ 539.2$$

$$h_i = 2503.5 \times \left(\frac{180\ 539.2}{165\ 494} \right)^{0.8} = 2684 \text{ W/(m}^2 \cdot {^\circ}\text{C)} \geq 1702.6 \text{ W/(m}^2 \cdot {^\circ}\text{C)}$$

Take $h_i = 1702.6 \text{ W/(m}^2 \cdot {^\circ}\text{C)}$

Heat transfer area required $A_{red} = 21.494 \text{ m}^2$

$$\% \text{ Excess heat transfer area} = \left(\frac{A_{pro.}}{A_{req}} - 1 \right) \times 100$$

$$= \left(\frac{25.8786}{21.494} - 1 \right) \times 100 = 20.4\%$$

Shell side pressure drop Δp_s :

For the condensation of saturated steam, shell side pressure drop is calculated by equation

$$\Delta P_s = 0.5 \times 8 \times J_f \left(\frac{D_s}{d_e} \right) \left(\frac{L}{B_s} \right) \frac{\rho u_s^2}{2} \quad (6.41)$$

Shell side Friction Factor, J_f :

To find this factor, first shell side flow area must be determined.

$$A_s = \frac{(P_t - d_o) D_s B_s}{P_t} \quad (6.29)$$

Let B_s = Baffle spacing = D_s = Shell inside diameter

$B_s = D_s = 0.438 \text{ m}$, $d_o = 25.4 \text{ mm}$, $P_t = 31.75 \text{ mm}$

$$A_s = \frac{31.75 - 25.4}{31.75} \times 0.438^2 = 0.03837 \text{ m}^2$$

$$\text{Shell side mass velocity } G_s = \frac{\dot{m}_s}{A_s} = \frac{0.371}{0.03837} = 9.67 \text{ kg/(m}^2 \cdot \text{s)}$$

$$\text{Density of steam } \rho_v = \frac{1}{\text{Specific volume}}$$

From Steam Tables specific volume of saturated steam at 700 kPa pressure, v_s

$$= 0.27268 \text{ m}^3/\text{kg}$$

$$\rho_v = 3.6673 \text{ kg/m}^3$$

$$u_s = \frac{G_s}{\rho_v} = \frac{9.67}{3.6673} = 2.64 \text{ m/s}$$

$$\text{Equivalent diameter, } d_e = \frac{1.1}{d_o} (P_t^2 - 0.907 d_o^2) \quad (6.29)$$

$$d_e = \frac{1.1}{0.0254} (0.03175^2 - 0.907(0.0254)^2)$$

$$d_e = 0.0183 \text{ m}$$

Shell side Reynolds number:

$$Re_s = \frac{d_e G_s}{\mu}$$

Viscosity of steam at 165°C and at 700 kPa pressure $\mu_v = 14.5 \times 10^{-6} \text{ (N} \cdot \text{s})/\text{m}^2$
(Table 3.302, of Ref. 2)

$$Re_s = \frac{0.0183 \times 9.67}{14.5 \times 10^{-6}} = 12204.2$$

$$J_f = 4.78 \times 10^{-2} \text{ (For 25% baffle cut)} \quad (\text{Ref. Fig. 6.15})$$

$$\Delta p_s = 0.5 \times 8 \times 4.78 \times 10^{-2} \left(\frac{0.438}{0.0183} \right) \left(\frac{2.4384}{0.438} \right) \times \frac{3.6673 \times 2.64^2}{2}$$

$$\Delta p_s = 325.6 \text{ Pa} = 0.326 \text{ kPa} << 13.8 \text{ kPa}$$

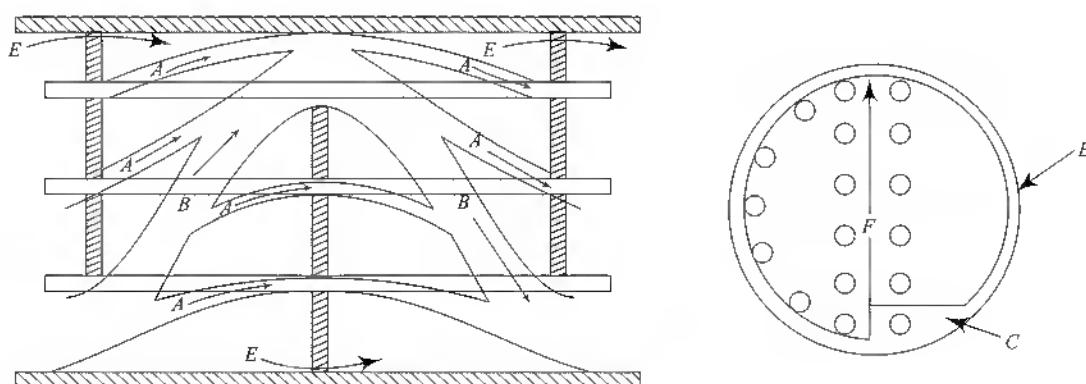
It is well within permissible limit.

6.7.3 Criteria of Selection between Kettle Type Reboiler and Thermosyphon Reboiler

- (i) Boiling fluid side (process fluid side) pressure drop in kettle type reboiler is negligible. While the same is high and significant in thermosyphon reboiler. Hence with vacuum distillation, kettle type reboiler is preferred. Thermosyphon reboiler is not preferred for use with absolute pressure below 0.3 bar. For example, in separation of the mixture of monochloroacetic acid and acetic acid by distillation, kettle type reboiler is the only choice. Cracking temperature of monochloroacetic acid is 120°C and is obtained as the bottom product. Vapour pressure of monochloroacetic acid at 118°C is 60 torr. Hence, to restrict the boiling point of monochloroacetic acid below 120°C, pressure drop in distillation column plus pressure drop in overhead condenser plus pressure drop in reboiler plus pressure drop in associated piping must be less than 60 torr (0.08 bar). So, kettle type reboiler is the only choice.
- (ii) If the viscosity of bottom product of distillation column is very high, then thermosyphon reboiler cannot be used. In such a case forced circulation reboiler is preferred.
In fatty acid separation in a distillation tower under vacuum, forced circulation reboiler is used due to viscous nature of fatty acids. However, for the vacuum and corrosive system with highly viscous bottom product kettle type reboiler is used. For example: separation of pure POCl_3 (phosphorous oxytrichloride) from treated crude POCl_3 , residue or bottom product of distillation is highly viscous. Thermosyphon reboiler cannot be used for this application.
- (iii) With atmospheric and high pressure distillation column thermosyphon reboilers are preferred as size and fixed cost of thermosyphon reboiler is less than the same of kettle type reboiler. Because of the fluid circulation, thermosyphon reboiler provides higher heat transfer coefficient than kettle type reboiler. Even for the same value of heat transfer area, fixed cost of kettle type reboiler is higher than the same of thermosyphon reboiler.
- (iv) If the ratio of vaporization rate required to mass flow rate of residue (bottom product of distillation column) is 0.8 or above, kettle type reboiler is preferred. Kettle type reboilers are also used as "Vaporizers" in industry. Thermosyphon reboiler cannot be used as vaporizer. Separation taking place in kettle type reboiler can be considered as equivalent to one theoretical stage. If Murphree efficiency of the column is 50% (approx.), it can result in reduction of two trays.

6.8 TINKER'S FLOW MODEL

All the latest methods (Ex. Bell's method), for the process design of without phase change heat exchangers are based on Tinker's flow model. If there is no phase change in shell side fluid then shell side heat transfer coefficient and shell side pressure drop in all recent methods are calculated based on this model. In

Fig. 6.27 Tinker's Flow Model^{4,13}

old methods like Kern's method it is assumed that entire shell side fluid is flowing across the tube bundle and between the baffles. But actually shell side fluid is flowing in various ways. In Tinker's flow model shell side flow is divided in five different streams.

1. Stream A

Stream A is the tube-to-baffle leakage stream or it is the fraction of shell side fluid flowing through the clearance between tube hole in baffle and tube outside diameter. This stream does not bypass the heat transfer area (outside area of tubes) and hence it does not create any adverse effect on the value of heat transfer coefficient. However, it makes the significant difference in pressure drop (loss). When stream A leaves this clearance it forms free flowing jet. Hence, boundary layer separation occurs and considerable friction loss or pressure drop takes place. Effect of stream A must be considered in the calculation of pressure drop.

2. Stream B

Stream B is the actual cross flow stream or it is the fraction of shell side fluid which is flowing across the tube bundle and between the baffles. In Kern's method and other old methods it is assumed that entire shell side fluid is flowing like stream B.

3. Stream C

Stream C is bundle to shell bypass stream or it is the fraction of shell side fluid flowing through the clearance area between shell inside diameter and tube bundle. Stream C is the main bypass stream. (bypassing the heat transfer area). This clearance area provides low pressure drop path for the shell side fluid. Hence, % of shell side fluid bypassed through this clearance area is quite significant. It is maximum with pull through floating head heat exchanger and minimum with fixed tube sheet heat exchanger. Amount of stream C can be reduced considerably by using sealing strips which are attached on inside surface of shell. They provide the partial blockage or the additional resistance in the path of stream C.

There is no stream designated as stream "D".

4. Stream E

Stream E is the baffle to shell leakage stream. It is a part of shell side fluid flowing through the clearance between the edge of baffle and shell wall. Like stream C, this stream is also bypassing the heat transfer area and hence reduces shell

side heat transfer coefficient. But amount of stream E is lesser than amount of stream C . Normally the clearance between baffle outside diameter and shell inside diameter is in the range of 1.6 to 4.8 mm while clearance between tube bundle and shell inside diameter is in the range of 10 to 100 mm.

5. Stream F

Stream F is the pass-partition bypass stream. In tube sheets where pass partition plates are attached, in that portion tubes can not be provided. In multipass heat exchangers one can find more number of gaps in tube bundle. Stream F is the fraction of shell side fluid flowing through these gaps. If the gap is vertical it provides low pressure drop path for fluid flow. Just like stream C and stream E , this stream is also bypassing the heat transfer area and reduces shell side heat transfer coefficient. Amount of stream F and its adverse effects are significant in multipass heat exchanger. To reduce the amount of this stream sometimes dummy tubes are used.

6.9 AIR COOLED HEAT EXCHANGERS AND AIR HEATERS

6.9.1 Introduction

Atmospheric air (having temperature varying from 20 to 45°C, depending on season) can be used as cooling medium if the process fluid is to be cooled up to 60°C. If it is to be cooled below 60°C, then atmospheric air cannot be used as cooling medium, as minimum driving force required for heat transfer in air-cooled heat exchanger is 10°C.

Advantages and disadvantages of air as a cooling medium over water as a cooling medium are given in previous section. (Sec. 6.3.2.3)

As shown in Fig. 6.28, air cooled heat exchanger consists of banks of finned tubes over which air is blown by forced draft fan or drawn by an induced draft fan. Forced draft fan is mounted below and induced draft fan is mounted above the tube bundle. Tube bundle consists of tubes with spiral wound transverse fins in the majority of applications. Forced draft unit consumes less power. Induced draft provides comparatively even distribution of air across the bundle since the

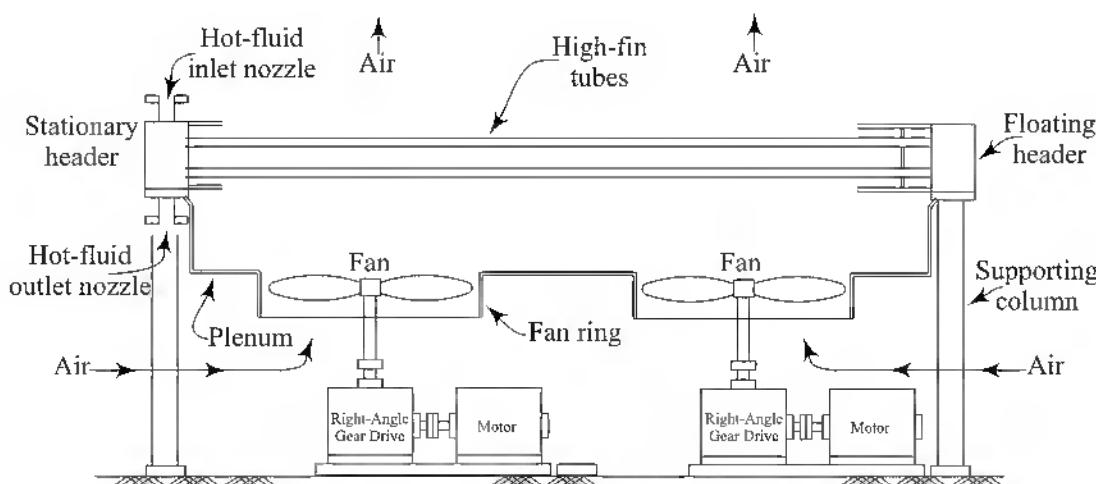


Fig. 6.28 Forced Draft Air Cooled Heat Exchanger

air velocity approaching the bundle is relatively low. Percentage recirculation of hot exhaust air is less in induced draft unit compared to the forced draft unit since the exist air velocity is several times that of forced draft unit. Induced draft arrangement gives more protection to heat transfer surface against rain fall, snow fall, sleet, etc.

If the difference between process fluid outlet temperature (required) and ambient-air temperature is less than 30°C, induced draft unit is preferred because of the less % recirculation of hot exhaust air. But if the same is more than 30°C, Forced draft unit is preferred as it consumes less power. 3/4 in (19.05 mm OD) and 1 in (25.4 mm OD) tubes are more commonly used. Fin spacing from 3.6 to 2.3 mm (7 to 11 per linear inch) and triangular pitch from 43 to 63.5 mm are common. Tube ends are left bare to permit the attachment or insertions of tubes into appropriate holes of box type headers. Fin material can be aluminium, copper, mildsteel, stainless steel, etc.

The same heat exchanger is also used for heating the air by saturated steam or cooling of the atmospheric air by chilled water. Hot air is used as drying medium in the dryers while chilled air is used for air conditioning.

For all above mentioned applications of the air heat exchangers, worst condition or design condition is different.

- (a) If atmospheric air is used as cooling medium then for the design calculation the maximum possible temperature of ambient air in the entire year must be considered.
- (b) If air is heated by saturated steam to get the hot air for dryer, then for the design calculations minimum possible temperature of ambient air in the entire year must be considered.
- (c) When atmospheric air is cooled to lower temperature (say to 14°C) by chilled water, then sizeable percentage of heat duty is utilized in the condensation of water vapour which is always present in atmospheric air. Hence for this case, for the design calculations atmospheric air with the maximum possible absolute humidity (and with corresponding temperature) must be considered.

Fins are used to increase the heat transfer area and thereby to decrease the size of heat exchanger required. For the heat transfer, fin area is not as effective as bare tube area. Hence, in design calculations to consider the effectiveness of fin area, fin efficiency is determined.

Design method for air side or finside is completely different from the same used for shell and tube heat exchanger, but for finding tube side heat transfer coefficient and tube side pressure drop same correlations are applicable that are used for a shell and tube heat exchanger.

6.9.2 Method for Finding Air Side or Fin Side Heat Transfer Coefficient and Pressure Drop

- (a) Air or finside heat transfer coefficient:

Find the equivalent diameter by using following equation

$$d_e = \frac{2(\text{fin area} + \text{bare tube area})}{\pi (\text{projected perimeter})} \quad (6.69)$$

For example:

Tube OD = 19.05 mm, Fin OD = 38 mm
 Tube pitch = 43 mm, Fin height = 9.475 mm
 Number of fins per liner inch = 8
 Fin thickness = 0.889 mm
 Type of fin = Spiral wound transverse fin
 Fin area per unit length of tube

$$\begin{aligned}
 &= \frac{\pi}{4} (\text{Fin OD}^2 - \text{Tube OD}^2) \times 2 \times \text{No. of fins per one metre of} \\
 &\quad \text{tube length} \\
 &= \frac{\pi}{4} (0.038^2 - 0.01905^2) \times 2 \times \frac{8}{25.4} \times 1000 \\
 &= 0.53486 \frac{m^2}{\text{m of tube length}}
 \end{aligned} \tag{6.70}$$

$$\begin{aligned}
 \text{Bare tube area/m of one tube} &= \pi d_o \times 1 - \pi d_o t_f \times n_f \\
 \text{where, } d_o &= \text{OD of bare tube, m} \\
 t_f &= \text{thickness of fin, m}
 \end{aligned} \tag{6.71}$$

$$n_f = \text{number of fins per 1 m of tube length} = 8 \times \frac{1000}{25.4}$$

Bare tube area

$$\begin{aligned}
 &= \pi \times 0.01905 \times 1 - \pi \times 0.01905 \times \left(\frac{0.889}{1000} \times 1 \right) \times 8 \times \frac{1000}{25.4} \\
 &= 0.04309 \text{ m}^2/\text{m of one tube}
 \end{aligned}$$

(b) Projected Perimeter:

$$\begin{aligned}
 \text{Projected perimeter} \\
 &= 2(\text{Fin OD} - \text{tube OD}) \times n_f - 2 \times (1 - t_f \cdot n_f) \tag{6.72} \\
 &= 2(0.038 - 0.01905) \times 8
 \end{aligned}$$

$$\begin{aligned}
 &\times \frac{1000}{25.4} - 2 \left(1 - 0.889 \times 10^{-3} \times 8 \times \frac{1000}{25.4} \right) \\
 &= 10.497 \text{ m/m of one tube}
 \end{aligned}$$

$$d_e = \frac{2 \times (0.53486 + 0.04309)}{\pi \times 10.497} = 0.03505 \text{ m}$$

Shell side or air side flow area:

$$a_s = \text{Air facing area} - \text{Area occupied by finned tubes}$$

$$\begin{aligned}
 \text{Air facing area} &= \text{Length of area} \times \text{width of area} \\
 &= \text{Tube length} \times \text{width of area}
 \end{aligned}$$

In majority of applications width of air facing area is kept equal to tube length.

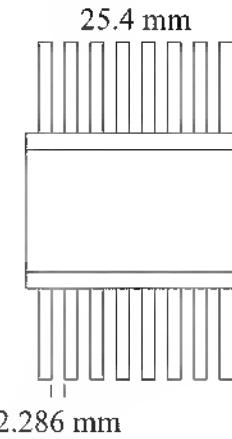


Fig. 6.29 Projection of Finned Tube

For example let tube length $L = 1500$ mm (in the previous example) and $W = L \approx 1500$ mm

$$a_s = WL - n_p \times d_o \times L - n_p \cdot t_f \cdot n_f L \quad (6.73)$$

where, n_p = Number of tubes in bank perpendicular to the direction of flow of air

n_f = Number of fins per 1 m of tube length

$$n_p = \frac{1500}{43} = 34.88 \approx 35$$

Let $W = 1510$ mm (revised) for $n_p = 35$ tubes in one bank

$$W = (n_p - 1) \times p_t + \text{Fin OD} + x$$

$$W = (35 - 1) \times 43 + 38 + x \text{ or } x = 10 \text{ mm}$$

Hence there will be 5 mm clearance on either side.

$$a_s = 1.51 \times 1.5 - 35 \times 0.01905 \times 1.5 - 35 \times (0.038 - 0.01905)$$

$$\times (0.889 \times 10^{-3}) \times 8 \times \frac{1500}{25.4}$$

$$a_s = 0.98631 \text{ m}^2$$

(c) After calculating d_e and a_s , calculate the air side mass velocity

$$G_s = \frac{\dot{m}_a}{a_s} \quad (6.74)$$

where, \dot{m}_a = Mass flow rate of air, kg/s

a_s = Air side or shell side flow area, m^2

(d) Calculate shell side or air side Reynolds number

$$Re = \frac{d_e G_s}{\mu} \quad (6.75)$$

where, d_e = Equivalent diameter, m

G_s = Mass velocity of air, $\text{kg}/(\text{m}^2 \cdot \text{s})$

μ = Viscosity of air at average temperature, $\text{kg}/(\text{m} \cdot \text{s})$

(e) Calculate air side Prandtl number

$$\text{Prandtl number for air, } Pr = \frac{C_p \mu}{k} \quad (6.76)$$

where, C_p = Specific heat of air, $\text{J}/(\text{kg} \cdot ^\circ\text{C})$

μ = Viscosity of air, $\text{kg}/(\text{m} \cdot \text{s})$

k = Thermal conductivity of air, $\text{W}/(\text{m} \cdot ^\circ\text{C})$

(f) All properties must be determined at average temperature on shell side.

Calculate fin side or air side heat transfer coefficient (clean and for 100% fin efficiency)

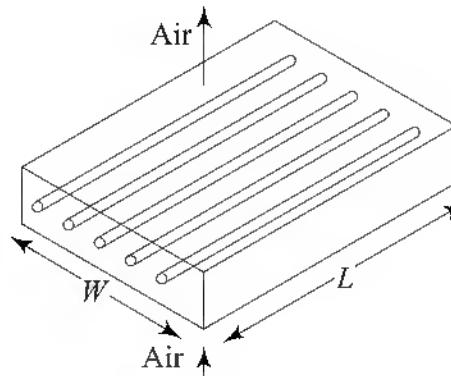


Fig. 6.30 Air-facing Area

$$h_f = J_f \frac{k}{d_e} Pr^{1/3} \quad (6.77)$$

where,
 h_f = Heat transfer coefficient, W/(m² · °C) (clean)
 J_f = Factor for heat transfer coefficient
 k = Thermal conductivity of air, W/(m · °C)
 d_e = Equivalent diameter, m
 Pr = Prandtl number of air

J_f , factor of heat transfer coefficient can be calculated by following equation.

$$J_f = 0.085 2072 Re^{0.7324} \quad (6.78)$$

(g) Calculate dirty fin side heat transfer coefficient, h'_f (for 100% fin efficiency)

$$\frac{1}{h'_f} = \frac{1}{h_f} + \frac{1}{h_{do}} \quad (6.79)$$

where,
 h_{do} = Shell side fouling coefficient, W/(m² · °C)
 h'_f = Dirty fin side heat transfer coefficient, W/(m² · °C)

(h) Calculate dirty fin side heat transfer coefficient based on inside heat transfer area and considering fin efficiency

$$h'_{f_i} = (\Omega \times A_f + A_o') \frac{h'_f}{A_i} \quad (6.80)$$

where,
 h'_{f_i} = Dirty fin side or Air side heat transfer coefficient based on inside heat transfer area, W/(m² · °C)
 Ω = Fin efficiency
 A_f = Total fin area, m²
 A_o' = Total bare tube area, m²
 A_i = Inside heat transfer area, m²

(i) Fin efficiency Ω can be determined from Fig. 6.31 in which graph is drawn

for $(r_e - r_b) \sqrt{\frac{h'_f}{k_m y_b}}$ vs Ω for the different values of $\frac{r_e}{r_b}$.

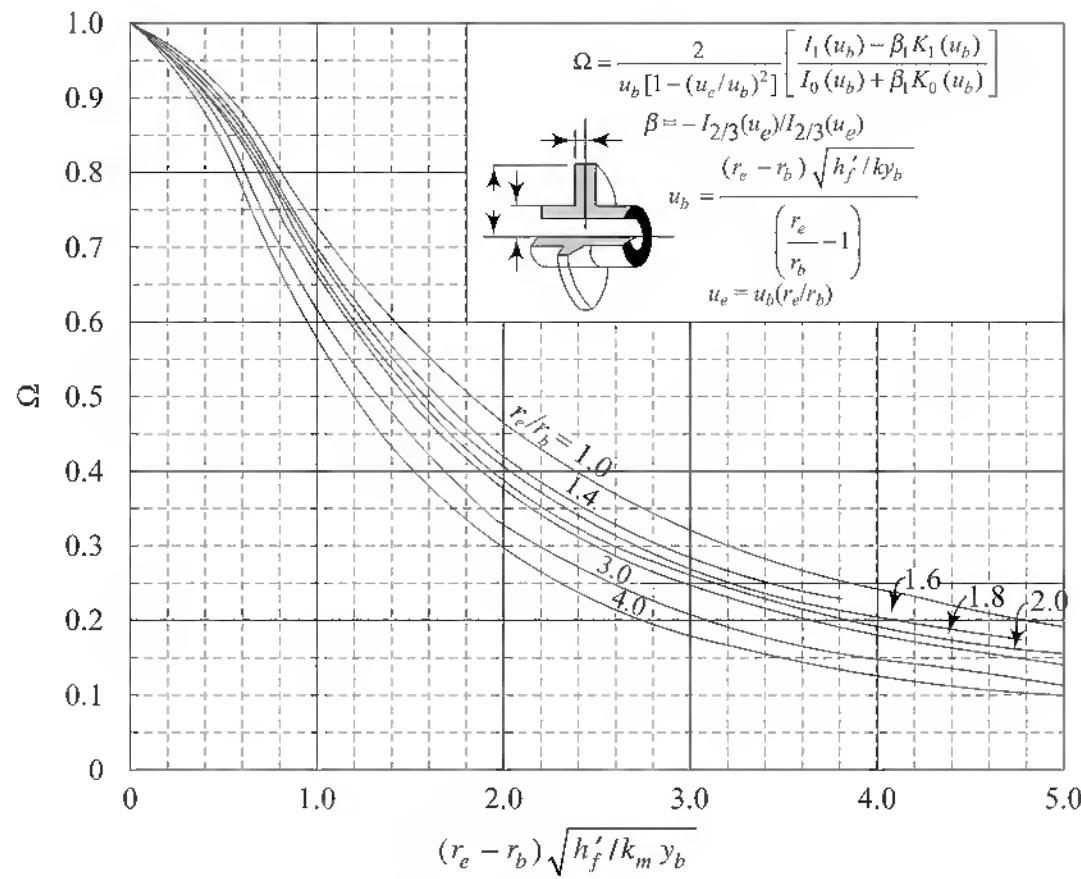
where, $r_e = \frac{\text{Fin OD}}{2}$, m, $r_b = \frac{\text{Tube OD}}{2}$, m

$$y_b = \frac{\text{Fin thickness}}{2}, \text{m}$$

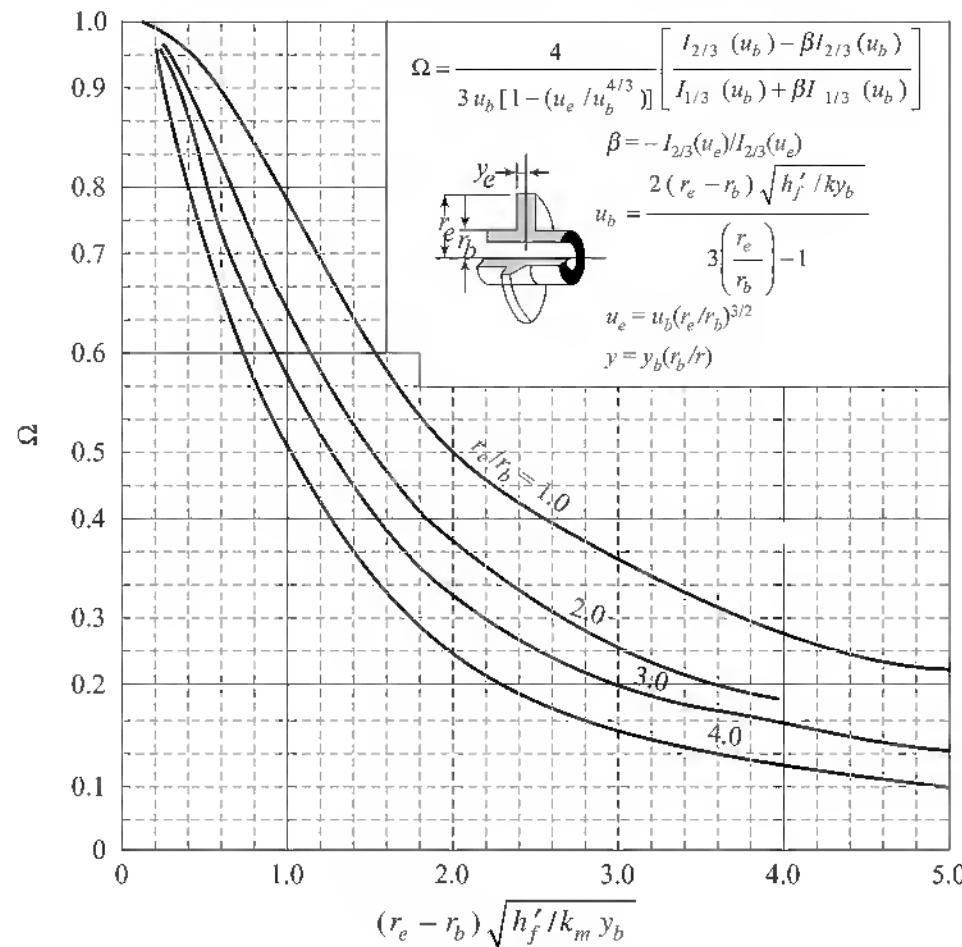
k_m = Thermal conductivity of fin material, W/(m · °C)

h'_f = Dirty fin side heat transfer coefficient determined by Eq. (6.79), W/(m² · °C)

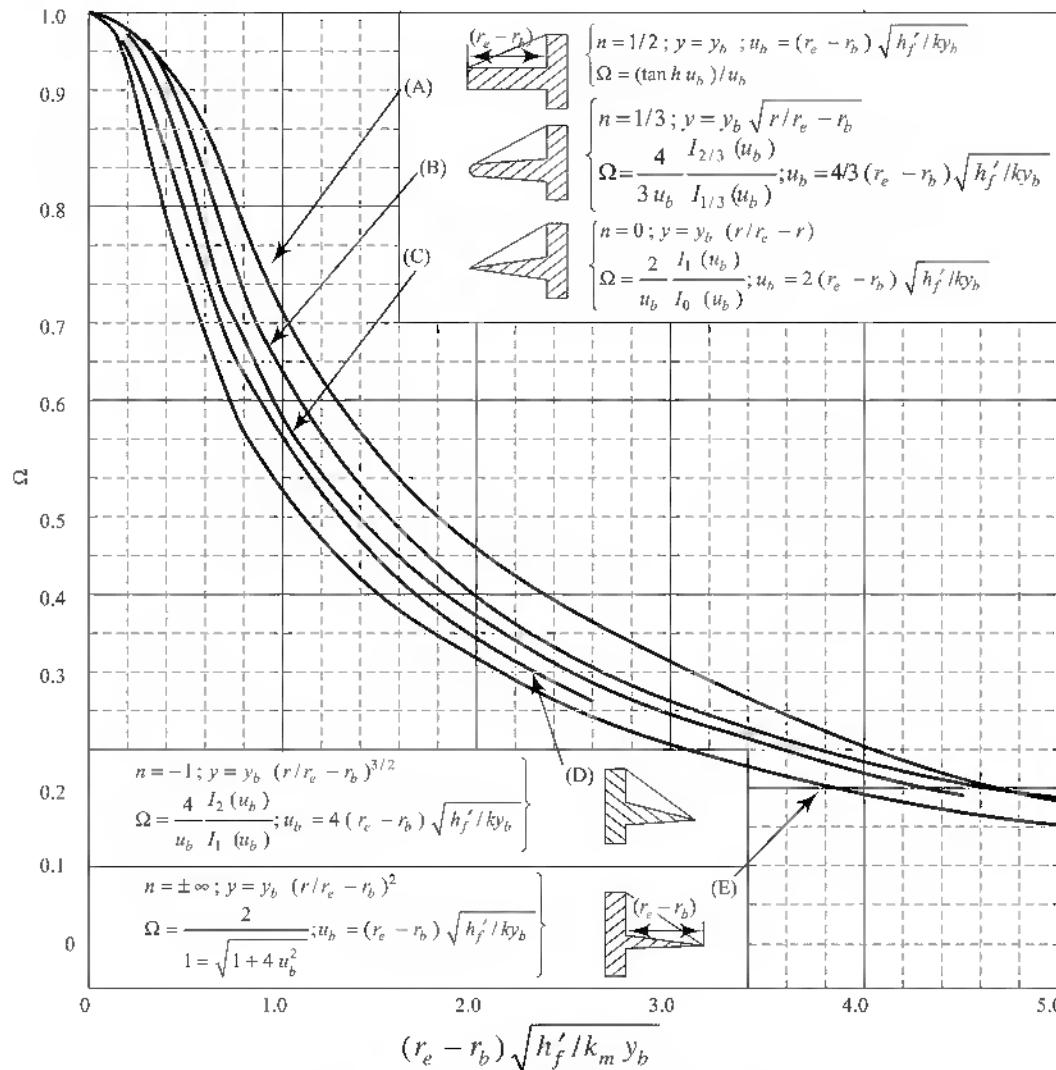
$I_n(u)$ = Modified Bessel function of the first kind and order n , dimensionless



(a) Efficiency of Annular Fins of Constant Thickness



(b) Efficiency of Annular Fins with Metal Area for Constant Heat Flux



(c) Fin Efficiency of Several Types of Straight Fin

$K_n(u)$ = Modified Bessel function of the second kind and order n , dimensionless

u = A function

β = Constant

- (j) Determine the tube side heat transfer coefficient h_i . For this determination, the same correlations are applicable which are used for shell and tube heat exchangers. Decide the tube side fouling coefficient h_f . Find the overall heat transfer coefficient based on inside heat transfer area, U_i

$$U_i = \frac{1}{\frac{1}{h_{fi}'} + \frac{1}{h_i} + \frac{1}{h_{id}}} \quad (6.81)$$

- (k) Then calculate the inside heat transfer area required

$$A_i = \frac{\phi_t}{U_i \Delta T_m} \quad (6.82)$$

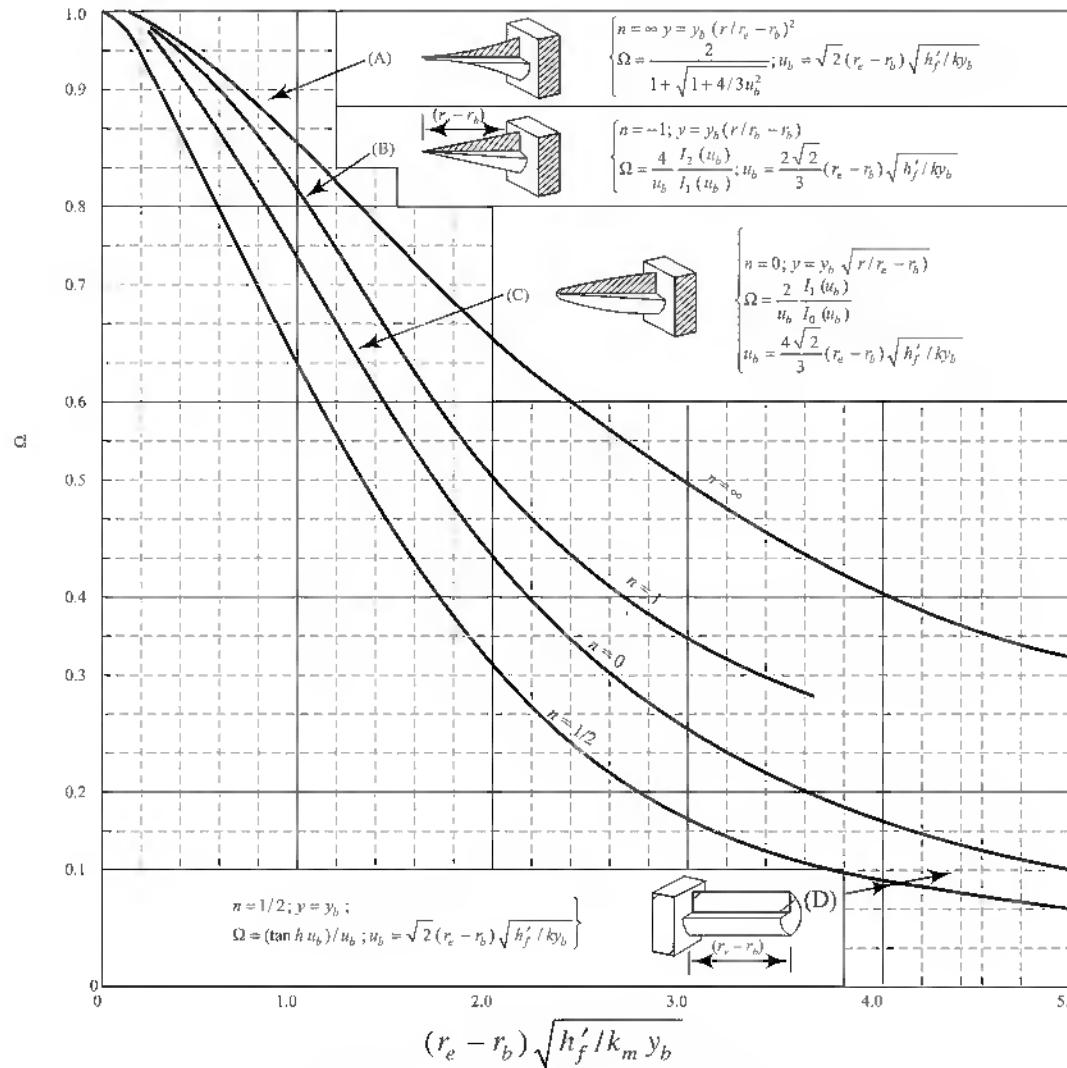


Fig. 6.31 Fin Efficiencies for Various Types of Fins⁵

where, ϕ_t = Total heat duty, W
 ΔT_m = Mean temperature difference, °C
 $\Delta T_m = F_t \Delta T_{ln}$
 where, F_t = LMTD correction factor for cross flow can be determined, from the Fig. (6.32)

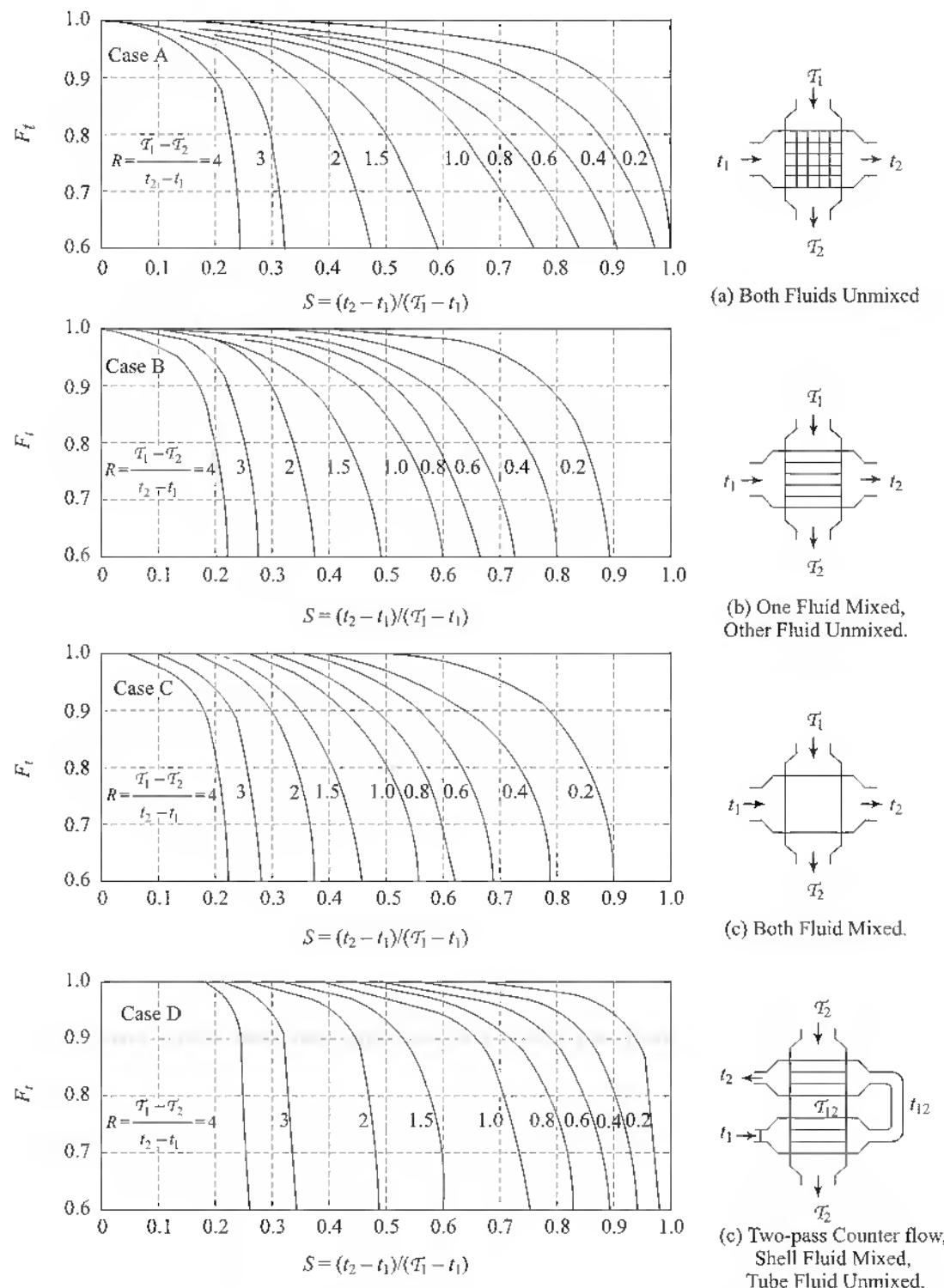
- (l) Heat transfer area provided (inside)

$$A_i = N_f \pi d_i L$$

Provided area should be greater than required area. Ideally provided area should be greater by 10 to 20% than the required area.

- (m) Calculate, the tube side pressure drop by using the same correlation that is used for shell and tube heat exchanger. It should be less than maximum allowable pressure drop.
 (n) Calculate the volumetric equivalent diameter by following equation.

$$D_{ev} = \frac{4 \times \text{net free volume}}{(A_f + A'_o) \left(\frac{n_p}{n_t} \right)} \quad (6.83 \text{ a})$$

Fig. 6.32 Cross Flow Temperature Difference Correction Factors⁵

Net free volume

$$= W \times L \times x' - n_p \times \frac{\pi}{4} d_o^2 L - n_p \times \frac{\pi}{4} (\text{Fin OD}^2 - d_o^2) \times t_f n_f L \quad (6.83 \text{ b})$$

where,

 W = Width of Air facing area, m L = Length of tube, m x' = Horizontal distance between two vertical bank (see Fig. 6.33)

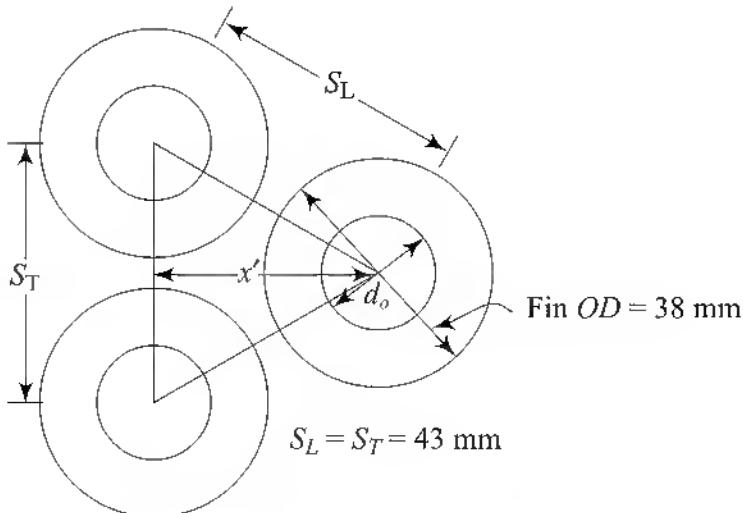


Fig. 6.33 Finned Tube Arrangement

For equilateral triangular pitch arrangement

$$x' = P_t \sin 60$$

n_t = Total number of tubes

n_p = Number of tubes in one bank, perpendicular to the direction of the flow of air

d_o = Outside diameter of (bare) tube, m

t_f = Thickness of fin, m

n_f = Number of fins per 1 m of tube length

A_f = Total fin area, m^2

A'_o = Total bare tube area, m^2

For the previous example:

$$\text{Net free volume} = 1.51 \times 1.5 \times 0.03724 - 35 \times \frac{\pi}{4} (0.01905)^2 \times 1.5$$

$$- 35 \times \frac{\pi}{4} (0.038^2 - 0.01905^2) \times (0.889 \times 10^{-3})$$

$$\times \left(8 \times \frac{1000}{25.4} \times 1.5 \right) = 0.0569 \text{ m}^3$$

$$A_f = (0.53486 \text{ m}^2/\text{1 m of one tube}) \times L \times n_t$$

$$= 0.53486 \times 1.5 \times n_t = 0.80229 \times n_t \text{ m}^2$$

$$A'_o = (0.04309 \text{ m}^2/\text{m of one tube}) \times L \times n_t = 0.064635 n_t \text{ m}^2$$

$$(A_f + A'_o) \frac{n_p}{n_t} = (0.80229 + 0.064635) \times 35 = 30.3424 \text{ m}^2$$

$$D_{ev} = \frac{4 \times 0.0569}{30.3424} = 0.0075 \text{ m}$$

Reynolds number for the calculation of pressure drop

$$Re_s = \frac{D_{ev} G_s}{\mu} \quad (6.84)$$

where, G_s = Mass velocity of air based on flow area a_s , $\text{kg}/(\text{m}^2 \cdot \text{s})$

μ = Viscosity of air at average temperature, $\text{kg}/(\text{m} \cdot \text{s})$

Calculate the air side or shell side pressure drop

$$\Delta p_s = \frac{f G_s^2 L_p}{D_{ev} \times \rho} \left(\frac{D_{ev}}{S_T} \right)^{0.4} \left(\frac{S_L}{S_T} \right)^{0.6} \quad (6.85)$$

Δp_s = Pressure drop, N/m^2

f = Air side friction factor can be calculated by following equation.

$$f = 1.085 \ 58 \times Re^{-0.128 \ 025} \quad (6.86)$$

S_L = Center to center distance to the nearest tube in the next bank, m (as shown in Fig. 6.33)

S_T = Pitch in transverse bank, m (as shown in Fig. 6.33)

L_p = Effective path length of pressure drop, m

L_p can be determined by following equation

$$L_p = x' \times \frac{n_t}{n_p} \quad (6.87)$$

Calculated pressure drop should be less than maximum allowable pressure drop.

Example 6.7

Mobil therm oil is used as heating medium in chemical industry. Its operating range is from -1.1°C to 316°C .

It is required to cool 9000 kg/h of mobil therm oil from 260°C to 200°C by using atmospheric air as a cooling medium in air cooler. Design the suitable air-cooler.

Properties of Mobil therm oil at 230°C temperature

Density, $\rho = 850 \text{ kg/m}^3$

Specific heat, $C_L = 2.5 \text{ kJ/(kg} \cdot ^\circ\text{C)}$

Viscosity, $\mu = 0.595 \text{ cP or mPa} \cdot \text{s}$

Thermal conductivity, $k = 0.1159 \text{ W}/(\text{m} \cdot ^\circ\text{C})$

Solution:

Heat duty required

$$\begin{aligned} \phi_t &= \dot{m} C_L \Delta t = 9000 \times 2.5 \times (260 - 200) \\ &= 1350 \ 000 \text{ kJ/h} \\ &\equiv 375 \text{ kW} \end{aligned}$$

Assume maximum possible temperature of atmospheric air in the area as 48°C .

Let outlet temperature of air = 75°C

Mass flow rate of Air required

$$\dot{m}_a = \frac{\phi_t}{Cp_a \Delta t} = \frac{375}{Cp_a (75 - 48)}$$

Specific heat of air at 61.5°C

$Cp_a = 0.25 \text{ kcal/(kg} \cdot ^\circ\text{C)} = 1.0467 \text{ kJ/(kg} \cdot ^\circ\text{C)}$ (Ref. 9)

$$\dot{m}_a = 13.2692 \text{ kg/s} \equiv 47 \ 769 \text{ kg/h}$$

$\Delta T_m'$ Mean temperature difference

$$\Delta T_m' = \Delta T_{ln} \cdot F_t$$

Oil temperatures are very much greater than air temperatures hence forced draft fan can be selected to reduce the power consumption. Atmospheric air will enter from the bottom of tube bundle. Hot oil is also introduced from the bottom of the header. Hence, overall there is a cocurrent contacts. But, actually there is cross flow contact with multipass flow of oil.

$$\Delta T_{ln} = \frac{(260 - 48) - (200 - 75)}{\ln\left(\frac{260 - 48}{200 - 75}\right)} = \frac{212 - 125}{\ln\left(\frac{212}{125}\right)} = 164.69 \text{ }^{\circ}\text{C}$$

$$R = \frac{T_1 - T_2}{t_2 - t_1} = \frac{260 - 200}{75 - 48} = 2.222$$

$$S = \frac{t_2 - t_1}{T_1 - t_1} = \frac{75 - 48}{260 - 48} = 0.12736$$

$$F_t = 0.98 \text{ (From Fig. 6.32 a)}$$

$$\Delta T_m' = 161.396 \text{ }^{\circ}\text{C}$$

Evaluation of h_o :

$$\text{Equivalent diameter } d_e = \frac{2(\text{fin area} + \text{bare tube area})}{\pi(\text{projected perimeter})} \quad (6.69)$$

Let tube OD = 19.05 mm,

Type of fin = Spiral wound transverse fin

Fin OD = 38 mm, Tube pitch = 43 mm (Equilateral triangular)

Number of fins per linear inch = 8

Fin thickness = 0.889 mm

Fin and tube material = mild steel

$$\text{Fin area/1 m of one tube} = 2 \times \frac{\pi}{4} (\text{fin OD}^2 - d_o^2) \times \text{No. of fins per 1 m length} \quad (6.70)$$

$$A_{f1} = 2 \times \frac{\pi}{4} (0.038^2 - 0.01905^2) \times 8 \times \frac{1000}{25.4}$$

$$A_{f1} = 0.53486 \text{ m}^2$$

$$\text{Bare tube area/1 m of one tube} = A_{o1} = \pi d_o \times 1 - \pi d_o t_f n_f \quad (6.71)$$

(where n_f = no. of fins/1 m of one tube)

$$A_{o1} = \pi \times 0.01905 \times 1 - \pi \times 0.01905 \times (0.889 \times 10^{-3}) \times 8 \times \frac{1000}{25.4}$$

$$A_{o1} = 0.04309 \text{ m}^2$$

Projected perimeter per 1 m of one tube

$$= 2(\text{fin OD} - d_o) \times n_f - 2(1 - t_f n_f) \quad (6.72)$$

$$= 2(0.038 - 0.01905) \times 8 \times \frac{1000}{25.4} - 2\left(1 - 0.889 \times 10^{-3} \times 8 \times \frac{1000}{25.4}\right) = 10.497 \text{ m}$$

$$d_e = \frac{2(0.53486 + 0.04309)}{\pi \times 10.497} = 0.03505 \text{ m}$$

Flow Area:

For the first trial calculation,

Let tube length = 900 mm = L

and width of air facing area, $W = 900 \text{ mm}$

Let n_p = number of tubes in one bank, perpendicular to the flow of air

$$n_p \equiv \frac{W}{pt} \equiv \frac{900}{43} \equiv 20.93$$

Let

$$n_p = 21$$

$$W = (n_p - 1) p_t + \text{Fin OD} + x$$

$$W = 20 \times 43 + 38 + x$$

$$x = 2 \text{ mm}$$

Let

$$W = 910 \text{ mm}$$

$$x = 12 \text{ mm} \quad (\text{Revised})$$

Flow area

$$a_s = WL - n_p \times d_o \times L - n_p (\text{fin OD} - \text{tube OD}) t_f n_f L \quad (6.73)$$

$$= 0.91 \times 0.9 - 21 \times 0.01905 \times 0.9 - 21(0.038 - 0.01905)$$

$$\times 0.889 \times 10^{-3} \times 8 \times \frac{1000}{25.4} \times 0.9$$

$$= 0.35867 \text{ m}^2$$

$$\text{Mass velocity, } G_s = \frac{\dot{m}_a}{a_s} = \frac{47769/3600}{0.35867} = 36.995 \text{ kg}/(\text{m}^2 \cdot \text{s})$$

$$\text{Reynolds number, } Re = \frac{d_e G_s}{\mu_a} \quad (6.75)$$

Viscosity of air at 61.5°C

$$\mu_a = 2000 \times 10^{-7} \text{ P} = 2000 \times 10^{-8} \text{ kg}/(\text{m} \cdot \text{s})$$

(Figure 3.42 of Ref. 2)

$$Re = \frac{0.03505 \times 36.995}{2000 \times 10^{-8}} = 64834$$

Factor for heat transfer coefficient

$$J_f = 0.0852072 Re^{0.7324} = 0.0852072(64833.74)^{0.7324} \quad (6.78)$$

$$= 284.865$$

Clean heat transfer coefficient, for 100% fin efficiency

$$h_f = J_f \frac{k}{d_e} Pr^{1/3} \quad (6.77)$$

Prandtl number of air at 61.5°C

$$Pr = \frac{C_{pa} \mu_a}{k_a}$$

Thermal conductivity of air at 61.5°C, $k = 0.028\ 784\ \text{W}/(\text{m} \cdot \text{k})$
(Table 3.314, of Ref. 2)

$$\begin{aligned} Pr &= \frac{1.0467 \times 2000 \times 10^{-8}}{0.028\ 784} \times 10^3 \\ &= 0.7273 \end{aligned}$$

$$\begin{aligned} h_f &= 284.865 \times \frac{0.028\ 784}{0.035\ 05} \times (0.7273)^{1/3} \\ &= 210.38\ \text{W}/(\text{m}^2 \cdot ^\circ\text{C}) \end{aligned}$$

Dirty fin side heat transfer coefficient

$$\frac{1}{h'_f} = \frac{1}{h_f} + \frac{1}{h_{do}} \quad (6.79)$$

$$h_{do} = 5000\ \text{W}/(\text{m}^2 \cdot ^\circ\text{C}) \quad (\text{From Table 6.10})$$

$$\frac{1}{h'_f} = \frac{1}{210.38} + \frac{1}{5000}$$

$$h'_f = 201.885\ \text{W}/(\text{m}^2 \cdot ^\circ\text{C})$$

Dirty fin side heat transfer coefficient based on inside heat transfer area and fin efficiency.

$$h'_{fi} = (\Omega \times A_f + A_o') \frac{h'_f}{A_i} \quad (6.80)$$

$$A_f = n_t A_{f1} L, \text{ where, } n_t = \text{Total number of tubes}$$

$$\text{Total fin area } A_f = n_t \times 0.53486 \times 0.9 = 0.481\ 374\ n_t\ \text{m}^2$$

$$A_o' = \text{Total bare tube area}$$

$$A_o' = n_t A_{o1} L = 0.043\ 09 \times 0.9 \times n_t = 0.038\ 781\ n_t$$

$$A_i = n_t \pi d_i L$$

Tube inside diameter, $d_i = 14.8336\ \text{mm}$ (For 14 BWG tube)

$$A_i = \pi(0.014\ 8336) \times 0.9 \times n_t = 0.041\ 94\ n_t\ \text{m}^2$$

Fin Efficiency (Ω):

$$\text{Evaluation of: } (r_e - r_b) \sqrt{\frac{h'_f}{k_m y_b}}$$

$$\text{where, } r_e = \frac{\text{fin OD}}{2} = \frac{38}{2} = 19\ \text{mm} = 0.019\ \text{m}$$

$$r_b = \frac{\text{Tube OD}}{2} = \frac{19.05}{2} = 9.525\ \text{mm} = 0.009\ 525\ \text{m}$$

$$\begin{aligned} k_m &= \text{Thermal conductivity of fin material (mild steel)} \\ &= 45\ \text{W}/(\text{m} \cdot ^\circ\text{C}) \end{aligned}$$

$$y_b = \frac{\text{Fin thickness}}{2} \quad (\text{For fins of uniform thickness})$$

$$= \frac{0.889 \times 10^{-3}}{2} = 4.445 \times 10^{-4} \text{ m}$$

$$(0.019 - 0.009525) \sqrt{\frac{201.885}{45 \times 4.445 \times 10^{-4}}} = 0.952$$

$$\frac{r_e}{r_b} = 1.99475, \Omega = 0.67 \quad (\text{from Fig. 6.31(a)})$$

$$h'_{fi} = \frac{(0.67 \times 0.481374 n_t + 0.038781 n_t) \times (201.885)}{0.04194 n_t}$$

$$= 1739.18 \text{ W}/(\text{m}^2 \cdot ^\circ\text{C})$$

Evaluation of h_i :

Tube side flow area, a_t

Let no. of tubes per pass = $n_p = 21$

$$a_t = 21 \times \frac{\pi}{4} d_i^2 = 21 \times \frac{\pi}{4} \times (0.0148336)^2 = 3.629 \times 10^{-3} \text{ m}^2$$

Tube side mass velocity,

$$G_t = \frac{\dot{m}}{a_t} = \frac{9000/3600}{3.629 \times 10^{-3}} = 688.9 \text{ kg}/(\text{m}^2 \cdot \text{s})$$

$$\text{Tube side velocity, } u_t = \frac{G_t}{\rho} = \frac{688.9}{850}$$

$$= 0.81 \text{ m/s}$$

Tube side Reynolds number

$$Re_t = \frac{d_i G_t}{\mu_0}$$

$$= \frac{0.0148336 \times 688.9}{0.595 \times 10^{-3}} = 17174.57$$

$$\text{Prandtl number } Pr = \frac{C_L \mu}{k} = \frac{2.5 \times 0.595 \times 10^{-3} \times 10^3}{0.1159}$$

$$= 12.8343$$

Using Dittus–Bolter equation

$$\frac{h_i d_i}{k_f} = 0.023 Re^{0.8} Pr^{0.33} \left(\frac{\mu}{\mu_w} \right)^{0.14} \quad (6.19)$$

$$h_i = 0.023 \times \frac{0.1159}{0.0148336} \times (17174.57)^{0.8} (12.8343)^{0.33}$$

$$h_i = 1019.15 \text{ W}/(\text{m}^2 \cdot ^\circ\text{C})$$

$$U_i = \frac{1}{\frac{1}{h_{ft}'} + \frac{1}{h_i} + \frac{1}{h_{id}}} \quad (6.81)$$

$$U_i = \frac{1}{\frac{1}{1739.18} + \frac{1}{1019.15} + \frac{1}{5000}} = 569.41 \text{ W/(m}^2 \cdot ^\circ\text{C)}$$

Heat transfer area (inside) required

$$A_{ireq} = \frac{\phi_t}{U_i \Delta T_m} = \frac{375 \times 1000}{569.41 \times 161.396} = 4.08 \text{ m}^2$$

Let heat transfer area provided

$$A_{ipro} = 1.1 \times A_{ireq} = 4.48855 \text{ m}^2$$

Heat transfer area per bank = $n_p \pi d_i L = 21 \times \pi \times 0.0148336 \times 0.9 = 0.88076 \text{ m}^2$

$$\text{Number of banks required} = \frac{4.48855}{0.88076} = 5.085$$

Let number of banks = 5

% Excess heat transfer area = 9.83%

Total number finned tubes $n_t = 21 \times 5 = 105$

Total fin area $A_f = 105 \times 0.53486 \times 0.9 = 50.544 \text{ m}^2$

Total bare tube area $A_o' = 105 \times 0.04309 \times 0.9 = 4.072 \text{ m}^2$

Total outside heat transfer area = 54.616 m^2

Air side Pressure Drop:

$$\text{Net free volume} = WLx' - n_p \times \frac{\pi}{4} d_o^2 L - n_p \times \frac{\pi}{4} (\text{Fin OD}^2 - d_o^2) t_f n_f L \quad (6.83 \text{ b})$$

where, x' = Horizontal distance between two vertical banks, mm

$x' = P_t \sin 60$ (For equilateral triangular pitch arrangement), mm

$$x' = 43 \text{ mm} \times \sin 60 = 37.239 \text{ mm}$$

$$\text{Net free volume} = 0.91 \times 0.9 \times 0.037239 - 21 \times \frac{\pi}{4} (0.01905)^2 \times 0.9 - 21$$

$$\times \frac{\pi}{4} (0.038^2 - 0.01905^2) \times 0.889 \times 10^{-3} \times \left(8 \times \frac{1000}{25.4} \right) \times 0.9 \\ = 0.02062 \text{ m}^3$$

Volumetric equivalent diameter

$$D_{ev} = \frac{4 \times \text{net free volume}}{(A_f + A_o') \left(\frac{n_p}{n_t} \right)} \quad (6.83 \text{ a})$$

$$= \frac{4 \times 0.02063}{(50.544 + 4.072) \times \frac{21}{105}} = 0.007555 \text{ m}$$

Reynolds number for calculating pressure drop

$$Re_s = \frac{D_{ev} G_s}{\mu} = \frac{0.007555 \times 36.995}{2000 \times 10^{-8}} = 13974.86$$

$$f = 1.08558 \times Re^{-0.128025} \quad (6.86)$$

$$f = 1.08558 \times (13974.86)^{-0.128025} = 0.31986$$

$$\Delta p_s = \frac{f G_s^2 L_p}{D_{ev} \rho} \left(\frac{D_{ev}}{S_T} \right)^{0.4} \left(\frac{S_L}{S_T} \right)^{0.6} \quad (6.85)$$

where, L_p = Effective path length for pressure drop, m

$$L_p = x' \frac{n_t}{n_p} = 0.037239 \times \frac{105}{21} = 0.1862 \text{ m}$$

$$\text{Density of Air } \rho = \frac{PM}{RT} = \frac{1 \times 29}{0.0821 \times (61.5 + 273)} = 1.056 \text{ kg/m}^3$$

$$\Delta p_s = \frac{0.31986 \times 36.995^2 \times 0.1862}{0.007555 \times 1.056} \left(\frac{0.007555}{0.043} \right)^{0.4} \times \left(\frac{0.043}{0.043} \right)^{0.6}$$

(For equilateral triangular pitch $S_L = S_T = P_t$)

$$\Delta p_s = 5096 \text{ N/m}^2 = 519.6 \text{ mm WC} > \Delta p_{smax} = 350 \text{ mm WC}$$

Discharge pressure of fans are generally less than 350 mm WC.

To decrease the pressure drop, flow area a_s must be increased

Let the revised tube length, $L = 1200 \text{ mm}$

width of flow area (air facing area), $W = 1200 \text{ m}$

Numbers of tube in one bank

$$n_p \approx \frac{W}{P_t} = \frac{1200}{43} = 27.9 = 28$$

Let

$$n_p = 28$$

$$W = (n_p - 1)p_t + \text{Fin OD} + x$$

$$W = 27 \times 43 + 38 + x = 1200 \Rightarrow x = 1 \text{ mm}$$

Let

$$W = 1210 \text{ mm and } x = 11 \text{ mm (Revised)}$$

$$a_s = WL - n_p d_o L - n_p (\text{fin OD} - d_o) t_f n_f L \quad (6.73)$$

$$= 1.21 \times 1.2 - 28 \times 0.01905 \times 1.2 - 28(0.038 - 0.01905)$$

$$\times 0.889 \times 10^{-3} \times 8 \times \frac{1200}{25.4}$$

$$= 0.63364 \text{ m}^2$$

$$\text{Mass velocity, } G_s = \frac{47769/3600}{0.63364} = 20.941 \text{ kg/(m}^2 \cdot \text{s})$$

$$\text{Reynolds number, } Re = \frac{d_e G_s}{\mu_a}$$

$$Re = \frac{0.035\ 05 \times 20.941}{2000 \times 10^{-8}} = 36\ 699.1$$

Factor for heat transfer coefficient

$$J_f = 0.085\ 2072 Re^{0.7324} = 187.77$$

Clean heat transfer coefficient, for 100% fin efficiency

$$\begin{aligned} h_f &= J_f \frac{k}{d_e} P_r^{1/3} \\ &= 187.77 \times \frac{0.028\ 784}{0.035\ 05} \times (0.7273)^{1/3} \\ &= 138.674 \text{ W/(m}^2 \cdot ^\circ\text{C)} \end{aligned} \quad (6.77)$$

Density finside coefficient

$$\frac{1}{h_f} = \frac{1}{h_f} + \frac{1}{h_{do}} \quad (6.79)$$

$$\frac{1}{h_f} = \frac{1}{138.674} + \frac{1}{5000}$$

$$h_f = 134.932 \text{ W/(m}^2 \cdot ^\circ\text{C)}$$

$$(r_e - r_b) \sqrt{\frac{h_f'}{k_m y_b}} = (0.019 - 0.0095\ 25) \sqrt{\frac{134.932}{45 \times 4.445 \times 10^{-4}}} \\ = 0.7782$$

$$r_e/r_b = 1.994\ 75, \Omega = 0.77 \text{ (Fig. 6.31(a))}$$

Dirty fin side heat transfer coefficient based on inside heat transfer area and fin efficiency

$$h_{fi}' = (\Omega \times A_f + A_o') \frac{h_f'}{A_i} \quad (6.80)$$

$$= (0.77 \times 0.534\ 86 \times 1.2 \times n_t + 0.043\ 09 \times 1.2 \times n_t) \frac{134.932}{\pi \times (0.014\ 336) \times 1.2 \times n_t}$$

$$= (0.77 \times 0.534\ 86 + 0.04309) \times \frac{134.932}{0.0466}$$

$$= 1317.27 \text{ W/(m}^2 \cdot ^\circ\text{C)}$$

Evaluation of h_i :

$$n_p = 28$$

$$a_t = 28 \times \frac{\pi}{4} (0.014\ 8336)^2$$

$$a_t = 4.84 \times 10^{-3} \text{ m}^2$$

$$G_t = \frac{\dot{m}}{a_t} = \frac{9000/3600}{4.84 \times 10^{-3}} = 516.53 \text{ kg/(m}^2 \cdot \text{s)}$$

$$u_t = \frac{G_t}{\rho} = \frac{516.53}{850} = 0.608 \text{ m/s},$$

$$Re = \frac{0.0148336 \times 516.53}{0.595 \times 10^{-3}} = 12877.3$$

$$h_i = 0.023 \times \frac{0.1159}{0.0148336} \times (12877.3)^{0.8} (12.8343)^{0.33}$$

$$= 809.445 \text{ W/(m}^2 \cdot ^\circ\text{C})$$

Overall coefficient based on inside heat transfer area

$$\frac{1}{U_i} = \frac{1}{h'_{fi}} + \frac{1}{h_i} + \frac{1}{h_{id}} = \frac{1}{1317.27} + \frac{1}{809.445} + \frac{1}{5000}$$

$$U_i = 455.67 \text{ W/(m}^2 \cdot ^\circ\text{C})$$

$$A_{i\text{req}} = \frac{\phi_i}{U_i \Delta T_m} = \frac{375 \times 1000}{455.67 \times 161.396} = 5.1 \text{ m}^2$$

Heat transfer area per one bank = $\pi d_p L \times n_p = 1.5658 \text{ m}^2$

Number of banks required = $\frac{5.1}{1.5658} = 3.2571$

Let number of banks = 4 = $\frac{n_t}{n_p}$

% Excess heat transfer area = $\frac{(4 - 3.2571)}{3.2571} \times 100 = 22.8 \%$

Total number of finned tubes $n_t = 28 \times 4 = 112$

Tube OD = 19.05 mm

Fin OD = 38 mm

No. of fins per inch = 8

Thickness of fin = 0.889 mm

Type of fin = spiral wound transverse fin

Tube length = 1200 mm

Width of air facing area = 1210 mm

Tube pitch = 43 mm (equilateral triangular)

Total inside heat transfer area, $A_i = 6.263 \text{ m}^2$ (provided)

Total fin area $A_f = 71.885 \text{ m}^2$

Total bare tube area, $A_o' = 5.7913 \text{ m}^2$

Air side Pressure Drop

$$\text{Net free volume} = WLx' - n_p \times \frac{\pi}{4} d_o^2 L - n_p \times \frac{\pi}{4} (\text{Fin OD}^2 - d_o^2) t_f n_f L \quad (6.83 \text{ b})$$

$$= 1.21 \times 1.2 \times 0.037239 - 28 \times \frac{\pi}{4} (0.01905)^2 \times 1.2 - 28$$

$$\times \frac{\pi}{4} (0.038^2 - 0.01905^2) \times 0.889 \times 10^{-3} \times 8 \times \frac{1200}{25.4} = 0.0365 \text{ m}^3$$

$$D_{ev} = \frac{4 \times \text{net free volume}}{(A_f + A_o') \frac{n_p}{n_t}} = \frac{4 \times 0.0365}{(71.885 + 5.7913) \times \frac{28}{112}} = 0.007518 \text{ m}$$

Reynolds number for calculating pressure drop

$$Re = \frac{D_{ev} G_s}{\mu} = \frac{0.007518 \times 20.941}{2000 \times 10^{-8}} = 7871.7$$

Friction factor for calculating pressure drop

$$f = 1.08558 \times Re^{-0.128025} = 1.08558 \times (7871.7)^{-0.128025} = 0.34425$$

Effective path length for pressure drop

$$L_p = x' \cdot \frac{n_t}{n_p} = 0.037239 \times \frac{112}{28} = 0.148956 \text{ m}$$

$$\Delta p_s = \frac{f G_s^2 L_p}{D_{ev} \rho} \left(\frac{D_{ev}}{S_T} \right)^{0.4} \left(\frac{S_L}{S_T} \right)^{0.6}$$

$$= \frac{0.34425 \times 20.941^2 \times 0.148956}{0.007518 \times 1.056} \times \left(\frac{0.007518}{0.043} \right)^{0.4} \times 1^{0.6}$$

$$= 1410 \text{ N/m}^2 \equiv 143.8 \text{ mm WC} < 350 \text{ mm WC} (\Delta p_{smax})$$

Tube Side Pressure Drop:

Tube side pressure drop can be calculated by following equation.

$$\Delta p_t = N_p \left(8 J_f (L/d_i) \left(\frac{\mu}{\mu_w} \right)^{-m} + 2.5 \right) \frac{\rho u_t^2}{2}$$

N_p = No. of tube side passes = 4

For $Re = 12877.3$ $J_f = 0.0045$ (From Fig. 6.13)

$L = 1.2 \text{ m}$, $d_i = 0.0148336 \text{ m}$

$$\rho = 850 \text{ kg/m}^3, u_t = 0.608 \text{ m/s}, \frac{\mu}{\mu_w} \cong 1$$

$$\Delta p_t = 4 \left(8 \times 0.0045 \left(\frac{1.2}{0.0148336} \right) \times 1 + 2.5 \right) \times \frac{850 \times 0.608^2}{2}$$

$$= 3401.25 \text{ N/m}^2 \equiv 3.401 \text{ kPa}$$

Calculated pressure drop is well within maximum allowable (68 kPa).

Hence to optimize the design, tube side velocity and tube side heat transfer coefficient can be increased. With special arrangement in header, number of tube side passes can be doubled.

Let number of tubes per pass = 14

$$a_t = 14 \times \frac{\pi}{4} (0.0148336)^2 = 2.4194 \times 10^{-3} \text{ m}^2$$

$$G_t = 1033.3 \text{ kg/(m}^2 \cdot \text{s}), Re = 25760.75, u_t = 1.2156 \text{ m/s}$$

$$h_t = 1409.6 \text{ W/(m}^2 \cdot ^\circ\text{C})$$

$$U_t = 599.313 \text{ W/(m}^2 \cdot ^\circ\text{C})$$

$$A_{req} = \frac{375 \times 1000}{599.313 \times 161.396} = 3.8769 \text{ m}^2$$

$$\text{No. of banks required} = \frac{3.8769 \text{ m}^2}{1.5658 \text{ m}^2} = 2.476$$

$$\text{Let no. of banks} = 3 = \frac{n_t}{n_p}$$

$$\% \text{ Excess area} = \frac{3 - 2.476}{2.476} \times 100 = 21.16\%$$

Total no. of finned tubes $n_t = 28 \times 3 = 84$

Tube OD = 19.05 mm, Fin OD = 38 mm, No. of fins per inch = 8

Thickness of fin = 0.889 mm, Type of fin = Spiral wound transverse fin

Tube length = 1200 mm

Width of Air facing area = 1210 mm

Tube pitch = 43 mm (equilateral triangular)

Total fin area, $A_f = 53.9139 \text{ m}^2$

Total bare tube area $A_o' = 4.34347 \text{ m}^2$

Number of tubes per pass = 14

Number of tube side passes = 6

Inside heat transfer area provided $A_i = 4.6974 \text{ m}^2$

Air Side Pressure Drop

$$De_v = \frac{4 \times 0.0365}{(53.9139 + 4.34347) \times \frac{28}{84}} = 0.007518 \text{ m (no change)}$$

$$Re_s = 7871.7, f = 0.34425 \text{ (no change)}$$

$$L_p = x' \frac{n_t}{n_p} = 0.037239 \times \frac{84}{28} = 0.11172 \text{ m (Revised)}$$

Revised air side pressure drop

$$\Delta p_s = 1410 \times \left(\frac{0.11172}{0.148956} \right) = 1057.53 \text{ N/m}^2 \equiv 1.058 \text{ kPa}$$

$$\equiv 107.85 \text{ mm WC} < \Delta p_{smax} (= 250 \text{ mm WC})$$

Tube Side Pressure Drop (Revised):

$$N_p = 6, Re_t = 25760.75, J_f = 0.0038$$

$$u_t = 1.2156 \text{ m/s}$$

$$\Delta P_t = 6 \left(8 \times 0.0038 \times \left(\frac{1.2}{0.0148336} \right) \times 1 + 2.5 \right) \times \frac{850 \times 1.2156^2}{2}$$

$$\Delta P_t = 18687 \text{ N/m}^2 \equiv 18.687 \text{ kPa} < 68 \text{ kPa}$$

6.10 PLATE HEAT EXCHANGERS^{2,4,10}

6.10.1 Introduction

The Plate Heat Exchanger (PHE) consists of a frame in which closely spaced metal plates are clamped between a head and a follower. The plates have circular holes in a corner and these holes are sealed around by gasket which also attach over the plate edges. Holes at the corner are known as ports. Typical plate and gasketed plate heat exchanger is shown in Fig. 6.34.

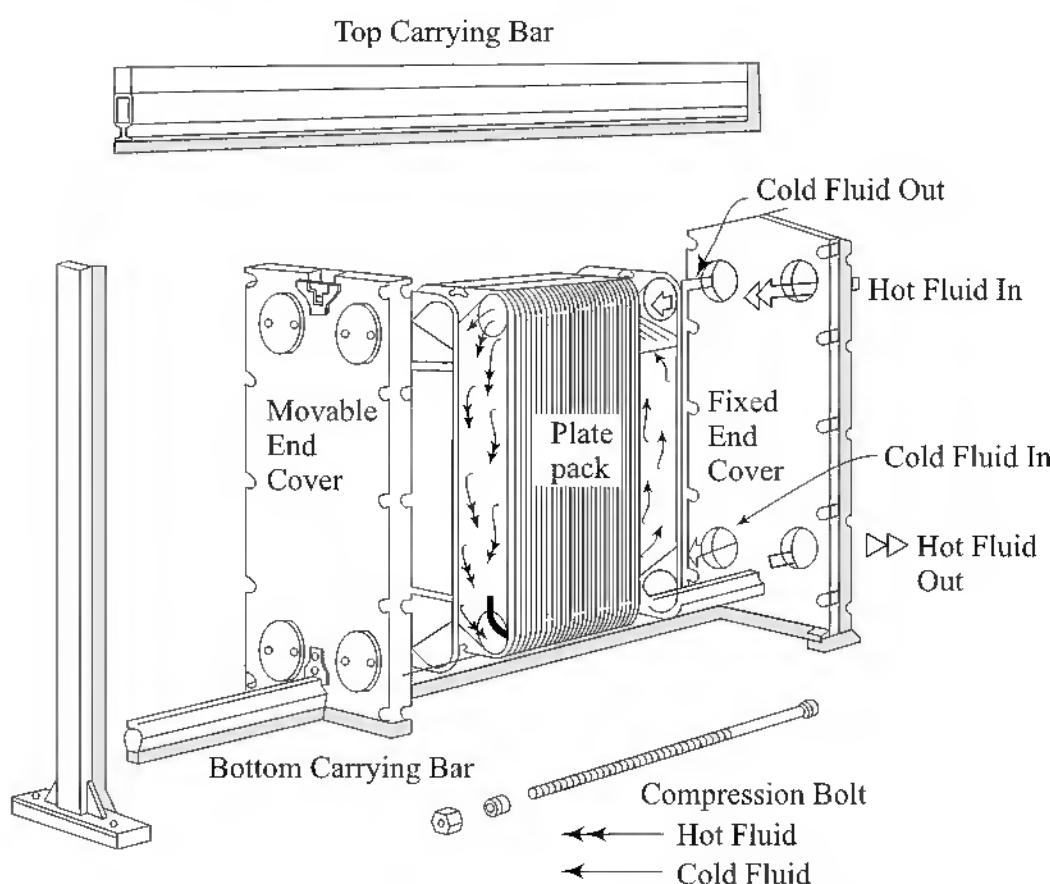
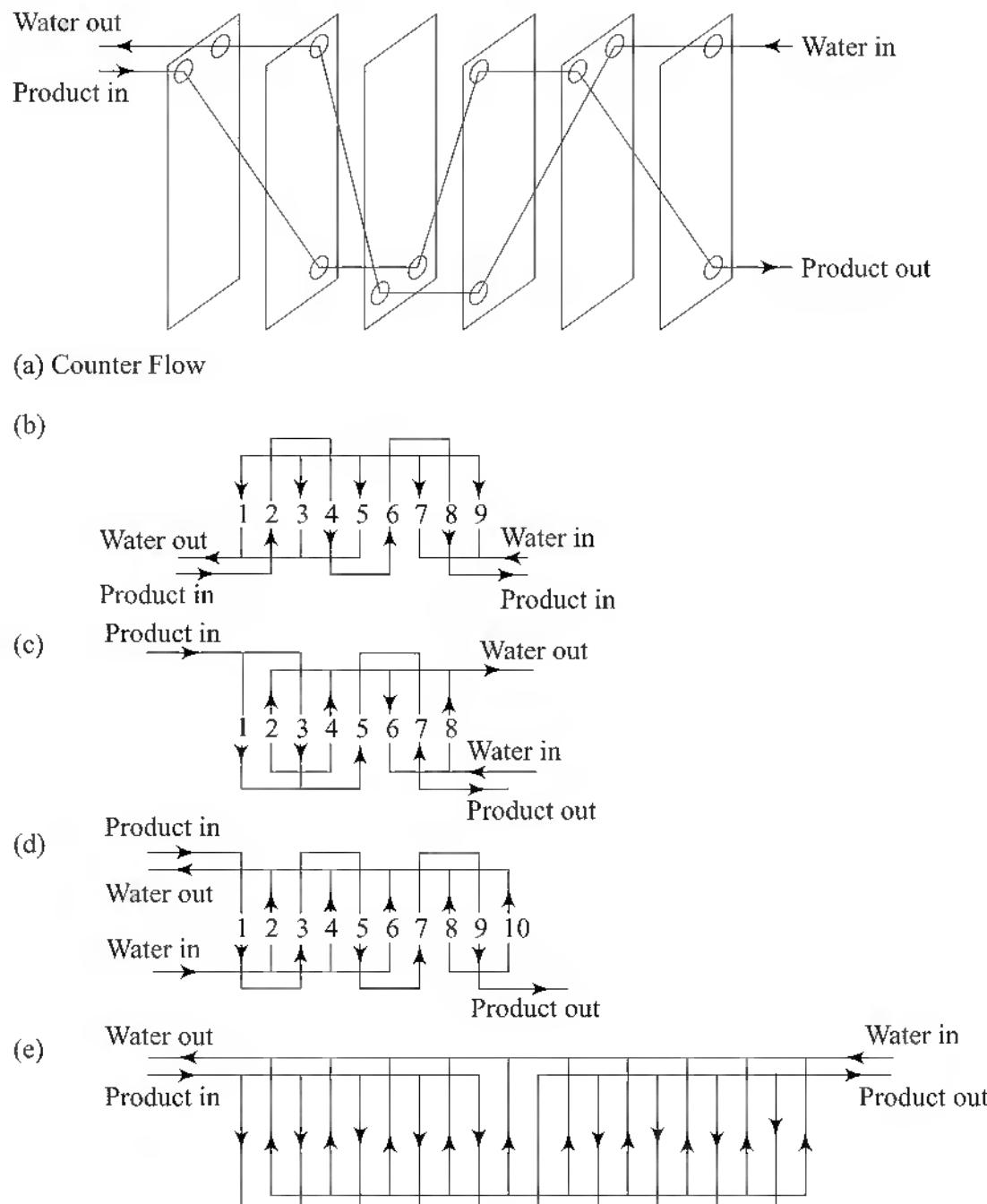


Fig. 6.34 Plate-and-frame Heat Exchanger. Hot Fluid Flows down between Alternate Plates and Cold Fluid Flows up between alternate plates.

Plates are pressed from stainless steel, titanium, Hastelloy C, Incoloy 825, nickel 200, Monel 400, aluminium brass, tantalum, etc. The thickness of plate ranges from 0.5 to 3 mm. The gap between the plates is ranging from 1.5 to 5 mm. This gap is maintained by gasket, which is sandwiched between two metal surfaces. Plate is having corrugated surface. Corrugated surface design strengthens the plates, increases the effective heat transfer area and produces turbulence in the fluids flowing through gaps. Plates are easily cleaned and replaced. The heat transfer area can be readily increased or decreased by adding or removing plates, respectively. Hot and cold fluids are allowed to flow in alternate channels. For any one plate, one side corrugated surface is in contact with cold fluid while the other side of corrugated surface is in contact with hot fluid. Variety of flow arrangements are possible.

**Fig. 6.35** Various Arrangements of Flow in Plate and Frame Heat Exchangers**Table 6.36** Gasket Materials for PHE

Gasket Material	Temperature limit, °C
Styrene butadine rubber	85
Medium Nitrile rubber	135
Butyl rubber	150
Fluorocarbon rubber	175
Compressed asbestos fiber	260

Heat transfer area per plate ranges from 0.03 to 1.5 m². Height to width ratio of plate ranges from 2 to 3. The upper limit of standard gasketed PHE is reported

as 650 m^2 of heat transfer surface. Total number of plates of this PHE is 400. Variety of gasket materials (Table 6.36) are used in plate size of this PHE having 2.8 m height and 1.1 m width.

6.10.2 Advantages and Disadvantages of Plate Heat Exchangers Over Shell and Tube Heat Exchangers

6.10.2.1 Advantages

- (a) Plate heat exchanger provides higher heat transfer coefficient as compared to shell and tube heat exchanger.
- (b) Maintenance and cleaning is easier with PHE as compared to shell and tube heat exchanger.
- (c) Fouling resistance is less with PHE.
- (d) Minimum driving force required for heat transfer is 1°C for PHE. While the same is 3 to 5°C for shell and tube heat exchanger.
- (e) PHE are more flexible. It is easy to increase or decrease the heat transfer area by adding or removing the plates.
- (f) With highly viscous liquid, PHE is better as it can be easily cleaned.

6.10.2.2 Disadvantages

- (a) Fixed cost of plate and hence of PHEs is higher. PHEs are not as widely used as shell and tube heat exchangers.
- (b) Gasketed PHE cannot be used if operating pressure is more than 30 bar or operating temperature is more than 250°C . While for the same case shell and tube heat exchanger can be used.
- (c) Internal leakage or mixing of two fluids is more common with PHE compared to shell and tube heat exchanger.
- (d) Liquid containing suspended particles tend to plug the flow area in PHE easily and so frequent cleaning becomes necessary. In shell and tube heat exchanger, chocking can be delayed /avoided by keeping higher velocity in tubes or by selecting bigger size tubes.

6.10.3 Design of Plate Heat Exchanger

General process design steps^{2,4,14,15} for PHE are same as that for shell and tube heat exchanger. Only differences are mentioned as follows:

1. LMTD correction factor (F_t) for PHE is determined from the value of number of transfer units, (NTU).

$$F_t = f(\text{NTU})$$

$$\text{where, } \text{NTU} = \frac{\Delta t}{\Delta T_{\ln}} \quad (6.88)$$

Δt = Temperature change required in process stream , $^\circ\text{C}$.

ΔT_{\ln} = Logarithmic mean temperature difference , $^\circ\text{C}$

For series flow, F_t can be taken as 0.95. For 1-1 pass and higher passes (2-2, 3-3, 4-4) curves of F_t vs NTU are available (refer Fig. 6.36).

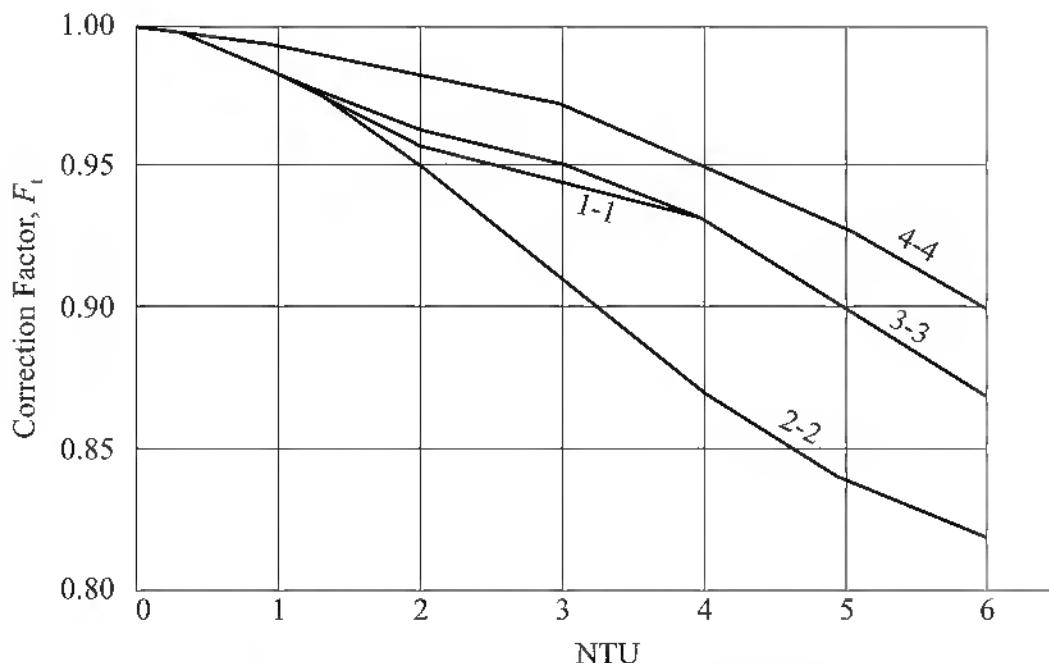


Fig. 6.36 Log Mean Temperature Correction Factor for Plate Heat Exchanger⁴
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2. If there is no phase change in the fluid, then forced convective heat transfer coefficient in conduits or plate film coefficient

$$\frac{h_p d_e}{k_f} = c Re^a Pr^b \left(\frac{\mu}{\mu_w} \right)^x \quad (6.89)$$

Reported values of constant and exponents are in the range of

$$c = 0.15 \text{ to } 0.4$$

$$a = 0.65 \text{ to } 0.85$$

$$b = 0.3 \text{ to } 0.45$$

$$x = 0.05 \text{ to } 0.2$$

Typical values are $c = 0.26$, $a = 0.65$, $b = 0.4$, and $x = 0.14$

where, h_p = Plate film coefficient, $\text{W}/(\text{m}^2 \cdot ^\circ\text{C})$

d_e = Equivalent diameter, m

$d_e = 2y$,

y = Gap between the plates, m

Re = Reynold number = $(d_e G_p) / \mu = (d_e u_p \rho) / \mu$

G_p = Mass velocity of fluid, $\text{kg}/(\text{m}^2 \cdot \text{s})$

$$= \dot{m}/A_f$$

\dot{m} = Mass flow rate of fluid per channel, kg/s

A_f = Cross sectional area of channel or gap (i.e. flow area), m^2

u_p = Channel velocity, m/s

3. Fouling coefficients for PHEs are given in Table 6.37. Fouling coefficients of PHEs are higher than the same for shell and tube heat exchangers.

Table 6.37 Fouling Coefficients for Plate Heat Exchangers

Fluid	Fouling coefficient, W/(m ² · °C)
Cooling water	10 000
Steam	100 000
Demineralized water	100 000
Soft water	50 000
Lubricating oil	6000
Light organic solvent	10 000
Process fluids	5000 – 20 000

4. Pressure drop in plate heat exchanger can be calculated by the following equation

$$\Delta p = \Delta p_p + \Delta p_{po} \quad (6.90)$$

where, Δp = Total pressure drop, Pa

Δp_p = Conduit or channel pressure drop, Pa

Δp_{po} = Port pressure drop, Pa

Pressure drop occurs, when fluid is flowing through conduit or channel

$$\Delta p_p = 8J_f \left(\frac{L_p}{d_e} \right) \frac{\rho u_p^2}{2} \quad (6.91)$$

where, d_e = Equivalent diameter for conduit, m

$d_e = 2y = 2 \times$ gap between plate, m

ρ = Density of fluid, kg/m³

u_p = Channel velocity, m/s

L_p = Path length, m

L_p = Plate length × number of passes

J_f = Friction factor = $f(Re)$

Value of J_f depends on the design of plate used. For the turbulent flow following approximate equation can be used.

$$J_f = 0.6 Re^{-0.3} \quad (6.92)$$

Δp_{po} = Pressure drop occurs due to the flow of fluid through ports

$$\Delta p_{po} = 1.3 \frac{\rho u_h^2}{2} \times N_p \quad (6.93a)$$

where, u_h = Velocity of fluid through the holes, m/s

$$= \frac{\dot{m}}{\rho A_h} \quad (6.93b)$$

$$A_h = \text{Area of hole} = \frac{\pi}{4} d_h^2, \text{ m}^2$$

d_h = Diameter of hole, m

N_p = Number of passes

Example 6.8

25 kg/s of ethanol liquid is to be cooled from 78°C to 40°C in gasketed plate heat exchanger. Operating pressure at inlet of heat exchanger is 2 atm g. Cooling water, available in plant at 32°C, is used as a cooling medium in heat exchanger. Design the suitable plate heat exchanger.

Solution:

$$\text{Heat duty, } \phi_t = \dot{m} C_L \Delta t$$

$$C_L = \text{Specific heat of ethanol liquid at average temperature (59°C), kJ/(kg} \cdot ^\circ\text{C)} \\ = 2.93 \text{ kJ/(kg} \cdot ^\circ\text{C)}$$

$$\phi_t = 25 \times 2.93 \times (78 - 40) = 2783.5 \text{ kW}$$

Let outlet temperature of cooling water = 40°C

$$\phi_t = 2783.5 \text{ kW} = \dot{m}_w \times 4.1868 \times (40 - 32)$$

$$\dot{m}_w = \text{Mass flow rate of cooling water} \\ = 83.1 \text{ kg/s} \equiv 301 \text{ m}^3/\text{h}$$

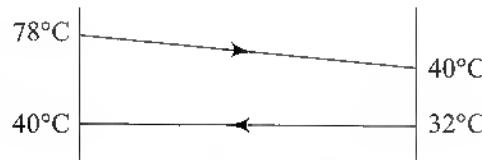


Fig. 6.37

Logarithmic mean temperature difference for the perfect counter current contact

$$\Delta T_{ln} = \frac{(78 - 40) - (40 - 32)}{\ln\left(\frac{38}{8}\right)} = 19.254^\circ\text{C}$$

Number of transfer unit (NTU) based on the maximum temperature difference

$$\text{NTU} = \frac{\Delta t}{\Delta T_{ln}} = \frac{78 - 40}{19.254^\circ\text{C}} = 1.9736 \quad (6.88)$$

For 1 : 1 pass arrangement, from Fig. 6.36

$$F_t = 0.96$$

$$\Delta T_m = F_t \Delta T_{ln} = 0.96 \times 19.254 = 18.484^\circ\text{C}$$

Assuming overall heat transfer coefficient

$$U_o = 2000 \text{ W/(m}^2 \cdot ^\circ\text{C)}$$

$$\phi_t = U_o A \Delta T_m$$

$$A = \frac{\phi_t}{U_o \Delta T_m} = \frac{2783.5 \times 10^3}{2000 \times 18.484} = 75.3 \text{ m}^2$$

Selecting the plate having effective width 0.5 m and effective length 1.5 m.

Effective area of one plate = $1.5 \times 0.5 = 0.75 \text{ m}^2$

For the first trial calculation

$$\text{Area provided} = 75.3 \text{ m}^2 \equiv \text{nos. of plates} \times 0.75 \text{ m}^2$$

No. of plates $\cong 100.4$

$$\text{Let no. of plates} = 101$$

$$\text{Area provided, } A_{pro} = 101 \times 0.75 = 75.75 \text{ m}^2$$

$$\text{Number of channels per pass} = (101 - 1)/2 = 50$$

$$\text{Let gap between successive plate, } y = 3 \text{ mm}$$

$$\text{Equivalent diameter } d_e = 2y = 6 \text{ mm} = 6 \times 10^{-3} \text{ m}$$

$$A_f = \text{Flow area} = \text{Cross sectional area of gap}$$

$$A_f = yW = 0.003 \times 0.5 = 1.5 \times 10^{-3} \text{ m}^2$$

Ethanol side heat transfer coefficient

$$\frac{h_p d_e}{K_p} = c Re^a Pr^b \left(\frac{\mu}{\mu_w} \right)^x \quad (6.89)$$

Values of c , a , b , and x depend on the plate design

$$\text{Let } c = 0.26, a = 0.65, b = 0.4 \text{ and } \frac{\mu}{\mu_w} \equiv 1$$

$$h_p \cdot \frac{d_e}{k_f} = 0.26 c Re^{0.65} Pr^{0.4}$$

$$\text{For the flow of ethanol, } Re = \frac{d_e G_p}{\mu}$$

$$\begin{aligned} \text{Mass velocity of ethanol, } G_{PE} &= \frac{\dot{m}_E}{\text{Total gap area}} \\ &= \frac{25}{50 \times 1.5 \times 10^{-3}} = 333.33 \text{ kg/(m}^2 \cdot \text{s)} \end{aligned}$$

$$\begin{aligned} \text{Channel velocity, } u_{PE} &= G_{PE}/\rho \\ \rho &= \text{Density of ethanol at } 59^\circ\text{C} = 775 \text{ kg/m}^3 \end{aligned}$$

$$u_{PE} = \frac{333.33}{775} = 0.43 \text{ m/s}$$

$$Re = \frac{6 \times 10^{-3} \times 333.33}{0.6 \times 10^{-3}} = 3333.3$$

Viscosity of ethanol at 59°C , $\mu = 0.6 \text{ cP}$

$$Pr = \frac{C_L \mu}{k}$$

Thermal conductivity of ethanol at 59°C , $k = 0.147 \text{ W/(m} \cdot {}^\circ\text{C)}$

$$Pr = \frac{2.93 \times 0.6 \times 10^{-3} \times 10^3}{0.147} = 11.96$$

$$h_{pe} = 0.26 \frac{k_f}{d_e} Re^{0.65} Pr^{0.4}$$

$$h_{pe} = 0.26 \times \frac{0.147}{6 \times 10^{-3}} \times (3333.3)^{0.65} (11.96)^{0.4}$$

$$h_{pe} = 3350.43 \text{ W/(m}^2 \cdot {}^\circ\text{C)}$$

Cooling water side heat transfer coefficient:

$$\text{Mass velocity of water, } G_{PW} = \frac{\dot{m}_w}{\text{Total gap area}}$$

$$G_p = \frac{83.1}{50 \times 1.5 \times 10^{-3}} = 1108 \text{ kg/(m}^2 \cdot \text{s)}$$

Density of water at 36°C, $\rho = 993.684 \text{ kg/m}^3$

$$h_{pw} = \frac{G_p}{\rho} = \frac{1108}{993.684} = 1.115 \text{ m/s}$$

$$\text{Reynold number } Re = \frac{d_e G_p}{\mu}$$

$$Re = \frac{6 \times 10^{-3} \times 1108}{0.72 \times 10^{-3}} = 9233$$

Viscosity of water at 36°C $\mu = 0.72 \text{ cP}$

Prandtl number for water at 36°C

$$Pr = \frac{C_{Lw} \mu}{k} = \frac{4.1868 \times 0.72 \times 10^{-3} \times 10^3}{0.6228}$$

Thermal conductivity of water at 36°C, $k = 0.6228 \text{ W/(m} \cdot ^\circ\text{C)}$

$$Pr = 4.84$$

$$h_{pw} = 0.26 \frac{k}{d_e} Re^{0.65} Pr^{0.4}$$

$$h_{pw} = \frac{0.26 \times 0.6228}{6 \times 10^{-3}} \times (9233)^{0.65} \times (4.84)^{0.4}$$

$$h_{pw} = 19168.66 \text{ W/(m}^2 \cdot ^\circ\text{C)}$$

Overall heat transfer coefficient:

$$\frac{1}{U} = \frac{1}{h_{pe}} + \frac{1}{h_{f_E}} + \frac{t_p}{k_p} + \frac{1}{h_{p_w}} + \frac{1}{h_{f_w}}$$

$$h_{pe} = 3350.43 \text{ W/(m}^2 \cdot ^\circ\text{C)}$$

h_{fe} = Fouling coefficient for ethanol = 10 000 W/(m² · °C) (from Table 6.37)

t_p = Plate thickness = 1 mm = 0.001 m

k_p = Thermal conductivity of plate material

Let the plate material be titanium.

$$k_p = 21 \text{ W/(m} \cdot ^\circ\text{C)}$$

$$h_{pw} = 19168.66 \text{ W/(m}^2 \cdot ^\circ\text{C)}$$

h_{f_w} = Fouling coefficient for cooling water in PHE
= 10 000 W/(m² · °C) (From Table 6.37)

$$\frac{1}{U} = \frac{1}{3350.43} + \frac{1}{10000} + \frac{0.001}{21} + \frac{1}{19168.66} + \frac{1}{10000}$$

$$U = 1671.52 \text{ W/(m}^2 \cdot ^\circ\text{C)}$$

$$\text{Area required} = \frac{\phi_t}{U\Delta T_m} = \frac{2783.5 \times 1000}{1671.52 \times 18.484} = 90 \text{ m}^2$$

Area required > Area provided

For the second trial calculations

No. of plates 121 with 2-2 pass arrangement

$$\text{No. of channels per pass} = \frac{121 - 1}{2 \times 2} = 30$$

$$\text{Area provided, } A_{\text{pro}} = 121 \times 0.75 = 90.75 \text{ m}^2$$

For NTU = 1.9736 $F_t = 0.95$ (From Fig. 6.36)

$$\Delta T_m = 0.95 \times 19.254 \text{ } ^\circ\text{C} = 18.2913 \text{ } ^\circ\text{C}$$

h_{PE} :

$$\text{Mass velocity, } G_{pe} = 333.33 \times \frac{50}{30} = 555.55 \text{ kg/(m}^2 \cdot \text{s)}$$

$$\text{Channel velocity, } u_{pe} = 0.43 \times \frac{5}{3} = 0.7167 \text{ m/s}$$

$$Re = \frac{d_e G_{PE}}{\mu} = \frac{6 \times 10^{-3} \times 555.55}{0.6 \times 10^{-3}} = 5555.5$$

$$h_{pe} = 3350.43 \times \left(\frac{5555.5}{3333.3} \right)^{0.65} = 4669.84 \text{ W/(m}^2 \cdot {^\circ}\text{C)}$$

h_{pw} :

$$\text{Mass velocity, } G_{pw} = 1108 \times \frac{5}{3} = 1846.67 \text{ kg/(m}^2 \cdot \text{s)}$$

$$\text{Channel velocity, } u_{pw} = 1.115 \times \frac{5}{3} = 18583 \text{ m/s}$$

$$Re = 9233.33 \times \frac{5}{3} = 15389$$

$$h_{pw} = 19168.66 \times \left(\frac{15389}{9233.33} \right)^{0.65} = 26717.5 \text{ W/(m}^2 \cdot {^\circ}\text{C)}$$

Overall coefficient

$$\frac{1}{U} = \frac{1}{4669.84} + \frac{1}{10000} + \frac{0.001}{21} + \frac{1}{26717.5} + \frac{1}{10000}$$

$$U = 2003.25 \text{ W/(m}^2 \cdot {^\circ}\text{C)}$$

$$\text{Area required, } A_{\text{req}} = \frac{\phi_t}{U\Delta T_m} = \frac{2783.5 \times 1000}{2003.25 \times 18.2913} = 75.9646 \text{ m}^2$$

$$\text{Area provided, } A_{\text{pro}} = 90.75 \text{ m}^2$$

$$\% \text{ Excess area} = \left(\frac{90.75}{75.9646} - 1 \right) \times 100 = 19.46\% \text{ (acceptable)}$$

Pressure drops

Ethanol side pressure drop

$$\Delta p = \Delta p_p + \Delta p_{po} \quad (6.90)$$

Channel pressure drop

$$\Delta p_p = 8J_f \left(\frac{L_p}{d_e} \right) \frac{\rho u_p^2}{2} \quad (6.91)$$

$$J_f = 0.6 Re^{-0.3}$$

$$= 0.6(5555.5)^{-0.3} = 0.045158 \quad (6.92)$$

Path length, L_p = Plate length \times No. of passes = $1.5 \times 2 = 3$ m

$$\Delta p_p = 8 \times 0.045158 \times \left(\frac{3}{0.006} \right) \times \frac{775 \times 0.7167^2}{2}$$

$$= 35953.5 \text{ N/m}^2 \equiv 35.954 \text{ kPa}$$

Let port diameter (hole diameter) = 120 mm

$$\text{Hole area} = \left(\frac{\pi}{4} \right) 0.12^2 = 0.01131 \text{ m}^2$$

u_h = velocity of ethanol through hole (port)

$$u_h = \frac{W}{\rho A_h} = \frac{25}{775 \times 0.01131}$$

$$= 2.8522 \text{ m/s}$$

Port pressure drop

$$\Delta p_{po} = 1.3 \frac{\rho u_h^2}{2} \times N_p$$

$$= 1.3 \times \frac{775 \times 2.8522^2}{2} \times 2 = 8196 \text{ N/m}^2 \equiv 8.196 \text{ kPa}$$

Ethanol side pressure drop

$$\Delta p = 35.954 + 8.196 = 44.15 \text{ kPa}$$

It may be noted that port pressure drop is 18.6 % of total pressure drop which is significant.

Cooling water side pressure drop

$$Re = 15389$$

$$J_f = 0.6 Re^{-0.3} = 0.6 \times (15389)^{-0.3} = 0.033265$$

$$L_f = \text{Plate length} \times \text{No. of passes} = 1.5 \times 2 = 3 \text{ m}$$

Channel pressure drop

$$\Delta p_p = 8J_f \left(\frac{L_p}{d_e} \right) \frac{\rho u_p^2}{2} \quad (6.91)$$

$$= 8 \times 0.033265 \times \left(\frac{3}{0.006} \right) \times \frac{993.684 \times 1.8583^2}{2}$$

$$= 228295.6 \text{ N/m}^2 \equiv 228.296 \text{ kPa} \quad (\text{very high})$$

Trial-2:

To decrease the cooling water side pressure drop, let number of cooling water side passes = 1 i.e. 2 : 1 arrangement. Ethanol side heat transfer coefficient, velocity and pressure drop are same as that for the previous case (2-2 arrangement).

For 2 : 1 arrangement

$$u_p = 0.7167 \text{ m/s}, h_{pe} = 4669.84 \text{ W/(m}^2 \cdot ^\circ\text{C}), \Delta p = 44.15 \text{ kPa} \quad (\text{ethanol side})$$

For cooling water,

No. of plates = 121, No. of passes = 1

For the flow of water,

$$\text{No. of channels per pass} = \frac{121 - 1}{2} = 60$$

$$\text{Mass velocity, } G_{pw} = \frac{1846.67}{2} = 923.33 \text{ kg/(m}^2 \cdot \text{s})$$

$$\text{Velocity of water through channel, } u_p = \frac{1.8583}{2} = 0.929 \text{ m/s}$$

$$Re = \frac{15389}{2} = 7694.5$$

$$h_{pw} = 26717.5 \times \left(\frac{1}{2}\right)^{0.65} = 17026.54 \text{ W/(m}^2 \cdot ^\circ\text{C})$$

Overall heat transfer coefficient

$$\frac{1}{U} = \frac{1}{2003.25} + \frac{1}{17026.54} - \frac{1}{26717.5}$$

$$U = 1921.26 \text{ W/(m}^2 \cdot ^\circ\text{C})$$

$$\text{Area required } A_{\text{req}} = \frac{\phi_t}{U \Delta T_m} \frac{2783.5 \times 1000}{1921.26 \times 18.2913} = 79.206 \text{ m}^2$$

$$\% \text{ Excess area} = \left(\frac{90.75}{79.206} - 1 \right) \times 100 = 14.575\%$$

Cooling water side pressure drop:

$$Re = 7694.5$$

$$J_f = 0.6 Re^{-0.3} = 0.6 \times (7694.5)^{-0.3} = 0.04095$$

Channel pressure drop

$$\Delta p_p = 8J_f \left(\frac{L_p}{d_e} \right) \times \frac{\rho u_p^2}{2} \quad (6.91)$$

Path length L_p = Plate length × No. of passes = 1.5 m

$$\begin{aligned} \Delta p_p &= 8 \times 0.04095 \times \left(\frac{1.5}{0.006} \right) \times \frac{993.684 \times 0.929^2}{2} \\ &= 35118.3 \text{ N/m}^2 \equiv 35.118 \text{ kPa} \end{aligned}$$

Hole or port diameter = 120 mm

Hole area = 0.011 31 m²

u_h = Velocity of water through hole (port)

$$u_h = \frac{W}{\rho A_h} = \frac{83.1}{993.684 \times 0.01131} = 7.3942 \text{ m/s (high)}$$

Increase the port size to 150 mm.

$$u_h = \frac{W}{\rho A_h} = \frac{\dot{m}_w}{\rho A_h} = \frac{83.1}{993.684 \times \frac{\pi}{4} (0.15)^2} = 4.732 \text{ m/s}$$

$$\Delta p_{pt} = \frac{\rho u_p^2}{2} \times N_p = 1.3 \times \frac{993.684 \times 4.732^2}{2} = 14462.7 \text{ N/m}^2 \equiv 14.463 \text{ kPa}$$

Pressure drop for the flow of cooling water $\Delta p = 35.118 \text{ kPa} + 14.463 \text{ kPa} = 49.58 \text{ kPa}$

Here also, port pressure drop is 29.2% of total pressure drop.

6.11 SPIRAL FLOW HEAT EXCHANGERS

6.11.1 Introduction

Spiral flow heat exchangers provide a spiral flow path for one or both (cold and hot) fluids being handled. They are of two types; (i) spiral tube exchanger and (ii) spiral-plate exchanger. These heat exchangers offer the following advantages over conventional shell and tube heat exchangers.

- (a) Compactness.
- (b) Centrifugal force increases the heat transfer coefficient particularly of highly viscous liquid slurries or sludges.
- (c) Spiral plate heat exchangers foul at much lower rate than the shell and tube heat exchanger because of the single flow passage and curved flow path which does not allow easy settlement of solids.
- (d) Relative ease of cleaning.
- (e) Spiral configuration reduces stress associated with differential thermal expansion.
- (f) Maintenance cost is less.

Disadvantages of spiral-tube and spiral-plate heat exchangers over shell and tube heat exchangers are as follows.

- (a) Spiral flow heat exchangers are more expensive (requires higher fixed cost) than shell and tube heat exchangers having the same heat transfer surface.
- (b) Design of spiral flow heat exchangers is not well established like the same of shell and tube heat exchanger.
- (c) Pressure drop (loss) is higher than the same in shell and tube heat exchanger.
- (d) Maximum design pressure for spiral plate heat exchanger is 10 bar g as the spiral construction limits the design pressure.
- (e) Gasket is special and assembly requires skill.

6.11.2 Spiral Plate Heat Exchanger

Spiral plate heat exchanger is fabricated from two relatively long strips of plate which are spaced apart and wound around an open split center to form a pair of concentric spiral passages. Spacing is maintained uniformly along the length of the spiral by spacer studs welded to plate. Commonly used material for the fabrication of spiral plate heat exchanger are carbon steel, stainless still. Hastelloys, nickel and nickel alloys, titanium, copper-alloys, etc.

Figure 6.38 shows spiral flow in both channels. Flow channels are closed by welding alternate channels at both sides of spiral plate.

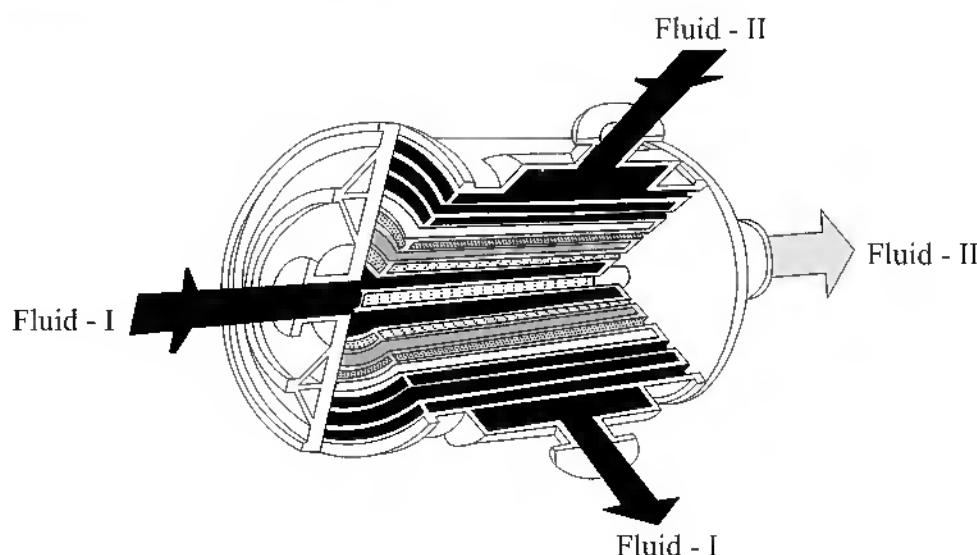


Fig. 6.38 Spiral Flow in Both Channels

Figure 6.39 shows flow is spiral in one channel, axial in other. One channel is completely open on both ends and other closed at both sides of plate.

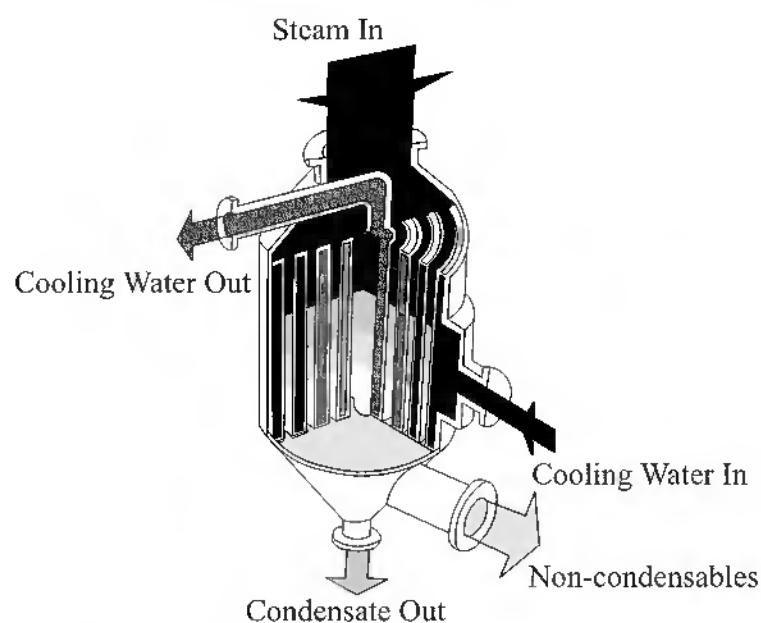


Fig. 6.39 Spiral Flow in One Channel, Axial in Another

In spiral plate heat exchangers following three types of flow arrangements are possible.

- Spiral flow in both channels (as shown in Fig. 6.38)
- Spiral flow in one channel and axial flow in other (as shown in Fig. 6.39)
- Combination flow (as shown in Fig. 6.40)

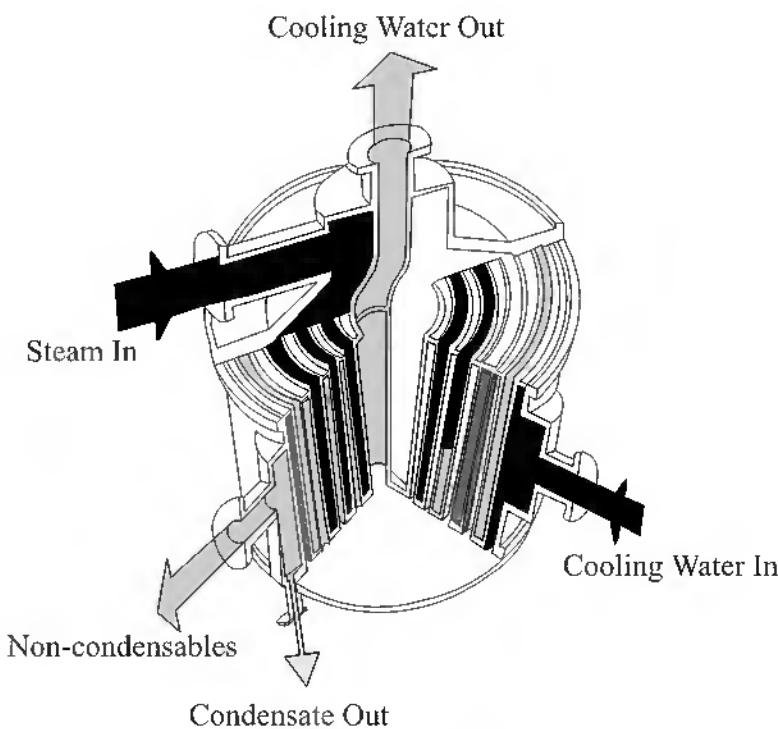


Fig. 6.40 Combination Flow

A. Spiral Flow in Both Channels

This type of flow arrangement is used for liquid-to-liquid services. With this type of flow arrangement spiral plate heat exchanger is covered by flat heads on both sides. In this arrangement usually two liquids flow counter currently with the cold liquid entering at the periphery and flowing toward the core and the hot liquid entering at the core and flowing toward the periphery. Use of this type spiral heat exchanger is common in cooling hot hydrogenated edible oil from deodorizer as it offers good heat transfer coefficient for viscous oil.

B. Spiral Flow in One Channel and Axial Flow in Other

This type of flow arrangement is used for condensing or boiling. With this type of flow arrangement spiral flow heat exchanger is covered by conical heads on one or both sides. Condensation or boiling takes place in axial direction. This arrangement is preferred where there is a large difference in the volumes of two fluids. In case of condensation, difference between volumetric flow rates of condensing vapour and cooling medium is always large. In case of boiling, if hot oil is used as heating medium, then also the difference between volumetric flow rate of boiling liquid and the same of hot oil is large.

C. Combination Flow

Combination flow is used to condense vapours. In this arrangement condensing vapour flows axially and then condensate flows spirally. This arrangement is used for condensation with subcooling. Part of the open spiral is kept closed at the top. Entering fluid (condensing vapour) flows axially through the center part of assembly in downward direction. Condensate at bottom flow spirally and coming out from the side bottom. With this arrangement spiral heat exchanger is equipped with conical head at top and flat cover at bottom.

6.11.3 Process Design Steps for Spiral Plate Heat Exchanger

General process design steps^{15,16} are as follows:

- (i) Calculate heat duty
- (ii) Select cooling medium or heating medium
- (iii) Based on energy balance calculate the mass flow rate of heating medium or cooling medium.
- (iv) Calculate the LMTD.
- (v) Assume the value of overall heat transfer coefficient for the first trial calculation.
- (vi) Determine the heat transfer area required based on the assumed value of overall heat transfer coefficient.

$$A = \frac{\phi_t}{U \times \Delta T_{ln}} = \text{Heat transfer area provided for the first trial calculation}$$

- (vii) Heat transfer area for spiral plate heat exchanger

$$A = 2 \times L \times H \quad (6.94)$$

where, A = Heat transfer area, m^2

L = Length of plate, m

H = Width of plate = Height or length of heat exchanger, m

Fix the value of H and find the value of L

$$L = \frac{A}{2 \times H} \quad (6.95)$$

Diameter of heat exchanger or diameter of outside spiral can be calculated by following equation.

$$D_s = [1.28 \times L(d_c + d_h + 2t) + c^2]^{1/2} \quad (6.96)$$

where, D_s = Outside spiral diameter, m

L = Length of plate, m

d_c = Channel spacing of cold side, m

d_h = Channel spacing of hot side, m

t = Plate thickness, m

c = Core diameter, m

Some dimensions of spiral plate heat exchanger are standardized and are as follows.

1. Plate width H : 101.6 mm (4 in), 152.4 mm (6 in),
304.8 mm (12 in), 457.2 mm (18 in),
609.6 mm (24 in), 762 mm (30 in)
914.4 mm (36 in), 1219.2 mm (48 in),
1524 mm (60 in) 1778 mm (72 in)
2. Core Diameter, C : 203.2 mm (8 in), 304.8 mm (12 in)
3. Channel spacing, d_c and d_h : 4.76 mm (3/16 in) (for 304.8 mm maximum width)
6.35 mm (1/4 in) (for 1219.2 mm maximum width)
7.94 mm (5/16 in), 9.525 mm (3/8 in),
12.7 mm (1/2 in), 19.05 mm (3/4 in),
25.4 mm (1 in)
4. Plate thickness, t : For carbon steel 3.175 mm (1/8 in),
4.76 m (3/16 in),
6.35 mm (1/4 in), 7.94 mm (5/16 in)
For stainless steel 14 to 3 US gauge

In spiral plate heat exchanger to arrive at the best compact design outside diameter of spiral D_s and plate width (H) are kept approximately equal.

(viii) Calculate the equivalent diameter of flow channel by following equation.

$$D_e = \frac{4 \times \text{flow area}}{\text{wetted perimeter}} = \frac{4[\text{spacing} \times \text{width}]}{2(\text{spacing} \times \text{width})}$$

For hot fluid

$$D_{eh} = \frac{4 \times (d_h \times H)}{2(d_h + H)} \quad (6.97)$$

For cold fluid

$$D_{ec} = \frac{4 \times (d_c \times H)}{2(d_c + H)} \quad (6.98)$$

(ix) Calculate the Reynolds number by following equation.

$$Re = \frac{2\dot{m}}{H \times \mu} \quad (6.99)$$

where, \dot{m} = Mass flow rate of fluid, kg/s

H = Width of plate, m

μ = Viscosity of fluid, kg/(m · s)

Reynolds number is calculated for both; cold fluid and hot fluid.

(x) Calculate the critical Reynolds number Re_c . Critical Reynolds number is the value of Reynolds number above which turbulent flow is achieved.

$$Re_c = 20\,000 \left(\frac{D_e}{D_s} \right)^{0.32} \quad (6.100)$$

where, Re_c = Critical Reynolds number

D_e = Equivalent diameter of flow channel, m

D_s = Outside diameter of spiral, m

- (xi) Calculate the cold fluid side and hot fluid side heat transfer coefficients by using the suitable correlations. Various correlations for calculating heat transfer coefficient for spiral plate heat exchangers are given as follows.

(a) For spiral flow with no phase change and $Re > Re_c$ (i.e. turbulent flow):

$$\frac{h}{C_p G} = \left(1 + 3.5 \left(\frac{D_e}{D_s} \right) \right) (0.023 Re^{-0.2} \times Pr^{-2/3}) \quad (6.101)$$

For spiral flow with, no phase change and, $Re < Re_c$ (i.e. laminar flow)

$$\frac{h}{C_p G} = 1.86 Re^{-2/3} Pr^{-2/3} \left(\frac{d}{D_s} \right)^{1/6} \left(\frac{\mu_f}{\mu_b} \right)^{-0.14} \quad (6.102)$$

where, h = Heat transfer coefficient, $\text{W}/(\text{m}^2 \cdot ^\circ\text{C})$

C_p = Specific heat of fluid, $\text{J}/(\text{kg} \cdot ^\circ\text{C})$

G = Mass velocity of fluid, $\text{kg}/(\text{m}^2 \cdot \text{s})$

Re = Reynolds number

$$Pr = \text{Prandtl number} = \frac{C_p \mu}{k}$$

μ = Viscosity of fluid, $\text{kg}/(\text{m} \cdot \text{s})$

k = Thermal conductivity of fluid, $\text{W}/(\text{m} \cdot ^\circ\text{C})$

d = Channel spacing, d_c or d_h , m

D_s = Outside spiral diameter, m

Here mass velocity is calculated by following equation

$$G = \frac{\dot{m}}{(d \times H)} \quad (6.103)$$

where, \dot{m} = Mass flow rate, kg/s

$d = d_c$ or d_h , m

H = Width of plate, m

- (c) For axial flow with, no phase change and, $Re > 10\,000$

$$\frac{h}{C_p G} = 0.023 Re^{-0.2} Pr^{-2/3} \quad (6.104)$$

- (xii) Calculate the overall heat transfer coefficient by following equation.

$$\frac{1}{U} = \frac{1}{h_c} + \frac{1}{h_h} + \frac{1}{h_{cd}} + \frac{1}{h_{hd}} + \frac{t}{k_m} \quad (6.105)$$

where, U = Overall heat transfer coefficient, $\text{W}/(\text{m}^2 \cdot {}^\circ\text{C})$
 h_c = Cold fluid side heat transfer coefficient, $\text{W}/(\text{m}^2 \cdot {}^\circ\text{C})$
 h_h = Hot fluid side heat transfer coefficient, $\text{W}/(\text{m}^2 \cdot {}^\circ\text{C})$
 h_{cd} = Cold fluid side fouling coefficient, $\text{W}/(\text{m}^2 \cdot {}^\circ\text{C})$
 h_{hd} = Hot fluid side fouling coefficient, $\text{W}/(\text{m}^2 \cdot {}^\circ\text{C})$
 t = Thickness of plate, m
 k_m = Thermal conductivity of plate material, $\text{W}/(\text{m} \cdot {}^\circ\text{C})$

(xiii) Calculate the heat transfer area required

$$A_r = \frac{\phi_r}{U \Delta T_{\ln}} \quad (6.106)$$

(xiv) Calculate the % excess heat transfer area. Ideally it should be in between 10 to 20%.

(xv) Calculate the cold fluid side and hot fluid side pressure drops by using the suitable correlations. Various correlations for calculating pressure drop for spiral flow heat exchanger are given as follows.

(a) Spiral flow with no phase change and $Re > Re_c$

$$\Delta p = 0.0789 \frac{L}{\rho} \left[\frac{W}{d_s H} \right]^2 \left[\frac{1.3 \mu^{1/3}}{(d_s + 0.0032)} \left(\frac{H}{W} \right)^{1/3} + 1.5 + \frac{16}{L} \right] \quad (6.107)$$

(b) Spiral flow with no phase change and $100 < Re < Re_c$

$$\Delta p = 36.84 \frac{L}{\rho} \left[\frac{W}{d_s H} \right] \left[\frac{1.035 \mu^{1/2}}{(d_s + 0.0032)} \left(\frac{H}{W} \right)^{1/2} \left(\frac{\mu_f}{\mu_b} \right) + 1.5 + \frac{16}{L} \right] \quad (6.108)$$

(c) Spiral flow with no phase change and $Re < 100$

$$\Delta p = 5.5256 \times 10^{-5} \left[\frac{L \rho u}{d_s^{2.75}} \right] \left(\frac{\mu_f}{\mu_b} \right)^{0.17} \left(\frac{W}{H} \right) \quad (6.109)$$

(d) Axial flow with no phase change and, $Re > 10\,000$

$$\Delta p = 253.85 \left(\frac{W}{L} \right)^{1.8} \left(0.0115 \mu^{0.2} \frac{H}{d_s} + 1 + 0.03 H \right) \quad (6.110)$$

where, Δp = Pressure drop, Pa

L = Total length of plate, m

ρ = Density of fluid, kg/m^3

$W = \dot{m}$ = Mass flow rate of fluid, kg/s

d_s = Channel spacing (d_c or d_h), m

H = Width of plate, m

μ = Viscosity of fluid, $\text{kg} / (\text{m} \cdot \text{s})$

μ_b = Bulk fluid viscosity, $\text{kg}/(\text{m} \cdot \text{s})$

Calculated pressure drop must be less than or equal to maximum allowable pressure drop or optimum pressure drop.

Exampel 6.9

Design a spiral flow plate heat exchanger for cooling the malamine suspension liquid from 47°C to 33°C by using cooling water as cooling medium based on the following data. Cooling water is available at 32°C.

Data

- (i) Composition and flow rate of melamine suspension liquid

Table 6.38

Component	kg/h	mole %	mass %
Water	16 052	74.2	57.21
Carbon dioxide	3151	5.49	11.23
Ammonia	3055	14.96	10.89
Urea	1329	1.84	4.74
Melamine	4470	2.95	15.93
Total	28 057	100	100

- (ii) Physical properties of melamine suspension liquid

- (a) Specific heat $C_L = 3.349 \text{ kJ/(kg} \cdot ^\circ\text{C)}$
- (b) Thermal conductivity $k = 0.4935 \text{ W/(m} \cdot ^\circ\text{C)}$
- (c) Viscosity

At 47°C, $\mu = 1.8 \text{ cP}$

At 33°C, $\mu = 2.3 \text{ cP}$

- (d) Density

At 47°C, $\rho = 1120 \text{ kg/m}^3$

At 33°C, $\rho = 1126 \text{ kg/m}^3$

- (iii) Suitable material of construction: SA: 240 Gr 316 L

- (iv) Maximum allowable pressure drops

For malamine suspension liquid = 1.5 bar

For cooling water = 1 bar

Solution:

Heat duty

$$\phi_t = \dot{m} C_L \Delta t = \left(28 057 \times \frac{1}{3600} \right) \times 3.349 \times (47 - 33) \\ = 365.41 \text{ kW}$$

Mass flow rate of cooling water:

Energy balance

$$\phi_t = \dot{m}_w C_{Lw} \Delta t = \dot{m}_w \times 4.1868 \times (36 - 32)$$

For the first trial calculations let the outlet temperature of cooling water = 36°C

$$\dot{m}_w = \frac{365.41}{4.1868 \times 4} = 21.819 \text{ kg/s} \equiv 78.55 \text{ m}^3/\text{h}$$

LMTD

$$\Delta T_{ln} = \frac{(47 - 36) - (33 - 32)}{\ln\left(\frac{47 - 36}{33 - 32}\right)}$$

$$\Delta T_{ln} = 4.17^{\circ}\text{C}$$

Assuming the value of overall heat transfer coefficient

$$U_o = 1500 \text{ W}/(\text{m}^2 \cdot ^{\circ}\text{C})$$

Heat transfer area

$$A = \frac{\phi_t}{U \Delta T_m} = \frac{365.41 \times 1000}{1500 \times 4.17} = 58.42 \text{ m}^2$$

Heat transfer area provided for the first trial calculations

$$A = 58.42 \text{ m}^2 = 2 \times L \times H \quad (6.94)$$

Let $H = 24 \text{ in} = 0.6096 \text{ m}$

$$L = \frac{58.42}{2 \times 0.6096} = 47.9167 \text{ m}$$

To get the best compact design of spiral plate heat exchanger, width of plate (or length of heat exchanger) should be approximately equal to outside spiral diameter (or outside diameter of heat exchanger.)

Let other diamensions of heat exchanger:

d_c = Channel spacing for cold side fluid = 12.7 mm

d_h = Channel spacing for hot side fluid = 6.35 mm

c = Core diameter = 203.2 mm

t = Plate thickness = 3.175 mm

Outside spiral diameter

$$\begin{aligned} D_s &= [1.28 \times L(d_c + d_h + 2t) + c^2]^{1/2} \\ &= [1.28 \times 47.9167(0.0127 + 0.00635 + 2 \times 0.003175) + 0.2032^2]^{1/2} \\ &= 1.2646 \text{ m} \end{aligned} \quad (6.96)$$

$$H \neq D_s$$

For second trial calculations let $H = 36 \text{ in} = 914.4 \text{ mm}$

$$L = 31.944 \text{ m}$$

$$\begin{aligned} D_s &= [1.28 \times 31.944(0.0127 + 0.00635 + 2 \times 0.003175) + 0.2032^2]^{1/2} \\ &= 1.04 \text{ m} \end{aligned}$$

$$H \approx D_s$$

$$H = 914.4 \text{ mm}, \quad D_s = 1040 \text{ mm}, \quad L = 31.944 \text{ mm}$$

$$d_c = 12.7 \text{ mm}, \quad d_h = 6.35 \text{ mm}, \quad C = 203.2 \text{ mm}$$

$$t = 3.175 \text{ mm}$$

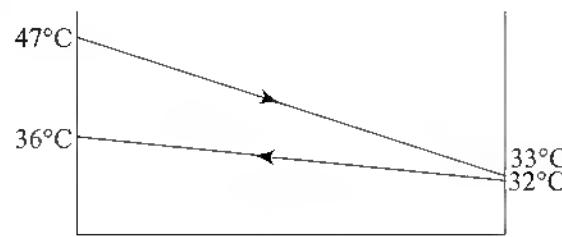


Fig. 6.41

Note: These dimensions of heat exchanger are not the final dimensions. These can be changed after heat transfer coefficient calculations and/or pressure drop calculations.

Hot fluid side heat transfer coefficient calculations:

Equivalent diameter

$$D_{eh} = \frac{4 \times (d_h \times H)}{2(d_h + H)} \quad (6.97)$$

$$D_{eh} = \frac{4 \times (6.35 \times 914.4)}{2(6.35 + 914.4)} = 12.6 \text{ mm}$$

Reynolds number,

$$Re = \frac{2 \dot{m}}{H \times \mu} \quad (6.99)$$

$$\left(\mu_{av} \frac{1.8 + 2.3}{2} = 2.05 \text{ cP} \right)$$

$$Re = \frac{2 \times 7.7936}{0.9144 \times 2.05 \times 10^{-3}} = 8315.3$$

Critical Reynolds number,

$$Re_c = 20\,000 \left(\frac{D_{eh}}{D_s} \right)^{0.32} = 20\,000 \left(\frac{12.6}{1040} \right)^{0.32} \\ = 4871.9$$

$Re > Re_c$ i.e. process fluid will flow in turbulent region

For spiral flow with, no phase change and $Re > Re_c$

$$\frac{h_h}{c_p G} = \left(1 + 3.5 \left(\frac{D_{eh}}{D_s} \right) \right) (0.023 Re^{-0.2} \times Pr^{-2/3}) \quad (6.101)$$

$$C_p = 3.349 \text{ kJ/(kg} \cdot ^\circ\text{C)}$$

$$\text{Prandtl number, } Pr = \frac{C_p \mu}{k}$$

$$= \frac{3.349 \times \frac{10^3}{1} \times (2.05 \times 10^{-3})}{0.4935} \\ = 13.912$$

$$\text{Mass velocity, } G = \frac{\dot{m}}{d_h \times H} = \frac{7.7936}{0.00635 \times 0.9144} \\ = 1342.234 \text{ kg/(m}^2 \cdot \text{s)}$$

$$\frac{h_h}{3.349 \times 10^3 \times 1342.234} = \left(1 + 3.5 \left(\frac{12.6}{1040} \right) \right) \times 0.023 \times 8315.3^{-0.2} \times 13.912^{-2/3}$$

$$h_h = 3063.9 \text{ W/(m}^2 \cdot \text{s)}$$

Cold fluid side heat transfer coefficient calculations

$$\begin{aligned} D_{e_c} &= \frac{4 \times (d_c \times H)}{2(d_c + H)} \\ &= \frac{4 \times (12.7 \times 914.4)}{2(12.7 + 914.4)} = 25.052 \text{ mm} \end{aligned} \quad (6.98)$$

Reynolds number,

$$Re = \frac{2\dot{m}}{H \times \mu} = \frac{2 \times 21.819}{0.9144 \times 0.738 \times 10^{-3}} \quad (6.99)$$

(Viscosity of water at 34°C = 0.738 cP)

$$Re = 64\,665.44$$

Critical Reynolds number,

$$\begin{aligned} Re_c &= 20\,000 \left(\frac{D_{e_c}}{D_s} \right)^{0.32} = 20\,000 \left(\frac{25.052}{1040} \right)^{0.32} \\ &= 6070.28 \end{aligned}$$

$$Re > Re_c$$

For spiral flow with, no phase change, and $Re > Re_c$

$$\begin{aligned} \frac{h_c}{C_p G} &= \left(1 + 3.5 \left(\frac{D_{e_c}}{D_s} \right) \right) (0.023 Re^{-0.2} Pr^{-2/3}) \quad (6.101) \\ C_p &= 4186.8 \text{ J/(kg} \cdot ^\circ\text{C)} \end{aligned}$$

Mass velocity $G = \frac{\dot{m}}{d_c \times H} = \frac{21.819}{0.0127 \times 0.9144} = 1878.9 \text{ kg/(m}^2 \cdot \text{s)}$

Prandtl number, $Pr = \frac{C_p \times \mu}{k} = \frac{4186.8 \times 0.738 \times 10^{-3}}{0.62}$

(Thermal conductivity of water at 34°C $k = 0.62 \text{ W/(m} \cdot k)$)

$$Pr = 4.9836$$

$$\begin{aligned} \frac{h_c}{4186.8 \times 1878.9} &= \left(1 + 3.5 \left(\frac{25.052}{1040} \right) \right) \times 0.023 \times 64665.44^{-0.2} \times 4.9836^{-2/3} \\ h_c &= 7336.76 \text{ W/(m}^2 \cdot ^\circ\text{C)} \end{aligned}$$

Overall heat transfer coefficient

$$\frac{1}{U} = \frac{1}{h_c} + \frac{1}{h_h} + \frac{1}{h_{e_d}} + \frac{1}{h_{h_d}} + \frac{t}{k_m} \quad (6.105)$$

where, $h_c = 7336.76 \text{ W/(m}^2 \cdot ^\circ\text{C)}$, $h_h = 3063.9 \text{ (m}^2 \cdot ^\circ\text{C)}$,

$$h_{e_d} = 15\,000 \text{ W/(m}^2 \cdot ^\circ\text{C)}$$

$$h_{h_d} = 15\,000 \text{ W/(m}^2 \cdot ^\circ\text{C)}$$

$$t = 0.003\ 175 \text{ m}$$

$$k_m = 16.26 \text{ W/(m} \cdot ^\circ\text{C)} \text{ (of stainless steel)}$$

$$\frac{1}{U} = \frac{1}{7336.76} + \frac{1}{3063.9} + \frac{1}{15\ 000} + \frac{1}{15\ 000} + \frac{0.003\ 175}{16.26}$$

$$U = 1263.78 \text{ W/(m}^2 \cdot ^\circ\text{C)}$$

Heat transfer area required, $A_r = \frac{\phi_t}{U \Delta T_{ln}}$

$$A_r = \frac{365.41 \times 1000}{1263.78 \times 4.17} = 69.34 \text{ m}^2$$

Required area, $A_r >$ Area provided, A_{pr} which is not acceptable.
For the second trial calculations let $L = 42 \text{ m}$

$$\left(L \approx \frac{69.34 \times 1.1}{2 \times H} \right)$$

Area provided $A_p = 2 \times L \times H$
 $A_p = 2 \times 42 \times 0.9144 = 76.81 \text{ m}^2$

Revised value of D_s (outside diameter of spiral)

$$D_s = [1.28 \times L(d_c + d_h + 2t) + c^2]^{1/2}$$

$$= [1.28 \times 42(0.0127 + 0.006\ 35 + 2 \times 0.003\ 175) + 0.2032^2]^{1/2}$$

$$= 1.186 \text{ m}$$

Critical Reynolds number for hot fluid

$$Re_c = 20\ 000 \left(\frac{12.6}{1186} \right)^{0.32} = 4671.34$$

$$Re > Re_c$$

Revised value of hot fluid side heat transfer coefficient

$$h_h = \frac{\left(1 + 3.5 \left(\frac{12.6}{1186} \right) \right)}{\left(1 + 3.5 \left(\frac{12.6}{1040} \right) \right)} \times 3063.9$$

$$h_h = 3048.56 \text{ W/(m}^2 \cdot ^\circ\text{C)}$$

Critical Reynolds number for cold fluid

$$Re_c = 20\ 000 \left(\frac{25.052}{1186} \right)^{0.32} = 5820.4$$

$$Re > Re_c$$

Revised value of cold fluid side heat transfer coefficient

$$h_c = \frac{\left(1 + 3.5 \left(\frac{25.052}{1186} \right) \right)}{\left(1 + 3.5 \left(\frac{25.052}{1040} \right) \right)} \times 7336.76 = 7266.53 \text{ W/(m}^2 \cdot ^\circ\text{C)}$$

Overall heat transfer coefficient

$$\frac{1}{U} = \frac{1}{3048.56} + \frac{1}{7266.53} + \frac{1}{15000} + \frac{1}{15000} + \frac{0.003175}{16.26}$$

$$U = 1259.07 \text{ W/(m}^2 \cdot ^\circ\text{C)}$$

Heat transfer area required

$$A_r = \frac{365.41 \times 1000}{1259.07 \times 4.17} = 69.6 \text{ m}^2$$

Area provided, $A_p = 76.81 \text{ m}^2 > A_r = 69.6 \text{ m}^2$

$$\% \text{ Excess heat transfer area} = \frac{76.81 - 69.6}{69.6} \times 100 = 10.36\% \text{ (acceptable)}$$

Malamine suspension liquid side pressure drop:

$$\Delta p = 0.0789 \frac{L}{\rho} \left[\frac{W}{d_s H} \right] \left[\frac{1.3 \mu^{1/3}}{(d_s + 0.0032)} \left(\frac{H}{W} \right)^{1/3} + 1.5 + \frac{16}{L} \right] \quad (6.107)$$

$L = 42 \text{ m}, \rho = 1123 \text{ kg/m}^3, W = 7.7936 \text{ kg/s}$

$d_s = d_h = 0.00635 \text{ m}, H = 0.9144 \text{ m}$

$\mu = 2.05 \times 10^{-3} \text{ kg/(m} \cdot \text{s)}$

$$\Delta p = 0.0789 \times \frac{42}{1123} \left[\frac{7.7936}{0.00635 \times 0.9144} \right]^2$$

$$\left[\frac{1.3(2.05 \times 10^{-3})^{1/3}}{0.00635 + 0.0032} \times \left(\frac{0.9144}{7.7936} \right)^{1/3} + 1.5 + \frac{16}{42} \right]$$

$$= 55.004.5 \text{ Pa} \equiv 55 \text{ kPa} \equiv 0.55 \text{ bar} < 1 \text{ bar} \quad (\text{Acceptable})$$

Cooling water side pressure drop

$$\Delta p = 0.0789 \frac{L}{\rho} \left(\frac{W}{d_s H} \right)^2 \left[\frac{1.3 \mu^{1/3}}{(d_s + 0.0032)} \left(\frac{H}{W} \right)^{1/3} + 1.5 + \frac{16}{L} \right] \quad (6.107)$$

$L = 42 \text{ m}, \rho = 1000 \text{ kg/m}^3, W = 21.819 \text{ kg/s}$

$d_s = d_c = 0.0127 \text{ m}, H = 0.9144 \text{ m}, \mu = 0.738 \times 10^{-3} \text{ kg/(m} \cdot \text{s)}$

$$\Delta p = 0.0789 \times \frac{42}{1000} \left[\frac{21.819}{0.0127 \times 0.9144} \right]^2$$

$$\left[\frac{1.3 \times (0.7378 \times 10^{-3})^{1/3}}{(0.0127 + 0.0032)} \left(\frac{0.9144}{21.819} \right)^{1/3} + 1.5 + \frac{16}{42} \right]$$

$$= 52.023.5 \text{ Pa} \equiv 0.52 \text{ bar} < 1 \text{ bar}$$

Comments: In the given case, malamine suspension liquid solution is a slurry solution. Hence, spiral plate heat exchanger is better choice as compared to shell and tube heat exchanger because of the following reasons.

- (i) Slurry solution must be allocated to shell side of shell and tube heat exchanger to facilitate the ease of flow. Shell and tube heat exchanger must be U-tube or floating

head heat exchanger to facilitate removal of tube bundle and the frequent cleaning. Also, malamine solution required the use of special material, SS: 316 L. Hence, shell, tubes, tube sheets, baffles, floating head and/or one of the heads must be of SS 316 L.

- (ii) Shell and tube heat exchanger provides less heat transfer coefficient for the slurry solution as compared to spiral plate heat exchanger. Also, fouling coefficients are lesser with shell and tube heat exchanger compared to spiral plate heat exchanger. Hence shell and tube heat exchanger requires more heat transfer area.
- (iii) Cleaning is easier with spiral plate heat exchanger but requires skill.
- (iv) Because of the compactness spiral plate heat exchanger will occupy less space as compared to shell and tube heat exchanger.
- (v) Here cooling water is available at 32°C. It can cool the malamine solution upto 33°C with spiral plate heat exchanger design, wherein driving force is only 1°C. Minimum driving force required for heat transfer in shell and tube heat exchanger is 3°C.

Based on (i) and (ii) it can be concluded that the cost difference between spiral plate heat exchanger and shell and tube heat exchanger is less.

6.12 BRAZED ALUMINIUM PLATE-FIN HEAT EXCHANGER

Typical brazed aluminium plate-fin heat exchanger handling multiple streams is shown in Fig. 6.42. This type of heat exchanger is preferred for low temperature (sub zero temperature) cryogenic applications as it offers the following advantages.

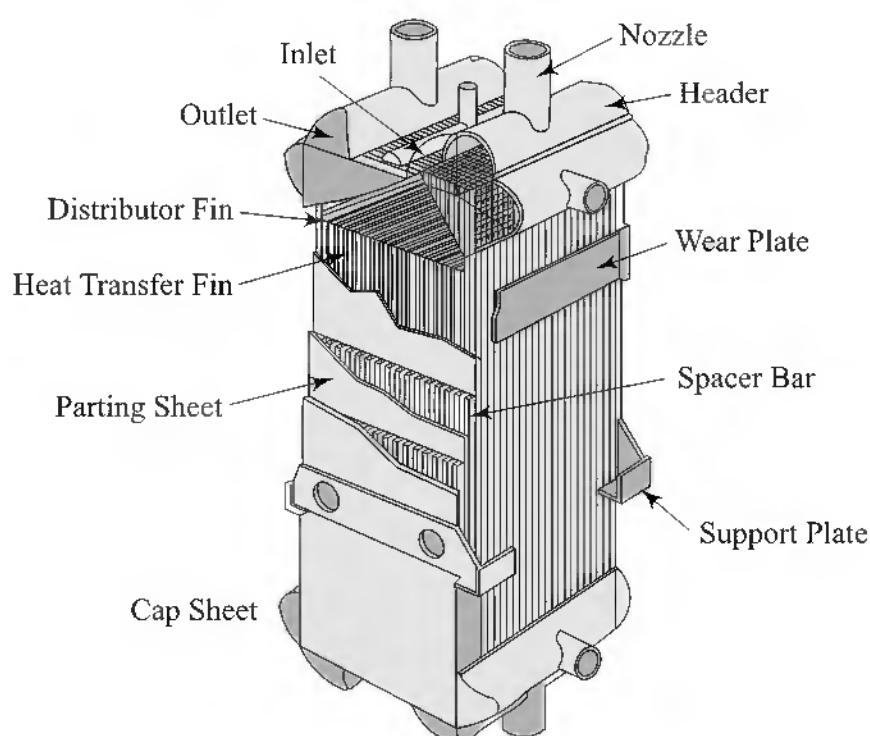


Fig. 6.42 Brazed Aluminium Plate-fin Heat Exchanger¹⁷

- (a) It can handle temperature approaches down to less than 2°C. In cryogenic duties operating cost is dominated by the cost of energy required to generate the low temperatures; hence such close temperature approach is very important.
- (b) It is the most compact and lightest heat exchanger and it provides the maximum heat transfer area density.
- (c) It can operate with a thermal effectiveness up to 98%.
- (d) It has multistream capability. All cold streams produced in process can be passed through single heat exchanger to cool the incoming warm streams.
- (e) It is an expensive heat exchanger.

6.12.1 Construction and Working

It consists of alternating layers of plates and corrugated fins. Flow passes are formed between the consecutive plates (plates are known as parting sheets). These sheets provide the primary surface and fins provide the secondary surface for heat transfer.

Brazed or corrugated fins hold the heat exchanger together. At the end of heat exchanger pads of finning are laid at an angle and serve as distributors, while in major part of heat exchanger finning are laid parallel to the axis. Streams are flowing truly in counter current directions. Each stream flows in number of layers, each of which is divided into numerous parallel, nearly rectangular sub-channels by the fins. Fin heights and frequencies decide the size of these sub-channels. Fin heights are in between 5 to 9 mm and fin frequencies in the main heat transfer region is 590 to 787 fins/m (15 to 20 fins/in). The equivalent hydraulic diameters of these subchannels are only a few millimeters. These small passages result in very high heat transfer area density in the range of 800 to 1500 m²/m³. For comparison, heat transfer area densities of shell and tube heat exchangers are about 50 to 250 m²/m³ and the same of plate and frame heat exchangers are about 200 to 1000 m²/m³. Such high area density and with aluminium construction make this heat exchanger the smallest as well as the lightest among all other type of heat exchangers. Typical size of such a heat exchanger can be 1.2 m wide, 1.2 m deep and 6.2 m long. This heat exchanger can also be used as vaporizer or condenser.

Brazed aluminium plate heat exchangers can be used at pressures up to 100 bar and within a temperature range of -269°C to 100°C. With appropriate alloys for the headers and nozzles, they can be used up to 200°C. However, the maximum permissible temperature for aluminium alloys decreases rapidly with increasing pressure.

6.13 HEAT EXCHANGER NETWORKING FOR ENERGY INTEGRATION

Energy requirement of a chemical plant can be considerably reduced by process integration. For example, in acetaldehyde plant, exothermicity of the reaction (e.g. partial oxidation of ethanol) is utilized as a part of heat duty required by the reboiler of the distillation column of the same plant. This integration reduces

saturated steam required for the reboiler of distillation column and eliminates the requirement of cooling medium for the reactor. Large scale chemical plant uses large number of heat exchangers. These heat exchanges are either heaters and vaporizers or coolers and condensers. Integration between few of them reduces hot utility requirement (e.g. requirement of saturated steam) as well as cold utility requirement. (e.g. requirement of cooling water). Systematic method is developed by Linnhoff and others¹⁸ for the energy integration via systematic heat exchanger networking which is known as *pinch technology*.

In heat exchanger networking via pinch technology, following facts are considered.

1. The heat transferred from a hot stream must be equal to heat transferred from cold stream (heat losses are neglected).
2. Heat can only be transferred from a hotter fluid to a colder fluid (second law of thermodynamics). Therefore, the temperature of a cold fluid must be less than that of a hot fluid at all points along the path of heat exchanger.

ΔT_m : In actual energy integration temperature of hot stream must be reasonably greater than the same of cold stream to get an acceptable rate of heat transfer. This acceptable minimum temperature difference is also called *pinch temperature difference*. Choice of pinch temperature difference ΔT_{min} is important. The lower value of ΔT_{min} decreases the energy requirement but increases the fixed cost of heat exchangers. Minimum suggested value of ΔT_{min} is 10°C. This implies that in any of the exchangers to be used in the network, the temperature difference between the hot stream and cold stream will not be less than 10°C. For any network, there will be a best value for the minimum temperature difference (ΔT_{min}) that will give the lowest total annual operating cost. It is usually in between 10°C to 20°C.

“Hot streams” and “Cold streams”: For heat exchanger networking total streams considered are divided in to two types. “Hot streams” are the streams that need cooling (heat sources) while “Cold streams” are the streams that need heating (heat sinks).

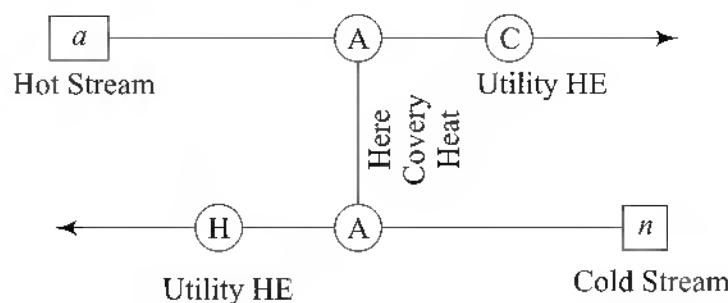
6.13.1 Representation of Heat Exchanger Network

Heat exchanger network is represented as a grid. The process streams are drawn as horizontal lines, with the stream numbers shown in square boxes, as shown in Fig. 6.45. Hot streams are drawn at the top of grid and flow from left to right. The cold streams are drawn at bottom, and flow from right to left.

Coolers and heaters are represented as circles; (C) and (H), respectively. Heat exchangers which are used to exchange the heat between the two process streams are marked by two circles and the two circles are connected by vertical line. Two circles with vertical line connect the two streams between which heat is being exchanged.

As shown in Fig. 6.43, in this case a is hot stream number, n is cold stream number. A is a heat exchanger exchanging the heat between hot stream a and cold stream n . Cooler C cooles the stream further after it leaves the heat exchanger A. Heater H heats the stream n after it leaves the heat exchanger A.

Calculations for finding minimum utility requirements for the given stream data:

**Fig. 6.43 Grid Presentation**

First step for this is selection of the value of minimum temperature difference, ΔT_{\min} between hot streams and cold streams. Then for the chosen value of ΔT_{\min} , minimum utility required can be determined by the following method, developed by Hohmann and Lockhart. This method is illustrated by following example.

Example 6.10

Synthesize the heat exchanger network for the following four process streams such that resulting heat exchanger network will require the minimum hot and cold utilities. Also, find the values of minimum utilities (hot and cold) required.

Table 6.39 Process Stream Data

Stream	Type	$\dot{m} C_p, \text{kW}/^\circ\text{C}$	$t_{in}, ^\circ\text{C}$	$t_{out}, ^\circ\text{C}$
1	Hot	10	250	130
2	Hot	8	160	90
3	Cold	7	60	160
4	Cold	6	110	260

Solution:

Select the value of $\Delta T_{\min} = 10^\circ\text{C}$. (Selected value of ΔT_{\min} , may not be equal to optimum temperature difference.) For $\Delta T_{\min} = 10^\circ\text{C}$, Table 6.40 is prepared by calculations, presented below.

Method for Preparing the Table:

The hottest temperature in the data is cold stream 4 outlet temperature (260°C). List it at the top of the cold stream temperature column (column 2). Note down the corresponding hot stream temperature (270°C), which is 10°C ($= \Delta T_{\min}$) hotter at the top of the hot stream temperature column. Next highest temperature is of hot stream 1 temperature; 250°C . Corresponding cold stream temperature based on $\Delta T_{\min} = 10^\circ\text{C}$ is 240°C and written second in column 2. Similarly, temperatures of hot streams and cold streams are written in descending order either in column 1 (if it is hot stream temperature) or in column 2 (if it is cold stream temperature). While corresponding temperatures for $\Delta T_{\min} = 10^\circ\text{C}$ are written in other columns.

There are seven temperature intervals in this problem. In column 3 available heat by hot streams for each temperature interval is calculated. No hot stream is available at 270°C , hence available heat for 7th interval is zero. Since hot stream temperature of 270°C is of academic interest only and so it is shown in brackets in column 1.

Table 6.40 Cascaded Heat Flow Calculations

1 Hot stream temperature °C	2 Cold streams temperature °C	3 Composite avail. heat kW	4 Hot streams casacated heat kW	5 Composite req'd heat kW	6 Cold streams casa'd heat kW	7 Grand composite net heat kW	8 Hot and cold streams Cascaded Heat kW	9 Adjusted Cascaded Heat, kW
(270°C)	260°C	0	0	120	0	-120	0	120
250°C	7 (240°C)	800	0	480	120	-120	-120	0
170°C	6 160°C	100	800	800	600	320	200	320
160°C	5 (150°C)	900	130	900	730	-30	170	290
130°C	4 (120°C)	540	390	1,440	1,120	150	320	440
120°C	3 110°C	80	130	130	1,250	-50	270	390
90°C	2 (80°C)	240	210	1,520	1,460	30	300	420
70°C	1 60°C	0	140	1,760	1,600	-140	160	280

For 6th interval (in column 3) available heat is calculated as $(\dot{m}C_p)_1 (250^\circ\text{C} - 170^\circ\text{C}) = 10 (250 - 170) = 800 \text{ kW}$. So, in sixth interval hot stream 1 is cooled from 250°C to 170°C. Stream 1 is also the only hot stream in interval 5, contributing another 100 kW, when it cools from 170°C to 160°C. Interval 4 has both hot streams 1 and 2. In interval 4 both streams are cooled from 160°C to 130°C. Hence available heat for interval 4 is calculated as follows

$$[(\dot{m}C_p)_1 + (\dot{m}C_p)_2] (160^\circ\text{C} - 130^\circ\text{C}) = ((10 + 8) \times 30) = 540 \text{ kW}$$

In column 4 cascaded heat available indicates cumulative or total heat available at temperature t , when hot streams cool from 250°C to temperature $t^\circ\text{C}$.

In column 5 required heat by cold streams for each temperature interval is calculated. Stream 4 is present only in interval 7 and requires 120 kW heat to increase its temperature from 240°C to 260°C, $(\dot{m}C_p)_4 (260 - 240) = 6(260 - 240) = 120 \text{ kW}$.

In interval 5 both streams 3 and 4 are present. Hence, required heat for interval 5 is $[(\dot{m}C_p)_3 + (\dot{m}C_p)_4] (160 - 150) = ((6 + 7) \times 10) = 130 \text{ kW}$. Cascaded required heat by cold streams at different temperatures are shown in column 6.

Column 7 represents the net available heat for each interval.

Net available heat (column 7) of any interval = Heat available with hot streams (column 3) in this interval – Heat required by cold streams (column 5) in the same interval

Column 8 represents the cascaded net available heat or cumulative net available heat at the end of each interval, starting from the 7th interval to 1st interval. Cascaded heat is the amount of heat available from the hot streams over that required by cold streams at any temperature t , as one starts the calculation from the highest temperature to the lowest temperature. Find the highest negative number in column 8. In this example it is -120 kW. It implies that 120 kW is the minimum amount of heat that must be supplied from hot utilities. The same amount of heat (120 kW) is added in each number of 8th column. This gives 9th column. The point in the last column where the value of adjusted cascaded heat is zero is the pinch point. Top number in last column (120 kW), is the minimum amount of heat that must be supplied from hot utilities and the bottom number (280 kW) is the minimum amount of heat that must be removed by cold utilities.

Grand Composite Curve (GCC):

It is the most important curve or graph to understand heat exchanger network synthesis problem. To draw this curve for the given problem, Table 6.40 must be prepared, based on given data. Then GCC is the plot of adjusted cascaded heat obtained in the last column (9th column) vs hot stream/cold stream temperatures. (Column 1 and 2). GCC for this example is shown in Fig. 6.44.

In this curve each interval is represented by line segment. Each interval is either consuming heat (heat sink) or producing heat (heat source). Interval which acts as a heat sink, represented by a line segment that moves down and to the left. Interval which acts as a heat source, represented by a line segment that moves down and to the right. For example 7th interval locally requires 120 kW of heat input (heat sink), hence it is represented by line segment which moves downward and to the left. Interval 6 produces 320 kW of heat (heat source), hence it is represented by line segment moving downward and to the right.

Whenever there is a heat source segment just above a heat sink segment right facing noze appear in the Grand Composite Curve. Such right facing nozes can be self integrated. While the bold segment above the pinch point requires heat from hot utilities and

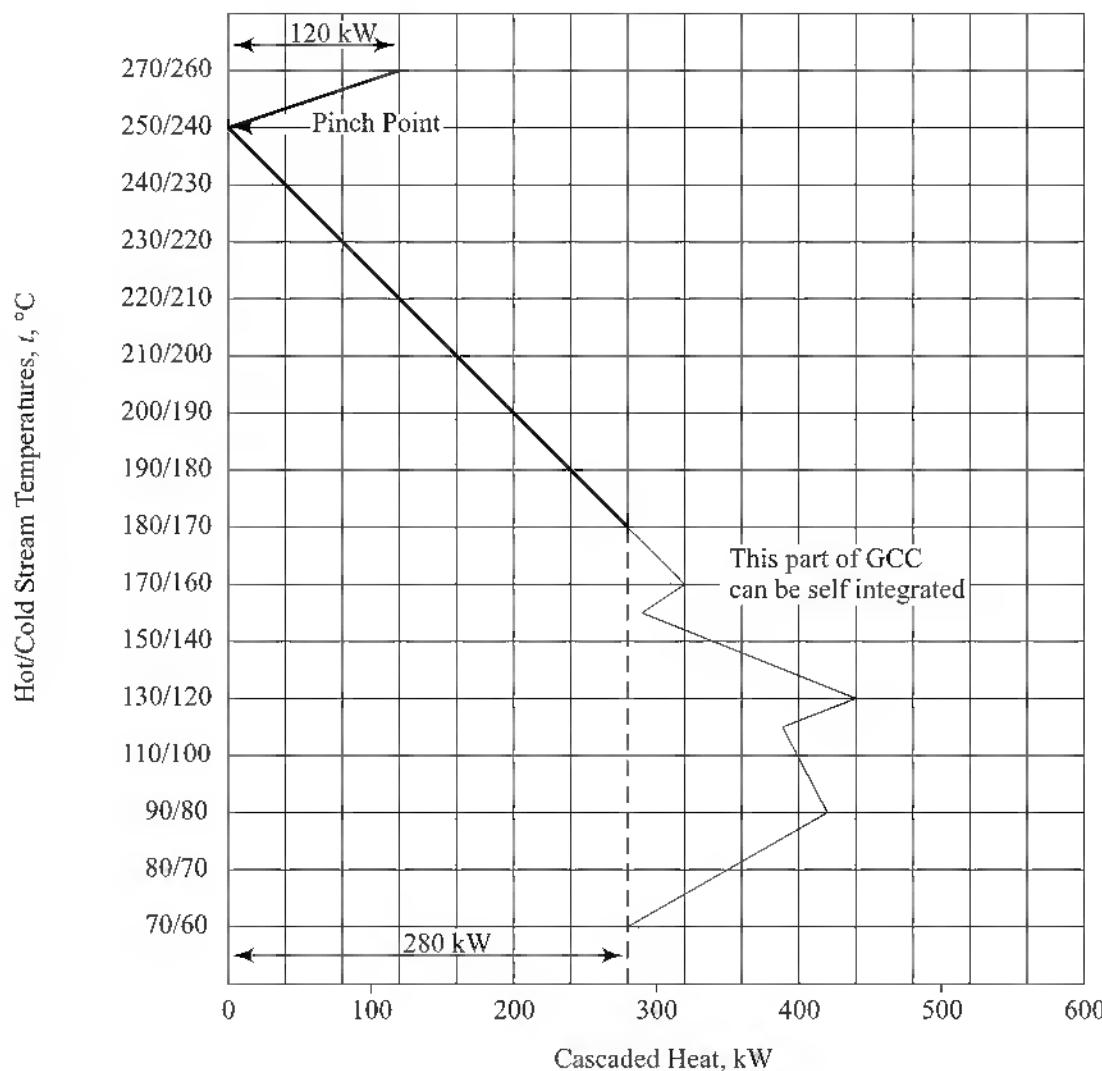


Fig. 6.44 Grand Composite Curve

bold segment below the pinch up to the dashed line represents the heat which must be removed by cold utilities.

Heat exchanger network synthesis for the maximum energy recovery:

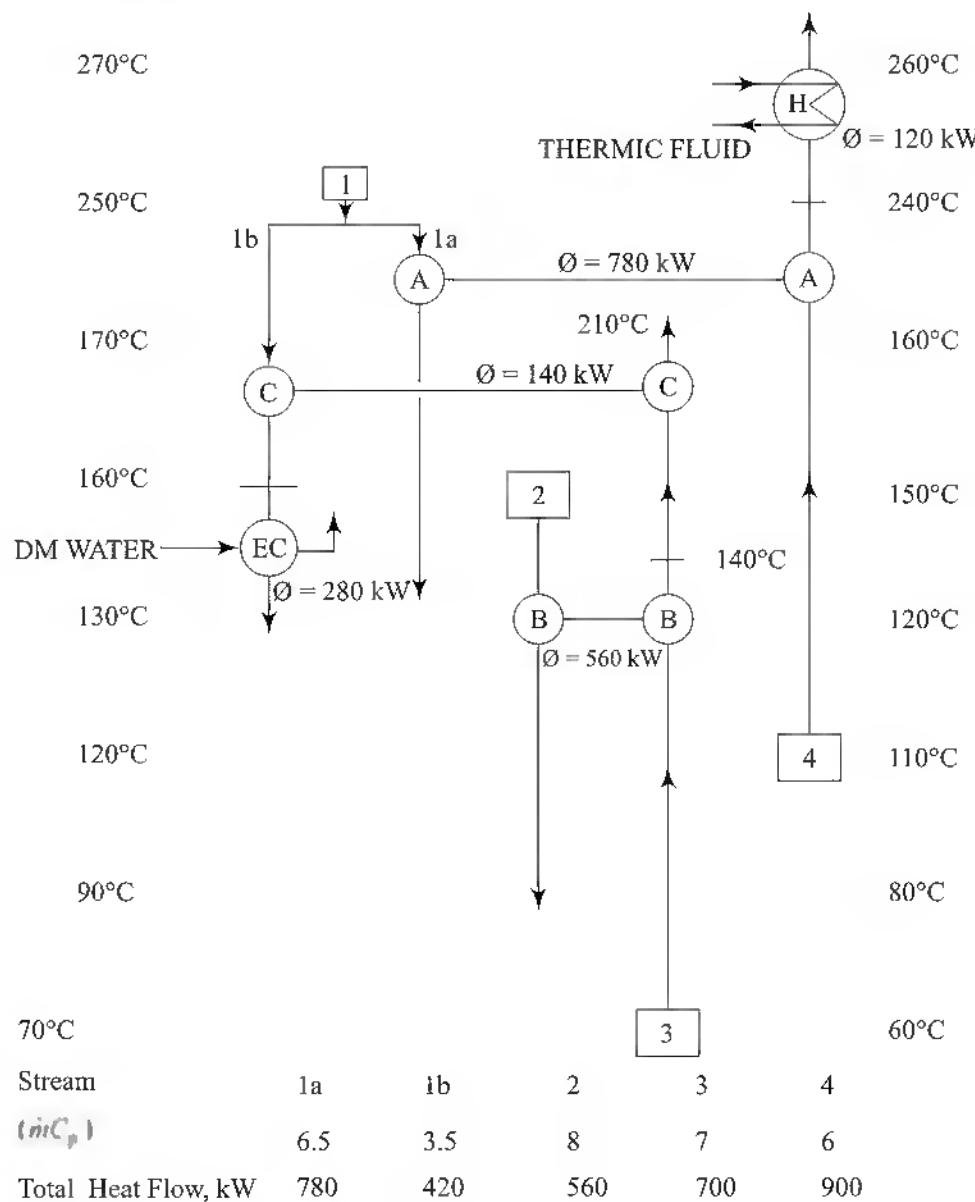
Network design for the maximum energy recovery and minimum use of hot and cold utilities can be divided in the following steps.

- Select the value of ΔT_{\min} .
- Prepare the table (like Table 6.40) for drawing the Grand Composite Curve.
- Calculate the minimum hot utility required and the minimum cold utility required. Identify the pinch point.
- For the maximum heat recovery do not use cold utilities above the pinch and do not use hot utilities below the pinch. Also, do not transfer heat across the pinch.
- Estimate the number of exchangers above the pinch equal to $N - 1$, where N is the number of streams above the pinch. Similarly estimate the number of exchangers below the pinch equal to $M - 1$, where M is the number of streams below the pinch.
- Invent a suitable network.

Applying the same steps to the given problem.

- $\Delta T_{\min} = 10^\circ \text{C}$ (selected)

- (b) GCC is drawn based on the value of ΔT_m (Fig. 6.44).
- (c) Minimum hot utility required is to supply 120 kW heat and the minimum cold utility required is for removing 280 kW heat. Pinch point occurs at 250°C hot stream temperature and equivalent cold stream temperature of 240°C.
- (d) Above the pinch only stream 4 exists. Hot oil (another stream) can be used to increase the temperature of stream 4 from 240°C to 260°C. Thus $N = 2$ which means one heat exchanger is needed for this heat duty. Heat duty of this heater is 120 kW. Below the pinch all four process streams and one cold utility stream exists ($M = 5$). Thus there is a need of four exchangers for this part or below the pinch.
- (e) One possible heat exchanger network for the maximum energy recovery is shown in Fig. 6.45.

**Fig. 6.45 Heat Exchanger Network**

Hot stream 1 contains $(\dot{m}C_p) \Delta t = 10 \times (250 - 130) = 1200$ kW heat. Part of this heat can be utilized to increase the temperature of stream 4 from 110°C to 240°C by using the heat exchanger A. Heat duty of heat exchanger A is $6 \times (240 - 110) = 780$ kW. Stream 1 is divided in two parts *a* and *b*. Stream *a* is used to heat stream 4.

$$(\dot{m}C_p)_{1a} = \frac{780}{(250 - 130)} = 6.5 \text{ kW/}^{\circ}\text{C}$$

$$(\dot{m}C_p)_{1b} = 10 - 6.5 = 3.5 \text{ kW/}^{\circ}\text{C}$$

Hot stream 2 contains $(\dot{m}C_p)\Delta t = 8 \times (160 - 90) = 560$ kW heat. The same can be used to increase the temperature of cold stream 3 from 60°C to 140°C , i.e. $[7 \times (140 - 60)] = 560$ kW. For this heat exchange, heat exchanger *B* is used.

To heat the stream 3 from 140°C to 160°C , heat duty required is $[7 \times (160 - 140)] = 140$ kW. Same can be furnished by hot stream 1b. This is done in heat exchanger *C*. Outlet temperature of hot stream 1 from heat exchange *C* is t_o .

Then

$$(\dot{m}C_p)_b (250 - t_o) = 140$$

$$3.5 (250 - t_o) = 140$$

$$t_o = 210^{\circ}\text{C}$$

Remaining heat of hot stream 1b is $[3.5 \times (210 - 130)] = 280$ kW. If there exists an opportunity to heat demineralised water for boiler feed make-up purpose, 280 kW heat exchange can be effected in an ecomiser (EC). If such an opportunity is not available, stream 1b can be cooled to 130°C with the help of cooling water in a cooler.

Stream 4, available at 240°C from heat exchanger, can be heated to the desired temperature of 260°C in a heater (*H*) with the help of circulating thermic fluid.

In all there will be 5 heat exchangers in the network; *A*, *B*, *C*, *H* and *EC*, the last two use hot and cold utilities, respectively.

Comments:

Here stream 1 is splitted in to two parts. Stream splitting has one advantage and two disadvantages.

Advantage: Stream splitting removes the use of one extra heat exchanger in network. Here if the stream 1 is not splitted, then total number of heat exchangers required in network will be 6 instead of 5.

Disadvantages:

- (i) In stream splitting, control of the flows in two branches is required.
- (ii) Splitting a stream means each branch has a lower flow rate than that for the entire stream. In case of no phase change heat transfer, lower flow rate means lower heat transfer coefficient and larger exchanger area.

Suggested heat exchanger network design is based on the value of $\Delta T_{min} = 10^{\circ}\text{C}$. It is not the optimum design. To arrive at the optimum network design detailed cost analysis is required for the different values of ΔT_{min} . The value of ΔT_{min} which gives the minimum total cost, is the optimum ΔT_{min} , and design of heat exchanger network based on that is optimum network design. A simple computer program can be developed for carrying out iterative calculations, varying ΔT_{min} values.

Example 6.11

Design the suitable heat exchanger network for atmospheric crude fractionation unit based on the following data^{19, 20}.

Solution:

1. Selection of pinch temperature difference: Fractions of crude petroleum oil provides relatively lower heat transfer coefficients. Hence, the optimum value of ΔT_m is relatively higher. Select the value of $\Delta T_m = 20^{\circ}\text{C}$.

Table 6.41 Table of Stream Data (Ref. 19, 20)

Stream No.	Name of stream	Temperature in, °C	Temperature out, °C
1	Fuel oil	349	90
2	Gas oil	341	65
3	Kerosene	268	38
4	Reflux	251	77
5	Heavy Naptha	235	38
6	Light Naptha (Main)	168	71
7	Light Naptha (Pre flash)	136	71
8	Desalted feed	15.6	121
9	Pre-flash feed	120	194
10	Crude tower feed	189	368

Table 6.42 $\dot{m}C_p$ Data of Streams (Ref. 19, 20)

Stream No.	Temperature range, °C			$\dot{m}C_p$, MW/°C
1	349	to	243	0.215
	243	to	213	0.197
	213	to	167	0.178
	167	to	90	0.168
2	341	to	210	0.105
	210	to	172	0.1
	172	to	111	0.087
	111	to	65	0.076
3	268	to	135	0.065
	135	to	38	0.054
4	251	to	169	0.105
	169	to	77	0.091
5	235	to	127	0.008
	127	to	38	0.007
6	168	to	136	0.6
	136	to	118	0.478
	118	to	108	0.41
	108	to	71	0.303
7	136	to	118	0.256
	118	to	108	0.21
	108	to	71	0.159
8	15.6	to	121	0.379
9	120	to	122	0.4
	122	to	163	0.422
	163	to	186	0.6
10	186	to	194	0.725
	189	to	237	0.477
	237	to	265	0.496
	265	to	368	0.66

2. Table is prepared for drawing Grand Composite Curve.

Sample calculation for available heat in column 3 for the interval of temperature change of hot streams from 214°C to 168°C: In this interval first five streams exist (all hot streams from 1 to 5). Cold stream 10 also exists in this interval. But, it cannot be considered in calculation of available heat (with hot streams).

Available heat within this interval

$$\begin{aligned} &= 0.197(214 - 213) + 0.178(213 - 168) \text{ (of Stream 1)} + (0.105(214 - 210) + 0.1(210 \\ &- 172) + 0.087(172 - 168)) \text{ (of Stream 2)} + (0.065(214 - 168)) \text{ (of Stream 3)} + [0.105 \\ &(214 - 169) + 0.091(169 - 168)] \text{ (of Stream 4)} + 0.008(214 - 168) \text{ (of Stream 5)} \\ &= 20.949 \text{ MW.} \end{aligned}$$

In column 4, available heat indicates the cumulative or total heat available from the hot streams at temperature t , when all hot streams (Stream No. 1 to 7) cool from the hottest temperature 388°C to the temperature $t^\circ\text{C}$.

In column 5 required heat by cold streams for each temperature interval are calculated.

Sample calculations for the required heat in column 5 for the interval of temperature from 148°C to 194°C of cold streams are as follows. In this interval two cold Streams No. 9 and Stream No. 10 exist. Required heat by cold streams in this interval

$$\begin{aligned} &= [(194 - 186) \times 0.725 + (186 - 163) \times 0.6 + (163 - 148) \times 0.422] \text{ (of Stream 9)} \\ &\quad + [(194 - 189) \times 0.477] \text{ (of Stream 10)} \\ &= 28.315 \text{ MW} \end{aligned}$$

In column 6, cascaded required heat by cold streams at temperature t indicates the total heat required by the cold streams to increase the temperature of cold streams from $t^\circ\text{C}$ to the maximum required temperatures :

Column 7 represents the net heat available for each interval.

$$\begin{aligned} \text{Net heat available (column 7)} &= \text{Heat available with hot streams (column 3)} \\ &\quad - \text{Heat required by cold streams (column 5)} \end{aligned}$$

Column 8 represents the cascaded or cumulative net heat available at any temperature t . The most negative number in column 8 is -60.344 MW. It implies that minimum amount of heat that must be supplied from the hot utilities. The same amount of heat, 60.344 MW is added in each number of 8th column. Resulting numbers are written in the last column (column 9) as adjusted heat in MW. The point in the last column where the adjusted cascaded heat is zero, represent the pinch point. Top most number in the last column, 60.344 MW is the minimum amount of heat that must be supplied from the hot utilities and the bottom most number of last column; 44.0974 MW is the minimum amount of heat that must be removed by cold utilities.

3. Grand Composite Curve (GCC):

For this example, GCC is shown in Fig. 6.46. Pinch point occurs at 168°C of hot streams temperature and 148°C of cold streams temperature.

4. Hot streams and cold streams exist above the pinch and below the pinch are shown in Fig. 6.47.

For the maximum heat or energy recovery cold utilities cannot be used above the pinch and hot utilities cannot be used below the pinch. Also, heat cannot be transferred across the pinch.

Figure 6.47 also shows heat load (heat available or required) above and below the pinch.

Table 6.43 Cascaded Heat Flow Calculations

1 Hot stream temper- ture °C	2 Cold stream temper- ature °C	3 Avai- able heat MW	4 Cas- caded heat MW	5 Requi- red heat MW	6 Cas- caded heat MW	7 Net heat MW	8 Cas- caded heat MW	9 Adju- sted cas- caded heat, MW
(388)	368	0	0	25.74	0	-25.74	0	60.344
349	(329)	0	0	25.74	25.74	-3.56	-25.74	34.604
341	(321)	1.72	1.72	5.28	31.02	-29.3	31.044	
		23.36	23.36	45.392	-22.032			
268	(248)	25.08	25.08	76.412	-1.773	-51.332	9.012	
251	(231)	6.545	6.545	8.318	84.73	-53.105	7.239	
235	(215)	7.696	7.696	7.632	0.064	-53.041	7.303	
		31.625	31.625					
214	194	10.08	10.08	10.017	92.362	0.063	-52.978	7.366
168	(148)	49.401	49.401	102.379	-7.366	-60.344	0	
		20.949	20.949	28.315				
141	121	70.35	70.35	130.694	16.151	-44.193	16.151	
		27.523	27.523	11.372				
(140)	120	97.873	97.873	142.066	0.24	-43.953	16.391	
		1.019	1.019	0.779				
136	(116)	98.892	98.892	142.845	2.56	-41.393	18.951	
		4.076	4.076	1.516				
90	(70)	102.968	102.968	144.361	30.731	-10.662	49.682	
		48.165	48.165	17.434				
77	(57)	151.133	151.133	161.795	4.043	-6.619	53.725	
		8.97	8.97	4.927				
71	(51)	160.103	160.103	166.722	1.32	-5.299	55.045	
		3.594	3.594	2.274				
65	(45)	163.697	163.697	168.996	-1.452	-6.751	53.593	
		0.822	0.822	2.274				
38	(18)	164.519	164.519	171.27	-8.586	-15.337	45.007	
		1.647	1.647	10.233				
		166.166	166.166	181.503	-9.096	-16.2466	44.0974	
		—	—	0.9096				
(35.6)	15.6	166.166	166.166	182.4126				

Sample calculation of heat load above the pinch for Stream No. 1:

$$\text{Heat load above the pinch for Stream No. 1} = \sum (mC_p)_i \Delta t_i$$

$$= 0.215 (349 - 243) + 0.197 (243 - 213) + 0.178 (213 - 168)$$

$$= 36.71 \text{ MW}$$

Above the pinch design:

For the maximum energy recovery cold utility cannot be used above the pinch. But hot utility can be used above the pinch. One of the possible network designs above the pinch

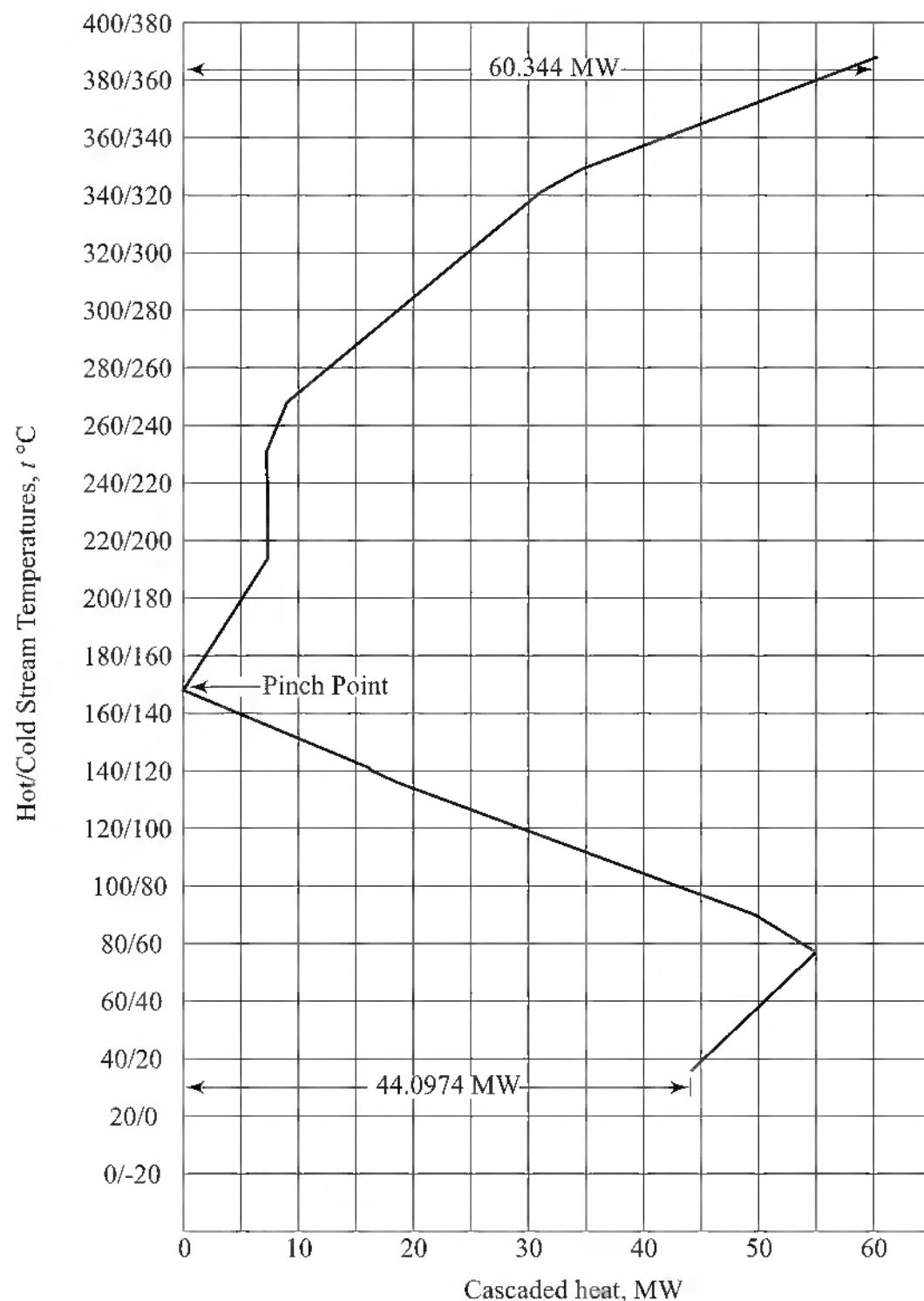


Fig. 6.46 Grand Composite Curve

is shown in Fig. 6.48. Here five hot streams and two cold steams exist above the pinch. Heat available with Stream No. 5 above the pinch is 0.536 MW and is very low compared to heat loads (heat available or required) of other streams existing above the pinch. Hence the heat exchange above the pinch heat load of Stream No. 5 is not considered but the same load is transferred or included in the design of network below the pinch. Here, Stream No. 9 (cold stream) is splitted into four streams to exchange the heat with four hot streams. Stream No. 10 (cold stream) is splitted into two steams to exchange the heat

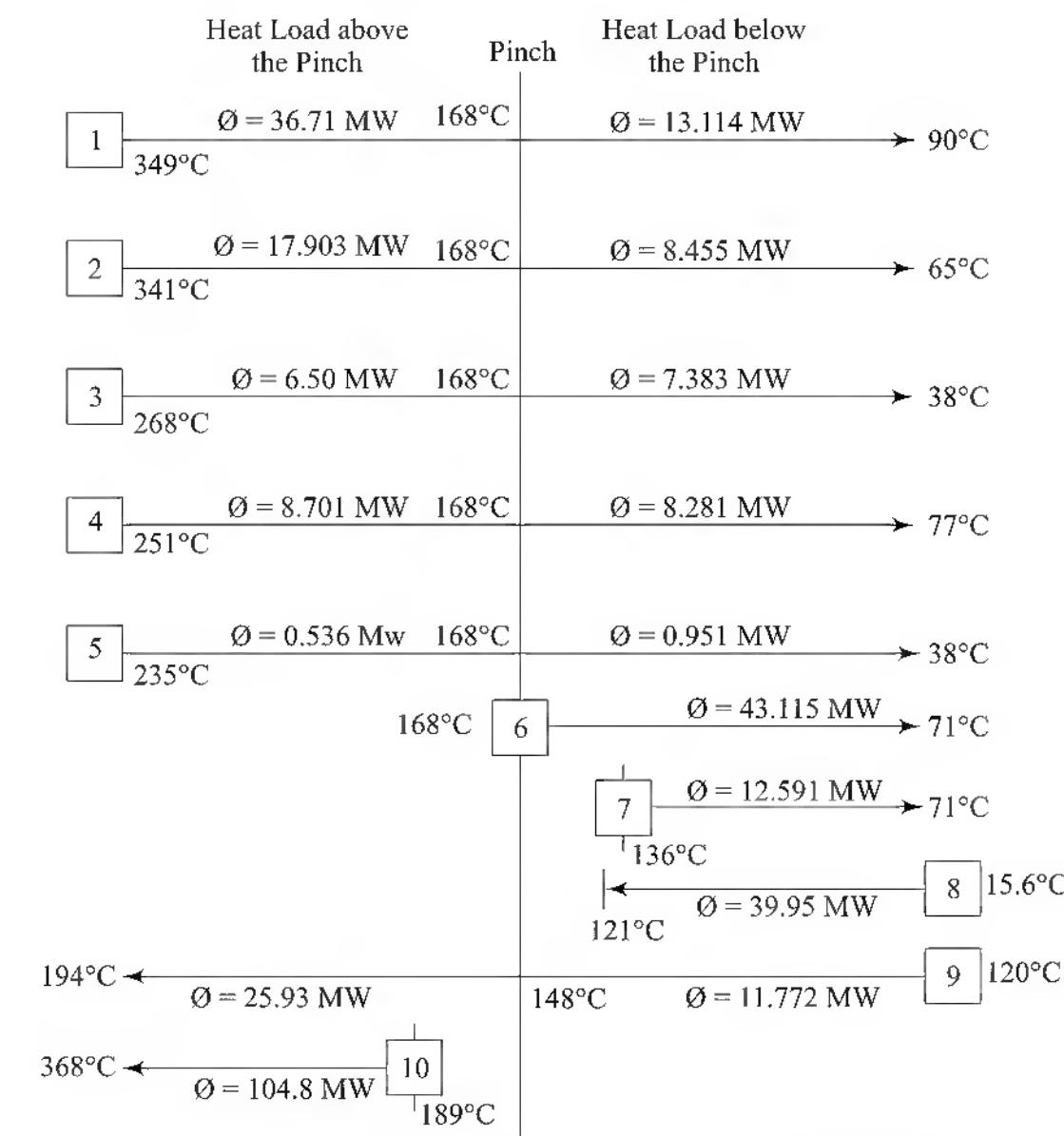


Fig. 6.47 Division of Hot Streams and Cold Stream Around Pinch Point

with two hot streams. Streams are splitted near the pinch while heater H is designed far away from the pinch.

Below the pinch design:

For the maximum energy recovery hot utility can not be used below the pinch Refer Fig. 6.49. If the stream splitting is required, then split it near the pinch. Design the heat exchangers which utilize the utilities far away from the pinch. Here, below the pinch, Stream No. 9 (cold stream) is again splitted at the pinch into four streams to exchange the heat with the same four streams, from Stream No. 1 to 4. Another cold stream, Stream No. 8, is also splitted into two streams to exchange the heat with Stream No. 1 and Stream No. 6. At the end, Stream No. 9 exchanges the heat with Stream No. 5. Total six coolers are designed, far away from the pinch, at the end of network to meet the process requirements.

Complete Design:

Complete design of heat exchanger network for the maximum energy recovery is obtained by merging the two network designs, above and below the pinch. To reduce the number of heat exchangers used for Stream No. 9 above the pinch, C, D, E and F are

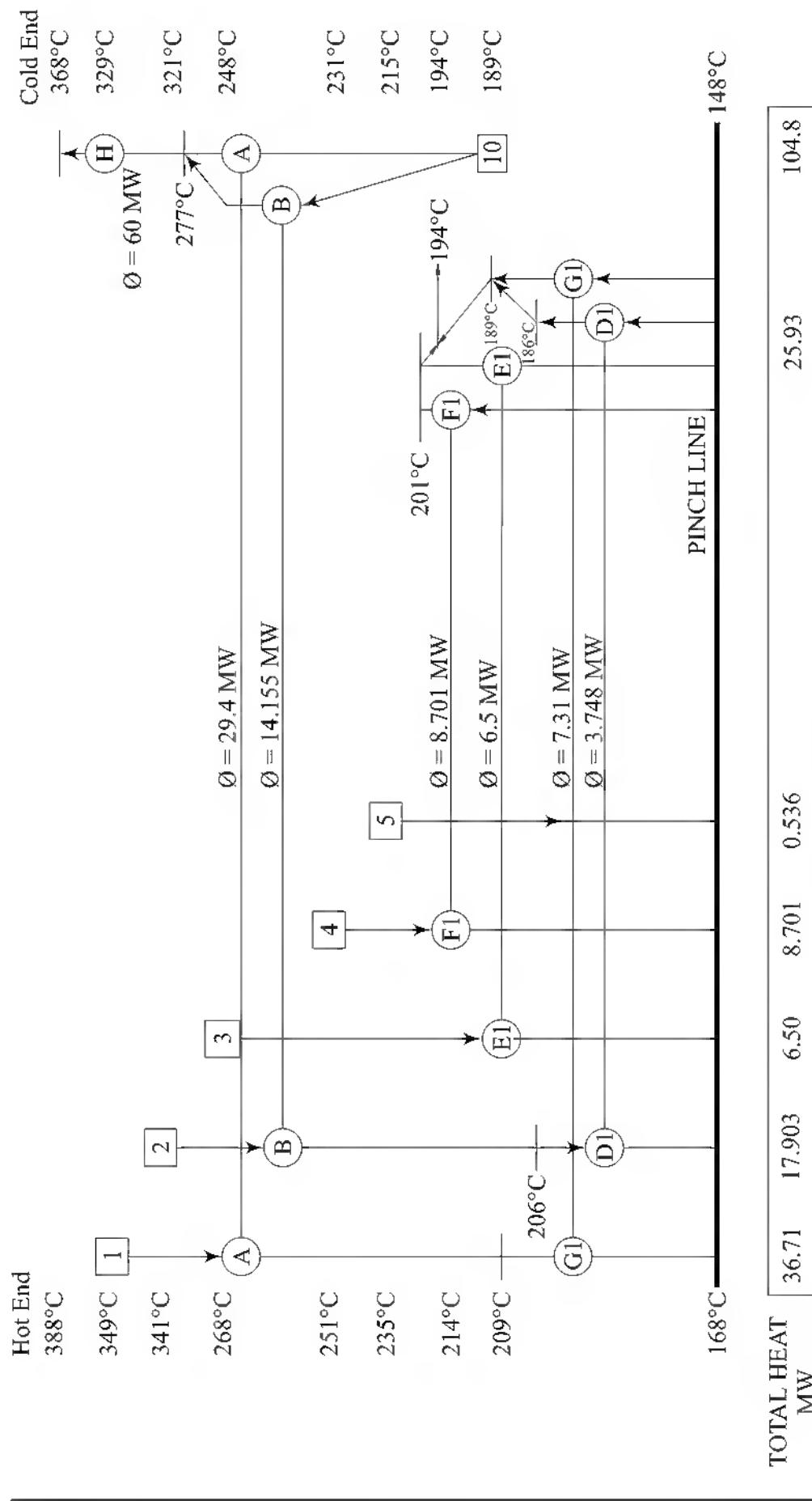
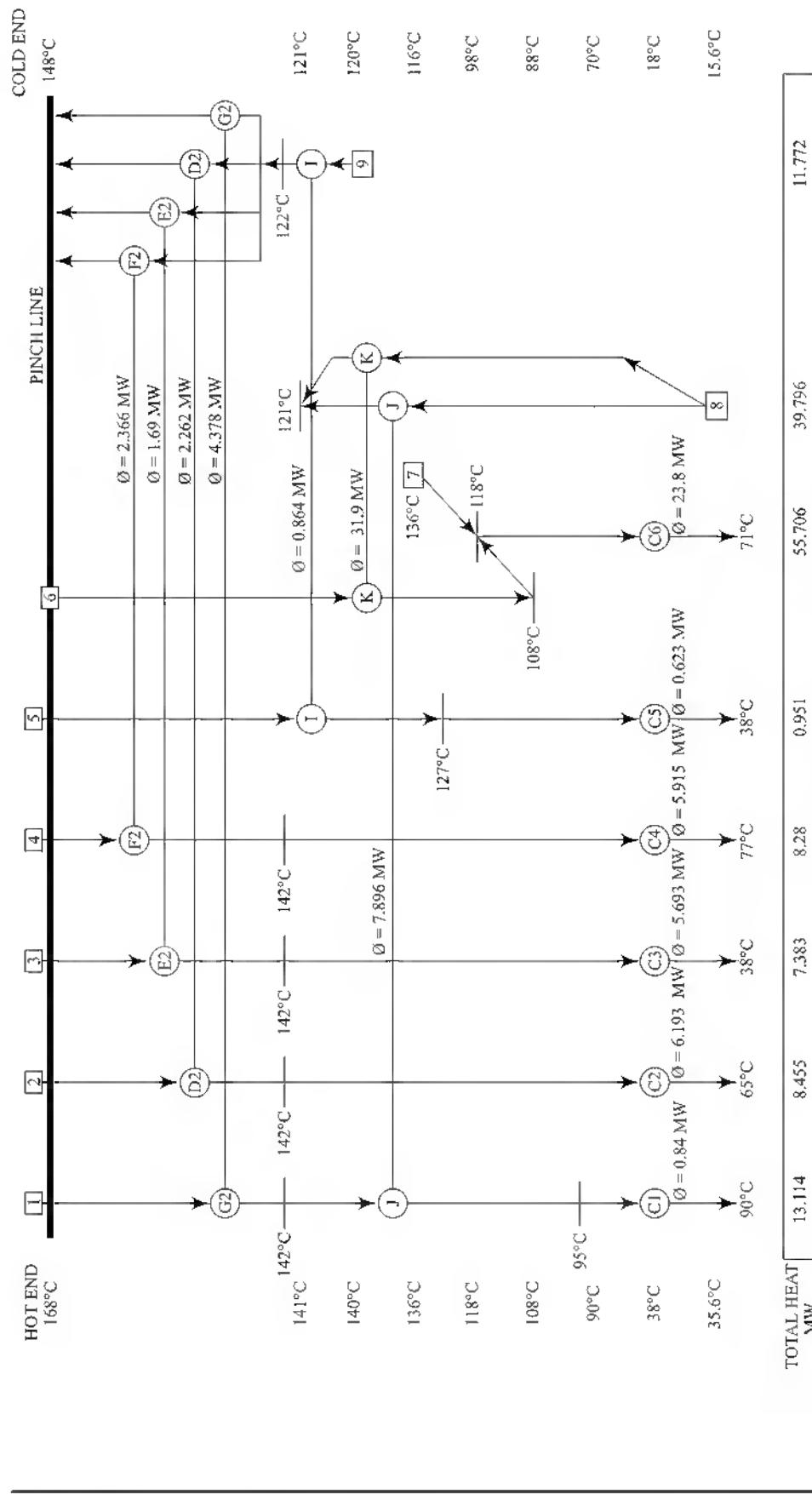
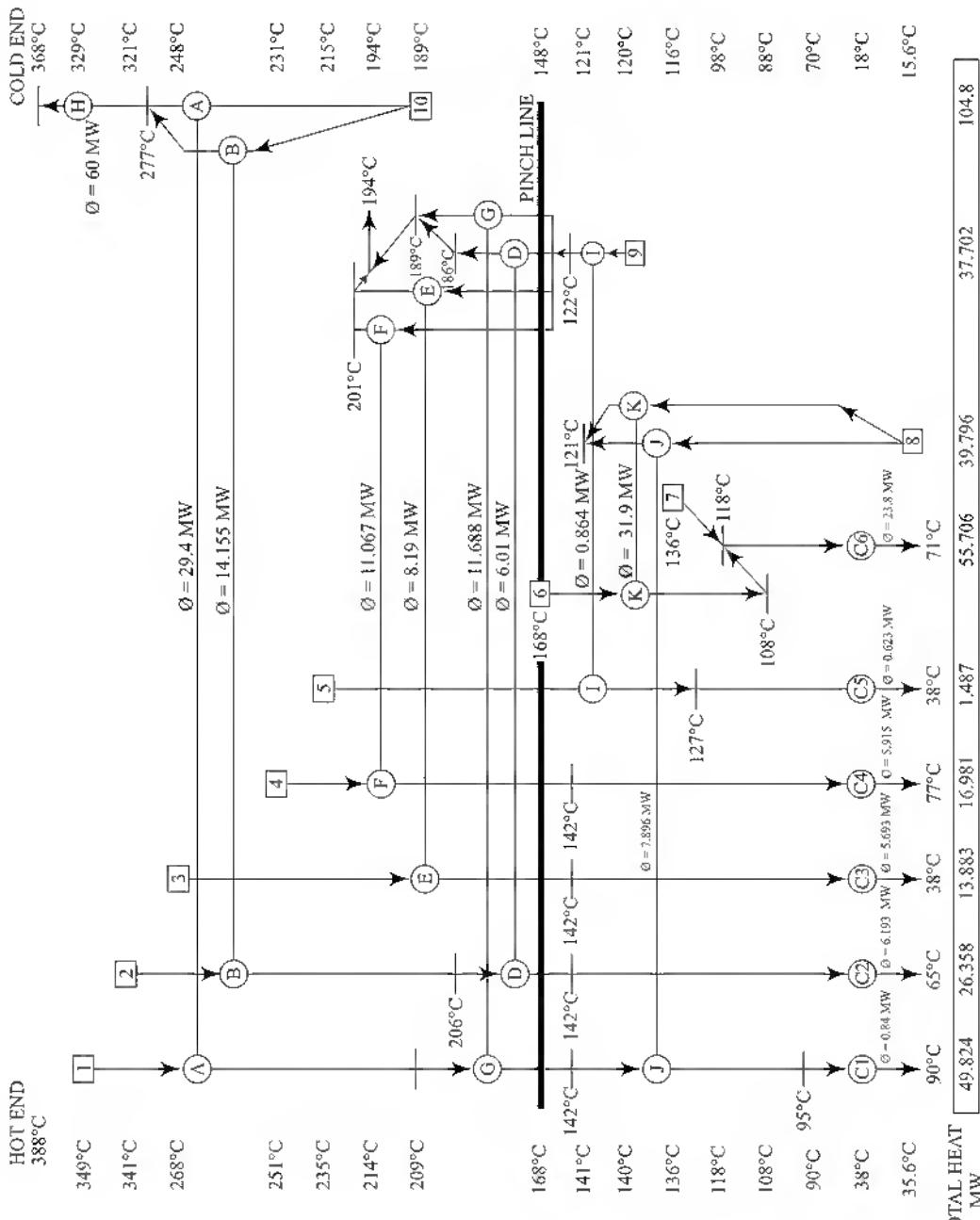


Fig. 6.48 Network Design Above the Pinch

**Fig. 6.49** Network Design below the Pinch

**Fig. 6.50** Combined Network

combined with C_2 , D_2 , E_2 and F_2 , respectively. C_2 , D_2 , E_2 and F_2 are the heat exchangers used below the pinch. For these combinations minor adjustments are done in the temperatures of branch streams. Combined design or completed design is shown in Fig. 6.50.

Note: Heat exchanger networking is normally carried on available heat sources/heat sinks. However, in actual practice, overriding considerations such as fouling, start-up/shut down conditions, etc. exclude some heat source/heat sink.

6.14 HEAT TRANSFER IN SOLIDS

Unlike heat transfer in liquids and gases, heat transfer in solids takes place chiefly by conduction. Cooling of solids is often required after a dryer, a calciner, a roaster or combustor. Metal powders, burnt lime, coal, etc. are cooled from high temperature (in excess of 500°C). Urea, furnace grade carbon black, etc. require cooling after drying. Pharmaceutical products (fine powder) require cooling after drying before cooling. In food processing, coffee beans are cooled after roasting. Beans, corn and peas are frozen by cooling with refrigerated air for preservation.

Cooling of hygroscopic solids is required for more than one reason. Granulated or prilled fertilizer and carbon black with some moisture may agglomerate on natural cooling due to moisture migration in silo. This results in lump formation. These lumps are quite hard and when broken by mechanical means produce dusty material making it unsuitable for end use. However, caking tendency is not observed in the same material if it is cooled (say below 60°C) in absence of moisture.

Several technologies are available for cooling solids. Although compact plate type heat exchangers are developed for cooling of solids, their application is limited due to cost and adherence of sticky solids to the heat exchanger surface.

Direct contact coolers with or without indirect cooling (with cooling water) are popular for cooling of solids. Adham²¹ has described various solids cooling technologies at length. Heat recovery from hot gases, coming out of the cooler is possible depending on temperature of gases and its possible use elsewhere in the plant. Chiefly, four types of coolers are in use for most applications of solids cooling.

- (a) Stationary cooler
- (b) Rotary cooler
- (c) Fluid bed cooler
- (d) Conveyor cooler

6.14.1 Stationary Coolers

Simple version of a stationary cooler (Fig. 6.51) has a bin with internal cooling surface. It is recommended for free flowing material and is unsuitable for wet, very fine and sticky powders. Overall heat transfer rate in the cooler is quite low and hence larger cooling surface is required.

Solids travel in a plug flow manner and a temperature gradient from top to bottom is observed. Stationary coolers are recommended for moderate inlet temperature (say 300 to 400°C) and for heat duties not exceeding 150 kW. Free

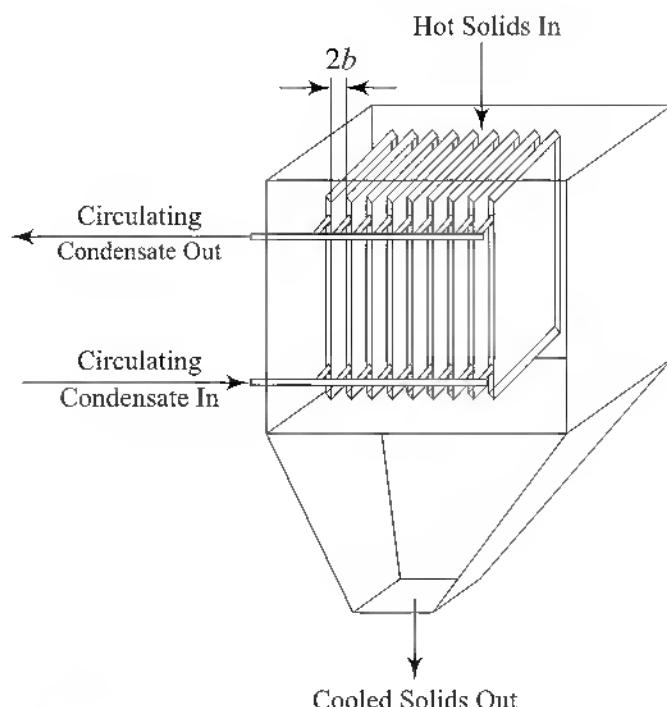


Fig. 6.51 Stationary Cooler with Water Cooled Plates

flowing powder up to $100 \mu\text{m}$ size can be cooled in such a cooler. Barring these limitations, stationary coolers are compact, require minimum or no auxiliary equipment, require no off-gas treatment and are simple to operate. Cooling up to 60 to 70°C should be possible with this cooler with circulating water maintained at a temperature of 30 to 35°C .

6.14.2 Rotary Coolers

This versatile cooler can handle different type of solids and can be used as direct or an indirect cooler with or without heat recovery. Although it can be used for a wide range of solids, it is a preferred equipment for coarse particles. In this types of coolers, hot air (or gas) flows in counter current direction of solids, increasing the thermal efficiency. Cooling coils can be embedded in the bed to provide indirect cooling. Heat from the hot air, coming out from the cooler, can be recovered in a number of ways.

Figure 6.52 is a simple sketch of a rotary cooler. Diamenter of the cooler is restricted below 3 m for the considerations of fabrication and transportation. Drive mechanism for the cooler also imposes restrictions on the dimensions. Length to diameter ratio is kept in the range of 4 to 10. To avoid passing of solids without coming in contact with air, depth of material is kept in the range of 30–40% of radius. Such equipment can be used for drying as well as cooling. In carbon black industry, ceramic industry, metal powder industry, etc., this equipment is popular.

6.14.3 Fluid Bed Cooler

Coarse small particles (below 2 – 2.5 mm) can be fluidized with air or gas. However, very fine powders (less than $10 \mu\text{m}$) resists smooth fluidization. So fluid-

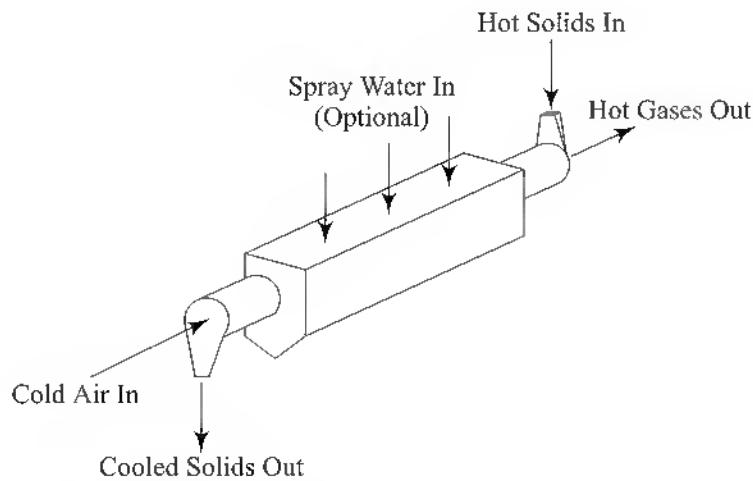
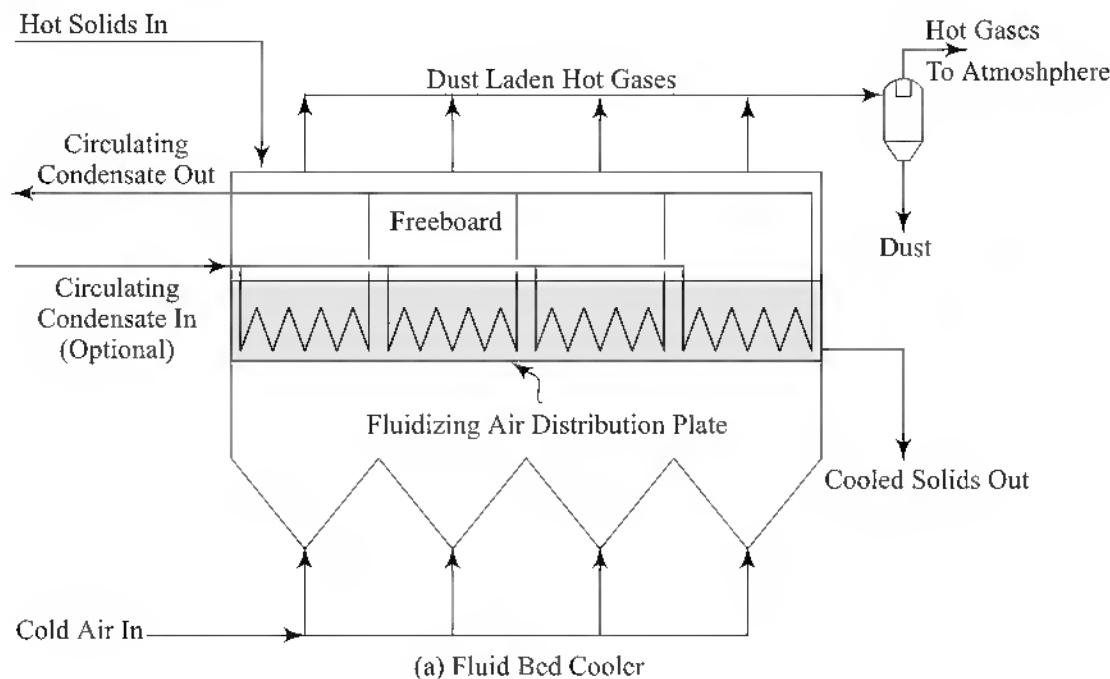
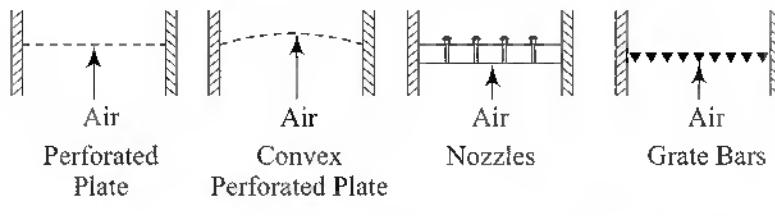


Fig. 6.52 *Rotary Cooler with Direct Air and Spray Water Cooling*

ized bed cooler can be considered for the intermediate size solids. In Fig. 6.53, 4-stage fluid bed cooler with embedded cooling coils is shown.



(a) Fluid Bed Cooler



(b) Typical Distributors for Fluidized Beds

Fig. 6.53 *Four Stage Fluidized Bed Cooler with Indirect Water Cooling*

Spray cooling is an option both in rotary as well as fluid bed coolers but it has to be executed with caution. Hot air wet bulb temperature should not be allowed to exceed 80°C and fluid or moving bed temperature is not allowed to fall below 150°C to prevent moisture absorption by the solids.

Fluid bed cooler can handle free flowing and non-sticky solids. Heat transfer rate is excellent in a fluid bed cooler among all solid-cooling equipments. However, it needs an auxiliary equipment such as a cyclone or a bag filter for off-gas treatments to trap fines. This equipment is relatively simple in operation and easy to maintain.

Removal of fines from the solids may be considered as an advantage. The product after cooling is free from dust and its particle size distribution is in the narrow range making it a preferred marketable product.

6.14.4 Conveyor Coolers

Variety of conveyor coolers are available. Vertical spiral cooler, moving grate cooler, vibrating pan cooler, screw conveyor coolers, etc. are a few of them. The screw conveyor cooler is normally used for indirect cooling of solids with cooling water which flows in the conveyor shell jacket and the hollow shaft. Screw conveyor coolers can be used for free flowing solids, normally coarse in nature (0.1–12 mm). Screw conveyor coolers can be advantageously used for cooling as well as conveying. Single or multiple screw designs are available but double screw design is popular up to a maximum length of 8.5 m.

Process design of cooling equipments of solids can be well explained by the following example.

Example 6.12

Urea prills from a prill tower in a fertilizer plant have a temperature of 100°C and moisture content of 1.5 to 2% (by mass). They need to be cooled to 60°C before sending to silo for storage to prevent caking and lump formation. Average sieve analysis of prills is given in Table 6.44.

Table 6.44 Distribution of Urea Prills by Size

Size of prills, mm	Mass%
+2.4	0.8
+2.0	9.0
+1.7	56.3
+1.4	26.6
+1.0	7.3

Physical properties of urea:

Particle density, $\rho_s = 1.335 \text{ kg/L}$

Bulk density, $\rho_{sb} = 0.758 \text{ kg/L}$ (without compaction)

Thermal conductivity, $k_s = 80 \text{ W/(m} \cdot ^\circ\text{C)}$

Heat capacity, $C_s = 1.775 \text{ kJ/(kg} \cdot ^\circ\text{C)}$

Cooling Mediums

- (a) **Circulating condensate** is proposed to be used in the stationary cooler to avoid possibility of fouling in the plates. Supply and return temperatures may be taken as 35 and 45°C, respectively. Hot condensate is passed through a shell and tube heat exchanger in which it is cooled to 35°C with the help of cooling water of 30°C which is recirculated.

- (b) **Ambient air** at 44°C is to be used in rotary and fluid bed coolers. Outlet air temperature from the coolers may be fixed to be 75°C.

Work out process design of stationary, rotary and fluid bed coolers to process 60 t/h urea prills. While there is hardly any change in moisture content in the stationary cooler, moisture content of prills gets reduced to 0.5 mass % in rotary and fluid bed cooler.

Overall heat balance:

$$\begin{aligned}\text{Average temperature of air} &= \frac{75 + 44}{2} \\ &= 59.5^\circ\text{C} \\ &\equiv 332.65 \text{ K}\end{aligned}$$

$$\begin{aligned}\text{Molar volume of air} &= \frac{22.4136 \times 332.65}{273.15} \\ &= 27.296 \text{ m}^3/\text{kmol}\end{aligned}$$

$$\text{Density of air, } \rho_a = \frac{28.96}{27.296} = 1.061 \text{ kg/m}^3$$

$$\begin{aligned}\text{Moisture removal of urea} &= 60000 (0.015 - 0.005) = 600 \text{ kg/h} \\ \text{Total heat load of prill cooling in rotary and fluid bed coolers,} &\end{aligned}$$

$$\begin{aligned}\phi_1 &= 60000 \times 1.775 (100 - 60) + 600 \times 2644 \\ &= 4260000 + 1586400 \\ &= 5846400 \text{ kJ/h} \\ &\equiv 1624.0 \text{ kW}\end{aligned}$$

Average heat capacity of air at 59.5°C,

$$C_{pa} = 1.005 \text{ kJ/(kg} \cdot ^\circ\text{C)}$$

Air is heated from 44°C to 75°C.

$$\begin{aligned}\text{Mass flow rate of air, } q_{ma} &= \frac{5846400}{1.005(75 - 44)} \\ &= 187655 \text{ kg/h}\end{aligned}$$

$$\begin{aligned}\text{Volumetric flow rate of air, } q_{va} &= 187655/1.061 \\ &= 176866 \text{ m}^3/\text{h}\end{aligned}$$

Viscosity of air at 59.5°C, $\mu_a = 0.072 \text{ kg}/(\text{cm} \cdot \text{h})$

In case of stationary cooler, heat lead,

$$\begin{aligned}\phi_2 &= 4260000 \text{ kJ/h (no evaporation load)} \\ &\equiv 1183.33 \text{ kW}\end{aligned}$$

Flow rate of circulating condensate,

$$\begin{aligned}q_{mc} &= \frac{4260000}{4.1868(45 - 35)} = 101748 \text{ kg/h} \\ &\equiv 101.7 \text{ m}^3/\text{h}\end{aligned}$$

Process design of stationary (bulk flow) Cooler:

Refer Fig. 6.48.

Assume $2b = 5 \text{ cm}$

$$b = 2.5 \text{ cm} \equiv 0.025 \text{ m}$$

In a stationary cooler, solids travel in plug flow and, therefore, a temperature gradient in bulk solids is observed. Following equation relating to the unsteady-state heat transfer from a solid, can be used to calculate cooling time of the solids.

$$\frac{t_2 - t_2'}{t_1 - t_2'} = \sum_{n=0}^{\infty} \frac{2}{(n+0.5)^2 \pi^2} e^{-\left((n+0.5)\frac{2\pi^2\alpha\theta}{b^2}\right)} \quad (6.111)$$

where,
 t_1 = Inlet solid temperature, °C
 t_2 = Outlet solid temperature °C
 t_1' = Inlet circulating condensate temperature, °C
 t_2' = Outlet circulating condensate temperature, °C
 α = Thermal diffusivity in solid, m^2/h
 $= k_s / (\rho_s \cdot C_s)$
 k_s = Thermal conductivity of solid, $kJ/(h \cdot m \cdot ^\circ C)$
 C_s = 1.775 $kJ/(kg \cdot ^\circ C)$ for urea
 θ = cooling time, h

Above equation assumes that convective transfer coefficient of cooling water and thermal conductivity of the plate material (SS in this case) is much larger than k_s . This assumption may be considered valid for urea prills.

$$\begin{aligned} k_s &= 80 \text{ W/(m} \cdot {^\circ}\text{C)} \\ &= 288 \text{ kJ/(h} \cdot \text{m} \cdot {^\circ}\text{C)} \end{aligned}$$

$$\begin{aligned} \alpha &= \frac{288}{1335 \times 1.775} \\ &= 0.12154 \text{ m}^2/\text{h} \end{aligned}$$

$$\begin{aligned} \text{LHS of Eq. (6.111)} &= \frac{60 - 45}{100 - 45} \\ &= 0.27273 \end{aligned}$$

Spreadsheet can be useful in solving Eq. (6.111). Substituting values of b and α in RHS of the equation and solving by trial and error,

$$\theta = 0.0023 \text{ h} = 8.28 \text{ s}$$

Hold-up required for solids cooling,

$$= \frac{60000 \times 8.28}{3600 \times 0.758} = 182.06 \text{ m}^3$$

Single silo or bin of such large operating volume with cooling plates would not be desirable for operational and maintenance reasons. Cooling height of at least 2 m is recommended.

Overall heat transfer coefficient of 175 $w/(m^2 \cdot {^\circ}\text{C})$ will be safe to design the cooling surface.

$$\Delta T_1 = 100 - 45 = 55^\circ\text{C}$$

$$\Delta T_2 = 60 - 45 = 15^\circ\text{C}$$

$$\Delta T_{lm} = \frac{55 - 15}{\ln\left(\frac{55}{15}\right)} = 30.79^\circ\text{C}$$

$$\text{Heating surface requirement, } A = \frac{4260\ 000}{30.79 \times 175 \times 3.6} \\ = 219.61 \text{ m}^2 \text{ (min.)}$$

Four or more silos may be provided. Distance between plates for cooling water flow should be kept at least 2 cm. Silos and plate coolers with HTA, exceeding 220 m², can be easily sized for the required heat duty.

For four silos,

$$\text{Hold-up volume of solids} = 182.06/4 = 45.515 \text{ m}^3$$

Assume cooling (plate) height = 3 m

$$\text{Cross-sectional area of silo} = 45.515/3 = 15.172 \text{ m}^2$$

Consider square construction of the silo.

$$\text{Each side} = \sqrt{15.172} = 3.895 \text{ say } 3.9 \text{ m}$$

$$\text{Plate length} = 3.5 \text{ m}$$

$$\text{Width between two plates} = 5 \text{ cm}$$

$$\text{No. of pairs of plates} = \frac{350}{5+5} \\ = 35$$

$$\text{HTA per pair of plates} = 3.5 \times 3 \times 2 \\ = 14 \text{ m}^2$$

$$\text{HTA per silo} = 14 \times 350 = 490 \text{ m}^2$$

$$\text{Total HTA} = 490 \times 4 = 1960 \text{ m}^2$$

$$\frac{\text{Available HTA}}{\text{Required HTA}} = \frac{1960}{219.61} = 8.92 \text{ (much in excess)}$$

In order to avoid clinging of urea dust, present in the feed of silo, a small quantity of dry air is injected from bottom. The exhaust air from the cooler is directed to prill tower bottom.

II. Process Design of Rotary Cooler

Assume diameter of rotary cooler as 2.75 m. Air is passed in countercurrent (horizontal) fashion to solids flow.

$$\text{Cross sectional area of cooler, } A_2 = (\pi/4)(2.75)^2 \\ = 5.939\ 57 \text{ m}^2$$

$$\text{Velocity of air} = \frac{176\ 866}{3600 \times 5.939\ 57} = 8.272 \text{ m/s}$$

This is a very high velocity and will blow dust and fine area prills. Normally in a rotary cooler, velocity of 1.5 m/s is preferred. This will call for six rotary coolers to be used in parallel.

$$\text{Flow of urea prills/cooler} = \frac{60\ 000}{6} \\ = 10\ 000 \text{ kg/h} \equiv 10 \text{ t/h}$$

Flow of air through each coolers,

$$q_{\text{var}} = \frac{176866}{6} \\ = 29477.7 \text{ m}^3/\text{h at inlet}$$

$$\text{Velocity of air at inlet} = \frac{29477.7}{3600 \times 5.93957} \\ = 1.379 \text{ m/s} (< 1.5 \text{ m/s})$$

Rotary cooler length can be calculated by the following equation.

$$L = N_t H_t \quad (6.112)$$

where, L = Rotary cooler length, m

N_t = Number of transfer units

H_t = Height (length) of transfer unit, m

$$N_t = \frac{\Delta t_G}{\Delta T_{ln}} \quad (6.113)$$

where, $\Delta t_G = t_{g,\text{out}} - t_{g,\text{in}}$, °C

$$\Delta T_{ln} = \frac{(t_{s,\text{in}} - t_{g,\text{out}}) - (t_{s,\text{out}} - t_{g,\text{in}})}{\ln \frac{(t_{s,\text{in}} - t_{g,\text{out}})}{(t_{s,\text{out}} - t_{g,\text{in}})}} \\ = \frac{(100 - 75) - (60 - 44)}{\ln \left(\frac{100 - 75}{60 - 44} \right)} = 20.17 \text{ °C}$$

$$N_t = \frac{75 - 44}{20.17} = 1.537$$

Preferred range of N_t is 1.5 to 2.5.

$$H_t = \frac{1000 G C_a}{U_v}$$

where, G = Mass flow rate per unit area of air, kg/(m² · s)

C_a = Heat capacity of air = 1.005 kJ/(kg · s)

U_v = Overall volumetric heat transfer coefficient, kJ/(m³ · s · °C)
= 237 $G^{0.67}/D$

D = Rotary cooler diameter, m

$$G = \frac{187655}{6} \times \frac{1}{3600 \times 5.93957} \\ = 1.46269 \text{ kg(m}^2 \cdot \text{s)} \\ U_v = 237 (1.46269)^{0.67}/2.75 \\ = 111.2 \text{ kJ/(m}^3 \cdot \text{s} \cdot \text{°C)}$$

$$H_T = \frac{1000 \times 1.46269 \times 1.005}{111.2}$$

$$= 13.2$$

$$L = 1.537 \times 13.2 = 20.29 \text{ m or } 20.3 \text{ m}$$

$$L/D = 20.3/2.75 = 7.38$$

Rotary coolers, commonly in use, have L/D ratio in the range of 4 to 10. Bed depth of about a meter is recommended in the cooler. Above design is for cooling with air only. If cooling surface is provided across the length for indirect cooling with cooling water, heat transfer rate can be estimated by considering overall heat transfer coefficient of 55–110 W/(m²·°C).

III. Process Design of Fluid Bed Cooler

For determining the fluidizing velocity, mean particle diameter (d_p) is required. Mean particle diameter,

$$d_p = \sum \frac{1}{m_i} \frac{d_i}{d_i} \quad (6.114)$$

where, d_p = Mean particle diameter and
 m_i = Mass fraction of particle diameter, d_i

$$\sum \frac{m_i}{d_i} = \frac{0.008}{2.4} + \frac{0.09}{2} + \frac{0.563}{1.7} + \frac{0.266}{1.4} + \frac{0.073}{1}$$

$$= 0.6425$$

$$d_p = 1/0.6425 = 1.556 \text{ mm}$$

For estimating fluidization velocity, use of following equation²² is recommended.

$$Re_m = \left[33.7^2 + \frac{5 \times 10^5 d_p^3 \rho_g (\rho_s - \rho_g) g}{\mu_g^2} \right]^{1/2} - 33.7 \quad (6.115)$$

where, Re_m = Reynolds number at minimum fluidization velocity
 d_p = 1.556 mm = 0.001 556 m
 ρ_g = 1.061 kg/m³
 ρ_s = 1.335 kg/L = 1335 kg/m³
 g = gravitational acceleration
 $= 9.81 \text{ m/s}^2$
 μ_g = 0.072 kg/(m · h)

$$Re_m = \left[1135.65 + \frac{5 \times 10^5 (0.001556)^3 1.061(1335-1.061)9.81}{(0.072)^2} \right]^{1/2} - 33.7$$

$$= (1135.65 + 5044.89)^{1/2} - 33.7$$

$$= 44.92$$

Minimum fluidization velocity is related with Reynolds number as

$$\begin{aligned} U_m &= \frac{Re_m \mu_g}{3600 \rho_g d_p} \\ &= \frac{44.92 \times 0.072}{3600 \times 1.061 \times 0.001556} \\ &= 0.54 \text{ m/s at ambient temperature} \end{aligned}$$

At the minimum velocity, fluidization starts. Terminal velocity is the velocity above which the solids particles will be pneumatically conveyed. Reynolds number (Re_t) at the terminal velocity is derived by

$$C_d Re_t^2 = 1.6 \times 10^7 g d_p^3 \frac{\rho_g (\rho_s - \rho_g)}{\mu^2} \quad (6.116)$$

where, C_d = Drug coefficient which depends on sphericity (ϕ_s) of the particles

$\phi_s = 0.6$ for broken solids and the same can be taken for urea prills

$C_d = 6$ for $\phi_s = 0.6$

$$\begin{aligned} Re_t^2 &= \frac{1.6 \times 10^7 \times (0.001556)^3 \times 1.061(1355 - 1.061)}{(0.072)^2 \times 6} \\ &= 26906 \\ Re_t &= 164 \end{aligned}$$

$$u_t = \frac{Re_t \mu_g}{3600 \rho_g d_p} = \frac{164 \times 0.072}{3600 \times 1.061 \times 0.001556} = 1.987 \text{ m/s}$$

Actual fluidization velocity will be between u_m and u_t . Select $u = 1 \text{ m/s}$ for normal fluidization.

Volumetric flow rate of air, $q_{va} = 176866 \text{ m}^3/\text{h}$

$$\begin{aligned} \text{Area of fluidization, } A_3 &= \frac{176866}{1 \times 3600} \\ &= 49.129 \text{ m}^2 \end{aligned}$$

Selected width, $w = 3 \text{ m}$

Length, $L = 49.129/3 = 16.376 \text{ m}$

To improve thermal efficiency, total length will be divided into a number of stages. Select 4 stages for fluidization.

$$\text{Length of each stage} = \frac{16.376}{4} = 4.094 \text{ m, say } 4.1 \text{ m}$$

Staging will be beneficial for crossflow of solids. Bed depth of 0.4 to 0.5 m is adequate in each stage. Assuming bed depth of 0.5 m, hold-up of urea in fluid bed cooler = $49.129 \times 0.5 \times 758$

$$= 18620 \text{ kg}$$

This means residence time (θ) of urea prills in the fluids bed cooler will be 18.62 min; or approximately 20 min.

Peak pressure drop in the bed²²,

$$\frac{\Delta p_p}{H \rho_b g} = \left[\frac{6.8}{\tan \gamma} \left(\frac{D_i}{D_c} \right) + 0.8 \right] - 34.4 \frac{d_p}{H} \quad (6.117)$$

where,

Δp_p = Peak pressure drop, kN/m² or kPa

H = Height of bed, m = 0.5 m

ρ_b = Bulk density, kg/m³ = 0.758 kg/L

g = 9.81 m/s²

γ = Angle of internal friction. For granular solids, consider it to be 55°.

D_i = Fluid inlet orifice diameter

= 2 cm = 0.02 m (assumed)

D_c = diameter of equivalent cross-section

$$= \sqrt{\frac{4}{\pi} (49.129)} = 7.909 \text{ m}$$

$$\begin{aligned} \frac{\Delta p_p}{0.5 \times 0.758 \times 9.81} &= \left[\frac{6.8}{\tan 55} \left(\frac{0.02}{7.909} \right) + 0.8 \right] - 34.4 \times \frac{0.001556}{0.5} \\ &= 0.812 - 0.107 = 0.705 \end{aligned}$$

$$\Delta p_p = 2.62 \text{ kPa} \equiv 267 \text{ mm WC}$$

In normal course, pressure drop will vary between 30 to 50% of peak pressure drop (i.e. 80–135 mm WC) across the bed. To this add friction loss in the ducts which will give the fan pressure requirement.

During fluidization, broken and prills with poor mechanical strength will break and form dust due to attrition. This is expected to give higher dust load than the other two types of coolers. Dust will be separated from exhaust air and will be allowed to fall in a dust dissolving tank for reprocessing. Urea prills, coming out from the fluid bed coolers will be of near uniform size and with definite mechanical strength which will not break during storage in silo.

EXERCISES

6.1 Select the correct answer.

- (a) For an exchanger with no phase change service design conditions are: Hot fluid is cooled in counter current manner from 213°C to 57°C with a cold fluid which is heated from 29.5°C to 40.5°C. Other parameters being maintained the same, if hot fluid inlet temperature is reduced to 201°C and cold fluid inlet temperature is increased to 35°C, then:
 - (i) Hot fluid and cold fluid outlet temperatures will change to 60°C and 45°C, respectively.
 - (ii) Hot fluid outlet temperature will change to 60°C while cold fluid outlet temperature will remain as 40.5°C.
 - (iii) Hot fluid outlet temperature will remain 57°C while cold fluid outlet temperature will change to 45°C or
 - (iv) Data not sufficient to predict.

- (b) In the heat transfer equation for heat exchanger, the heat transfer rate appears to be directly proportional to the heat transfer area. Hence (other parameters remaining the same) for double the heat transfer area, the heat transfer rate will be
- doubled
 - more than doubled **or**
 - less than doubled.
- (c) A shell and tube, horizontally installed heat exchanger with 2-tube passes has pressure gauges (P_1 and P_2) installed, respectively on the inlet pipe to the lower nozzle and on the outer pipe from the upper nozzle of the channel. The pressure gauges are approximately 1.4 m apart. The difference in the readings ($P_1 - P_2$) is 0.42 bar. Each nozzle accounts for 0.07 bar pressure drop. Estimate the difference in pressure reading (as $P_1 - P_2$) for the following cases.
- Tube side passes are changed from 2 to 4 and
 - Tube side flow rate is doubled.

[Answer: (a) 1.42 bar, (b) 0.85 bar **or** (c) 0.42 bar]

- 6.2 A fire tube waste-heat boiler generate steam at a pressure of 10.34 bar g. It is designed for a gas flow of 80 000 kg/h from an incinerator. Gas is designed to be cooled from 600°C to 260°C. On a particular day, the gas flow is 55 000 kg/h and inlet temperature to the boiler is 500°C and steam is generated at 10.34 bar g. What will be the expected gas outlet temperature if the overall heat transfer coefficient is proportional to mass flow rate of gas to the power of 0.8? Assume other properties of flue gases to be nearly the same.

Assume overall thermal efficiency of the boiler to be 95% in both the cases. Average heat capacity of fine gases may be taken as 1 kJ/(kg · K). Demineralized water enters steam drum at 35°C. Calculate steam generation from the boiler in both the cases.

Data: Saturation temperature of steam at 10.34 bar g = 185.5°C

Enthalpy of saturated steam at 10.34 bar g = 2780.8 kJ/kg

Enthalpy of water at 35°C = 146.6 kJ/kg

- 6.3 120 000 kg/h of ethanol is to be cooled from 78°C to 40°C by cooling water as coolant. Cooling water enters at 32°C and leaves at 40°C. Design shell and tube heat exchangers for the different tube lengths 1.5 m, 3 m, 4.5 m and 6 m.
- 6.4 3000 kg/h of nearly pure saturated Phosphorous Oxychloride (POCl_3) vapour at 15 kPa g is to be condensed and cooled to 60°C by cooling water which is available in plant at 32°C. Design the suitable shell and tube heat exchanger for the given duty using following data.

Data:

Tube material: Graphite

Tube OD : 25 mm

Tube ID : 20 mm

Thermal conductivity of graphite: 150 W/(m · K)

Molar mass of POCl_3 : 153.4 kg/kmol

Specific heat of liquid POCl_3 : 0.92 kJ/(kg · °C)

Latent heat of vaporization of POCl_3 : 229 kJ/kg

Thermal conductivity of liquid POCl_3 : 0.173 W/(m · °C)

Viscosity of liquid POCl_3 = 1.1 cP

Liquid density of POCl_3 = 1675 kg/m³

Condensation temperature of POCl_3 at 15 kPa g = 110°C

Viscosity of POCl_3 vapour, μ_v = 1000×10^{-7} Poise

- 6.5 Design vertical thermosyphon reboiler for acetic acid dehydration column based on following data :

(a) Feed is liquid at 30°C.

Feed flow rate = 12 000 kg/h

Feed composition = 80% acetic acid and 20% water (by mass)

(b) Distillate is saturated liquid at 100.6°C

Flow rate of distillate = 3000 kg/h

Distillate composition = 20% acetic acid and 80% water (by mass)

(c) Residue is nearly pure saturated acetic acid at 138°C and at 179.5 kPa a.

(d) Reflux ratio, $R = 4.2$.

(e) Latent heat of vaporization

$$\lambda_{AA} = 25\ 732 \text{ kJ/kmol at } 293.8 \text{ K}$$

$$\lambda_W = 40\ 677 \text{ kJ/kmol at } 373.15 \text{ K}$$

(f) Critical temperature of acetic acid = 592.71 K

Critical temperature of water = 647.11 K

(g) Heat capacity equation for liquids

$$C_{ml} = a + bT + cT^2 + dT^3$$

where, C_{ml} = Liquid heat capacity, kJ/(kmol · K)

T = Absolute temperature, K

Table 6.45 Heat Capacity Data⁴

	a	$b \times 10^3$	$c \times 10^6$	$d \times 10^9$
Acetic acid	-36.0814	604.681	-393.957	-561.602
Water	18.2964	472.118	-1338.78	1314.24

- 6.6 50 000 kg/h cold air is required at 18°C temperature. Chilled water with 6°C supply temperature and 8°C return temperature is to be used as cooling medium. Design a suitable finned tube heat exchanger with box type shell.

Hint: Atmospheric air contains water vapour. Hence condensation load of water vapour must be considered in the calculation of heat duty. Air saturated with water vapour at 40°C can be considered as design condition.

- 6.7 Synthesize the heat exchanger network for the following five process streams such that resulting heat exchanger network will require the minimum hot and cold utilities. Also find the values of minimum utilities (hot and cold) required.

Table 6.46 Process Stream Data²³

Stream	Type	$\dot{m} \text{ Cp, kW/}^\circ\text{C}$	$t_{in}, {}^\circ\text{C}$	$t_{out}, {}^\circ\text{C}$
1	Hot	228.5	159	77
2	Hot	20.4	267	88
3	Hot	53.8	343	90
4	Cold	93.3	26	127
5	Cold	196.1	118	265

- 6.8 Design a kettle type reboiler which must provide 2000 kg/h of ethylene glycol vapour to a distillation column at 30 kPa a pressure at bottom.

REFERENCES

1. TEMA, 8th Ed., Standard of the Tubular Exchanger Manufacturers Association, New York, USA, 1988.
2. Perry, R. H. and Green D., *Perry's Chemical Engineers' Handbook*, 6th Ed., McGraw-Hill, USA, 1984.
3. *Pfaudler Heat Exchanger Databook*, Pfaudler International GmbH, Basel, Switzerland.
4. Sinnott, R. K., *Coulson and Richardson's Chemical Engineering*, Vol. 6, Revised 2nd Ed., Asian Publishers Books Pvt. Ltd., New Delhi, 1998.
5. Kern, D. Q., *Process Heat Transfer*, McGraw-Hill, USA, 1950.
6. Ludwig, E. E. *Applied Process 3rd Design for Chemical and Petrochemical Plants*, Vol. 3, 3rd Ed. Gulf Publishing, USA, 2001.
7. Smith, R.A., *Vaporisers: Selection, Design and Operation*, Longmans, UK, 1986.
8. Dadyburjor, D. B., *Chem. Engg. Progr.*, 74(4), 1978, p. 86.
9. Bhatt, B.I. and S. M. Vora, *Stoichiometry*, 4th Ed., Tata McGraw-Hill Publishing Co. Ltd., New Delhi, 2004.
10. Hughmark, G. A., *Chem. Eng. Progr.* 60(7), 1964, p. 59.
11. Fair, J. R., *Chem. Engg.*, 70(14), July, 1963, p. 119.
12. Frank, O. and Prickett, R. D., *Chem. Engg.*, 80(20), September 3, 1973, p. 107.
13. Saunders, E. A. D., *Heat Exchangers*, 1st Ed., Longmans, UK, 1988.
14. Buonopane, R. A., Troupe, R. A. and J. C. Morgan, *Chem. Engg. Progr.*, 59(7), 1963, p. 57 to 61.
15. Bhatia, M. V. and P. N. Cheremisinoff, *Process Equipment Series, Heat Transfer Equipment*, Vol. 2, Technomic Publishing Company, Inc., USA, 1980.
16. Minton, P. E., *Chem. Engg.*, 77(10) May 4, 1970, p. 103.
17. Wadekar, V. V., *Chem. Eng. Progr.* 96(12), 2000, p. 39 to 49.
18. Linhoff, B., Townsend, D. W. and G. W. Hewitt, *User Guide on Process Integration for the Efficient Use of Energy*, Rev. Ed., Institution of Chemical Engineers, London, UK, 1994.
19. Bagajewicz, M. and J. Shuncheng, *Ind. Eng. Chem. Res.*, 40(2), 2001, 617–626.
20. Bagajewicz, M. and J. Soto, *Ind. Eng. Chem. Res.*, 40(2), 2001, p. 627–634.
21. Adham, K., *Chem. Engg. Progr.*, 96 (8), 2000, p. 37.
22. McKetta J.J. and W.A. Cunningham, (Ed.) *Encyclopedia of Chemical Processing and Design*, Vol. 23, Marcel Dekker, Inc., USA, 1985, p. 51 and 76 to 79.
23. Linhoff, B. and T. N. Tjoe, *Chem. Engg.*, 93(8), April 28, 1986, p. 47 to 60.
24. Chopey, N. P., *Handbook of Chemical Engineering Calculations*, 2nd Ed., McGraw-Hill, Inc., USA, 1994, Ch-7.
25. McAdams, W. H., *Heat Transmission*, 2nd Ed., McGraw-Hill, Inc., USA 1942.

Chapter

7



Design of Liquid-Liquid Extractor

7.1 INTRODUCTION

Liquid-liquid extraction is the separation of the components of a liquid solution by contacting it with another insoluble liquid solvent. One of the components or certain group of the components of the solution (feed) is selectively soluble in the solvent. It is called *solute*. After the contact, components of liquid feed are distributed between two immiscible liquids; called extract phase and raffinate phase. Solvent rich phase is called extract phase and solvent lean phase is called raffinate phase, respectively. A component which is left behind in the raffinate phase after extraction is called *diluent*.

Solvent extraction finds many applications in industry. Take an example of separation of solution containing 10% by mass acetic acid and 90% by mass water. Separation of these two is very difficult by distillation as this mixture provides very low relative volatility. In liquid-liquid extraction, acetic acid–water solution is contacted with a solvent such as ethyl acetate. The solvent makes the immiscible or partially miscible liquid mixture with water. Acetic acid is selectively extracted in ethyl acetate. At the end of single stage extraction two immiscible liquid layers are obtained. Ethyl acetate rich layer is called extract phase and water rich layer is called raffinate phase.

Liquid–liquid extraction is an important unit operation and finds applications in refinery, pharmaceutical industries, metallurgical industries and other industries. In refinery, after distillation, liquid-liquid extraction is the most widely used unit operation.

Invariably extraction will have to be followed by another unit operation, such as distillation for separation of solute from the extract phase.

7.2 IMPORTANT INDUSTRIAL APPLICATIONS OF LIQUID-LIQUID EXTRACTION^{1, 2}

- (i) Separation of 10% to 30% acetic acid in aqueous solution by using ethyl acetate or ethyl ether as solvent.
- (ii) Recovery of uranium from ore rich liquor. Solvent for this case is alkyl phosphate in kerosene.
- (iii) Separation of long-chain fatty acids from vegetable oil by using propane as solvent.
- (iv) Propane deasphalting. Separation of lube oil from residuum (residue of vacuum distillation column of refinery) by using propane as solvent.
- (v) Separation of tantalum and niobium. It involves dissolution of this mixutre in hydrofluoric acid, followed by extraction with Methyl Isobutyl Ketone (MIBK). Similarly liquid–liquid extraction is used for the metal separations such as uranium–vanadium, hafnium–zirconium and tungsten–molybdenum mixtures.
- (vi) Phosphoric acid and boric acid are finally purified by liquid–liquid extraction.
- (vii) Liquid–liquid extraction is used for the separation of the mixture of aromatic and paraffinic hydrocarbons of nearly the same molar mass (e.g. a mixture of benzene and hexane). For this case, solvents used are liquid sulphur dioxide, diethylene glycol or sulfolane. Extractive distillation is also used for the same application.
- (viii) Many pharmaceutical products are produced in the form of complex mixture. From this mixture, pure product is separated by liquid–liquid extraction; e.g. penicillin, resorcinol, etc.
- (ix) In the petroleum refinery the separation of pure benzene and toluene from aromatic rich feed stock is carried out by extraction. In this case sulfolane is used as solvent.
- (x) For dearomatization of raw hexane fraction (or for the production of food grade hexane) using N–methyl pyrrolidone (NMP) as solvent.
- (xi) For dearomatization of the straight run kerosene fraction (or for the production of superior kerosene) using sulfolane as solvent.
- (xii) For the purification of lube oil using furfural or NMP as solvent.
- (xiii) In an aqueous effluent containing very high COD, organic solvent (such as toluene or xylene) is effectively used to extract out COD causing components.
- (xiv) In the manufacturing process of MTBE, methanol is separated from the mixture of methanol and C₄ stream by using water as solvent.
- (xv) For extraction of unsaturated fatty acids from a mixture of fatty acids using fulfural as solvent.

7.3 PHASE EQUILIBRIUM

Distribution coefficient or partition coefficient K or m is defined as

$$K \text{ or } m = \frac{\gamma}{x} = \frac{\text{mass fraction of solute in extract phase}}{\text{mass fraction of solute in raffinate phase}} \quad (7.1)$$

In liquid-liquid extraction, all the components are present in both extract and raffinate phases at equilibrium. At equilibrium, activity of any component is same in both phases.

$$\bar{A}_r = \bar{A}_e \Rightarrow \gamma_r x = \gamma_e y \quad (7.2)$$

where, \bar{A}_r = activity of component in raffinate phase

\bar{A}_e = activity of component in extract phase

γ_r, γ_e = activity coefficients of components in raffinate/extract phase

y = mole fraction of component in extract phase

x = mole fraction of component in raffinate phase

$$K' = \frac{y}{x} = \frac{\gamma_r}{\gamma_e} \quad (7.3)$$

where K' = distribution coefficient in mole fraction units

Large collections of experimental equilibrium data are now available for liquid-liquid systems. One source is "Liquid-Liquid equilibrium Data Collection" by Sorenson and Arit, DECHEMA, Frankfurt, Germany. In *Perry's Chemical Engineers' Handbook*, (Ref. 3) distribution coefficients for a few ternary systems are given (Table 15.5 of 6th Ed.)

7.4 DESIRABLE SOLVENT PROPERTIES OR CHOICE OF SOLVENT

7.4.1 Selectivity

The relative separation or selectivity is defined as the ratio of ratios of solute to nonsolute components in extract phase to the same in raffinate phase at equilibrium. It is represented as α or β .

For all useful extractions, $\alpha > 1$. If $\alpha = 1$, it means no separation or no extraction. It represents the separation power of solvent. Higher the selectivity, lesser is the number of theoretical stages required for the desired separation. Dilute solute concentration generally gives the highest selectivity.

7.4.2 Distribution Coefficient

Distribution coefficient K or m is the ratio of mass fraction of solute in extract phase to mass fraction of solute in raffinate phase at equilibrium. $K = Y/X$ at equilibrium as defined in Eq. (7.1).

It is not necessary that K should be greater than 1. K can be less than 1. But higher value of K is desirable because higher the distribution coefficient, lesser is the amount of solvent required for desired separation. Unfortunately solvents which provide the higher value of K normally provide poor value of α .

7.4.3 Recoverability

It is always necessary to recover the solvent from extract phase for reuse. Hence, selected solvent must be easily recoverable from the extract phase. In most of the cases, solvent is separated from extract phase by distillation. If selected solvent is

to be separated or recovered by distillation than it should not form an azeotrope with the solute and also it should provide higher value of relative volatility. Lesser value of latent heat of vaporization of solvent is also desirable for low cost of recovery. Water is not an attractive solvent for extraction as it forms azeotrope or non-ideal solution with many organics and it has very high value of latent heat of vaporization.

7.4.4 Capacity

Capacity of a solvent to extract the solute is measured in terms of maximum solubility of the solute in solvent or in terms of solubility limit. Higher the solubility limit, lesser is the amount of solvent required. Solute should be significantly soluble in the selected solvent.

7.4.5 Insolubility of Solvent

Low solubility of solvent in the raffinate phase is desirable. Solvent which is more insoluble is preferred. More insoluble solvent can separate wide range of composition of feed mixture. Also, it gives less solvent loss and adds less impurity in the final raffinate. Generally, a solvent which is more insoluble provides higher selectivity.

7.4.6 Density

Higher density difference between solvent and diluent or extract and raffinate phase is desirable. It makes the decantation easier. In most of the systems, density difference decreases to zero at plait point, but in some system it can become zero at an intermediate solute concentration and can invert the phases at higher concentration. For such a system, in the desired range of separation in the extractor, if density difference passes through zero then use of continuous contact equipment like packed tower is not preferred but mixer-settler type stage wise extractor can be used. Other option is to use two continuous contact extractors in series.

7.4.7 Interfacial Tension

A high interfacial tension promotes rapid coalescence and generally requires high mechanical energy for agitation to produce small droplets. Conversely, low interfacial tension requires lesser mechanical energy for agitation to produce small droplets but at the same time it provides slow coalescence rates. Usually interfacial tension decreases with increase in solute concentration and increase in mutual solubility and falls to zero at plait point. Very high and very low interfacial tension of selected solvent is not desirable, otherwise higher collision rate is more important, hence moderately high interfacial tension is preferred.

7.4.8 Chemical Reactivity

The solvent should be inert towards the other components of extraction system and to the common materials of construction.

7.4.9 Viscosity, Vapour Pressure and Freezing Point

All these properties of solvent should be low for ease in handling, transportation and storage.

7.4.10 Toxicity and Flammability

The solvent should be nontoxic, non flammable and of low cost.

7.5 DESIGN OF COUNTER CURRENT MULTISTAGE EXTRACTOR

Process design of extractor can be divided in the following steps:

- (i) Selection of suitable solvent and determination of liquid-liquid equilibrium data. Actually both these steps are interrelated.
- (ii) Determine the minimum amount of solvent required for the desired separation.
- (iii) Determine or decide the actual amount of solvent. Actual amount of solvent must be the optimum amount of solvent. Then complete and finalise the material balance.
- (iv) Determine the number of theoretical stages (for stage wise extractor) or number of transfer units (for differential continuous contact extractor) required for the desired separation.
- (v) Find the tower diameter of tower type extractor or carry out the process design of agitator for agitated vessel type extractor and also decide the height and diameter of agitated vessel.
- (vi) Find the stage efficiency for stage wise extractor or height of transfer units for differential or continuous contact type extractor.
- (vii) Find the actual number of stages for stage wise extractor or height of tower for tower type extractor.
 - (a) First step is explained in previous Sec. 7.4.
 - (b) S_m : Minimum amount of solvent is the maximum amount of solvent for which infinite number of stages are required for desired separation.

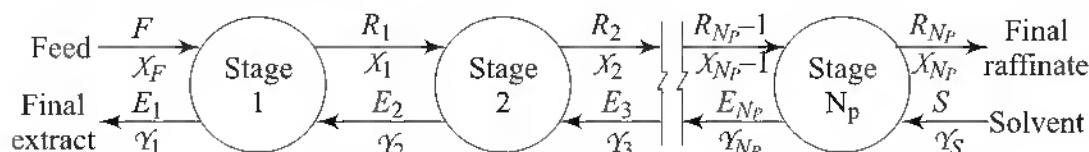


Fig. 7.1 Multistage Counter Current Extraction

$$F + S_m = E_{lm} + R_{NP} \quad (7.4)$$

$$R_{NP} - S_m = F - E_{lm} = \Delta R_m \quad (7.5)$$

ΔR_m is a difference point. It represents net flow outward at the last stage N_p and is equal to net flow inward at first stage. On an equilateral triangular coordinates, when operating line from ΔR matches with tie line, infinite number of stages are required for the desired separation. To find S_m , extend the tie lines and allow them to intersect \overrightarrow{SR}_{NP} . A line joining the equilibrium compositions of extract and raffinate phases is called a tie line.

Among all such points, a point that is farthest from S , if all points are on the left hand side of S or a point which is nearest to S , if all points are on the right hand side of S , represents ΔR_m , the difference point for minimum solvent.

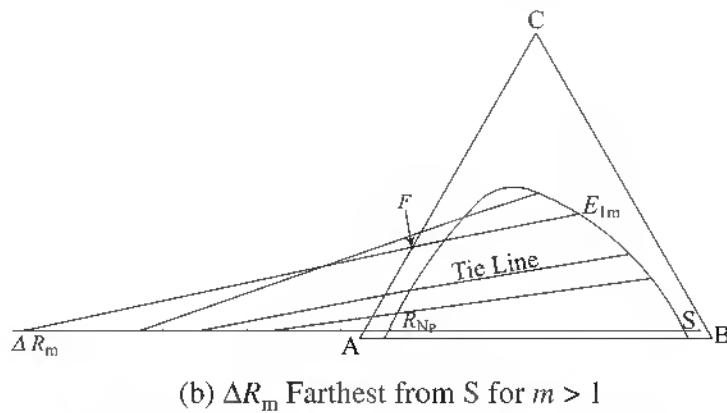
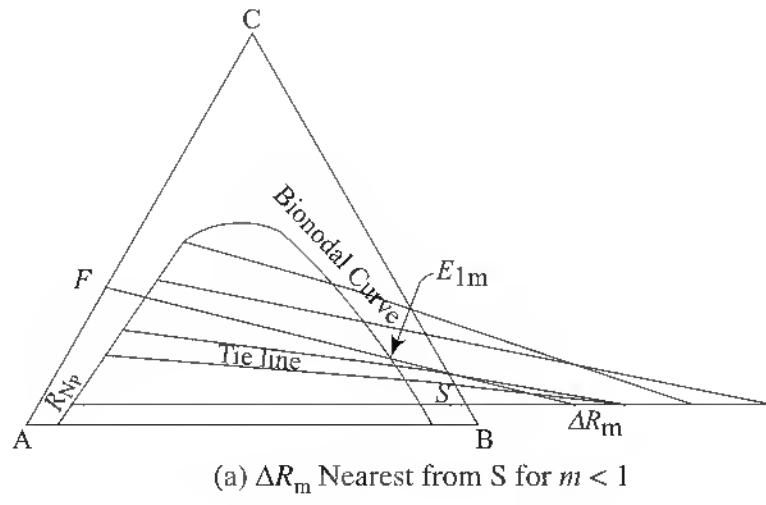


Fig. 7.2 Minimum Solvent for Counter Current Extraction⁴

In most of the cases but not in all cases, tie line which is on extension passes through F gives the location of ΔR_m . After finding the location of ΔR_m , locate E_{1m} . Draw the line $E_{1m} R_{NP}$ which intersects line FS at X_{Mm} .

$$F + S_m = M_m \quad (7.6)$$

$$F X_F + S_m Y_S = X_{Mm} M_m = X_{Mm} (F + S_m) \quad (7.7)$$

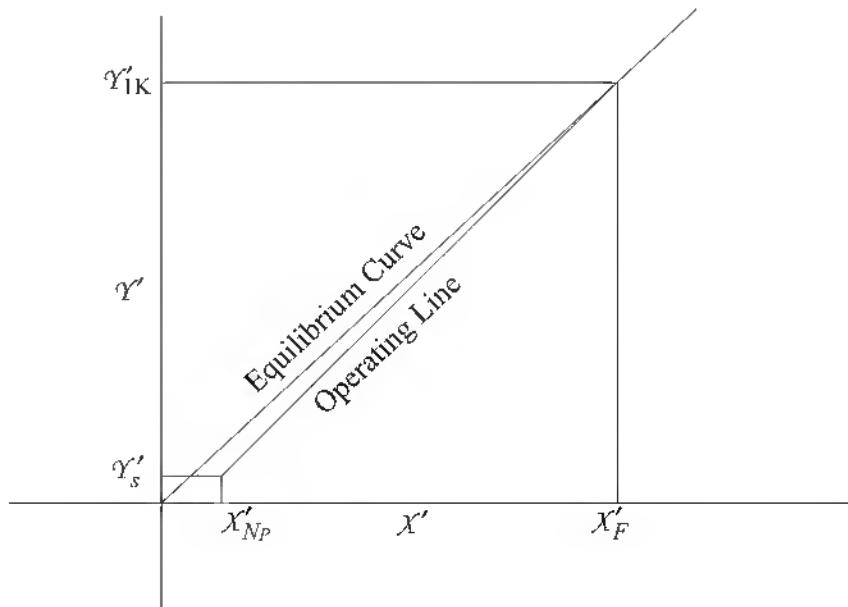
By substituting the value of X_{Mm} from the graph, one can find S_m using Eq. (7.7).

If A and B are insoluble liquids or can be considered as insoluble liquids at operating temperature and in desired concentration change, then solute balance is given by following equation.

$$A(X'_F - X'_{NP}) = B(Y'_1 - Y'_S) \quad (7.8)$$

where,

$$X' = \frac{\text{kg of solute (C)}}{\text{kg of nonsolute (A)}} \quad \text{and} \quad Y' = \frac{\text{kg of solute (C)}}{\text{kg of solvent (B)}}$$

**Fig. 7.3** Determination of B_m

$$\frac{A}{B} = \frac{Y'_1 - Y'_s}{X'_F - X'_{NP}} \quad (7.9)$$

$$\frac{A}{B_m} = \frac{Y'_{1k} - Y'_s}{X'_F - X'_{NP}} \quad (7.10)$$

where, B_m = Minimum amount of solvent

Y'_{1k} is an equilibrium with X'_F . From equilibrium data, one can find Y'_{1k} that is in equilibrium with X'_F . Then from Eq. (7.10), one can find B_m . Operating line starts from point (X'_{NP}, Y'_s) .

(iii) To decide the actual amount of solvent, find the number of theoretical stages required for the desired separation for the different values of actual amount of solvent, like

$$S = 1.1 S_m, S = 1.2 S_m, S = 1.3 S_m \dots \text{and so on.}$$

On increasing the values of S from S_m , value of N will decrease. In initial range, N will significantly decrease with increase in S , but after a certain increase in S , N may not decrease appreciably.

For example, in this case (Fig. (7.4)) one can say optimum value of S is very close to $1.4 S_m$.

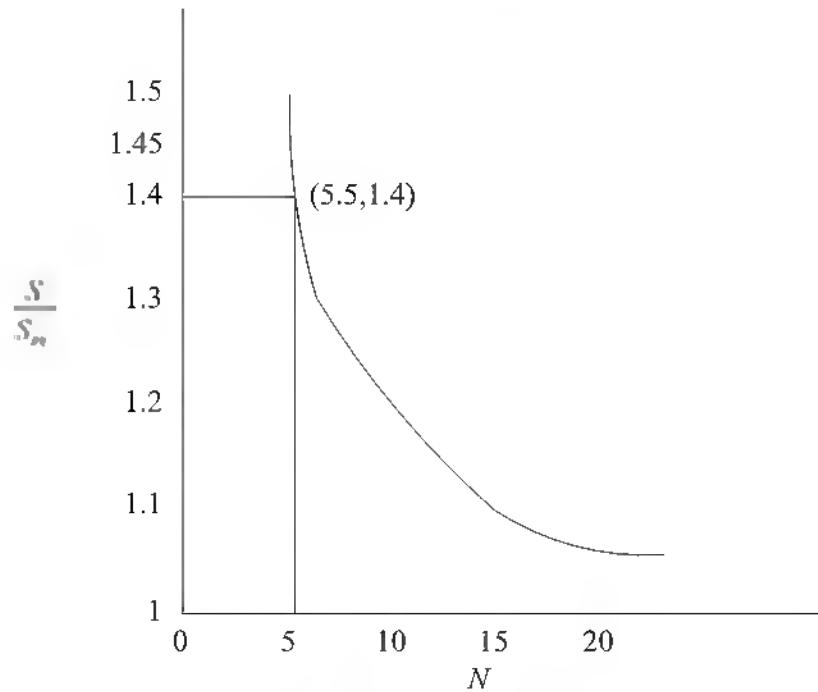
Ideally actual amount of solvent = S_{opt}

But to find optimum values of S detailed costing calculations are required.

(iv) How to find number of theoretical stages for desired separation N from the value of S :

(a) If A and B are not completely insoluble liquids:

Refer Fig. (7.1).

**Fig. 7.4** Determination of S_{opt}

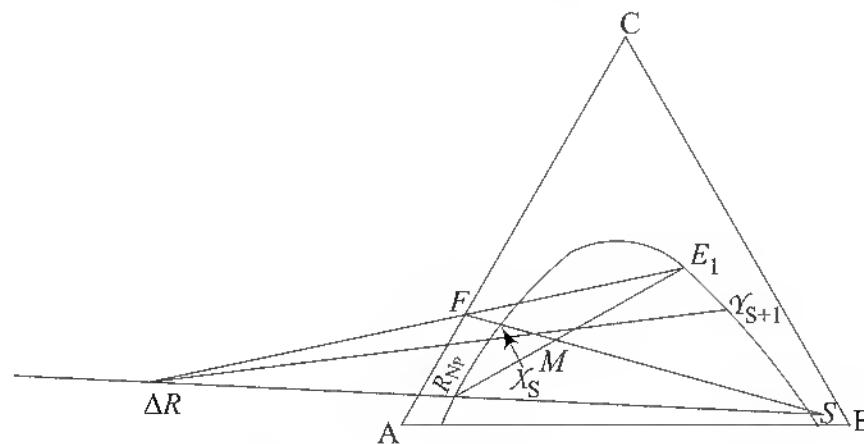
Overall Material Balance.

$$F + S = E_I + R_{NP} \quad (7.11)$$

Solute balance:

$$FX_F + S\gamma_s = E_I\gamma_I + X_{NP}R_{NP} = M\chi_M \quad (7.12)$$

$$F - E_I = R_{NP} - S = \Delta R = \text{Net flow outward} = \text{Net flow inward} \quad (7.13)$$

**Fig. 7.5** Determination of Operating Curve

Procedure:

Find $\chi_m = \frac{FX_F + S\gamma_s}{F + S}$. Locate point M on graph which lies on \overline{FS} line.

Extend the line $\overline{R_{NP}, M}$ that will intersect the binodal curve at E_1 . Draw the lines $\overline{E_1 F}$ and $\overline{R_{NP} S}$. These will intersect at the point ΔR . Draw the random lines from ΔR that give the points of operating curve (χ_s, γ_{s+1}) .

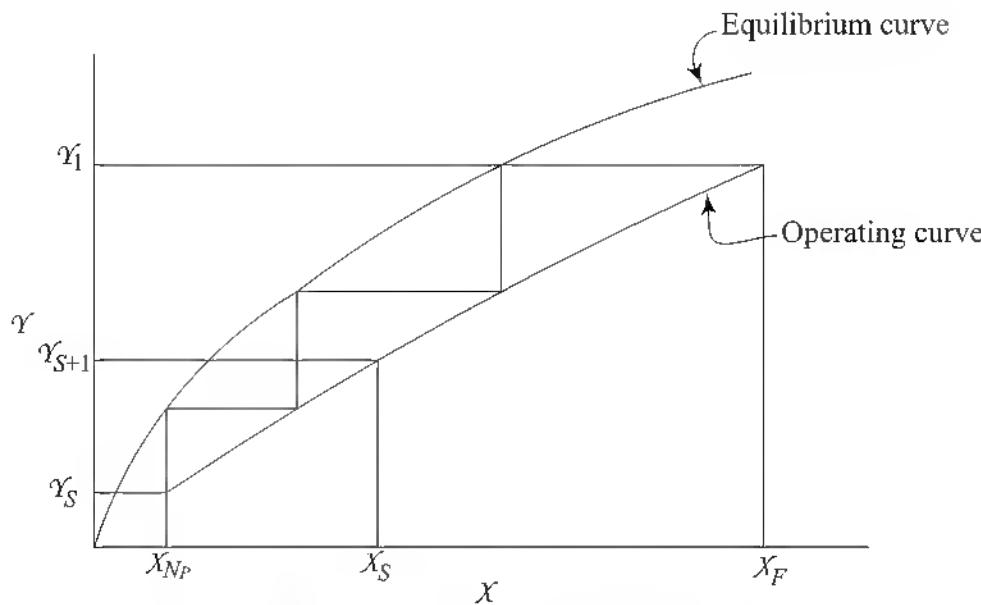


Fig. 7.6 Determination of Equilibrium Stages

Operating curve starts from point (X_F, Y_1) and terminates at point (X_{NP}, Y_s) . Start the stagewise construction from (X_F, Y_1) to (X_{NP}, Y_s) in between equilibrium curve and operating line and find number of theoretical stages, required for the desired separation.

- (b) If A and B are immiscible liquids then overall material balance will be

$$A(X'_F - X'_{NP}) = B(Y'_1 - Y'_s) \quad (7.14)$$

By using Eq. (7.14), for the given actual amount of solvent B and its composition Y'_s , one can find Y'_1 . Operating line starts at $(X'_F - Y'_1)$ and terminates at (X'_{NP}, Y'_s) .

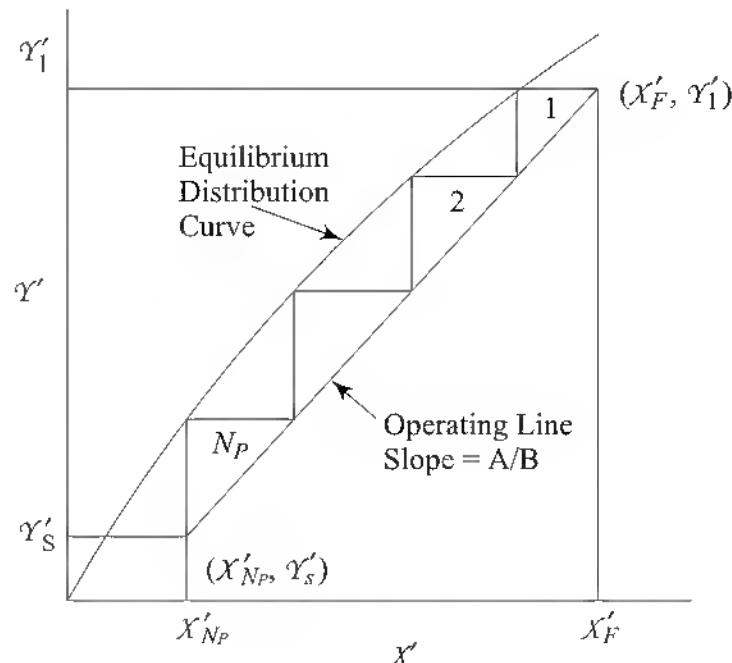


Fig. 7.7 Counter Current Extraction with Insoluble Solvent

Stagewise construction starts from point. (X'_F, Y'_1) and terminates at the point (X'_{NP}, Y'_s) of the operating line.

In a special case, where equilibrium curve is also the straight line then the number of theoretical stages required for the desired separation can be obtained from Eq. (7.16 a). or Eq. (7.16 b) (Ref. 3).

$$m' = \frac{Y'}{X'} = \text{slope of equilibrium curve}$$

Ratio of the slope of equilibrium curve to the slope of operating line is given by following equation.

$$\varepsilon = \frac{m'B}{A} \quad (7.15)$$

If, $\varepsilon \neq 1$

$$N = \frac{\ln \left[\left(\frac{X'_F - Y'_s/m'}{X'_{NP} - Y'_s/m'} \right) \left(1 - \frac{1}{\varepsilon} \right) + \frac{1}{\varepsilon} \right]}{\ln \varepsilon} \quad (7.16 \text{ a})$$

When, $\varepsilon = 1$

$$N = \frac{X'_F - Y'_s/m'}{X'_{NP} - Y'_s/m'} - 1 \quad (7.16 \text{ b})$$

Multistage counter current extraction requires less number stages for the given amount of solvent than the cross current extraction or it requires less amount of solvent for the fixed number of stages than cross current extraction.

Number of Transfer Units (Ref. 3)

In case of differential or continuous contact type extractor, instead of number of theoretical stages, number of transfer units are determined. Then the working height of continuous contact type extractor is given by equation

$$Z = N_t H_t \quad (7.17)$$

where, N_t = Number of transfer units

H_t = Height of transfer units

Number of overall mass transfer units based on raffinate phase is given by equation.

$$N_{toR} = \int_{X_{NP}}^{X_F} \frac{dX}{X - X^*} + \frac{1}{2} \ln \left(\frac{1 - X_{NP}}{1 - X_F} \right) + \frac{1}{2} \ln \left(\frac{X_{NP} (r - 1) + 1}{X_F (r - 1) + 1} \right) \quad (7.18)$$

where, X_F = Mass fraction of solute in feed

X_{NP} = Mass fraction of solute in final raffinate

r = Ratio of molar masses of non solute to solute

X^* = For any point (X_a, Y_a) of operating curve. X_a^* is the value of X that is in equilibrium with Y_a .

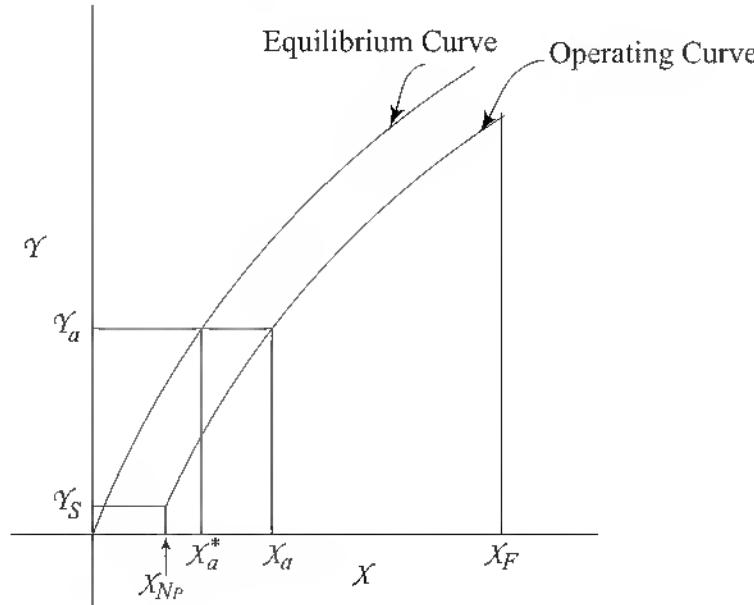


Fig. 7.8 Determination of Number of Transfer Units

If solute component or fraction of feed (*A*) and solvent (*B*) can be considered as completely immiscible liquid mixture, then it is better to use mass ratio concentrations, rather than mass fractions. For this special case N_{toR} is given by equation

$$N_{toR} = \int_{X'_{NP}}^{X'_F} \frac{dX'}{X' - X'^*} + \frac{1}{2} \ln \left(\frac{1 + r X'_{NP}}{1 + r X'_F} \right) \quad (7.19)$$

where, X'_F = Mass ratio of solute to non solute in feed

X'_{NP} = Mass ratio of solute to non solute in final raffinate

X'^* = For any point (X'_a, Y'_a) of operating line X'^* is the value of X' that is in equilibrium with Y'_a

r = Ratio of molar masses of non solute to solute

In another special case where operating line and equilibrium curve both are straight lines, number of overall mass transfer units based on raffinate phase (N_{toR}) is given by equation,

$$N_{toR} = \frac{\ln \left[\left(\frac{X'_F - Y'_s/m'}{X'_{NP} - Y'_s/m'} \right) \left(1 - \frac{1}{\varepsilon} \right) + \frac{1}{\varepsilon} \right]}{\left(1 - \frac{1}{\varepsilon} \right)} \quad (\text{if } \varepsilon \neq 1) \quad (7.20)$$

or

$$N_{toR} = [(X'_F - Y'_s/m') / (X'_{NP} - Y'_s/m')] - 1. \quad (\text{if } \varepsilon = 1) \quad (7.21)$$

where, $\varepsilon = \frac{m'B}{A}$ and $m' = \frac{Y'}{X'}$

Example 7.1

A feed stream having flow rate of 200 kg/h and containing 20 mass % acetic acid in water is to be extracted at 25°C with 400 kg/h of recycled MIBK (Methyl IsoButyl Ketone) that contains 0.1 per cent acetic acid and 0.01 per cent water. The aqueous raffinate is to be extracted down to 1 per cent acetic acid. How many theoretical stages will be required and what will the extract composition be? Extraction is to be carried out in counter current manner.

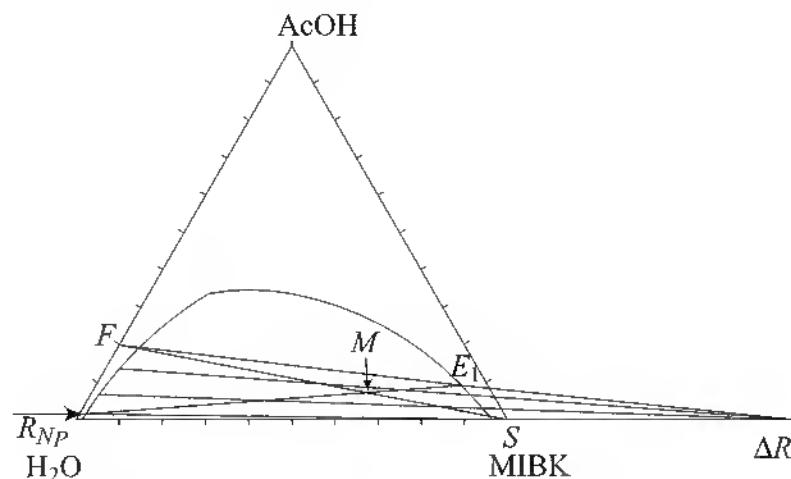
Table 7.1 Equilibrium Data of Water – Acetic acid – Methyl Isobutyl Ketone at 25°C

Mass % in Raffinate				Mass % in Extract			
Water	Acetic acid	MIBK	χ'	Water	Acetic acid	MIBK	γ'
98.45	0	1.55	0	2.12	0	97.88	0
95.46	2.85	1.7	0.0299	2.8	1.87	95.33	0.0196
85.8	11.7	2.5	0.1364	5.4	8.9	85.7	0.1039
75.7	20.5	3.8	0.2708	9.2	17.3	73.5	0.2354
67.8	26.2	6.0	0.3864	14.5	24.6	60.9	0.4039
55	32.8	12.2	0.5964	22	30.8	47.2	0.6525
42.9	34.6	22.5	0.8065	31	33.6	35.4	0.9492

(Table 15.1 of Ref. 3)

χ' = mass ratio of solute to diluent (C/A) in raffinate phase

γ' = mass ratio of solute to extraction solvent (C/B) in extract phase

**Fig. 7.9 Ternary Diagram for AcOH-H₂O-MIBK System³**

Solution:

$$F + S = E_I + R_{NP} = M \quad (7.11)$$

$$200 + 400 = M = 600 \text{ kg/h}$$

$$\chi_m = \frac{F\chi_F + S\gamma_s}{F + S}$$

$$= \frac{(200 \times 0.2) + (400 \times 0.001)}{600} = 0.06733, \quad \gamma_i = 0.088$$

(From Fig. 7.9)

$$M\mathcal{X}_M = E_I \mathcal{Y}_I + \mathcal{X}_{NP} R_{NP}$$

$$40.4 = 0.088(600 - R_{NP}) + 0.01R_{NP}$$

$$R_{NP} = 158.97 \text{ kg/h}, E_I = 441.03 \text{ kg/h}$$

Random lines from ΔR give the points of operating line ($\mathcal{X}_s, \mathcal{Y}_{s+1}$).

\mathcal{X}_s	0.2	0.14	0.08	0.065	0.01
\mathcal{Y}_{s+1}	0.085	0.06	0.035	0.025	0.001

Equilibrium data:

\mathcal{X}	0	0.0285	0.117	0.205	0.262	0.328	0.346
\mathcal{Y}^*	0	0.0187	0.089	0.173	0.246	0.308	0.336

Stepwise construction from \mathcal{X}_{sF} (\mathcal{X}_s corresponds to \mathcal{X}_F) to \mathcal{X}_{NP} gives number of theoretical stages required for desired separation.

From graph, $N = 4.62$

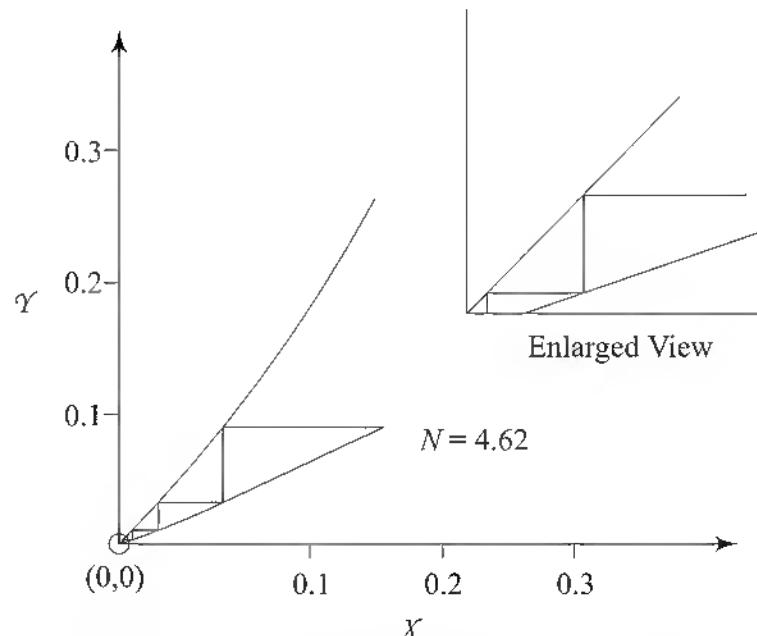


Fig. 7.10 Determination of Number of Stages for Example 7.1

Example 7.2

Solve Example 7.1 considering MIBK (B, solvent) and water (A, non-solute component) of feed as completely immiscible liquids and equilibrium curve as a straight line in terms of mass ratio concentrations.

Solution:

Overall solute balance

$$A(\mathcal{X}'_F - \mathcal{X}'_{NP}) = B(\mathcal{Y}'_1 - \mathcal{Y}'_s) \quad (7.14)$$

$$A = 200(1 - 0.2) = 160 \text{ kg/h}$$

$$B = 400(1 - 0.001) = 399.6 \text{ kg/h}$$

$$\mathcal{X}'_F = \frac{20}{80} = 0.25 \quad \mathcal{X}'_{NP} = \frac{1}{99} = 0.0101$$

$$\mathcal{Y}'_1 = ? \quad \mathcal{Y}'_s = \frac{0.1}{(100 - 0.1 - 0.01)} = 0.001$$

$$160(0.25 - 0.0101) = 399.6(\mathcal{Y}'_1 - 0.001)$$

$$\gamma_1' = 0.097 \frac{\text{kg acetic acid}}{\text{kg MIBK}}$$

$$X_F' = 0.25,$$

For operating range $X' < 0.25$ slope of equilibrium curve

$$m' = \left[\left(\frac{0.0196}{0.0299} \right) \times \left(\frac{0.1039}{0.1364} \right) \times \left(\frac{0.2354}{0.2708} \right) \right]^{1/3} = 0.7571$$

$$\varepsilon = \frac{m'B}{A} = 0.7571 \times \frac{399.6}{160} = 1.89$$

$$N = \frac{\ln \left[\left(\frac{X_F' - Y_s'm'}{X_{NP}' - Y_s'm'} \right) \left(1 - \frac{1}{\varepsilon} \right) + \frac{1}{\varepsilon} \right]}{\ln \varepsilon} \quad (7.16 \text{ a})$$

$$N = \frac{\ln \left[\left(\frac{0.25 - (0.001/0.7571)}{0.0101 - (0.001/0.7571)} \right) \left(1 - \left(\frac{1}{1.89} \right) \right) + \left(\frac{1}{1.89} \right) \right]}{\ln 1.89}$$

$$N = 4.13$$

Example 7.3

For the extraction, described in Example 7.1, calculate

- (a) N_{toR} considering MIBK and water as partially miscible liquids.
- (b) N_{toR} considering MIBK and water as completely immiscible liquids and equilibrium curve as straight line (in terms of mass ratio concentration).

Solution:

(a) Number of overall mass transfer units based on raffinate phase is given by equation

$$N_{toR} = \int_{X_{NP}}^{X_F} \frac{dX}{X - X^*} + \frac{1}{2} \ln \left(\frac{1 - X_{NP}}{1 - X_F} \right) + \frac{1}{2} \ln \left(\frac{X_{NP}(r-1)+1}{X_F(r-1)+1} \right) \quad (7.18)$$

$$X_F = 0.2, X_{NP} = 0.01$$

Data for equilibrium curve:

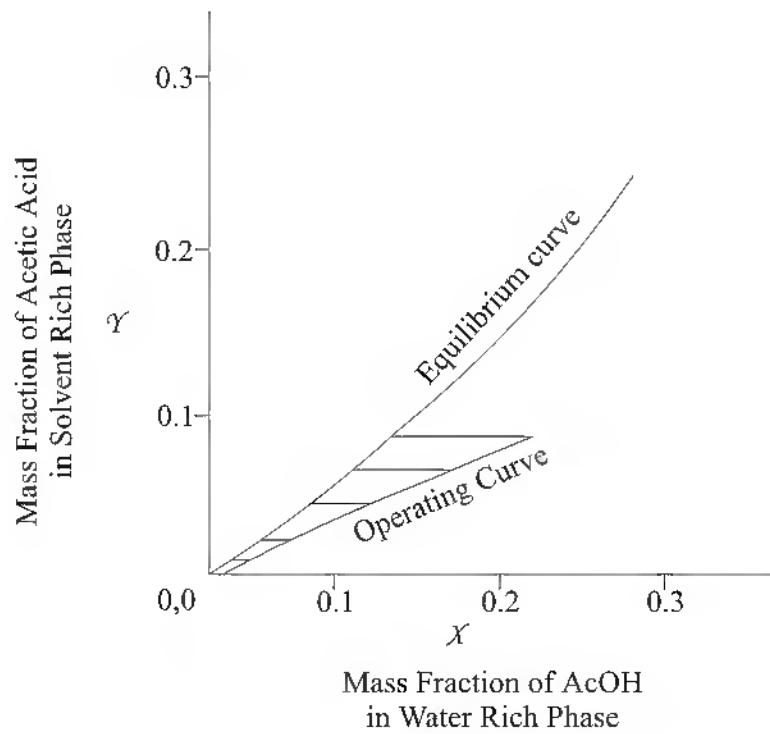
X	0	0.0285	0.117	0.205	0.262	0.328	0.346
γ	0	0.0187	0.089	0.173	0.246	0.308	0.336

Data for operating curve:

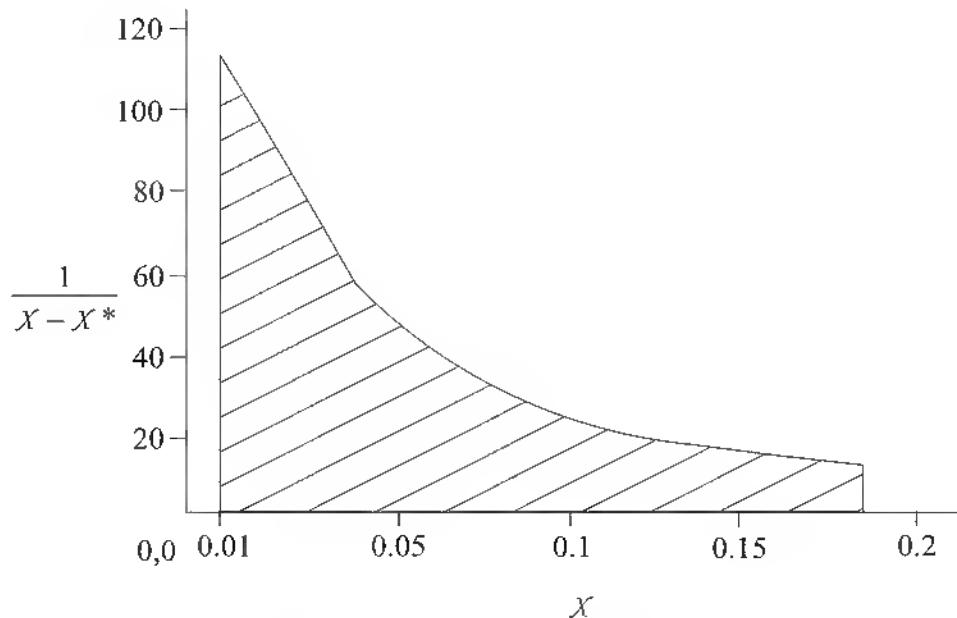
X_s	0.2	0.14	0.08	0.01
γ_{s+1}	0.085	0.06	0.035	0.001

Table 7.2 Data for Finding N_{toR} [from Fig. (7.11)]

X	0.2	0.15	0.1	0.05	0.025	0.01
X^*	0.113	0.089	0.063	0.032	0.014	0.001
$X - X^*$	0.087	0.061	0.037	0.018	0.011	0.009
$1/(X - X^*)$	11.494	16.393	27.027	55.555	90.909	111.111

**Fig. 7.11** Equilibrium Data for AcOH – Water – MIBK System

From Fig. (7.12), $\int_{0.01}^{0.2} \frac{dX}{X - X^*} = 1.7236$ square units = 7.0351 (Integral value)

**Fig. 7.12** Determination of N_{toR}

$$r = \frac{\text{Molar mass of nonsolute}}{\text{Molar mass of solute}} = \frac{18}{60} = 0.3$$

$$N_{toR} = 7.0351 + \frac{1}{2} \ln\left(\frac{1 - 0.01}{1 - 0.2}\right) + \frac{1}{2} \ln\left(\frac{0.01(0.3 - 1) + 1}{0.2(0.3 - 1) + 1}\right)$$

$$N_{toR} = 7.0351 + 0.10655 + 0.0719 = 7.21355$$

(b) If water (A) and MIBK (B) are immiscible liquids and equilibrium curve is straight line (in mass ratio concentration) for the given operating range

$$N_{toR} = \frac{\ln \left[\left(\frac{x'_F - y'_s/m'}{x'_{NP} - y'_s/m'} \right) \left(1 - \frac{1}{\varepsilon} \right) + \frac{1}{\varepsilon} \right]}{\left(1 - \frac{1}{\varepsilon} \right)} \quad (7.20)$$

$$x' \frac{\text{kg of Acetic acid}}{\text{kg of water}} \quad 0 \quad 0.0299 \quad 0.1364 \quad 0.2708$$

$$y' \frac{\text{kg of Acetic acid}}{\text{kg of MIBK}} \quad 0 \quad 0.0196 \quad 0.1039 \quad 0.2354$$

$$x'_F = 0.25, m' = \left[\left(\frac{0.0196}{0.0299} \right) \times \left(\frac{0.1039}{0.1364} \right) \times \left(\frac{0.2354}{0.2708} \right) \right]^{1/3}$$

$$= 0.7571$$

$$\varepsilon = \frac{m'B}{A} = 0.7571 \times \frac{399.6}{160} = 1.89$$

$$N_{toR} = \frac{\ln \left[\left(\frac{0.25 - (0.001/0.7571)}{0.0101 - (0.001/0.7571)} \right) \left(1 - \left(\frac{1}{1.89} \right) \right) + \frac{1}{1.89} \right]}{\left[1 - \left(\frac{1}{1.89} \right) \right]}$$

$$= 5.584$$

Example 7.4

For the separation of dimethylformamide (DMF) from its dilute solution in water, liquid–liquid extraction may require lower operating cost than atmospheric distillation. Methylene chloride is considered to be the best solvent for extracting DMF from its aqueous solution.

DMF–water solution having a flow rate of 1000 kg/h and containing 20% DMF by mass is to be counter currently extracted with methylene chloride to reduce the DMF concentration to 1% in the final raffinate. Determine (a) the minimum amount of solvent which can be used and (b) the number of theoretical stages if actual amount of solvent is double than the minimum required.

Data: Phase equilibrium equation⁵ is given by equation $y = 0.5555 x$ at 25° C where, y and x are mass fractions of solute.

Solution:

Mutual solubilities of water (A) and methylene chloride (B) in each other at room temperature are very low. Hence, to simplify the calculations, water (A) and methylene chloride (B) may be assumed as completely insoluble liquids.

(a) For completely immiscible liquids, solute balance is given by the equation

$$A(x'_F - x'_{NP}) = B(y'_1 - y'_s) \quad (7.14)$$

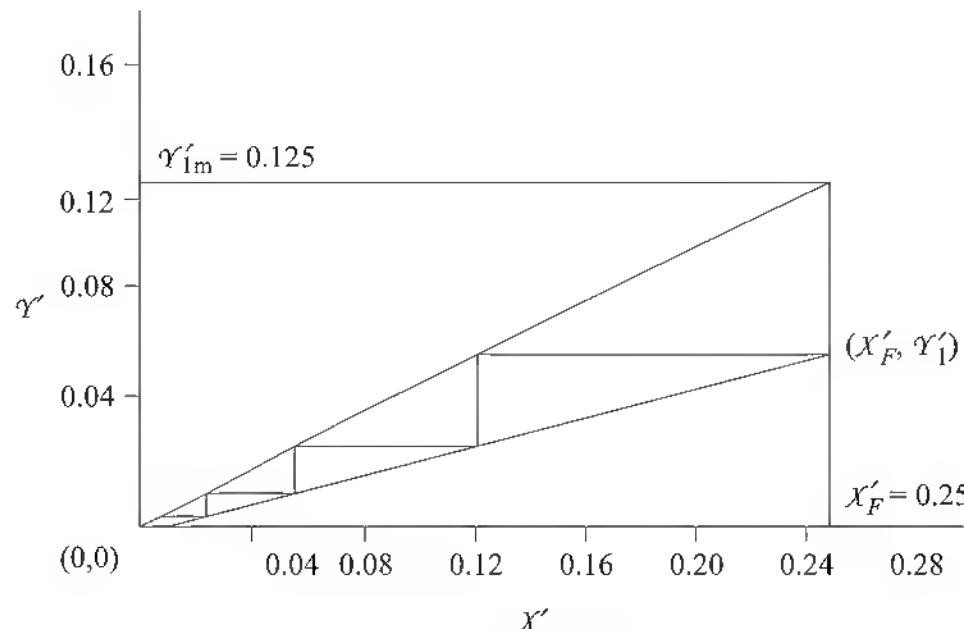


Fig. 7.13 Determination of Equilibrium Stages for Example 7.4

Table 7.3 Determination of Data for Equilibrium Line

γ'	0	0.01389	0.0278	0.042	0.05555	0.0694	0.0833	0.1111	0.1389	0.16665
x'	0	0.025	0.05	0.075	0.1	0.125	0.15	0.2	0.25	0.3
γ'_s	0	0.014	0.0286	0.04384	0.0588	0.0746	0.09	0.125	0.1613	0.2
x'_s	0	0.0256	0.0526	0.081	0.1111	0.1429	0.1765	0.25	0.3333	0.4286

From graph $\gamma'_{1m} = 0.125$ [From Fig. (7.13)]

Solute balance:

$$A(X'_F - X'_{NP}) = B_m(\gamma'_{1m} - \gamma'_s)$$

$$A = F(1 - X_F) = 1000(1 - 0.2) = 800 \text{ kg/h}, X'_{NP} = 0.01/0.99 = 0.0101$$

$$800(0.25 - 0.0101) = B_m(0.125 - 0)$$

$$B_m = 1535.36 \text{ kg/h}$$

(b) Actual amount of solvent $B = 2 \times 1535.36 = 3070.72 \text{ kg/h}$

Solute balance:

$$A(X'_F - X'_{NP}) = B(\gamma'_{1m} - \gamma'_s)$$

$$800(0.25 - 0.0101) = 3070.72 (\gamma'_{1m} - 0)$$

$$\gamma'_{1m} = 0.0625$$

From Fig. 7.13, $N = 3.85$

If equilibrium curve is considered as a straight line in terms of mass ratio concentration, then number of theoretical stages required for desired separation is given by following equation

$$N = \frac{\ln \left[\left(\frac{X'_F - \gamma'_s/m'}{X'_{NP} - \gamma'_s/m'} \right) \left(1 - \frac{1}{\varepsilon} \right) + \frac{1}{\varepsilon} \right]}{\ln \varepsilon} \quad (7.16 \text{ a})$$

$$\varepsilon = \frac{m' B}{A}$$

$$m' = \left[\left(\frac{0.014}{0.0256} \right) \left(\frac{0.04384}{0.081} \right) \left(\frac{0.0746}{0.1429} \right) \left(\frac{0.1613}{0.3333} \right) \right]^{1/4}$$

$$m' = 0.523$$

$$\varepsilon = \frac{0.523 \times 3070.72}{800} = 2$$

$$N = \frac{\ln \left[\left(\frac{0.25}{0.0101} \right) \left(1 - \frac{1}{2} \right) + \frac{1}{2} \right]}{\ln (2)}$$

$$N = 3.687$$

Example 7.5

For the liquid–liquid extraction system, given in Example 7.4, calculate N_{toR} for the actual amount of solvent $B = 2 B_m$.

Solution:

Water (A) and methylene chloride (B) are considered as completely immiscible liquids. Also, as shown in Fig. 7.13, equilibrium curve is a straight line in terms of mass ratio concentration. With these assumptions, formula for finding N_{toR} is

$$N_{toR} = \frac{\ln \left[\left(\frac{x'_F - \gamma'_s m'}{x'_{NP} - \gamma'_s m'} \right) \left(1 - \frac{1}{\varepsilon} \right) + \frac{1}{\varepsilon} \right]}{\left(1 - \frac{1}{\varepsilon} \right)} \quad (7.20)$$

$$B = 2B_m = 3070.72 \text{ kg/h}, \quad A = 800 \text{ kg/h}, \quad x'_F = 0.25, \quad \gamma'_s = 0, \quad m' = 0.523$$

$$\varepsilon = \frac{0.523 \times 3070.72}{800} = 2, \quad x'_{NP} = 0.0101$$

$$N_{toR} = \frac{\ln \left[\left(\frac{0.25 - 0}{0.0101 - 0} \right) \left(1 - \frac{1}{2} \right) + \frac{1}{2} \right]}{\left(1 - \frac{1}{2} \right)}$$

$$N_{toR} = 5.11$$

7.6 INDUSTRIALLY IMPORTANT EXTRACTORS

Many types of extractors are available for achieving mass transfer (Ref. 3, 4, 6, 7, 8, 9) each has its own particular advantage. As per general classification, there are two fundamental types of extractors; stagewise and differential. The stagewise extractors consist of discrete units, in each of which the phases are brought into

contact, mixed, allowed to separate and sent to next unit. Differential extractors provide continuous contact and mass transfer along the full length of the device and phases are separated only at the end.

7.6.1 Industrially Important Extractors can be Divided into Four Major Categories

- (i) Mixer-settlers (stage wise)
- (ii) Unagitated columns (differential)
- (iii) Agitated columns (differential)
- (iv) Centrifugal extractor (differential)

Operation, construction and design of different extractors are given in various books like *Mass Transfer Operations* by Treybal (Ref. 4), *Perry's Chemical Engineers' Handbook* (Ref. 3), *Unit Operations* by G .G. Brown (Ref. 7), etc.

Design of a few type of extractors are discussed here.

7.6.2 Design of Mixer-Settler

- (a) Mixer-settler type extractors have the following advantages over other type of extractors.
 - (i) Provide better contacting of extract and raffinate phases and provide higher rate of mass transfer.
 - (ii) Higher efficiency or attain separation closer to equilibrium.

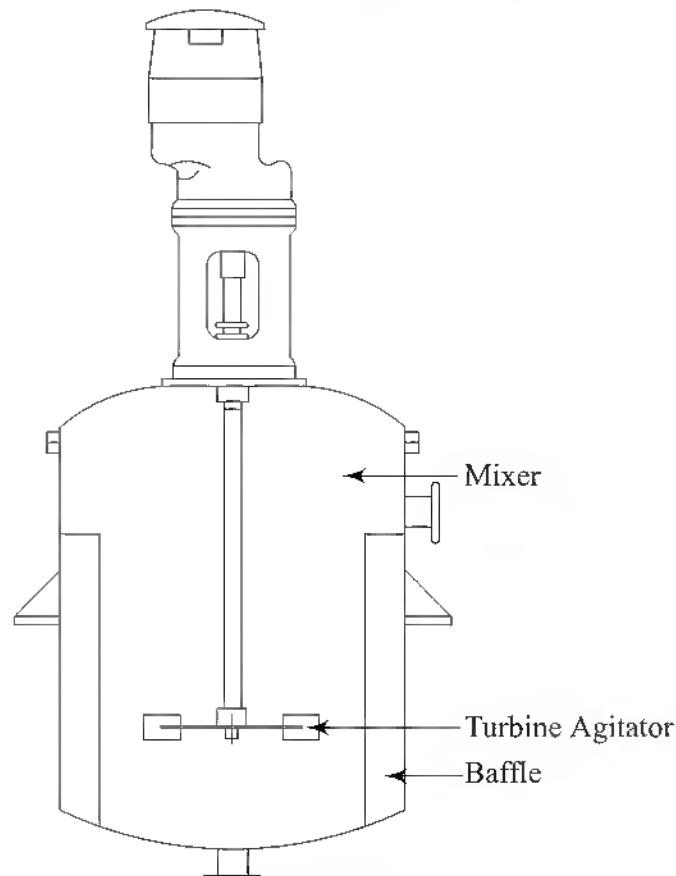


Fig. 7.14 Baffled Mixing Vessel

- (iii) Reliable scale-up.
 - (iv) Requires low head room.
 - (v) Low fixed cost.
 - (vi) Provides more flexibility stage wise.
- (b) These type of extractors have the following disadvantages over other types of extractors.
- (i) Require large hold up.
 - (ii) Require high power cost.
 - (iii) High solvent inventory.
 - (iv) Require large floor space, etc.

If number of theoretical stages required for desired separation is less than or equal to four, mixer-settler is probably the most economical among all types of extractors. For this application, turbines and propellers are used as agitators. Among all type of agitators, pitch blade turbine agitator [refer Fig. 10.2 (iv)] are found to be most effective for mixing of two immiscible liquids. For smaller agitated vessel mixer propellers are selected. For large mixers, turbines are selected.

7.6.3 Process Design of Mixer

It includes two steps:

- (i) Determining the volume of the mixer.
- (ii) Process design of agitator; e.g. deciding shape, factors and power requirement.
- (i) Mixer settlers are operated in batch wise mode and also in continuous manner. For batch mixer, volume of mixer is decided by the total volume of liquid mixture, charged to the mixer. For continuous mixer, volume of the mixer depends on hold up time.

Ideally, holdup time in a continuous mixer is the time required to reach equilibrium. It also depends on the rate of mass transfer. It can be decided on the basis of pilot plant or lab scale experiments of the given system.

(ii) Process Design of Agitator (Ref. 1 and 10)

The work of Miller and Mann and of Olney and Carlson have established that to calculate power required for agitating two liquid phases one can use the power data available for agitating single phase liquids, if density and viscosity of two phase liquid mixture are determined by following equations.

$$\rho_{av} = \rho_C Z_C + \rho_D Z_D \quad (7.22)$$

$$\mu_{av} = \frac{\mu_C}{Z_C} \left(1 + \frac{1.5 \mu_D Z_D}{\mu_D + \mu_C} \right) \quad (7.23)$$

where, ρ_{av} = Average density of two phase liquid mixture, kg/m^3

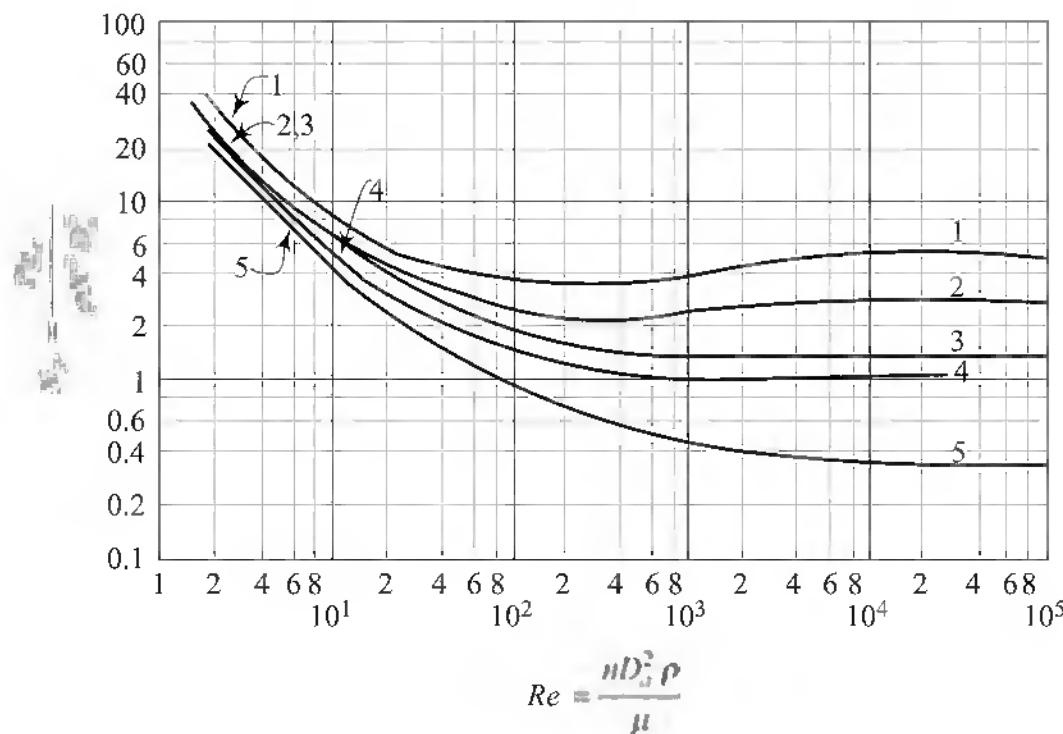
ρ_C = Density of continuous phase, kg/m^3

ρ_D = Density of dispersed phase, kg/m^3

Z = Volume fraction of appropriate phase

μ_C = Viscosity of continuous phase, cP or $\text{mPa} \cdot \text{s}$

μ_D = Viscosity of dispersed phase, cP or $\text{mPa} \cdot \text{s}$

**Fig. 7.15** Agitator Power Correlations³.

- Curve: 1 Six Flat Blade Turbine, $D_a/W_i = 5$, Four Baffles Each $D_T/12$, (Refer Fig. 10.2 (v))
 Curve: 2 Six Flat Blade Open Turbine, $D_a/W = 8$, Four Baffles Each Equal To $D_T/12$, (Refer Fig. 10.2 (ii))
 Curve: 3 45° Pitched Blade Turbine, $D_a/W_i = 8$, Four Baffles, Each $D_T/12$, (Refer Fig. 10.2 (iv)). But with six blades)
 Curve: 4 Propeller Pitch Equal To $2D_a$, Four Baffles, Each $D_T/12$, (Refer Fig. 10.2 (i))
 Curve: 5 Propeller Pitch Equal To D_a , Four Baffles, Each $D_T/10$, (Refer Fig. 10.2 (i))
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Dispersed phase is a liquid phase which forms the droplets and are dispersed in a continuum of the other. Other phase is called continuous phase. Ordinarily the liquid flowing at the smaller volume rate in the extractor will be dispersed in another. For batch operation, the liquid in which the impeller is immersed when at rest is usually continuous. Normally dispersed phase has higher viscosity and lower interfacial tension. In continuous extraction interchanging of dispersed phase and continuous phase is possible.

Power data for agitating single phase liquids in baffled vessel are available in the form of graph N_p vs Re [Fig. (7.15)]. The same can be used to calculate the power required for mixing two phase liquid mixture.

$$N_p = \frac{P g_c}{\rho_{av} n^3 D_a^5} \quad (7.24)$$

$$Re = \frac{n D_a^2 \rho}{\mu_{av}} \quad (7.25)$$

where, N_p = Power number
 Re = Reynolds number

P = Power required for agitation, W

g_c = Newton's law of gravitational constant, ($g_c = 1$ for SI system)

n = Impeller rotational speed, s^{-1}

D_a = Impeller diameter, m

ρ_{av}, μ_{av} = Density and viscosity of two phase liquid mixture, can be determined by Eqs (7.22) and (7.23), respectively.

7.6.4 Process Design of Settlers (Ref.: 3, 4, 11)

Settlers are also known as decanters. They are used to separate two immiscible liquids or two partially miscible liquids. The mixture of liquids leaving a mixer, is a cloudy dispersion which must be settled, coalesced and separated into its liquid phases. It requires sedimentation and coalescence of the drops of the dispersed phase. Decanters used are of the various shape as shown in Fig. (7.16). It can be horizontal or vertical.

Volume of settler is estimated by taking hold-up time equal to 5 to 10 min. Laboratory experiments for deciding hold-up time could prove valuable.

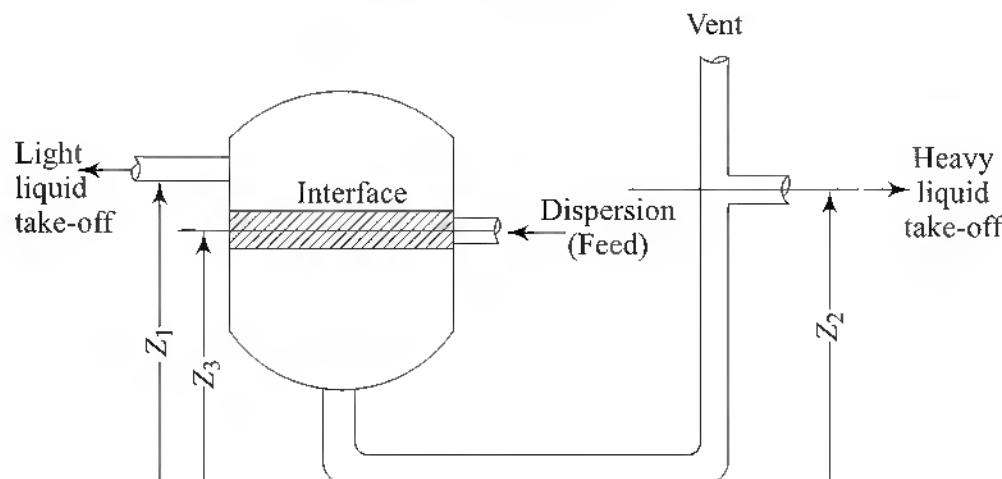
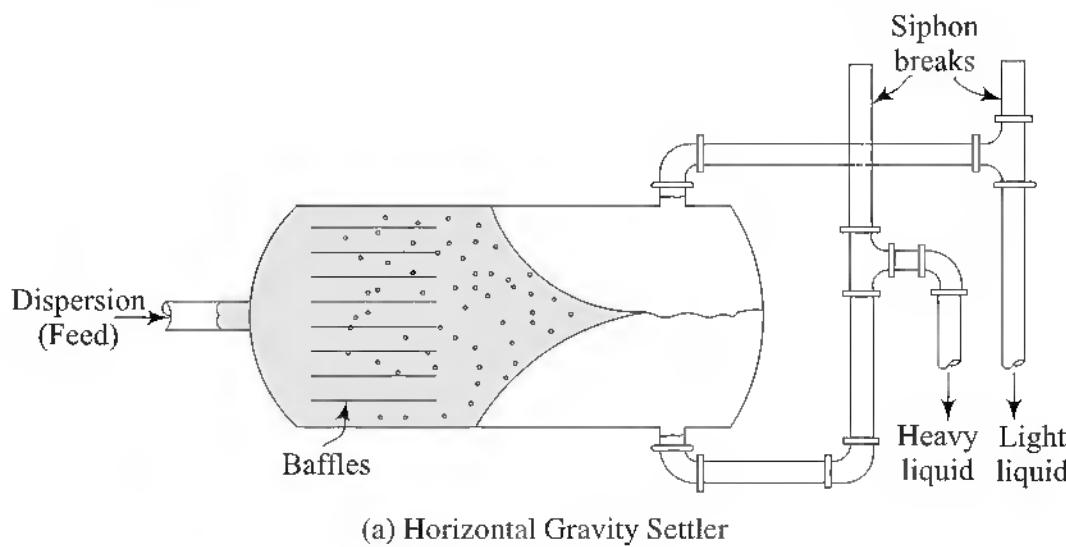


Fig. 7.16

Diameter or size of the decanter is decided on the basis of fact that velocity of continuous phase should be less than settling velocity of the droplets of dispersed phase. Velocity of continuous phase is calculated by equation;

$$u_c = \frac{L_c}{A_i} < u_d \quad (7.26)$$

where,

u_c = Velocity of continuous phase, m/s

L_c = Continuous phase volumetric flow rate, m^3/s

A_i = Area of interface, m^2

u_d = Settling velocity of the dispersed phase droplets, m/s

Stokes law is used to determine the settling velocity of the droplets.

$$u_d = \frac{d_d^2 g (\rho_d - \rho_c)}{18 \mu_c} \quad (7.27)$$

where,

d_d = Droplet diameter, m

u_d = Settling velocity of the dispersed phase droplet, m/s

g = Acceleration due to gravity = 9.81 m/s^2

ρ_c = Density of continuous phase, kg/m^3

ρ_d = Density of dispersed phase, kg/m^3

μ_c = Viscosity of continuous phase, $\text{N} \cdot \text{s}/\text{m}^2$ or $\text{kg}/(\text{m} \cdot \text{s})$

Droplet size (d_d) can be assumed equal to $150 \mu\text{m}$. In decanter, feed droplet sizes are normally well above $150 \mu\text{m}$. If the calculated settling velocity is greater than $4 \times 10^{-3} \text{ m/s}$, then a figure of $4 \times 10^{-3} \text{ m/s}$ is to be used.

For vertical decanter, take $L = 2 D_i$, thickness of dispersion band = $0.1 L$ and residence time of droplets in the dispersion band = $0.1 L / u_d$. The residence time of droplets in dispersion band should be 2 to 5 min.

For a horizontal cylindrical decanter, the interfacial area A_i depends on the position of the interface.

$$A_i = WL \quad (7.28)$$

$$W = 2 \sqrt{2rZ - Z^2} \quad (7.29)$$

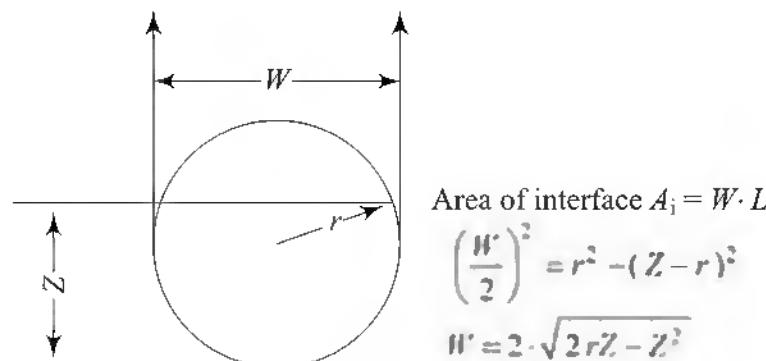


Fig. 7.17 Geometry of Horizontal Separator

where, W = Width of interface, m

Z = Height of the interface from the base of the vessel, m

L = Length of cylinder, m

r = Radius of cylinder, m

In the approximate method, for horizontal decanter,

$$\text{take } L/D_i = 4 \text{ and } D = 8.4 \sqrt{q_c + q_d} \quad (7.30)$$

where, D_i = Inside diameter, m

q_d, q_c = Volumetric flow rate, m^3/s (for horizontal decanter)

Example 7.6

For the extraction system described in Example 7.1, design a mixer-setter type extractor. Following data are obtained from pilot study.

- (i) Residence time (hold-up time) of liquid mixer in pilot plant mixer, $\theta = 6 \text{ min}$
- (ii) Stage efficiency = 0.92
- (iii) Type of agitator = 45° pitch blade turbine
- (iv) Shape factors of pilot plant mixer: $D_a/W_i = 8$, four baffles, $D_T/J = 12$, six blades, $H/D_T = 1$, $D_a/D_T = 0.33$
where, D_a = Diameter of agitator, m
 D_T = Inside diameter of mixing vessel, m
 J = Width of baffle, m
 H = Depth of liquid tank, m
 W_i = Width of agitator blade, m
- (v) Power required per unit volume in pilot plant = 220 W/m^3

Solution:

Treybal¹ demonstrated that for geometrically similar agitated vessels with equal holding time and equal power per unit volume on the two scales, the stage efficiency is likely to increase on scale-up. Based on this proposition, residence time in commercial scale mixer is 6 min and stage efficiency is 0.92.

Total number of mixer settler = $4.62/0.92 \approx 5$

Working volume of mixer = Hold up time \times Volumetric flow rate of liquid mixture

$$V_w = \theta \times q_v$$

$$q_v = (\text{mass flow rate of feed} + \text{mass flow rate of final extract})/\rho_{av}$$

$$\rho_{av} = \rho_c Z_C + \rho_D Z_D$$

$$\rho_{MIBK} = 801 \text{ kg/m}^3, \rho_{water} = 1000 \text{ kg/m}^3, \rho_{Acetic\ acid} = 1049 \text{ kg/m}^3$$

This mixer-settler will be operated in continuous manner. If 1st mixer is considered, then extract phase (MIBK rich phase) is the lighter phase and feed (water rich phase) is the heavier phase.

$$\rho_F = \frac{1}{\left(\frac{w_1}{\rho_1} + \frac{w_2}{\rho_2} \right)} \quad (7.31)$$

where w_1, w_2 = Mass fractions,

ρ_1, ρ_2 = Densities of components

$$\rho_F = \frac{1}{\left(\frac{0.2}{1049} + \frac{0.8}{1000} \right)} = 1009.4 \text{ kg/m}^3$$

$$\rho_E = \frac{1}{\left(\frac{0.088}{1049} + \frac{0.892}{801} + \frac{0.02}{1000} \right)} = 821.36 \text{ kg/m}^3$$

Density of extract, $\rho_E = 821.36 \text{ kg/m}^3$

Volumetric flow rate of feed = $200/1009.4 = 0.198 \text{ m}^3/\text{h}$

Volumetric flow rate of final extract = $441.03/826.36 = 0.5337 \text{ m}^3/\text{h}$

Final extract is having higher volumetric flow rate than feed. Hence, feed or water-rich layer is the dispersed phase and final extract (solvent rich layer) is the continuous phase.

$$Z_d = \frac{200/1009.4}{\left(\frac{200}{1009.4} + \frac{441.03}{821.36} \right)} = 0.27$$

$$Z_C = 1 - Z_d = 0.73$$

$$\rho_{av} = \rho_C Z_C + \rho_D Z_D = 821.36 \times 0.73 + 1009.4 \times 0.27 = 872.13 \text{ kg/m}^3$$

$$q_v = (200 + 441.03) / 872.13 = 0.735 \text{ m}^3/\text{h}$$

$$\frac{H}{D_T} = 1, \quad V_w = 0.735 \frac{6}{60} = 0.0735 \text{ m}^3$$

$$\frac{\pi}{4} D_T^2 H = 0.0735$$

$$\frac{\pi}{4} D_T^3 = 0.0735 \text{ m}^3$$

$$D_T = 0.454 \text{ m}$$

For 45° pitch blade turbine, $D_a/D_T = 0.33$, where D_a = diameter of agitator, m

$$D_a = 0.1498 \text{ m}$$

Power required per unit volume = 220 W/m^3

$$P = 220 \times 0.0735 = 16.17 \text{ W}$$

For 1st trial calculations, let tip velocity of agitator, $v = 200 \text{ m/min} = \pi D_a n$

$$\pi \times 0.1498 \times n = 200$$

$$n = 424.98 \text{ rpm} \approx 425 \text{ rpm}$$

For pure components $\mu_{\text{MIBK}} = 0.5 \text{ mPa} \cdot \text{s}$, $\mu_{\text{water}} = 0.8 \text{ mPa} \cdot \text{s}$, $\mu_{\text{Acetic acid}} = 1.1 \text{ mPa} \cdot \text{s}$

$$\frac{1}{\mu_m} = \frac{w_1}{\mu_1} + \frac{w_2}{\mu_2} + \frac{w_3}{\mu_3}$$

$$\frac{1}{\mu_c} = \frac{0.088}{1.1} + \frac{0.02}{0.8} + \frac{0.892}{0.5} = 1.889$$

$$\mu_c = 0.5294 \text{ mPa} \cdot \text{s}$$

$$\frac{1}{\mu_d} = \frac{w_{AA}}{\mu_{AA}} + \frac{w_{\text{water}}}{\mu_{\text{water}}} = \frac{0.2}{1.1} + \frac{0.8}{0.8} = 1.1818$$

$$\mu_d = 0.846 \text{ mPa} \cdot \text{s}$$

$$\mu_{av} = \frac{0.5294}{0.73} \left(1 + \frac{1.5 \times 0.27 \times 0.846}{(0.846 + 0.5294)} \right) = 0.9059 \text{ mPa} \cdot \text{s}$$

$$Re = \frac{n D_a^2 \rho_{av}}{\mu_{av}} = \frac{(425/60) \times 0.1498^2 \times 872.13}{0.9059 \times 10^{-3}} = 153\,025$$

$$N_p = 1.5 \quad [\text{From Fig. (7.15)}]$$

$$N_p = \frac{P g_c}{\rho_{av} n^3 D_a^5} = 1.5$$

$$P = \frac{1.5 \times 872.13 \times \left(\frac{425}{60}\right)^3 \times 0.1498^5}{1}$$

$$P = 35 \text{ W} >> 16.17 \text{ W}$$

Hence, decrease the value of tip velocity to match this value.

Let for second trial calculation, tip velocity = 155 m/min

$$n = \frac{155}{(\pi \times 0.1498)} = 329.36 \text{ rpm} \approx 329 \text{ rpm}$$

$$Re = 153\,025 \times \frac{329}{425} = 118\,459$$

$$N_p = 1.5 \quad (\text{From Fig. 7.15})$$

$$P = \frac{1.5 \times 872.13 \times \left(\frac{329}{60}\right)^3 \times 0.1498^5}{1} = 16.27 \text{ W}$$

This is close to 16.17 W.

Use 45° pitch blade turbine, having diameter $D_a = 0.1498 \text{ m}$ and rotational speed, $n = 330 \text{ rpm}$.

(b) Process design of settler:

(i) Let position of settler be horizontal.

Approximate diameter of horizontal settler

$$D = 8.4 \sqrt{q_c + q_d} \quad (7.30)$$

$$q_c = \frac{441.03/3600}{826.36} = 1.4825 \times 10^{-4} \text{ m}^3/\text{s}$$

$$q_d = \frac{200/3600}{1009.4} = 5.50382 \times 10^{-5} \text{ m}^3/\text{s}$$

$$D = 8.4 \sqrt{q_c + q_d} = 0.1197 \approx 0.12 \text{ m}$$

This equation is valid for $L/D_i = 4$.

Length of horizontal decanter $L = 0.48$ m

D and L by other method:

Settling velocity of the droplets of dispersed phase

$$u_d = \frac{d_d^2 g (\rho_d - \rho_c)}{18 \mu_c} \quad (7.27)$$

$$\begin{aligned} d_d &= 150 \times 10^{-6} \text{ m}, \quad g = 9.8 \text{ m/s}^2, \quad \rho_d = 1009.4 \text{ kg/m}^3 \\ \rho_c &= 821.36 \text{ kg/m}^3, \quad \mu_c = 0.5294 \text{ mPa} \cdot \text{s} \end{aligned}$$

$$\begin{aligned} u_d &= \frac{(150 \times 10^{-6})^2 \times 9.81 \times (1009.4 - 821.36)}{18 \times 0.5294 \times 10^{-3}} \\ &= 0.004355 \text{ m/s} \end{aligned}$$

$$A_{\min} = \frac{L_c}{u_d} = \frac{q_c}{u_d} = \frac{(0.5337/3600)}{0.004355} = 0.034 \text{ m}^2$$

$$A_{\min} = (W_i L)_{\min}$$

If the position of interface is fixed at the centre line of settler then $W_i = D_i$, let $L = 4D_i$.

$$A_{\min} = 4 D_i^2 = 0.034 \text{ m}^2$$

$$D_{i\min} = 0.0922 \text{ m} < D_i = 0.12 \text{ m}, \quad L = 0.48 \text{ m}$$

To fix the position of interface at centre line of decanter $Z_i = D_i/2$, locations of outlet nozzles can be determined based on the pressure balance.

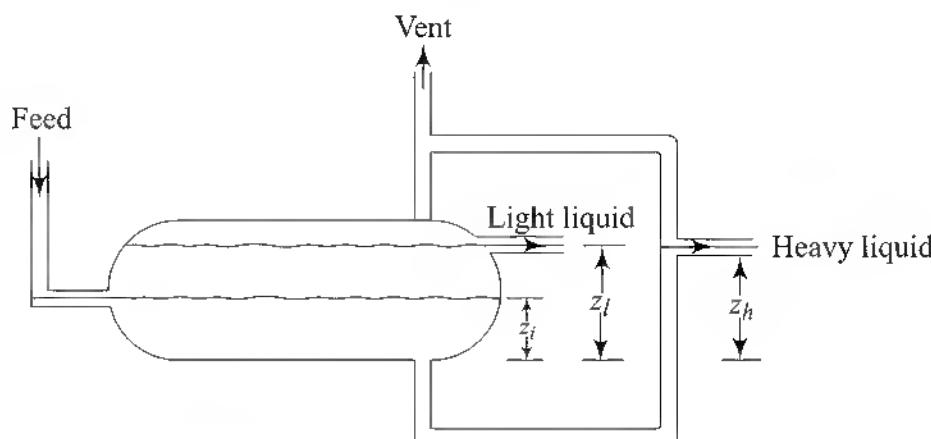


Fig. 7.18 Location of Nozzles for Horizontal Decanter

Pressure balance:

$$(z_h - z_i) \rho_h = (z_l - z_i) \rho_L$$

$$\rho_h = \text{density of heavier phase} = 1009.4 \text{ kg/m}^3$$

$$\rho_L = \text{density of lighter phase} = 821.36 \text{ kg/m}^3$$

$$z_i = D_i/2 = 0.12/2 = 0.06 \text{ m}$$

Let $z_l = 0.8 D_i = 0.8 \times 0.12 \text{ m} = 0.096 \text{ m}$

$$(z_h - 0.06) \times 1009.4 = (0.096 - 0.06) \times 821.36$$

$$z_h = 0.0893 \text{ m}$$

(b) Vertical decanter (other option)

$$A_{i\min} = (\pi/4) D_{i\min}^2 = 0.034 \text{ m}^2$$

$$D_{i\min} = 0.208 \text{ m}$$

Let $D_i = 0.25 \text{ m}$ $L = 2 \times 0.25 = 0.5 \text{ m}$

Width of dispersion bend = 0.1 $L = 0.05 \text{ m}$

Residence time of the droplets of dispersed phase in dispersion band,

$$\theta = \frac{0.05}{u_d} = \frac{0.05}{0.004355} = 11.481 \text{ s}$$

As per the guidelines, this should be at least 120 s. For 120 second residence time, $L = 5.226 \text{ m}$ (very high). This guide line is given for higher flow rates (1500 kg/h to 600 kg/h).

Hence, let $D_i = 0.25 \text{ m}$ and $L = 1 \text{ m}$ which means $\theta = 23 \text{ seconds}$.

To fix the interface at the centre of vertical decanter, locations of nozzles must be determined.

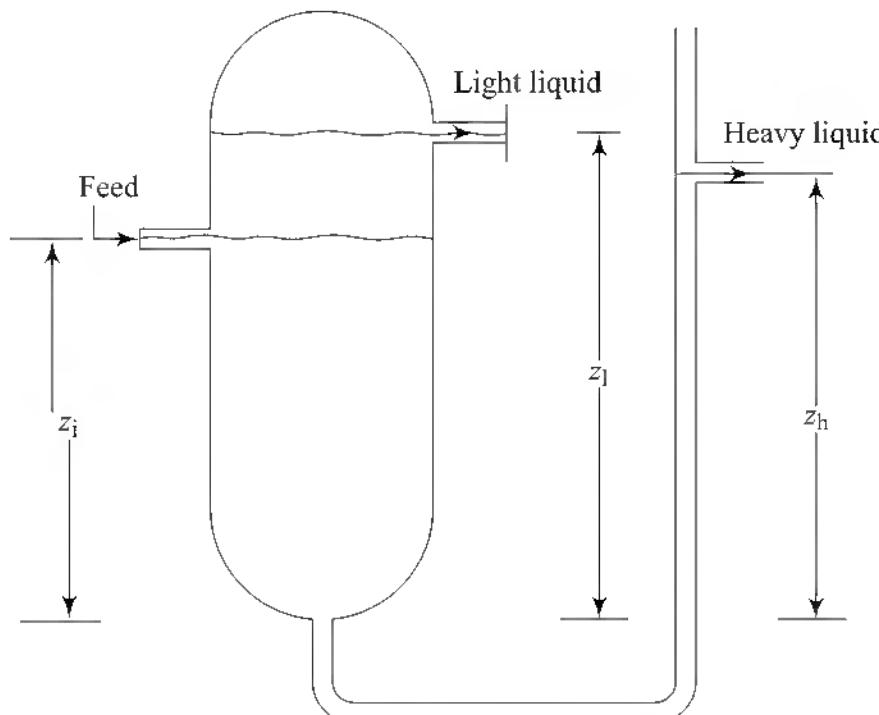


Fig. 7.19 Location of Nozzles for Vertical Decanter

Pressure balance:

$$(z_l - z_i) \rho_L g + Z_i \rho_h g = z_h \rho_h g$$

$$z_h = (z_l - z_i) \frac{\rho_L}{\rho_h} + z_i$$

$$z_i = 0.5 \text{ m}, \quad z_l = 0.9 \text{ m} \quad L = 0.9 \text{ m}$$

$$z_h = (0.9 - 0.5) \times \frac{821.36}{1009.4} + 0.5 = 0.8255 \text{ m}$$

Two criteria may be used for scale-up of agitated vessel; (a) constant tip velocity and (b) constant power per unit volume.

Criteria of constant tip velocity is used for miscible liquids where heat is transferred from coil or jacket or where suspended solids are involved.

Criteria of constant power per unit volume can be used for immiscible liquid systems gas-liquid systems, emulsions and pastes.

7.6.5 Process Design of Packed Tower Type Extractor (Ref.: 3, 6, 7, 8)

In packed tower type extractor, standard commercial packings, used in vapour-liquid system, are used. This includes structured packings, Raschig and Pall rings, Berl and Intalox saddles and other random packings. Packing reduces the vertical back mixing of the continuous phase and thereby reduces height required for mass transfer. It is recommended to redistribute the dispersed phase at about every 1.5 to 3 m distance to generate new droplets. Size of packing should preferably be less than one-eighth of the tower diameter to minimize the wall effect. Flooding velocity in packed tower is the velocity of dispersed phase or continuous phase at which dispersed phase is coming out from the outlet pipe of continuous phase or vice versa.

(a) Tower diameter: Diameter of packed tower is determined based on value of flooding velocity. To find the flooding velocity in packed tower many correlations are available. One of the best correlations; Crawford-Wilke correlation, is represented in Fig. 7.20.

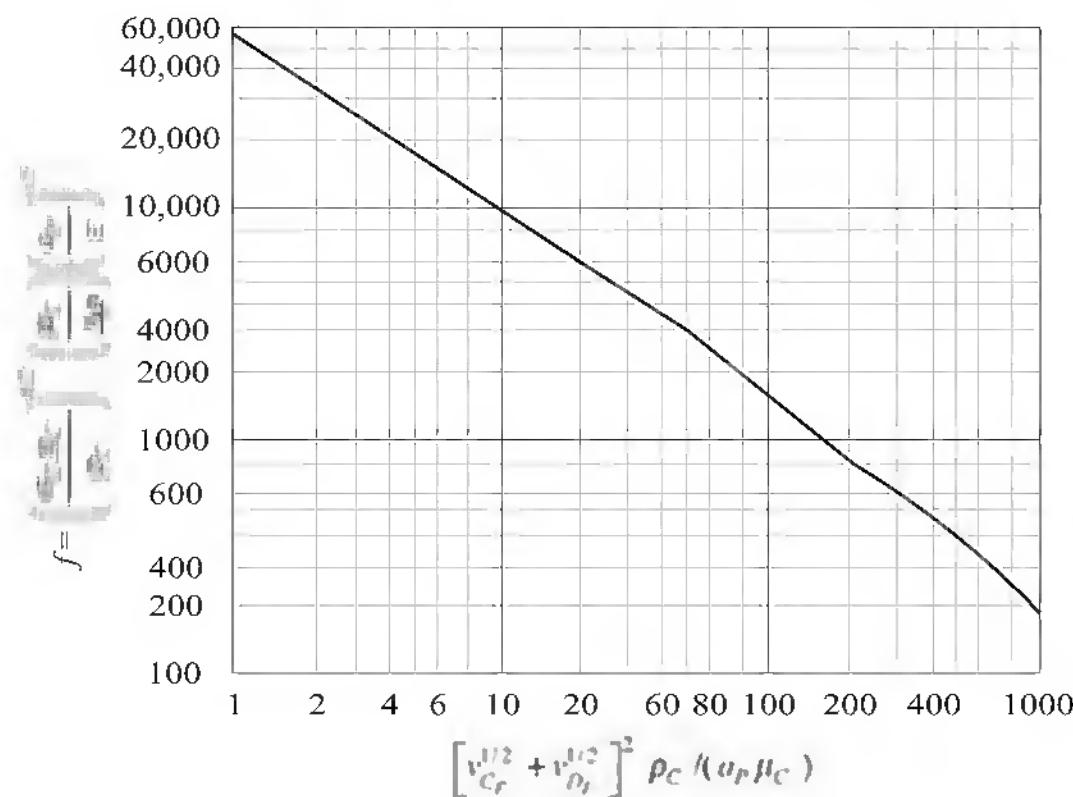


Fig. 7.20 Flooding in Packed Towers, Crawford-Wilke Correlation³
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where, σ_c = Interfacial tension of continuous phase, lbf/ft
 g_c = Newton's law gravitational constant
 $= 4.18 \times 10^8$ (lb · ft)/(lbf · h²)

ρ_c = Density of continuous phase, lb/ft³

μ_c = Viscosity of continuous phase, lb/(ft · h)

$\Delta\rho$ = Difference in density, lb/ft³

a_p = Specific packing surface, ft²/ft³

ϵ = Fraction void volume in packed section

v_{cF}, v_{DF} = Superficial flooding velocity, ft/h

Actual velocity of the continuous phase and the same of the dispersed phase should be less than or equal to 50% of the flooding values.

- (b) Height of packed tower: Height of packed tower H is given by equation.

$$H = N_{toR} \cdot H_{toR} = N_{toE} \cdot H_{toE} \quad (7.32)$$

Value of H_{toR} or H_{toE} must be decided by actual experiments at pilot plant scale. Value of H_{toR} in packed tower depends on type of system, type of packings, size of packings, tower diameter, etc. Hence it is difficult to find the suitable correlation for finding H_{toR} for the given system with the given type and size of packing and given diameter of packed tower.

For example, equation for finding H_{toR} for acetic acid-water-MIBK system, in which water rich phase is the raffinate phase and MIBK rich phase is the extract phase, is given by equation¹.

$$H_{toR} = 0.2347 \left(\frac{v_C}{v_D} \right)^{0.648} \quad (7.33)$$

where, H_{toR} = Height of equivalent overall transfer unit based on raffinate phase, m

v_D = Actual superficial velocity of dispersed phased, m/s

v_C = Actual superficial velocity of continuous phase, m/s

This equation was derived by Sherwood *et al*¹ for the packed tower having 0.09 m diameter, 12.7 mm carbon rings at 25°C for the certain range of flow rates. Hence, the use of this equation for the same system with larger diameter packed tower, different type and size of packing and for different values of flow rates, may not be reliable. So the equation of H_{toR} or H_{toE} must be determined on the same system at pilot plant scale with similar type of equipment and with similar conditions like same values of superficial velocities, same operating temperature, etc. However, Eq. (7.33) may be used for the preliminary design of the pilot equipment.

- (c) **Criteria of Selection:** If number of theoretical stages required for desired separation is less and limited floor area is available in the plant than packed tower can be selected as extractor. Packed tower consumes much less power than mixer settler.

Example 7.7

For the extraction system described in Example 7.1, design the packed tower type extractor.

- (a) Tower Diameter:

Let type of packing: SS Pall rings

Size: 16 mm (for 1st trial calculations)

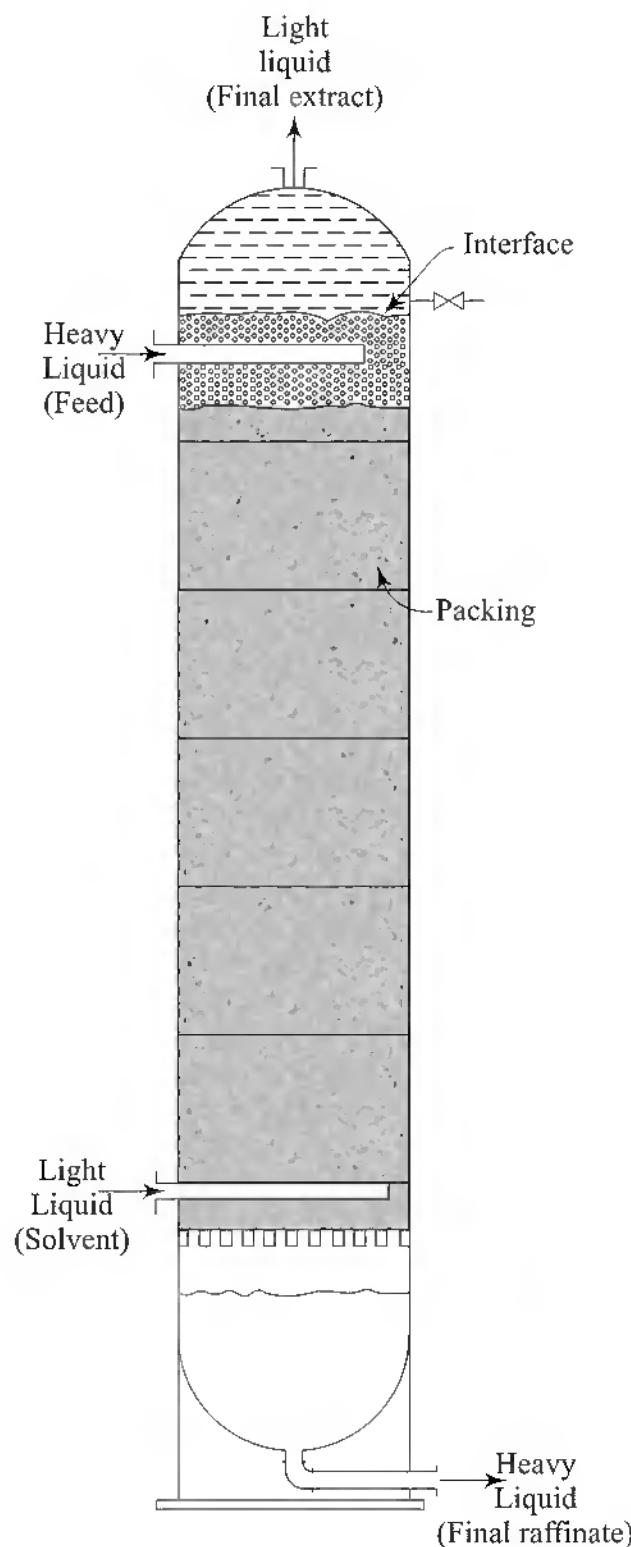


Fig. 7.21 Packed Extraction Tower, Light Liquid Dispersed.

From Table 9.2 of Chapter 9,

$$a_p = \text{specific packing surface} = 340 \text{ m}^2/\text{m}^3$$

$$\epsilon = \text{fraction of void volume} = 0.92$$

To find the flooding velocities, first the value of following function must be determined.

$$f = \left(\frac{\sigma_C g_C}{\rho_C} \right)^{0.2} \left(\frac{\mu_C}{\Delta \rho} \right) \left(\frac{a_p}{\epsilon} \right)^{1.5} \quad (7.34)$$

where, σ_C = Surface tension of continuous phase, lbf/ft

From Example 7.6, volumetric flow rate of feed = $0.198 \text{ m}^3/\text{h}$

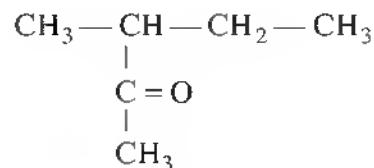
Volumetric flow rate of final extract = $0.5337 \text{ m}^3/\text{h}$.

Volumetric flow rate of extract is very much greater than the same of feed, hence extract is the continuous phase and feed is the dispersed phase.

Final extract contains 89.2% MIBK, 8.8% acetic acid and 2% water by mass.

Surface Tension of MIBK:

Structure of MIBK:



(Molar mass = $12 \times 6 + 16 + 12 = 100$)

For hydrogen bonded compound, Sugden equation can be used to predict surface tension.

$$\sigma^{1/4} = [\mathcal{P}] (\rho_L - \rho_G)$$

where,

σ = Surface tension, dyn/cm

ρ_L = Liquid density, mol/cm³

$[\mathcal{P}]$ = Temperature independent parachor value

For MIBK,

$$\rho_L = 801 \text{ kg/m}^3$$

$$\rho_G = \frac{pM}{RT} = \frac{1 \times 100}{(273 + 30)} \times \frac{273}{1 \times 22.414}$$

$$\rho_G = 4 \text{ kg/m}^3$$

$$\rho_L = 801 \times \frac{10^3}{1} \times \frac{1}{10^6} \times \frac{1}{100} = 0.008 \text{ mol/cm}^3$$

$$\rho_G = 4 \times \frac{10^3}{1} \times \frac{1}{10^6} \times \frac{1}{100} = 4 \times 10^{-5} \text{ mol/cm}^3$$

From Table 3.343, Ref. 3,

$$[\mathcal{P}] = 171.9 + 55.5 + 29 = 256.4$$

$$\sigma^{1/4} = 256.4 (0.008 - 4 \times 10^{-5}) = 2.0409$$

$$\sigma = 17.35 \text{ dyn/cm}$$

For acetic acid,

$$\rho_L = 1049 \text{ kg/m}^3$$

$$\rho_G = \frac{pM}{RT} = \frac{1 \times 60}{(273 + 30)} \times \frac{273}{1 \times 22.414} = 2.41 \text{ kg/m}^3$$

$$\rho_L = 1049 \times \frac{10^3}{1} \times \frac{1}{10^6} \times \frac{1}{60} = 0.01748 \text{ mol/cm}^3$$

$$\rho_G = 2.41 \times \frac{10^3}{1} \times \frac{1}{10^6} \times \frac{1}{60} = 4.016 \times 10^{-5} \text{ mol/cm}^3$$

$$\begin{aligned} [\mathcal{P}] &= 55.5 + 73.8 = 129.3 \\ \sigma^{1/4} &= 129.3 (0.01748 - 4.016 \times 10^{-5}) \\ \sigma &= 25.86 \text{ dyn/cm} \end{aligned}$$

For water, $\sigma = 71.4 \text{ dyn/cm}$

Surface tension of liquid mixture is given by following:

$$\sigma_m^{1/4} = \sum \sigma_i^{1/4} x_i \quad (7.35)$$

where x_i = mole fraction of component i in liquid phase

$$x_{\text{MIBK}} = \frac{0.892/100}{\left(\frac{0.088}{60} + \frac{0.892}{100} + \frac{0.02}{18} \right)} = 0.7758$$

$$x_{\text{AcOH}} = \frac{0.088/60}{\left(\frac{0.088}{60} + \frac{0.892}{100} + \frac{0.02}{18} \right)} = 0.1276$$

$$x_{\text{H}_2\text{O}} = 0.0966$$

$$\sigma_c^{1/4} = 17.35^{1/4} \times 0.7758 + 25.86^{1/4} \times 0.1276 + 71.4^{1/4} \times 0.0966$$

$$\begin{aligned} \sigma_c &= 21.443 \text{ dyn/cm} \\ &\equiv 0.021443 \text{ N/m} \end{aligned}$$

From Example 7.6:

$$\rho_c = 821.36 \text{ kg/m}^3, \mu_c = 0.5294 \times 10^{-3} \text{ kg/(m} \cdot \text{s)}$$

$$g_c = 1, \Delta\rho = \rho_d - \rho_c = 1009.4 - 821.36 = 188.04 \text{ kg/m}^3$$

To use Fig. 7.20 (Crawford – Wilke correlation) all variables must be in US customary units.

$$\sigma_c = 0.021443 \times \frac{1}{4.44822} \times \frac{1}{3.28} = 1.47 \times 10^{-3} \text{ lbf/ft}$$

$$g_c = 4.18 \times 10^8 \text{ lb} \cdot \text{ft}/(\text{lbf} \cdot \text{h}^2)$$

$$\rho_c = 821.36 \times \frac{1}{1.601846 \times 10} = 51.276 \text{ lb/ft}^3$$

$$\mu_c = 0.5294 \times 10^{-3} \times \frac{1}{4.133789 \times 10^{-4}} = 1.28 \text{ lb/(ft} \cdot \text{h)}$$

$$\Delta\rho = 188.04 \times \frac{1}{16.01846} = 11.739 \text{ lb/ft}^3$$

$$a_p = 340 \text{ m}^2/\text{m}^3 = 340 \times \frac{0.3048}{1} = 103.632 \text{ ft}^2/\text{ft}^3$$

$$\epsilon = 0.92$$

$$f = \left(\frac{1.47 \times 10^{-3} \times 4.18 \times 10^8}{51.276} \right)^{0.2} \left(\frac{1.28}{11.739} \right) \left(\frac{103.632}{0.92} \right)^{1.5} = 852.81$$

From Fig. 7.20 and from Example 7.6,

$$(v_{CF}^{1/2} + v_{DF}^{1/2})^2 \rho_C / (a_p \mu_C) = 175$$

$$(v_{CF}^{1/2} + v_{DF}^{1/2})^2 = \frac{175 a_p \mu_C}{\rho_C} = \frac{175 \times 103.632 \times 1.28}{51.276} = 452.72 \text{ ft/h}$$

$$q_c = 1.4825 \times 10^{-4} \text{ m}^3/\text{s}$$

$$q_d = 5.50382 \times 10^{-5} \text{ m}^3/\text{s}$$

$$\frac{v_{CF}}{v_{DF}} = \frac{1.4825 \times 10^{-4}}{5.50382 \times 10^{-5}} = 2.6936$$

$$((2.6936 v_{DF})^{1/2} + v_{DF}^{1/2})^2 = 452.72$$

$$(2.6412 v_{DF}^{1/2})^2 = 452.72 \times \frac{1}{3600} \times \frac{0.3048}{1} = 0.03833$$

$$(2.6412)^2 v_{DF} = 0.03833$$

$$v_{DF} = 0.0055 \text{ m/s} \text{ and } v_{CF} = 0.0148 \text{ m/s}$$

Let actual velocities be

$$v_C = 0.5 v_{CF} \text{ and } v_D = 0.5 v_{DF}$$

$$v_D = 0.00275 \text{ m/s}, v_C = 0.0074 \text{ m/s}$$

$$v_C = \frac{q_c}{(\pi/4) D_i^2}$$

$$\frac{\pi}{4} D_i^2 = \frac{1.4825 \times 10^{-4}}{0.0074} = 0.02 \text{ m}^2$$

$$D_i = 0.16 \text{ m}$$

Let $D_i = 0.2 \text{ m}$

Revised values of actual velocities based on final value of inside diameter.

$$v_C = 0.0047 \text{ m/s}, v_D = 0.00175 \text{ m/s}$$

Height of packing:

Total height of packing $H = N_{toR} \cdot H_{toR}$

$$N_{toR} = 7.21355 \quad (\text{From Example 7.3})$$

H_{toR} for the given system

$$H_{toR} = 0.2347 (v_C/v_D)^{0.648}$$

Where H_{toR} is in m.

$$H_{toR} = 0.2347 \left(\frac{0.0047}{0.00175} \right)^{0.648} = 0.4452 \text{ m}$$

$$H = 7.21355 \times 0.4452 = 3.211 \text{ m}$$

Results: Type of packing: 16 mm, SS Pall rings, inside diameter of tower: 0.2 m, total height of packing section = 3.2 m

7.6.5.1 How to Find H_{toR} or H_{toE} from Experiment (Ref. 11, 12)

To find the H_{toR} from the experiment at pilot plant or at lab scale, one can use the following equation for the packed tower type extractor.

$$H_{toR} = \frac{\text{Superficial velocity of raffinate}}{K_{Ra}} = \frac{V_R}{K_{Ra}} \quad (7.36)$$

where, K_{Ra} = Overall mass transfer coefficient based on raffinate phase

$$K_{Ra} = \frac{\text{kmol of solute transferred/second}}{\text{volume of packing} \times \Delta C_{1m}}$$

ΔC_{1m} = Logarithmic mean driving force

$$\text{Similarly, } H_{toE} = \frac{V_E}{K_{Ea}} \quad (7.37)$$

$$K_{Ea} = \frac{\text{kmol of solute transferred/second}}{\text{volume of packing} \times \Delta C_{1m}}$$

Example 7.8

In the extraction of acetic acid from an aqueous solution by benzene in a pilot plant scale packed tower type extractor, following data were measured.

- (i) Cross sectional area of tower = 0.0045 m²
- (ii) Height of packing = 1.4 m
- (iii) Concentration of acetic acid in feed = 0.69 kmol/m³
- (iv) Concentration of acetic in final raffinate = 0.684 kmol/m³
- (v) Flow rate of extract phase = 5.6×10^{-6} m³/s
- (vi) Concentration of acetic acid in solvent = 0.004 kmol/m³
- (vii) Concentration of acetic acid in final extract = 0.0115 kmol/m³

Determine H_{toE} from above data.

Equilibrium relationship for this system¹² is $C_B^* = 0.0247 C_w$,

where C_B^* , C_w = concentration of acid, kmol/m³

Solution:

Total acetic acid transferred to extract (benzene) phase

$$\begin{aligned} &= q_{VE} (C_{B2} - C_{B1}) \\ &= 5.6 \times 10^{-6} (0.0115 - 0.004) = 4.2 \times 10^{-8} \text{ kmol/s} \end{aligned}$$

Equilibrium relationship $C_B^* = 0.0247 C_w$

where, C_B^* = Concentration of acid in benzene phase, kmol/m³

C_w = Concentration of acid in water phase, kmol/m³

Equilibrium concentration of acetic acid in benzene phase at inlet and outlet,

$$C_{B1}^* = 0.0247 \times 0.684 = 0.0169 \text{ kmol/m}^3$$

$$C_{B2}^* = 0.0247 \times 0.69 = 0.0171 \text{ kmol/m}^3$$

Driving force at bottom, $\Delta C_1 = C_{B1}^* - C_{B1} = 0.0169 - 0.004 = 0.0129 \text{ kmol/m}^3$

Driving force at top, $\Delta C_2 = C_{B2}^* - C_{B2} = 0.0171 - 0.0115 = 0.0056 \text{ kmol/m}^3$

Logarithmic mean driving force

$$\Delta C_{lm} = \frac{\Delta C_1 - \Delta C_2}{\ln\left(\frac{\Delta C_1}{\Delta C_2}\right)} = \frac{0.0129 - 0.0056}{\ln\left(\frac{0.0129}{0.0056}\right)}$$

$$\Delta C_{lm} = 0.008748 \text{ kmol/m}^3$$

$$H_{toE} = \frac{\text{Superficial velocity of extract phase}}{\text{Overall mass transfer coefficient based on extract}}$$

$$H_{toE} = \frac{v_E}{K_{Ea}} \quad (7.37)$$

$$v_E = \frac{\text{Volumetric flow rate of extract}}{\text{Cross sectional area}} = \frac{5.6 \times 10^{-6}}{0.0045}$$

$$= 1.244 \times 10^{-3} \text{ m/s}$$

$$K_{Ea} = \frac{\text{kmol transferred}}{(\text{Volume of packing} \times \Delta C_{lm})}$$

$$K_{Ea} = \frac{4.2 \times 10^{-8}}{(1.4 \times 0.0045 \times 0.008748)} = 7.62 \times 10^{-4} \text{ s}^{-1}$$

$$H_{toE} = \frac{1.244 \times 10^{-3}}{7.62 \times 10^{-4}} = 1.6325 \text{ m}$$

Example 7.9

For the extraction system described in Examples 7.4 and 7.5, design the packed tower type extractor.

Solution:

Feed flow rate $F = 1000 \text{ kg/h}$. It contains 80% DMF and 20% water. At steady state it is contacted with final extract E_1 .

$$E_1 = B(1 + \gamma'_1)$$

$$E'_1 = 3070.72(1 + 0.0625) = 3262.64 \text{ kg/h}$$

$$\gamma'_1 = \frac{\gamma'_1}{1 + \gamma'_1} = \frac{0.0625}{1 + 0.0625} = 0.0588$$

$$\rho_F = \frac{1}{\left(\frac{w_1}{\rho_1} + \frac{w_2}{\rho_2}\right)}$$

$$\rho_{\text{DMF}} = 944.5 \text{ kg/m}^3, \quad \rho_{\text{water}} = 1000 \text{ kg/m}^3$$

$$\rho_F = \frac{1}{\left(\frac{0.8}{944.5} + \frac{0.2}{1000} \right)} = 955.1 \text{ kg/m}^3$$

$$\rho_{\text{Extract}} = \frac{1}{\left(\frac{w_1}{\rho_1} + \frac{w_2}{\rho_2} \right)}$$

$$\rho_{\text{CH}_2\text{Cl}_2} = 1336 \text{ kg/m}^3$$

$$\rho_{\text{DMF}} = 944 \text{ kg/m}^3$$

$$\rho_E = \frac{1}{\left(\frac{0.0588}{944.5} + \frac{(1-0.0588)}{1336} \right)} = 1304.2 \text{ kg/m}^3$$

$$q_{VF} = \frac{F}{\rho_F} = \frac{1000}{955.1} = 1.047 \text{ m}^3/\text{h}$$

$$q_{VE} = \frac{E_1}{\rho_E} = \frac{3262.64}{1304.2} = 2.502 \text{ m}^3/\text{h}$$

Volumetric flow rate of extract phase is greater than volumetric flow rate of feed. Hence, extract phase is continuous phase and feed is dispersed phase.

Tower Diameter:

Let type of packing: SS pall rings, size = 25 mm

a_p = surface area of packing = $205 \text{ m}^2/\text{m}^3$

ϵ = fraction of void volume = 0.94

To find the flooding velocities by Crawford – Wilke correlation, first the following function must be determined.

$$f = \left(\frac{\sigma_c g_c}{\rho_c} \right)^{0.2} \left(\frac{\mu_c}{\Delta\rho} \right) \left(\frac{a_p}{\epsilon} \right)^{1.5} \quad (7.34)$$

$$\sigma_c^{1/4} = \sum x_i \sigma_i^{1/4} \quad (7.35)$$

$$\sigma_{\text{DMF}} = 35.2 \text{ dyn/cm}$$

Molar mass of DMF, $M = 73.09$

$\rho_{\text{CH}_2\text{Cl}_2}$:

$$\sigma^{1/4} = [P] (\rho_L - \rho_G) \quad (\text{Sudgen's equation})$$

ρ_L, ρ_G in mol/cm³

$$[P] = 40 + 55.2 \times 2 = 150.4$$

(Table 3–343, of Ref. 3)

$$\rho_L = 1336 \text{ kg/m}^3$$

$$\rho_G = \frac{pM}{RT} = \frac{1 \times 85}{(273 + 25)} \times \frac{273}{1 \times 22.414} = 3.474 \text{ kg/m}^3$$

$$\rho_L = 1336 \times \frac{10^3}{1} \times \frac{1}{10^6} \times \frac{1}{85} = 0.0157 \text{ mol/cm}^3$$

$$\rho_G = 3.474 \times \frac{10^3}{1} \times \frac{1}{10^6} \times \frac{1}{85} = 4.087 \times 10^{-5} \text{ mol/cm}^3$$

$$\sigma^{1/4} = 150.4 (0.0157 - 4.087 \times 10^{-5}) = 2.355$$

$$\sigma = 30.765 \text{ dyn/cm}$$

Mole fraction of DMF in extract phase,

$$x_1 = \frac{0.0588 / 73.09}{\left(\frac{0.0588}{73.09}\right) + \left(\frac{1 - 0.0588}{85}\right)} = 0.0677$$

$$\sigma^{1/4} = x_1 \sigma_{\text{DMF}}^{1/4} + (1 - x_1) \sigma_{\text{CH}_2\text{Cl}_2}^{1/4}$$

$$\sigma_c^{1/4} = 0.0677 \times 35.2^{1/4} + (1 - 0.0677) \times 30.765^{1/4}$$

$$\sigma_C = 31.05 \text{ dyn/cm} \equiv 0.031 \text{ N/m}$$

μ_c :

$$\frac{1}{\mu_c} = \frac{w_1}{\mu_1} + \frac{w_2}{\mu_2}$$

$$\mu_{\text{DMF}} = 0.802 \text{ mPa} \cdot \text{s}, \quad \mu_{\text{CH}_2\text{Cl}_2} = 0.6 \text{ mPa} \cdot \text{s}$$

$$\frac{1}{\mu_c} = \frac{0.0588}{0.802} + \frac{(1 - 0.0588)}{0.6}$$

$$\mu_c = 0.61 \text{ mPa} \cdot \text{s} \equiv 0.61 \times 10^{-3} \text{ kg} / (\text{m} \cdot \text{s})$$

$$\Delta\rho = \rho_c - \rho_d = 1304.2 - 955.1 = 349.1 \text{ kg/m}^3$$

To use Fig. (7.20), all variables must be in US customary units.

$$\sigma_C = 0.03105 \times \frac{1}{4.448222} \times \frac{1}{3.28} = 2.128 \times 10^{-3} \text{ lbf/ft}$$

$$g_c = 4.18 \times 10^8 \text{ lb} \cdot \text{ft} / (\text{lbf} \cdot \text{h}^2)$$

$$\rho_C = 1304.2 \times \frac{1}{1.601846 \times 10^1} = 81.42 \text{ lb/ft}^3$$

$$\mu_C = 0.61 \times 10^{-3} \times \frac{1}{4.133789 \times 10^{-4}} = 1.4756 \text{ lb/(ft} \cdot \text{h)}$$

$$\Delta\rho = 349.1 \times \frac{1}{16.01846} = 21.7936 \text{ lb/ft}^3$$

$$a_p = 205 \text{ m}^2/\text{m}^3 = 205 \times \frac{0.3048}{1} = 62.484 \text{ ft}^2/\text{ft}^3$$

$$\epsilon = 0.94$$

$$f = \left(\frac{\sigma_c g_c}{\rho_c} \right)^{0.2} \left(\frac{\mu_c}{\Delta \rho} \right) \left(\frac{a_p}{\epsilon} \right)^{1.5} \quad (7.34)$$

$$= \left(\frac{2.128 \times 10^{-3} \times 4.18 \times 10^8}{81.42} \right)^{0.2} \left(\frac{1.4756}{21.7936} \right) \times \left(\frac{62.484}{0.94} \right)^{1.5} = 235.66$$

From Fig. (7.20),

$$(v_{CF}^{1/2} + v_{DF}^{1/2})^2 \rho_c / (a_p \mu_c) = 600$$

$$(v_{CF}^{1/2} + v_{DF}^{1/2})^2 = 600 \times \frac{62.484 \times 1.4756}{81.42} = 679.45$$

$$\frac{v_{CF}}{v_{DF}} = \frac{q_{VC}}{q_{VD}} = \frac{q_{VE}}{q_{VF}} = \frac{2.502}{1.047} = 2.39$$

$$(2.39 v_{DF})^{1/2} + v_{DF}^{1/2} = 679.45$$

$$6.482 v_{DF} = 679.45$$

$$v_{DF} = 104.82 \text{ ft/h and } v_{CF} = 250.52 \text{ ft/h}$$

or $v_{DF} = 104.82 \times \frac{1}{3600} \times \frac{0.3048}{1} = 0.009 \text{ m/s}$

$$v_{CF} = 0.0215 \text{ m/s}$$

Let $v_D = 0.5 v_{DF} = 0.0045 \text{ m/s,}$
 $v_C = 0.01075 \text{ m/s}$

$$\frac{\pi}{4} D_i^2 = \frac{q_{VC}}{v_C} = \frac{2.502/3600}{0.01075} = 0.065 \text{ m}^2$$

$$D_i = 0.288 \text{ m}$$

Let $D_i = 0.3 \text{ m} = 300 \text{ mm}$

Packing height:

$$H = N_{toR} \times H_{toR}$$

$$N_{toR} = 5.345$$

Suitable correlation for H_{toR} is not available in open literature for the given system. It must be determined by pilot plant study. Here H_{toR} is determined by the same equation that is derived for acetic acid – water – MIBK system.

$$H_{toR} = 0.2347 \left(\frac{v_C}{v_D} \right)^{0.648} = 0.2347 \left(\frac{0.01075}{0.0045} \right)^{0.648}$$

$$H_{toR} = 0.41 \text{ m}$$

$$H = 5.345 \times 0.41 = 2.19 \text{ m}$$

Let $H = 2.5 \text{ m}$

Resulting data:

Type of packing: 25 mm SS Pall rings

Inside Diameter of tower = 300 mm

Height of packing section = 2.5 m

7.7 SUPERCRITICAL EXTRACTION (SCE) (REF. 13 TO 20)

In liquid–liquid extraction solvents like water, ethylene glycol, chloroform, hexane, ethyl acetate, etc. are contacted with feed mixture to separate the desired product. Desired product is transferred to liquid solvent. Then desired product is to be separated from solvent. In short by liquid–liquid extraction from a given feed solution one can make new solution (solvent + solute). If separation of new solution is easier than the same of the given solution, then extraction is called useful extraction.

Separation of solute from solvent is normally done by distillation. It consumes large amount of energy. Also it is difficult to get 100% pure solute; absolutely free from solvent by distillation.

In supercritical extraction, supercritical fluid is used as a solvent. Until now it has been discussed that the fluid can exist as liquid or gas. But in addition to that a fluid can also exist as supercritical fluid.

Supercritical fluid is any fluid at a temperature higher than critical temperature and at a pressure higher than critical pressure.

In Fig. 7.22 and Fig. 7.23, phase diagrams of carbon dioxide and water are given. Supercritical regions for both the compounds are shown in the figures.

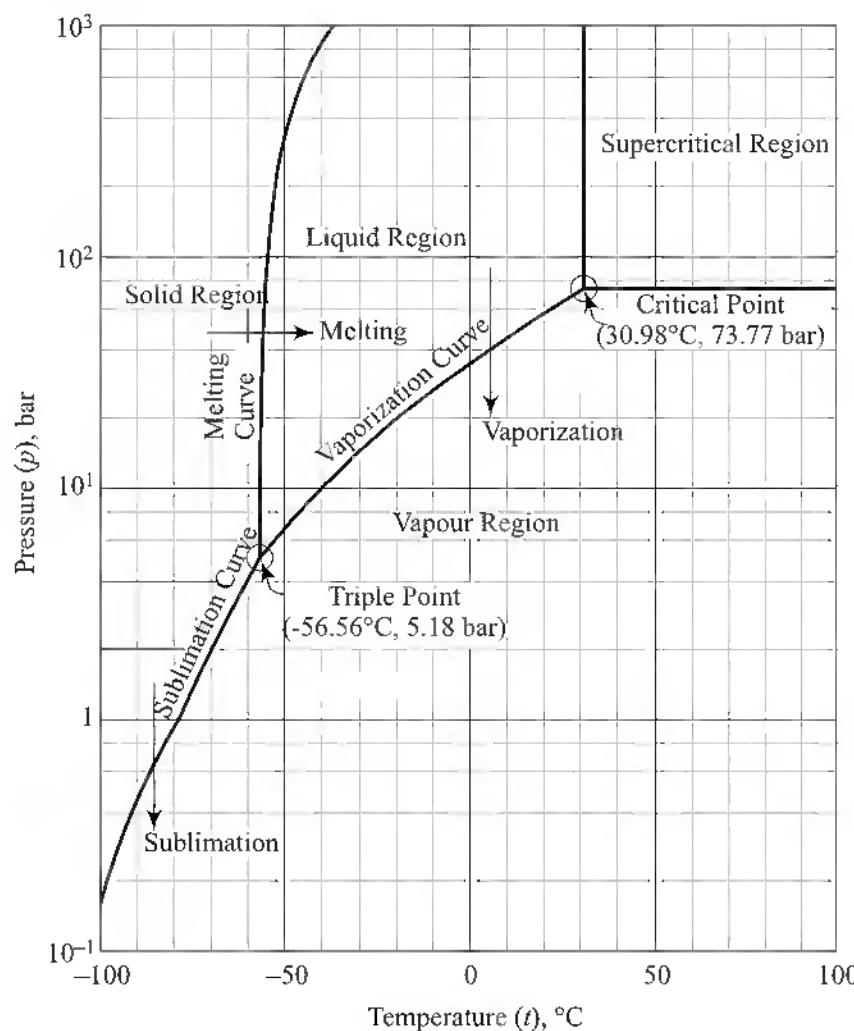


Fig. 7.22 Phase Diagram (p - t Diagram) of Carbon Dioxide

Based on Data Contained in Thermodynamic Properties of Carbon Dioxide, by: R. Span and W. Wagner, *J. Phys. Chem. Ref. Data*, **25**(6), p. 1509, 1996.

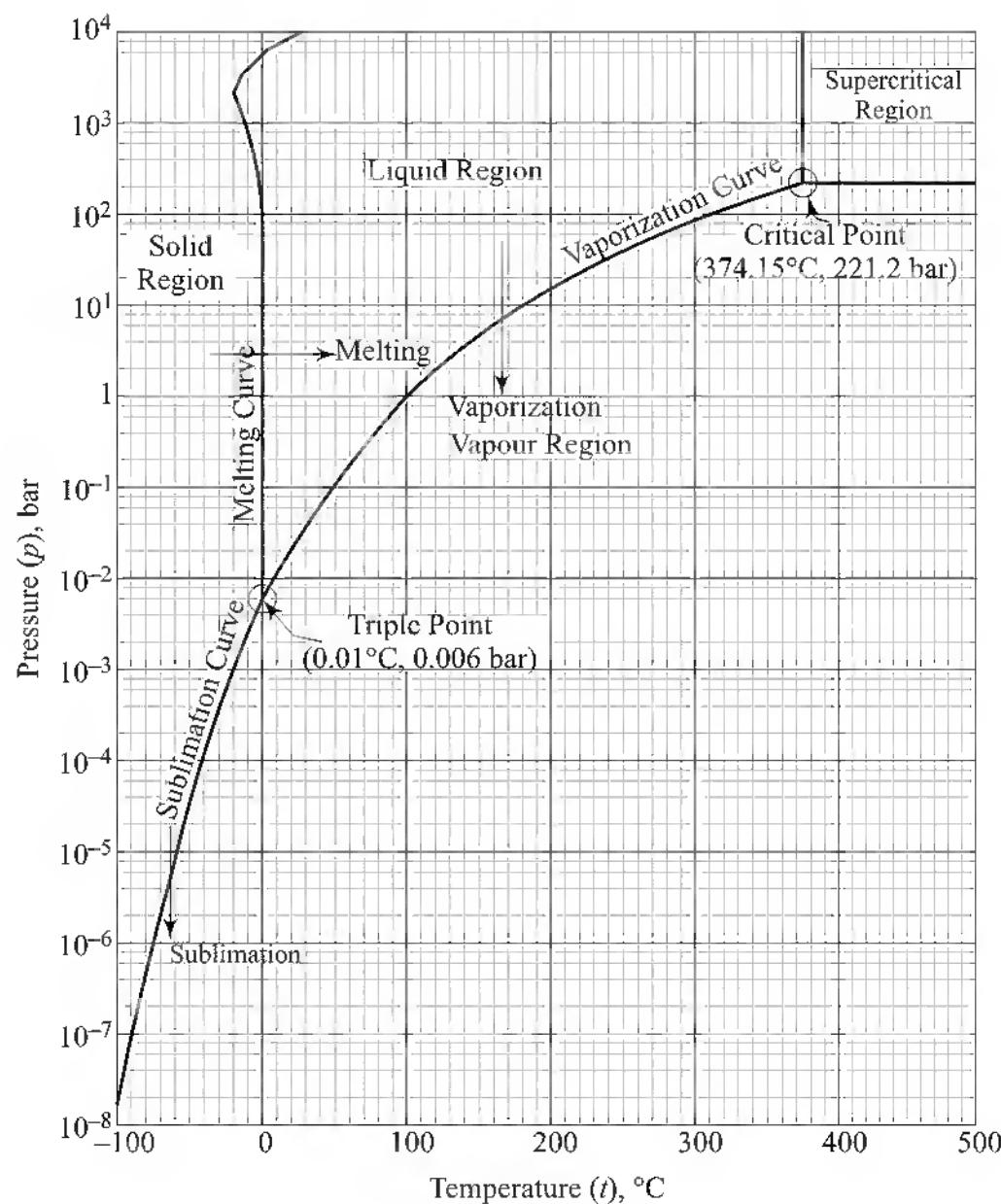


Fig. 7.23 Phase Diagram (p - t Diagram) of Water

Based on Data Contained in

1. Vacuum Technology, Its Foundations, Formulae & Tables, by Leybold AG, Germany, 1987.
2. 1980 SI JSME STEAM TABLES, Japan.
3. Publication No. NISTIR 5078 of National Institute of Standards & Technology, USA, 1998.

7.7.1 Properties of Supercritical Fluid Solvents (SCFS) (Ref. 13, 14)

- (i) In supercritical state, density of fluid is just like liquid density and hence solvent power for extraction is also equivalent to liquid solvents. In supercritical region density of the fluid is sensitively changing with pressure. Slight change in pressure creates the dramatic change in density.
- (ii) Viscosities of SCFS are equivalent to viscosities of gases. Hence they consume less power for transportation. Also they provide lesser resistance to heat transfer and mass transfer.

- (iii) Molecular diffusivities of SCFS are intermediate to liquids and gases.
 D_L is in the order of $C \times 10^{-9} \text{ m}^2/\text{s}$ (D_L = diffusivity of liquid).
 D_G is in the order of $C \times 10^{-5} \text{ m}^2/\text{s}$ (D_G = diffusivity of gas).

7.7.1.1 Advantages and Disadvantages of SCF Solvents Over Liquid Solvents

(a) Advantages

- (i) SCF solvents are easily and completely separated from extracted products. This means SCF solvents give the product, nearly 100% free from solvent.
- (ii) Reduction in pressure or increase in temperature separates the SCFS from extracted products. Hence they require lesser energy for separation. Hence, with SCE, near 100% recovery of solvent from extracted product is possible with low cost, compared to liquid–liquid extraction with liquid solvents.
- (iii) SCF solvent has very low viscosity and higher diffusivity compared to liquid solvent. Hence, it provides lesser resistance to mass transfer and also it provides lesser friction during transportation.
- (iv) SCFS provides very high value of selectivity, compared to conventional liquid solvents.
 Selectivity,

$$\alpha = \frac{\text{mass of solute}/\text{mass of non solute component of extract phase}}{\text{mass of solute}/\text{mass of non solute component of raffinate}}$$

$$\alpha = \frac{(C/A)_{\text{extract}}}{(C/A)_{\text{raffinate}}}$$

Extraction is possible only if $\alpha > 1$. If $\alpha \gg 1$, implies lesser number of theoretical stages are required for desired separation. Hence with SCF solvents, lesser number of theoretical stages are required for desired separation. Consequently they require lesser height of extractor as compared to conventional liquid solvents.

- (v) With common SCFS like CO_2 , solvent recovery is possible at room temperature or at temperature close to the room temperature. While the same is not possible with conventional liquid solvent.
- (vi) Common SCFS like CO_2 is easily available with relatively low price as compared to conventional liquid solvent.
- (vii) SCFS like CO_2 (dry) is non corrosive and nontoxic in nature.

(b) Disadvantages

- (i) It is found by experiments that supercritical fluids, provide poor value of distribution coefficient for extraction as compared to conventional liquid solvents.

Distribution coefficient,

$$m = \frac{\text{mass fraction of solute in extract phase at equilibrium}}{\text{mass fraction of solute in raffinate phase at equilibrium}} \quad (7.1)$$

Hence solvent/feed ratio, required with SCF solvents, is very much higher than conventional liquid solvents.

- (ii) Except the few solvents like CO_2 , for other solvent like hexane, ethylene glycol, etc, it is very difficult to achieve the supercritical state of fluid due to the practical limitations. Choice of solvent is narrow with SCFS.
- (iii) Certain SCFS are flammable (like propane, butanes, etc.) and hence adequate safety measures are required in design.

Properties of CO_2 , H_2O and *n*-hexane in supercritical region are given in Ref. 21. Properties of ethane, propane and butanes in supercritical region can be found in Ref. 22.

Table 7.4 List of Supercritical Solvents and Their Properties²³

Fluid	t_c ($^{\circ}\text{C}$)	p_c (bar)	Remarks
Carbon dioxide, (CO_2)	31.00	73.77	Most Favourable
Dinitrogen monoxide, (N_2O)	36.45	72.40	Not stable
Xenon, (Xe)	16.60	58.68	High price, poor availability
Ethane, (C_2H_6)	32.27	48.80	Inflammable
Ethylene, (C_2H_4)	9.19	50.39	Inflammable
Water, (H_2O)	374.15	221.20	High temperature and pressure operation, corrosive, high latent heat of vaporization
Propane (C_3H_8)	96.67	42.47	Inflammable
<i>n</i> -Butane (C_4H_{10})	152.03	37.97	Inflammable
<i>i</i> -Butane (C_4H_{10})	135.00	36.48	Inflammable

Among all these solvents, CO_2 and propane are used in commercial plants. Water is also an useful SCF but requires a very high pressure operation.

CO_2 is the most preferred as SCE solvent. Reasons are:

- (i) It has low critical temperature ($t_c = 31.0^{\circ}\text{C}$).
- (ii) It has relatively low critical pressure ($p_c = 73.77$ bar).
- (iii) It is nontoxic and non-corrosive in nature (when dry).
- (iv) It is easily available at low cost. It is available as a byproduct from many industries; such as fermentation of molasses, ammonia plant, etc. It is present in substantial amount in atmosphere as green house gas.

7.7.1.2 Advantages and Disadvantages of SCE Over Conventional Unit Operations

(a) Advantages

- (i) It consumes lesser energy compared to liquid–liquid extraction and distillation, combined together. Hence, it requires lesser operating cost as compared to extraction and distillation. It requires only electricity as a utility to drive a compressor and a positive displacement liquid pump. With CO_2 or N_2O , it also requires chilled water or brine for condensation.
- (ii) Compared to the liquid–liquid extraction it gives purer products, nearly 100% free from solvent or foreign impurities.

- (iii) SCF provides very high value of selectivity as compared to conventional liquid solvents. Hence supercritical extractor requires lesser number of theoretical stages and lesser height, compared to liquid–liquid extractor.
- (iv) Compared to (atmospheric) distillation, supercritical extraction can be operated at lesser temperature, closer to room temperature. Liquid–liquid extraction requires higher temperature in solvent recovery step. This advantage is beneficial particularly for heat sensitive products.
- (v) Products obtained by biotechnology route is usually a dilute solution (< 10%) in water. For the separation of desired product from water by distillation requires very high amount of energy and operating cost. While for the same separation, SCFE consumes very less energy and has low operating cost.

Several examples of herbal and heat-sensitive products are given in Ref. 20 with the use of CO_2 as SCF.

One research paper mentions that operating cost required to separate ethanol from dilute solution by distillation is 5 times higher than the same by SCFE while currently fixed cost of distillation unit is one half that of the SCFE unit.

(b) Disadvantages

- (i) It requires higher capital investment as this operation is carried out at high pressure. Fixed cost of compressor is relatively higher than cost of other equipments. Also the cost of the extractor is high. There are only a few suppliers of the technology and equipments of the plant of SCFE at present. Hence the specialization of technology further increases fixed cost of plant at present.
- (ii) It provides very low value of distribution coefficient hence large amount of solvent is required for separation.
- (iii) Limited experience with its use in pilot and commercial applications. However, now pilot studies with CO_2 as SCF are available at certain research institutes with sufficient data for scale-up.
- (iv) Limited knowledge of design data. Also thermodynamics of supercritical system is not fully understood till date.

7.7.2 Flow Sheet of Batch Supercritical Extraction (Ref. 13, 14)

Figure (7.24) is the flow sheet of SC Extraction plant which is operated in batch wise manner. Operational steps are as follows.

- (i) In bath extraction, feed is initially charged before starting carbon dioxide circulation.
- (ii) In CO_2 vessel, CO_2 is in compressed gas form but $p < p_c$ and temperature is equal to room temperature.
- (iii) From CO_2 vessel, compressed gas passes through chilled water condenser or brine condenser where CO_2 is liquefied.
- (iv) Liquefied CO_2 is pumped by metering pump (a positive displacement pump) to high pressure. At the discharge of pump, CO_2 is liquid with $p > p_c$ and $t < t_c$.

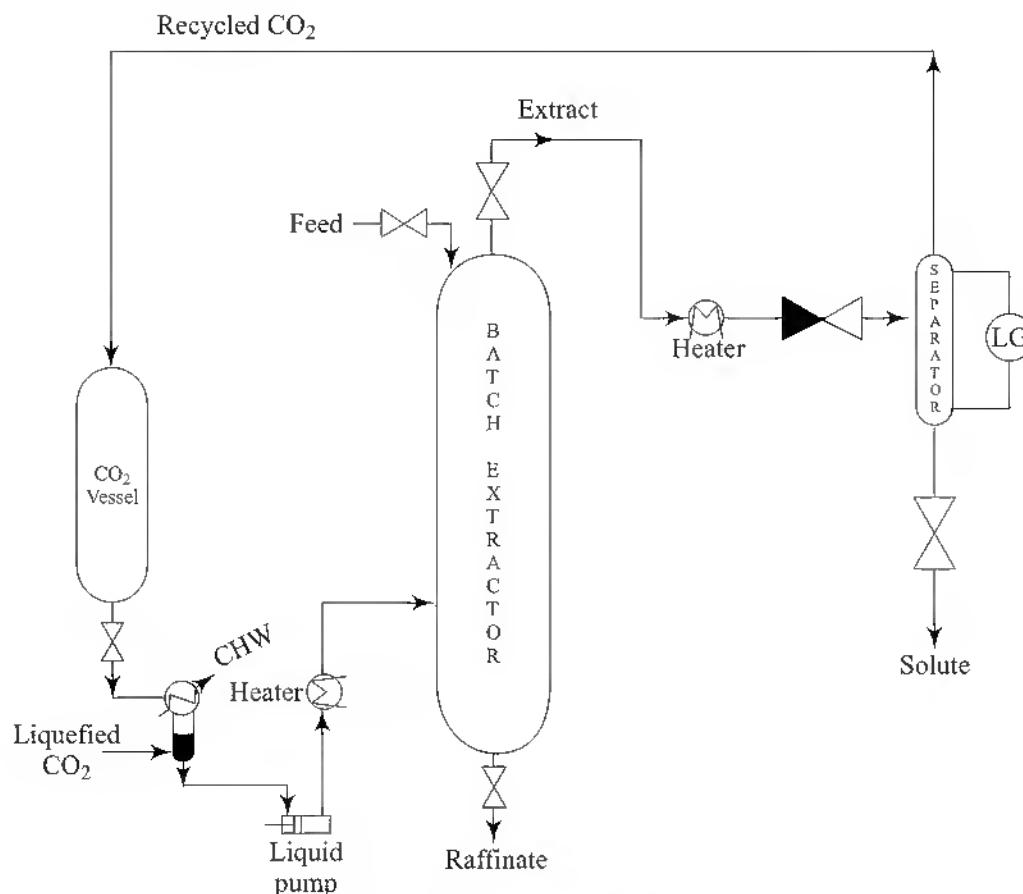


Fig. 7.24 Flow Sheet of Batch SCE Plant

- (v) Compressed liquefied CO₂ which is in liquid state at $p > p_c$ and at temperature less than t_c is converted to SCF by increasing the temperature above critical temperature.
- (vi) Supercritical CO₂ is contacted with feed which was initially charged in the batch extractor.
- (vii) From the top of the extractor, extract phase (SC CO₂ + solute) is obtained. It is heated to the temperature at which soluble solute is separated into insoluble solid or liquid.
- (viii) Then pressure is decreased and SC CO₂ is compressed to gaseous CO₂ with $p < p_c$ and at temperature which is close to t_c .
- (ix) CO₂ is circulated till the desired degree of extraction is obtained. A level indicator can be provided on the separator.
- (x) Once desired degree of extraction is obtained, system is depressurized. Raffinate phase is separated from the extractor at atmospheric pressure. New feed then can be charged to the extractor.

7.7.3 Application of SCE (Ref. 13 to 20)

SCE finds its application in food, pharmaceutical, petroleum refineries, solid fuel industries and polymer industries. Extensive research work is going on other areas of applications.

A. Commercial applications

- (a) In decaffeination of coffee

- (b) In deasphalting of oil
- (c) In extraction of essence from fragrant materials
- (d) In the separation of aromas and oils from spices
- (e) In spray painting and coating
- (f) In chromatographic analysis of polymer
- (g) Supercritical fluid polymerization
- (h) In extraction of herbs
 - (i) In purification of active pharmaceutical intermediates (API)
 - (j) In isolation of eicosapentanoic acid (EPA) from fish oils
 - (k) Purification of a vitamin intermediate
 - (l) Purification of a reactive multifunctional acrylic monomer
- (m) Extraction of cholesterol from butter
- (n) Extraction of oligomers from high vacuum sealants.
- (o) Extraction of sterols from deodorized distillate of edible/non-edible oils.

B. Applications under Research and Development

- (a) Extraction of various chemicals from coal and wood
- (b) Extraction of organic chemicals from its dilute solution in water. (This application is useful in biotechnology route and also in the recovery of chemicals from wastewater stream)
- (c) In separation of liquid hydrocarbon mixtures which are difficult to separate by conventional techniques
- (d) Separation of aromatics from waste solid material such as tobacco dust
- (e) In regeneration of adsorbent materials
- (f) Extraction of oil from oil – containing conventional and unconventional seeds, e.g. soybeans, jojoba beans, coriander, etc.
- (g) In deodorization of vegetable and animal oils.
- (h) In extraction of oil from potato chips
- (i) In recovery of citrus oil from peelings
- (j) Extraction of anticancer drugs and other substances from plants (herbs)
- (k) In refining of pyrethrum to obtain pyrethrin insecticides
- (l) Destruction in hazardous waste and production of biocrude from organic waste (water is used as SC fluid)
- (m) In extraction of chemicals from used automotive tires
- (n) In separation of monoglycerides from the mixture of glycerides
- (o) In paper deacidification and preservation
- (p) In plastic recycling
- (q) In textile dyeing

Many other applications are under R&D and can be found from literature.

7.7.4 Use of Cosolvents and Surfactants (Ref. 15)

One of the major limitations of SCE is availability of very few suitable solvent. Most suitable solvent is supercritical CO_2 which is nonpolar in nature.

Many nonvolatile polar substances cannot be dissolved easily in nonpolar SC CO_2 . Most of the organic compounds are polar in nature, e.g. ethanol, ethylene glycol, etc.

Polar solvents have high critical temperature. Hence they involve very high cost in extraction. Also other problems like thermal cracking may take place of

such solvent as SC solvent results in its rejection. It is found by experiment that an addition of small amount of polar liquid solvent (1 to 10 mole %) in SC CO₂, dramatic improvement in the solubility can be achieved.

Take for example case of hydroquinone which dissolves very little in pure SC CO₂. Addition of 2.8 mole % methanol increases hydroquinone's solubility by a factor of 6. Addition of 2 mole % tributyl phosphate (TBP) increases hydroquinone's solubility by a factor of 250.

Hence TBP is much more effective than methanol as cosolvent. Selection of the correct cosolvent requires detailed knowledge of chemistry and molecular structure of polar and non-polar compounds. Hence, selection of right cosolvent is very important.

7.7.5 Few Commercial Applications

It will be interesting to study two case studies of supercritical extraction/processing.

7.7.5.1 Decaffeination of Coffee (Ref. 17)

Coffee industry is an important food grade industry. With industrialization and increasing demand of refreshers, there has been an increase in consumption of coffee in most of the parts of world. Coffee seeds contain 1–3% caffeine. Taking large dosages of caffeine produce adverse effect on human body, but at moderate level no major harmful effects are observed.

Pure caffeine is a white powder, soluble in water and various organic solvents. Hot water is an excellent solvent for decaffeination. There are many plants in the world which extract caffeine from raw coffee. One of them is coffee decaffeination plant in Houston, USA, built by Kraft General Foods Ltd. The process, used by Kraft, is shown in Fig. (7.25).

Process Description The moist green coffee beans are charged to an extraction vessel having 7 ft (2.13 m) diameter and 70 ft (21.33 m) height. Carbon dioxide is charged from the bottom of extractor in supercritical state. From supercritical extract, caffeine is then recovered by counter current water wash, carried out at the same pressure.

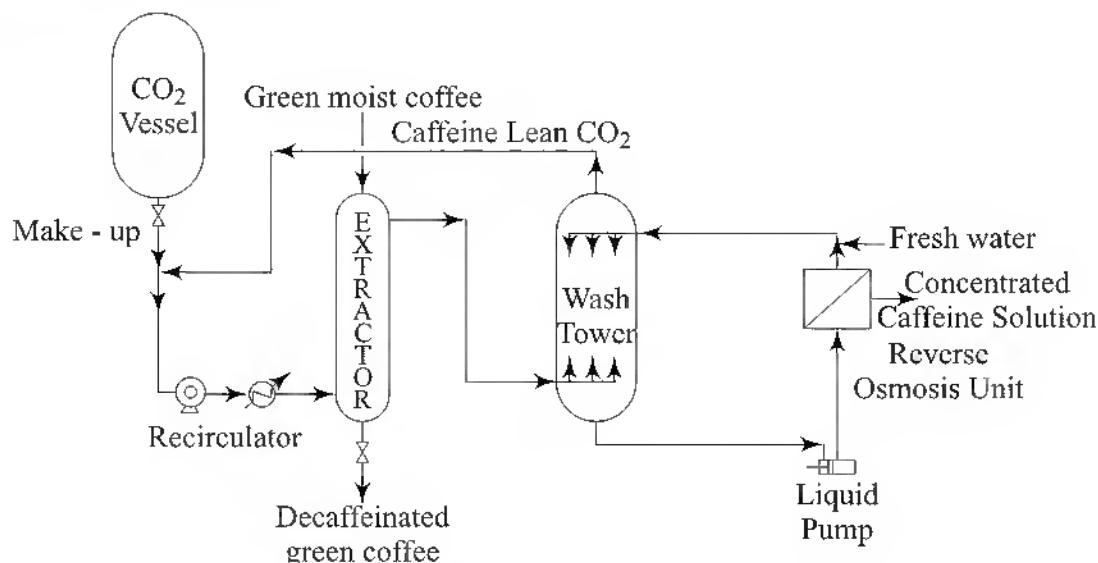


Fig. 7.25 Decaffeination Process

The depleted carbon dioxide is recycled to the extractor. Caffeine rich aqueous stream is concentrated by reverse osmosis.

The Lipton tea decaffeination process is similar to Kraft process (Lipton tea decaffeination plant is in Germany.) except that the caffeine is removed from the carbon dioxide by passing the solution through a bed of activated carbon.

The caffeine recovery from the extract is the key part of the process. As the decaffeinated green coffee is finally contacted with solvent, equilibrium distribution of caffeine between beans and solvent decide the percentage of unrecovered caffeine in the beans.

If caffeine concentration in the solvent (SC CO_2) is reduced to 0.002 mass % in recovery step, then it gives equilibrium concentration of caffeine remaining in the processed beans to about 0.16 mass%.

Hence, it is very much necessary to remove caffeine from extract phase as much as possible. One way is to reduce the pressure of SC extract to atmospheric pressure, but it is not an economical option.

It is suggested not to try to reduce the concentration of caffeine from coffee below 0.16% as it is not economical. Hence the selection of recovery process and maximum extraction of caffeine is very important.

7.7.5.2 ROSE Process (Deasphalting of Oil) (Ref. 24)

Solvent extraction has been used to separate valuable components from heavy oil and residuals from less desirable material. One of them is propane deasphalting. In the refinery, bottom product of vacuum distillation column is known as residuum. Propane deasphalting is used to separate the residuum into three fractions; (i) lube oil (ii) resin and (iii) asphalt. It uses liquid propane as solvent to extract lube oil and resin from residuum and to reject high molar mass asphalt, as raffinate. Solvent recovery in conventional propane deasphalting consists of series of flash vessels and steam stripping. Entire propane is recovered as vapour at lower pressure. It is converted into high pressure liquid propane by compression and liquefaction and recycled back to extractor. Thus, energy consumption in conventional propane deasphalting is very high and it demands the use of new technique. The ROSE (Residuum Oil Supercritical Extraction) process is a new process developed by Kerr-McGee's R&D group. ROSE process produces identical products from residuum (lube oil, resin and asphalt) but it consumes less energy.

Details of three products obtained from residuum are as follows:

- (i) Asphalt: It is the heaviest, most viscous and the most hydrogen deficient fraction of crude petroleum oil. At room temperature it is a dark brown to black friable solid. Its ring and ball softening point ranges from 70° to 200°C . Carbon to hydrogen ratio is 9 to 11.6 by mass. It is extremely difficult to remove sulphur, nitrogen or metals from asphalt. Hydrotreating and cracking catalysts are rapidly coked, deactivated and poisoned by asphalt.
- (ii) Resin: It is an intermediate fraction, lighter than asphalt and heavier than deasphalts oil or lube oil. Resin can be hydrotreated, hydrocracked, thermally-cracked or even coked to produce more volatile products. Resins are less viscous, lower in metals, sulphur and carbon. Resins have less C/H ratio than asphaltene.

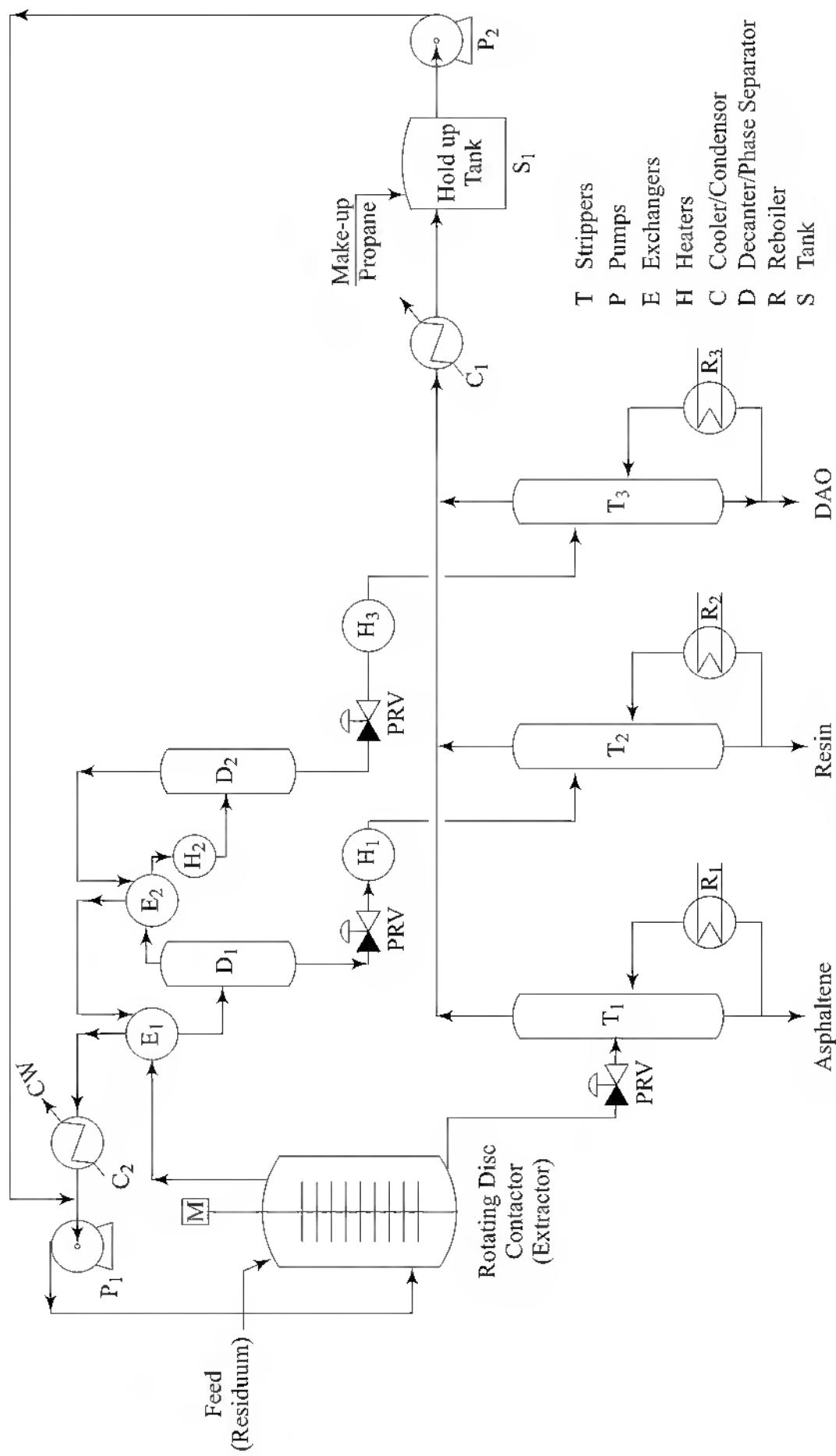


Fig. 7.26 ROSE Process

- (iii) DAO or Lube Oil: Deasphated oil or lube oil is a clean product containing substantially lower metals and carbon residue than either asphalt or resin. DAO can be sent for hydrotreating, hydrocracking or catalytic cracking, without any further purification.

7.7.5.3 Process Description

The residuum is charged to extractor where it is contacted with liquid propane at elevated pressure. In ROSE process also, extraction is liquid-liquid extraction, similar to conventional propane deasphalting. It is not the supercritical extraction. As an extractor, either Mixer-settler or Rotating Disc Contractor (RDC) is used. From the extractor, asphalt is obtained as raffinate phase, but it is contaminated by very less amount of propane. Propane vapour is separated from raffinate phase by flashing and stripping in tower T_1 which is a flash vessel cum stripper.

Extract phase, containing large amount of liquid propane, resin and lube oil, is passed through heater E_1 where it is heated to the temperature at which resin becomes insoluble and is separated from the extract phase. Propane rich layer and resin layer are separated in decanter D_1 . Traces of propane from resin layer is separated by flashing and stripping in the tower T_2 .

The remaining propane-oil solution is heated to higher temperature by two heaters E_2 and H_2 in series. At this temperature liquid propane is converted into supercritical propane. DAO or lube oil is not soluble in supercritical propane, hence it is separated from propane in decanter D_2 . Traces of propane from DAO or lube oil is separated by flashing and stripping in tower T_3 . Supercritical propane from D_2 is passed through heat exchanges E_2 and E_1 , where its sensible heat is utilized for heating. Then in heat exchanger C_2 it is liquefied. Liquified propane is recycled back to extractor by pump P_1 . Difference between discharge pressure and suction pressure of pump P_1 is relatively low. Pump P_1 develops only differential pressure necessary to overcome system pressure drop.

Small amount of propane vapour, stripped off from the towers T_1 , T_2 and T_3 , is condensed in condenser C_1 . Condensate is sent to tank S_1 . This intermediate hold-up tank is maintained at lower temperature and at lower pressure, where make up liquefied propane is also added. Small amount of propane from this tank (7 to 15% of the total required in extractors) is pumped by pump P_2 and sent to suction line of pump P_1 . Pressure difference around pump P_2 is quite higher than the same around pump P_1 .

Thus in ROSE process, extraction is carried out in subcritical region or as liquid-liquid extraction. Only properties of supercritical fluid are utilized in solvent recovery step.

Advantages of Rose process over conventional propane deasphalting are as follows.

- (i) 85 to 93% of the extraction solvent is recovered as supercritical fluid in the oil separator without evaporation and pressure reduction. Also, the concept of energy integration is applied in two heat exchangers. (E_1 and E_2) to save the utilities. Hence energy required by ROSE process is substantially lower than the same required for conventional propane deasphalting.

- (ii) ROSE process requires less maintenance cost.
 - (iii) Increase in propane to feed ratio improves the quality of products. With ROSE process, higher solvent to feed ratio is affordable, hence it results in better quality of products.
- ROSE process is competitive, well established and commercially proven technology. It is licensed by more than 15 companies worldwide.

7.7.5.4 Conclusion

In the early stages of development of the Supercritical Fluid Extraction, the requirement of expensive equipments, lack of proper and appropriate techniques were the main hurdles of adoption and commercialization of the technology.

But now in the changed scenario of improvement in the SCE techniques, increasing cost of energy and increasing cost of conventional solvents makes this unit operation more feasible and attractive. Still there is a large scope for the development of supercritical extraction technology. More research work is required to improve the distribution coefficient of SCE system. In future, it is certain that this unit operation will be applied in many processes.

EXERCISES

- 7.1 A feed stream having flow rate of 1000 kg/h and containing 40 mass % styrene and 60 mass % ethyl benzene is to be extracted with ethylene glycol as solvent. Extraction is to be carried out in multistage counter current extractor. Final raffinate is to be extracted down to 1 percent styrene. Determine (a) minimum amount of solvent required for the desired separation and (b) number of theoretical stages required for desired separation if the actual amount of solvent is 5000 kg/h.

Table 7.5 Liquid-liquid Equilibrium Data³ of Ethyl Benzene-Styrene-Ethylene Glycol at 25°C

Mass % in raffinate			Mass % in extract		
Ethyl benzene	Styrene	Ethylene glycol	Ethyl benzene	Styrene	Ethylene glycol
90.56	8.63	0.81	9.85	1.64	88.51
80.4	18.67	0.93	9.31	3.49	87.2
70.49	28.51	1.00	8.72	5.48	85.8
60.93	37.98	1.09	8.07	7.45	84.48
53.55	45.25	1.2	7.35	9.25	83.4
52.96	45.84	1.2	7.31	9.49	83.2
43.29	55.32	1.39	6.3	12.00	81.7
41.51	57.09	1.4	6.06	12.54	81.4

(From Table 15.2 of Ref. 3)

- 7.2 For the extraction described in Exercise 7.1, if packed tower is used as extractor then (i) find the value of N_{toR} for $S = 5000 \text{ kg/h}$ and (ii) determine the tower diameter.
- 7.3 A feed mixture containing 60 mass % methyl cyclohexane and 40 mass % *n*-heptane is contacted with pure aniline as solvent. Extraction is carried out in counter current manner in four mixer-settler vessels. Stage efficiency of mixer-settler may be assumed equal to 1. Solvent to feed ratio (mass basis) is kept 4. Feed flow rate is 200 kg/h. Find the resultant product flow rates and product compositions.

Table 7.6 Equilibrium Data

Hydrocarbon phase, mass %		Aniline rich phase, mass %	
Methyl cyclohexane	<i>n</i> -Heptane	Methyl cyclohexane	<i>n</i> -Heptane
0.0	92.6	0.0	6.2
9.2	83.1	0.8	6.0
18.6	73.4	2.7	5.3
22.0	69.8	3.0	5.1
33.8	57.6	4.6	4.5
40.9	50.4	6.0	4.0
46.0	45.0	7.4	3.6
59.0	30.7	9.2	2.8
67.2	22.8	11.3	2.1
71.6	18.2	12.7	1.6
73.6	16.0	13.1	1.4
83.3	5.4	15.6	0.6
88.1	0.0	16.9	0.0

(Ref. : 7, p. 325)

(Hint: Assume *n*-heptane and aniline as completely immiscible liquids to simplify the calculations.)

- 7.4 Design a mixer settler for Exercise 7.3.

Residence time required in mixer to achieve equilibrium = 8 min

Type of agitator of mixer = 45° pitched blade turbine

Tip velocity of agitator = 200 m/min

- 7.5 A feed mixture containing 20 mass % acetic acid and 80 mass % water is contacted with nearly pure butyl acetate as solvent to recover acetic acid. Extraction is carried out in packed tower. Feed flow rate is 1000 kg/h. Final raffinate leaving the packed tower should contain less than or equal to 1% (by mass) acetic acid. Determine the following:

(i) Minimum amount of solvent required for desired separation.

(ii) Value of N_{toR} for actual amount of solvent $S = 2 S_m$.

(iii) Tower diameter required for packed tower.

(iv) Determine the height of packing. Assuming that Eq. (7.33) is valid for acetic acid-water-butyl acetate system for finding H_{toR} .

Data:

Table 7.7 Liquid-Liquid Equilibrium Data (mass %) at 25°C.

Solvent rich layer			Water rich layer		
Water	Butyl acetate	Acetic acid	Water	Butyl acetate	Acetic acid
1.8	94.8	3.4	90.5	0.8	8.7
3.7	84.6	11.5	78.9	1.1	20.0
4.7	82.0	13.3	75.6	1.2	23.2
7.8	73.0	19.2	68.0	1.6	30.4
8.9	68.0	23.1	65.0	2.3	32.7
14.7	56.0	29.3	56.3	5.6	38.1

REFERENCES

1. Treybal R. E. *Liquid Extraction*, McGraw-Hill Book Company, Inc., USA, 1951.
2. Rawat B. S. *Aromatic Extraction*, Indian Institute of Petroleum, Dehradun, 2000.
3. Perry R. H. and Green D. *Perry's Chemical Engineers' Handbook*, 6th Ed., McGraw-Hill Book Company, USA, 1984.
4. Treybal R. E. *Mass Transfer Operations*, 3rd Ed., McGraw-Hill Book Company, USA, 1980.
5. *DMF Recovery and Purification*, Technical Catalogue of Dupont Co., USA.
6. Nanoti. S. M., *Industrial Extraction Equipment Selection and Process Design*, Indian Institute of Petroleum, Dehradun, 2000.
7. Brown G. G., *Unit Operations*, John Wiley and Sons, Inc., New York, 1950.
8. Schweitzer, *Handbook of Separation Techniques for Chemical Engineers*, McGraw-Hill Book Company, USA, 1979.
9. McKetta. J. J., *Encyclopedia of Chemical Processing and Design*, Vol. 21, Marcel Dekker Inc., USA, 1984.
10. Nagata, S., *Mixing*, Kodansha Ltd., Japan, 1975.
11. Coulson, J. M. and Richardson, J. F., *Chemical Engineering*, Volume 2, Pergamon Press, New York, USA, 1987.
12. Backhurst, J. R. and Harker J. H., *Chemical Engineering*, Volume 5, Pergamon Press, New York, USA, 1989.
13. Awasthi, A., Trivedi, R. A., *Chem. Engg. World*, Vol. **XXXII**, No. 10 Oct. 97, p. 65.
14. Bork, M., *Chem. Engg. World*, Vol. **XXXII**.
15. Chrastil, J., *J. Phys. Chem.*, **86** 1982, p. 3012
16. De Hann, A. B., De Graauw, J., *Ind. Eng. Chem. Res.*, **30** 1991, p. 2463.
17. Kohn P. M. and Savage P. R., *Chem. Engg.*, **86** (6), March 12, 1979, p. 41.
18. Laheire, R. J., Fair J. R., *Ind. Engg. Chem. Res.*, **26**, 1987, p. 2086.
19. Moore S. and Samdini S. *Chem. Engg.*, **32**, March 1994.
20. Mukhopadhyay, M. *Natural Extracts using Supercritical Carbon Dioxide*, CRC Press LLC, USA, 2000.
21. Bhatt, B. I., *Design Databook, Properties of Steam, Selected Refrigerants, Hexane and Brines*, CBS Publishers and Distributors, New Delhi, 2007.
22. Younglove, B. A. and J. F. Ely, *J. Phy. Chem. Ref. Data*, **16**(4), 1987, p. 577.
23. Bhatt, B. I. and S. M. Vora, *Stoichiometry*, 4th Ed., Tata McGraw-Hill Publishing Co. Ltd., 2004.
24. Nelson, R. R., *Chem. Engg. World*, **24**(7), 1989, p. 47.

Chapter

8



Process Design of Distillation Columns

8.1 INTRODUCTION

In simple terms, vaporization followed by condensation is known as distillation. It is a method of separating components of solution which are relatively volatile. Distillation is the most widely used separation method in chemical industries. Simple distillation or batch distillation was known in first century. Multistage counter current distillation was invented in 1830 by Aeneas Coffey of Dublin¹. He used tray tower for separating 95.6% ethanol (by mass) from its dilute solution in water. But as of today 100% reliable method for designing of distillation column, which can be applied to all systems without any limitation, is not available. All methods have limitations. Hence process design engineers use more than one method for the design of distillation column. For an entirely a new mixture, it is better to verify the design of its distillation column first on the pilot plant scale column before applying it at commercial or large scale.

8.2 CRITERIA OF SELECTION

For a given mixture, if all the components are relatively volatile and are required to be separated in pure form, then distillation is the first choice.

If the relative volatility of the two adjacent components is very high, then evaporation is preferred. For example, concentration of orange juice.

If relative volatility of two adjacent components is very low: close to 1 (e.g. 10 to 30%, by mass acetic acid in water) or equal to 1 (e.g. azeotropic mixture, 95.6% by ethanol in water), special type of distillation like Azeotropic Distillation or Extractive Distillation is used. For the same case, liquid–liquid extraction, membrane separation or crystallization may be more economical. Examples: (a) For the separation of pure acetic acid from 10% (by mass) aqueous acetic acid solution, liquid–liquid extraction using ethyl acetate as solvent is more economical than azeotropic distillation of the same solution using butyl acetate or ethyl acetate as an entrainer. (b) For the separation of pure ethanol from its azeotropic mixture with water. [95.6% ethanol (by mass) in ethanol – water solution], pervapouration unit consumes less than 20% of the energy, than that used in azeotropic distillation. Also for the same separation, pervapouration unit requires low pressure steam

(to maintain 80°C temperature) which can be obtained by flashing the condensate of high pressure steam. (c) For the separation of *p*-xylene from the mixed xylenes, crystallization is used.

If the separation of only one component is required rather than a separation of all the components of the mixture, then absorption, stripping or liquid–liquid extraction may be more economical than distillation.

Examples:

- (a) For the separation of penicillin from the complex mixture, liquid–liquid extraction is used.
- (b) For the removal of hydrogen and methane from aromatics, stripping is used.
- (c) For the removal of propane from the mixture called asphalt, stripping is used. Volatility of propane is very high compared to volatilities of other components of the asphalt.
- (d) For the removal of hydrogen sulphide from the natural gas mixture, absorption using ethanolamines as solvent is used.
- (e) For removal of ammonia from lean aqueous solution, steam stripping is preferred.
- (f) If all components of air are to be separated in pure form (nitrogen, oxygen, argon, etc.) cryogenic distillation is preferred. But if only nitrogen in pure form is to be separated from air then pressure swing adsorption (PSA) is preferred.

8.3 SELECTION OF EQUIPMENT FOR DISTILLATION^{1,2}

In most of the cases, distillation is carried out either in a tray tower or in a packed tower. Criteria of selection between tray tower and packed tower are as follows.

- (a) For high vacuum distillation, a packed tower is selected. Packed tower generally provides lower pressure drop as compared to the tray tower. For example in the distillation of the mixture of monochloroacetic acid and acetic acid, absolute operating pressure in the reboiler should be less than 50 torr a to avoid thermal cracking of bottom product (monochloroacetic acid). For this case, packed tower should be selected. Packed tower provides lower residence time for liquid as compared to tray tower. The rate of thermal cracking is directly proportional to residence time. For highly heat sensitive product, decrease in boiling point means improvement in the quality. The rate of thermal cracking increases exponentially with the temperature. For very high vacuum distillation, short path distillation is used. Example: Separation of vitamin E from its crude mixture (such as soybean oil deodorizer distillate) is carried out in short path distillation equipment. Operating absolute pressure in short path distillation can be as low as 0.05 mbar or 50 µbar a.
- (b) For very small tower diameter packed tower is preferred. With large tower diameter, tray tower is preferred. With very small diameter (less than 150 mm), fabrication of trays is difficult while with large diameter, liquid distribution is difficult in the packed tower.
- (c) For handling higher ratio of liquid flow rate to vapour flow rate, packed towers are preferred. For very low liquid flow rate, tray tower is selected. But for very low L/V ratio, tray tower is selected. Packed tower requires minimum wetting rate to work efficiently.

- (d) For corrosive system, packed tower with plastic packings like polypropylene packings, glass fibre reinforced plastic packings, etc. is more economical than tray tower made from special metal alloys. Example: For the separation of methyl iodide and acetic acid, packed tower with glass fibre reinforced plastic packings is more economical than tray tower which should be entirely made from Hastalloy-B.
- (e) If the side products are to be drawn from the distillation column, like the distillation columns of refinery, tray tower is preferred.

8.4 DISTILLATION COLUMN DESIGN

The design of a distillation column can be divided into the following steps.

- (a) If the feed mixture contains more than two components, then select two key components from all the components of feed mixture.
- (b) Decide the operating pressure of distillation column. At the operating pressure, find the vapour–liquid equilibrium data.
- (c) Specify the degree of separation required. In case of a binary distillation, decide the top (distillate) and the bottom (residue) product compositions. In case of multicomponent distillation, decide the distribution of key components in distillate and residue. Also, find the distribution of non key components in the same. Complete the material balance.
- (d) Determine the minimum reflux ratio R_m .
- (e) Determine the optimum reflux ratio R and find the number of theoretical or equilibrium stages required for the desired separation.
- (f) Select the type of tower: tray tower or packed tower (as discussed in Sec. 8.3). Select the type of tray in tray tower or type of packing in packed tower.
- (g) Find tower diameter and pressure drop across the tower. Decide the top disengagement and bottom disengagement spaces.
- (h) Determine the tray efficiency and find the actual number of trays in case of tray tower or find HETP (height equivalent to theoretical plate) and decide the height of packing in packed tower. In case of packed tower, design the liquid distributor, redistributor, packing support, etc.
- (i) Select the material of construction for all parts. Design the suitable insulation system.
- (j) Design the condenser and reboiler.
- (k) Decide the control methodology for the distillation column, line sizes and locations of all nozzles.
- (l) Mechanical design of distillation column which also includes design of skirt support for the distillation column.

Calculation and equations for the first five steps are different for binary and multicomponent distillation. In this chapter first eight steps are discussed in detail.

8.4.1 Step I: Selection of Key Components^{1,2}

If the feed mixture to distillation column contains only two components, then it is called binary distillation. In many cases feed mixture actually contains more than

two components, but it is considered as binary mixture because the amounts of other components (except two key components) are negligible. Step 1 is not valid for binary mixture or binary distillation.

When a feed mixture to distillation column contains more than two components, it is called multicomponent distillation. In such a distillation, it is not possible to obtain more than one pure component in single column. For the separation of N' component mixture by distillation, if all the components are to be separated in pure form, then at least $N' - 1$ distillation columns are required.

In multicomponent distillation, one cannot specify the complete composition of the top and bottom products independently. For example: A feed mixture to distillation column is 100 kmol/h containing 20 kmol/h ethane, 30 kmol/h propane and 50 kmol/h of butane.

One cannot fix distillate composition: 20 kmol/h ethane, 29 kmol/h propane, 1 kmol/h butane and Residue composition: 1 kmol/h propane, 49 kmol/h of butane.

One can fix that distillate will contain 29 kmol/h of propane and 1 kmol/h of butane, and residue will contain 1 kmol/h of propane and 49 kmol/h of butane.

Thus one can fix only distribution of two components of feed mixture in distillate and residue but not the distribution of all components³. Before starting the design of multicomponent distillation, two key components are selected. Key components are the two components of the feed mixture between which one likes to make the sharp separation. Two key components are further divided as light key component and heavy key component. More volatile key component is called light key and less volatile component is called heavy key.

Light key component: It is a component of the feed mixture which is desired to be kept out of the bottom product. It is the component of feed which is present in residue in important amount, while components lighter than light key are either not present or present in very small amount. If all components of feed are present in residue with sufficient concentrations, then most volatile component of residue is the light key component.

Heavy key component: It is a component of feed mixture which is desired to be kept out of the top product. It is the component of feed which is present in distillate in important amount, while all components heavier than the heavy key are not present or present in negligible amount. If all components of feed are found in distillate with sufficient concentrations, then the least volatile is the heavy key component.

If the light key and heavy key components are selected as per the order of volatility then they are known as adjacent keys. If any other component lies in between them, then they are known as split keys.

Components of the feed mixture other than key components are known as non-key components. The non key components which are distributed in top and bottom products are known as distributed components. Any component which does not appear in either top or bottom product up to significant extent is known as non distributed component.

8.4.2 Step 2: Selection of Operating Pressure and Determination of Vapour–Liquid Equilibrium Data

8.4.2.1 Selection of Operating Pressure

In many cases operating pressure in distillation column is fixed by the temperature of the cheapest cooling medium (cooling water), which must be able to condense the distillate vapour. For this, bubble point of distillate (in case of total condenser) should be sufficiently greater than cooling water temperature. In case of partial condenser, bubble point of reflux (is equal to dew point of distillate vapour) should be sufficiently greater than temperature of cooling water. But if, this operating pressure (based on cooling water temperature) is calculated to be very high, then chilled water or brine can be considered as cooling medium for the overhead condenser. Operating pressure in distillation column is always less than the critical pressure of top product or temperature of cooling medium of overhead condenser cannot be greater than critical temperature of top product. In many cases, distillation is carried out under vacuum to avoid the thermal degradation of heat sensitive product or to facilitate the use of cheaper heating medium like saturated steam in reboiler or to alter the vapour-liquid equilibrium data.

Examples for the Selection of Operating Pressure:

- (i) Distillation of acetaldehyde–ethanol–water mixture is carried out at about 3 atm g pressure. Pure acetaldehyde is the top product. Normal boiling point (t_B) of acetaldehyde is 20.2°C. To facilitate the use of cooling water as a cooling medium in the overhead condenser, boiling temperature or condensation temperature of acetaldehyde should be sufficiently greater than the design temperature of cooling water from cooling tower (say 32°C). Also the product acetaldehyde is stored in bullet type storage tanks which are exposed to atmosphere. Hence, to reduce the cost of storage or to avoid the use of insulation around storage tank and to avoid the use of any refrigeration system for the storage tank, boiling point of acetaldehyde should be sufficiently greater than atmospheric temperature (50°C–maximum). Considering all these aspects, condensation temperature (is also equal to boiling temperature of pure component) of acetaldehyde is fixed at around 60°C. For getting 60°C as a condensation temperature of acetaldehyde, required operating pressure at the top of distillation column is around 3.6 atm g (vapour pressure of acetaldehyde at 60°C).
- (ii) For the separation of methyl chloride from the mixture of chloromethanes, normal boiling point or condensation temperature of pure methyl chloride is –24°C. At 10 atm a pressure, its condensation temperature is 47.3°C. Hence to facilitate the use of cooling water as a cooling medium in overhead condenser, this distillation column can be operated at 10 atm a pressure. Similarly in case of distillation of ammonia (Example 8.7), saturation pressure of ammonia at 40°C is 1555.5 kPa a. Hence, the column is operated at about 15 bar g to facilitate condensation of ammonia at 40°C in the condenser with cooling water at 32°C.

- (iii) For the separation of ethane from the mixture of ethane, propane and butanes by distillation, cooling water cannot be used as a cooling medium in the overhead condenser because critical temperature of ethane ($t_c = 32.3^\circ\text{C}$) is not sufficiently greater than the temperature of cooling water (say 32°C). Minimum driving force required for heat transfer in shell and tube type overhead condenser is 3 to 5°C . At 10°C , vapour pressure of ethane is 30 atm a, hence chilled water (at 5.7°C) can be used as a cooling medium in the overhead condenser, but the required operating pressure is 30 atm a, which is high particularly for larger diameter column. At 20 atm pressure, condensation temperature of pure ethane is -6.4°C , hence suitable brine system can be used as a cooling medium in the overhead condenser, but it will require higher cost of refrigeration and insulation. In the plant, if this column is following to demethanizer column, then top product stream of demethanizer can be used as a cooling medium in the overhead condenser of deethanizer column and operating pressure can be reduced to very low value, close to atmospheric. Optimum solution should therefore be worked out.

Vacuum Distillation: For heat sensitive material or to facilitate the use of cheaper heating medium in reboiler or to alter the vapour–liquid equilibrium data, distillation column is operated under vacuum. Advantages and disadvantages of vacuum distillation are:

Advantages:

- (a) It prevents thermal decomposition of heat sensitive material. Example: In separation of monochloroacetic acid and acetic acid by distillation, monochloroacetic acid is obtained as bottom product. Boiling point of monochloroacetic acid must be restricted below 120°C , otherwise thermal degradation can take place. If the boiling point of monochloroacetic acid is kept at 109°C , then operating pressure at bottom of column must be equal to 40 torr (absolute).
- (b) It allows the use of more economical heating medium such as saturated steam against hot oil in high capacity plant. Example: For the separation of ethylene glycol–water mixture by distillation, ethylene glycol is obtained as bottom product. Its normal boiling point is 197°C . Use of saturated steam as a heating medium could be economical up to 180°C . Hence, this column is better to be operated under vacuum to facilitate the use of saturated steam against the use of hot oil.
- (c) It requires fewer stages or lower reflux ratio or both in many cases where relative volatility increases with decrease in the operating pressure.
- (d) In some case vacuum breaks the azeotrope and make the separation easier. Example: If the distillation of ethanol water mixture is carried out below 70 torr absolute pressure, azeotrope does not form.
- (e) It reduces undesirable side reactions, if they are taking place. Example: Distillation of ethyl benzene-styrene mixture under vacuum reduces polymerization.
- (f) Vacuum distillation provides safety in the distillation of toxic or hazardous material. Example: For the separation of nearly 100% pure nitric acid from

the azeotropic mixture of nitric acid and water, extractive distillation is used. This extractive distillation uses concentrated sulphuric acid as solvent. Material of construction of this column is borosilicate or high quality glass. This column is operated under slight vacuum for safety purpose. Here, vacuum also facilitates the use of saturated steam in reboiler.

- (g) It increases product recovery when dealing with maximum temperature constraints. Example: Refinery vacuum tower.

Disadvantages of Vacuum Distillation:

- It requires larger column diameter and larger diameter vapour line.
- Because of the reduction in condensation temperature, costlier cooling source may be required for condensation.
- Presence of air as a non-condensable reduces the efficiency of main condenser.
- Increases the possibility of contamination of products by air or other components of atmosphere.

8.4.2.2 Determination of Vapour–Liquid Equilibrium (VLE) Data

VLE data must be determined for the given mixture for distillation and at the operating pressure of distillation column. To find the VLE data, there are three options. Priority wise these three options are given as follows:

- VLE data can be determined by experiment. If the operating pressure is atmospheric or vacuum, then VLE data-experiment is easier. Only difficult part is analysis. But if VLE data are required at high pressure, then this experiment is difficult. This is the most reliable option or method for finding the VLE data for the given mixture because the given mixture for distillation may contain some unknown impurities which may not be considered or ignored in other two options or methods given in the coming section.
- Find the VLE data for the given system from the literature, if they are available at the same operating pressure. In the literature VLE data are available for the several thousand binary and many multicomponent systems. All these data were determined by actual experiments. Major sources for these data are as follows.
 - **Vapour–Liquid and Liquid – Liquid Equilibrium Data Collection**, DECHEMA Chemistry Data Series⁴, Volume–1, Parts 1 to 10, by Gmehling and Onken (1977).
 - **Vapour–Liquid Equilibrium Data**⁵ by Chu, Ju–chin et al (1956).
 - **Vapour–Liquid Equilibrium Data Bibliography**⁶ by Wichterle, Linek and Hala, Elsevier, The Netherlands, 1976.
 - **Vapour–Liquid Equilibrium Data at Normal Pressures**⁷ by Boublík, Polak, Wichterle ad Hala, Pergamon, Oxford, UK, 1968
 - **Perry's Chemical Engineers Handbook**, 7th Edition by Perry and Green, McGraw-Hill, USA, 1998.
 - **Azeotropic Data**⁸, 3 Vol., 2nd Ed. by Gmehling, J., Wiley-VCH, Germany, 2004.
- VLE data can be determined theoretically by using phase equilibrium equation. In the given feed mixture for distillation, if all components are from the same group and operating pressure for distillation is atmospheric then

usually vapour–liquid equilibrium for such mixture can be assumed as ideal vapour–liquid equilibrium. Example: Feed mixture of benzene and toluene at atmospheric pressure.

For the ideal VLE, Raoult's law is applicable.

$$y_i p_t = x_i p_i^{\text{sat}} \quad (\text{Raoult's law}) \quad (8.1)$$

where, y_i = Mole fraction of component i in vapour phase

x_i = Mole fraction of component i in liquid phase

p_i = Operating pressure, kPa

p_i^{sat} = Vapour pressure of pure liquid i at the given temperature, kPa

General form of vapour–liquid equilibrium equation is $y_i = K_i x_i$ (8.2)

where, K_i = Equilibrium constant for component i

K – values for some hydrocarbons for the range of operating pressure from 101.3 kPa to 6000 kPa and for the range of operating temperature from -70°C to 200°C , can be obtained from Dadyburjor chart (Refer Fig. 6.20).

For non ideal system VLE data is determined by following equation.

$$y_i \theta_i p_t = x_i \gamma_i p_i^{\text{sat}} \quad (8.3)$$

where, θ_i = Fugacity coefficient of component i

γ_i = Activity coefficient of component i

Fugacity coefficient θ_i represents the deviation of vapour phase from ideal gas mixture. If the operating pressure is close to atmospheric pressure then vapour phase can be assumed as ideal gas and θ_i is unity. θ_i can be calculated from an appropriate equation of state like Redlich – Kwong equation, Peng–Robinson equation, etc.

Activity coefficient γ_i represents the deviation of the behaviour of component in given solution from the behaviour of same component in ideal solution. γ_i is a function of temperature and liquid composition.

$$\gamma_i = f(x_i, T) \quad (8.4)$$

γ_i is not dependent on pressure.

Activity coefficient γ_i can be determined by van Laar equation, UNIFAC method or UNIQUAC method or by NRTL equation. Group contribution method like UNIFAC method or UNIQUAC method are more reliable, but group contribution parameters are available for limited number of groups in literature.

For example,

For the mixture of ethane and propane, if VLE data are required at 10 atm a pressure, then $\theta_i \neq 1$, but γ_i can be assumed equal to 1 for both components.

For the mixture of ethanol–ethyl iodine, if VLE data are required are at 1 atm pressure, then for both components

$$\theta_i \approx 1, \text{ but } \gamma_i \neq 1.$$

8.4.3 Step 3: Degree of Separation

In case of binary distillation, specify x_D and x_w considering equilibrium limitations in mind, (where x_D = mole fraction of more volatile component in distillate and x_w = mole fraction of more volatile component in residue). Find the molar flow rate of distillate D and molar flow rate of residue W , by using following two material balance equations.

$$F = D + W \quad (8.5)$$

$$F z_F = D x_D + W x_w \quad (8.6)$$

where z_F represents mole fraction of more volatile component in feed. Since feed can be saturated liquid, saturated vapour or a mixture of liquid and vapour, symbol z_F is used. If it is only liquid, symbol x_F can be used.

To establish the material balance in multicomponent distillation is not easy. In case of multicomponent distillation, specify the distribution of two key components in distillate (top product) and in residue (bottom product), considering equilibrium limitations in mind. Exact distribution of non-key components in top product and bottom predict is not possible at the starting of design. But, the same can be determined approximately by Hegstebeck and Geddes equation⁹.

$$\log \left(\frac{d_i}{b_i} \right) = A + C \log \alpha_i \quad (8.7)$$

where, d_i = Moles of i^{th} component in distillate

b_i = Moles of i^{th} component in residue

α_i = Relative volatility of i^{th} component with respect to heavy key component

A, C = Constants

Material balance calculations (approximate) for multicomponent distillation is illustrated by following example.

Example 8.1

Feed mixture to a distillation column contains 30% *n*-hexane, 32% *n*-propane, 20% *n*-butane and 18% *n*-pentane (by mole). Total flow rate of the feed is 100 kmol/h. If butane and pentane are selected as light key and heavy key components, respectively then (a) fix the operating pressure of distillation column and (b) find the product compositions.

Solution:

Generally the mixture of propane and butane is stored and sold as LPG (Liquefied Petroleum Gas). Hence in that case, top product (mixture of propane and butane) must be in liquid phase or a total condenser must be used. To facilitate the use of cooling water as cooling medium in overhead total condenser, bubble point temperature of distillate must be sufficiently greater than design temperature of cooling water from cooling tower (say 32°C). Also to facilitate the storage of liquid distillate without using insulation and refrigeration system, bubble point temperature must be sufficiently greater than atmospheric temperature.

Let bubble point of distillate = 60°C

For the first trial calculations, assuming that non-key components, propane will appear in distillate only and hexane will appear in residue only. For the first trial calculations material balance is assumed as follows.

Table 8.1 Material Balance of Distillation Column (1st Trial Calculations)

Component	Distillate, kmol/h	Residue, kmol/h
<i>n</i> -Propane	32.0	—
<i>n</i> -Butane	19.8	0.2
<i>n</i> -Pentane	0.2	17.8
<i>n</i> -Hexane	—	30.0
Total	52.0	48.0

Bubble point temperature of distillate = 60°C. At bubble point, $\sum K_i x_i = 1$

$$K_{Pr} x_{Pr} + K_{Pe} x_{Pe} + K_{Bu} x_{Bu} = 1$$

$$x_{Pr} = \frac{32}{52} = 0.6154, x_{Pe} = \frac{0.2}{52} = 0.0038, x_{Bu} = \frac{19.8}{52} = 0.381$$

$$0.6154 K_{Pr} + 0.0038 K_{Pe} + 0.381 K_{Bu} = 1 \quad (a)$$

Here values of equilibrium constants are to be determined at the temperature 60°C and operating pressure p_t which is unknown. Hence by trial and error calculations find the value of p_t which satisfy Eq. (a). Values of equilibrium constants are determined by using Fig. 6.20.

$$p_t = 1500 \text{ kPa and } t = 60^\circ\text{C}$$

$$K_{Pr} = 1.4, K_{Pe} = 0.21, K_{Bu} = 0.52$$

$$\sum K_i x_i = 0.6154 \times 1.4 + 0.0038 \times 0.21 + 0.381 \times 0.52 = 1.06$$

At 1600 kPa and 60°C,

$$K_{Pr} = 1.35, K_{Pe} = 0.2, K_{Bu} = 0.5$$

$$\sum K_i x_i = 0.6154 \times 1.35 + 0.0038 \times 0.2 + 0.38 \times 0.5 = 1.022 \approx 1$$

Operating pressure at top of column = 1600 kPa

Assume pressure drop in column = 30 kPa

Operating pressure at the base of column = 1630 kPa

To decide the distribution of non-key components Hegstebeck-Gedde's equation can be used.

$$\log \left(\frac{d_i}{b_i} \right) = A + C \log \infty_i \quad (8.7)$$

$$\text{where, } \infty_i = \infty_{\text{avi}} = (\infty_{i,\text{top}} \cdot \infty_{i,\text{bottom}})^{1/2} \quad (8.8)$$

To find out the average value of relative volatility, top and base (bottom) temperatures of distillation column are to be determined.

For distillation column with total condenser, composition of overhead vapour and distillate are same. Top most temperature of distillation column is equal to dew point temperature of overhead vapour.

Top most temperature:

$$\text{At dew point, } \sum \frac{y_i}{K_i} = 1$$

$$\frac{0.6154}{K_{Pr}} + \frac{0.381}{K_{Bu}} + \frac{0.0038}{K_{Pe}} = 1$$

At operating pressure $p_t = 1600$ kPa and at temperature t , values of equilibrium constants must be determined, where t is unknown.

For the first trial calculations, Let $t = 75^\circ\text{C}$

$$\text{At } 75^\circ\text{C, } K_{Pr} = 1.65, K_{Bu} = 0.65, K_{Pe} = 0.26$$

$$\sum \frac{y_i}{K_i} = \frac{0.6154}{1.65} + \frac{0.381}{0.65} + \frac{0.0038}{0.26} = 0.974$$

$$\text{At } 72^\circ\text{C, } K_{Pr} = 1.55, K_{Bu} = 0.6, K_{Pe} = 0.25$$

$$\sum \frac{y_i}{K_i} = \frac{0.6154}{1.55} + \frac{0.381}{0.6} + \frac{0.0038}{0.25} \approx 1$$

Bottom most temperature of distillation column is the bubble point of residue. Operating pressure at the base of column $p'_t = 1630 \text{ kPa}$.

At bubble point $\sum K_i x_i = 1$

$$\text{For residue, } x_{\text{Bu}} = \frac{0.2}{48} = 0.00417, x_{\text{Pe}} = \frac{17.8}{48} = 0.37, x_{\text{He}} = \frac{30}{48} = 0.625$$

For the first trial calculations, let $t = 150^\circ\text{C}$ (and at 1630 kPa)

$$K_{\text{Bu}} = 1.9, K_{\text{Pe}} = 0.95, K_{\text{He}} = 0.52$$

$$\sum K_i x_i = 0.00417 \times 1.9 + 0.37 \times 0.95 + 0.625 \times 0.52 = 0.684$$

$$\text{Let } t = 180^\circ\text{C}. K_{\text{Bu}} = 2.5, K_{\text{Pe}} = 1.3, K_{\text{He}} = 0.79$$

$$\sum K_i x_i = 0.00417 \times 2.5 + 0.37 \times 1.3 + 0.625 \times 0.79 = 0.99 \approx 1$$

Table 8.2 Relative Volatilities

Feed Components	$\infty_{72^\circ\text{ C}}$	$\infty_{180^\circ\text{ C}}$	∞_{avi}
n-Propane	$\frac{1.55}{0.25} = 6.20$	$\frac{4.25}{1.3} = 3.270$	4.500
n-Butane (LK)	$\frac{0.6}{0.25} = 2.40$	$\frac{2.5}{1.3} = 1.923$	2.150
n-Pentane (HK)	1.00	1.000	1.000
n-Hexane	$\frac{0.11}{0.25} = 0.44$	$\frac{0.79}{1.3} = 0.6077$	0.517

At 72°C , $K_{\text{He}} = 0.11$ and at 180°C , $K_{\text{Pr}} = 4.25$.

For butane (light key component):

$$\log\left(\frac{d_i}{b_i}\right) = A + C \log \infty_i$$

$$\log\left(\frac{19.8}{0.2}\right) = A + C \log (2.15)$$

$$1.9956 = A + 0.3324 C$$

For pentane (heavy key component)

$$\log\left(\frac{0.2}{17.8}\right) = A + C \log (1)$$

$$-1.94939 = A + 0$$

Solving two equations

$$A = -1.94939$$

$$C = 11.8682$$

For propane:

$$\log\left(\frac{d_i}{b_i}\right) = -1.94939 + 11.8682 \log (4.5)$$

$$\log\left(\frac{d_i}{b_i}\right) = 5.803$$

$$\left(\frac{d_i}{b_i} \right) = 635\ 331 \text{ (very high)}$$

$$\begin{aligned} d_i &= 32 \text{ kmol/h} \\ b_i &= 5 \times 10^{-5} \text{ kmol/h} \approx 0 \text{ kmol/h} \end{aligned}$$

For hexane:

$$\log \left(\frac{d_i}{b_i} \right) = -1.949\ 39 + 11.8682 \log (0.517)$$

$$\left(\frac{d_i}{b_i} \right) = 4.47 \times 10^{-6}$$

$$\begin{aligned} b_i &= 30 \text{ kmol/h} \\ d_i &= 1.34 \times 10^{-4} \text{ kmol/h} = 0 \text{ kmol/h} \end{aligned}$$

Propane does not appear in the residue and hexane does not appear in the distillate. Hence, assumption made before deciding the distribution of components is valid. No correction is required. So Table 8.1 shows the final product composition.

8.4.4 Step 4: Minimum Reflux Ratio

Determine the minimum reflux ratio R_m . It is the maximum reflux ratio for which infinite number of trays are required for the desired separation. It corresponds to the minimum condenser cooling load and the minimum reboiler heating load required for the desired separation. In other words on decreasing reflux ratio, a value of reflux ratio obtained for which desired separation become impossible, is called minimum reflux ratio R_m .

8.4.4.1 Determination of R_m for Binary Distillation

- (a) In case of binary distillation with ideal vapour-liquid equilibrium curve or with the equilibrium curve which is always concave downward, R_m can be calculated by any of the following equations¹.
 - (i) For feed liquid at the bubble point or with, the saturated liquid feed (i.e. for $q = 1$)

$$R_m = \frac{1}{\infty - 1} \left[\frac{x_D}{x_F} - \frac{\infty(1 - x_D)}{1 - x_F} \right] \quad (8.9)$$

where, ∞ = Average relative volatility

x_D = Mole fraction of more volatile component in distillate

x_F = Mole fraction of more volatile component in feed liquid

$$q = \frac{H_G - H_F}{H_G - H_L} \quad (8.10)$$

H_G = Enthalpy of saturated vapour, kJ/kmol

H_F = Enthalpy of feed, kJ/kmol

H_L = Enthalpy of saturated liquid, kJ/kmol

- (ii) For the saturated vapour feed or for feed vapour at dew point (i.e. for $q = 0$)

$$R_m = \frac{1}{\infty - 1} \left[\frac{\infty x_D}{y_F} - \frac{1 - x_D}{1 - y_F} \right] - 1 \quad (8.11)$$

where, y_F = Mole fraction of more volatile component in feed vapour

- (iii) For any condition of feed (for any value of q), R_m can be determined by solving the following equation.

$$\frac{R_m z_F + q x_D}{R_m (1 - z_F) + q (1 - x_D)} = \frac{\infty [x_D (q - 1) + z_F (R_m + 1)]}{(R_m + 1) (1 - z_F) + (q - 1) (1 - x_D)} \quad (8.12)$$

- (b) In case of binary distillation with ideal or non ideal vapour-liquid equilibrium curve, minimum reflux ratio R_m can be determined by McCabe – Thiele method (Ref. 1, 10). Following assumptions are made in McCabe – Thiele method.

- (i) For any equilibrium stage, molar latent heat of vaporization is equal to molar latent heat of condensation.
- (ii) Heat losses and heat of solution are negligible.
- (iii) Total condenser is used but there is no subcooling. Condenser removes all the latent heat from the overhead vapour but does not cool the resulting liquid further.

Because of the first two assumptions, molar flow rate of liquid and molar flow rate of vapour in each section (enriching section and stripping section) remain constant throughout the section. Because of the third assumption, composition of overhead vapour is equal to composition of distillate and also equal to composition of reflux.

$$L, G, \bar{L}, \bar{G} = \text{constant}$$

$$x_D = y_i = x_o$$

where, L, G = Molar flow rates of liquid and vapour respectively, in rectification or enriching section, kmol/h.

\bar{L}, \bar{G} = Molar flow rate of liquid and vapour respectively in stripping or exhausting section, kmol/h.

x_D = Mole fraction of more volatile component in overhead vapour

x_o = Mole fraction of more volatile component in reflux.

Method for determining R_m by McCabe Thiele method:

- (i) Draw the vapour – liquid equilibrium curve, y vs x . Also, draw the 45° diagonal ($x = y$) line.
- (ii) Mark the points $x = y = x_D$, $x = y = z_F$ and $x = y = x_w$ on the 45° diagonal line.
- (iii) Draw the q -line. It starts from the point $x = y = z_F$. The equation of q -line is

$$y = \frac{q}{q - 1} x - \frac{z_F}{q - 1} \quad (8.13)$$

Slope of q -line is $\frac{q}{q - 1}$.

- (iv) Draw the line from $x = y = x_D$ point to the point of intersection of q -line and equilibrium curve. Extend the same line to y -axis. Intercept of this line gives the value of $\frac{x_D}{R_m + 1}$ from which the value of R_m can be calculated. But this procedure is valid for ideal vapour-liquid equilibrium curves and also for non ideal vapour – liquid equilibrium curves except the curves as shown in Figs 8.1(b) and (c). For the nonideal VLE curve like the Fig. 8.1(b) tangent line of equilibrium curve decides the value of R_m . For the nonideal VLE curve as shown in Fig. 8.1(c), a tangent operating line of stripping section may set the value of R_m .

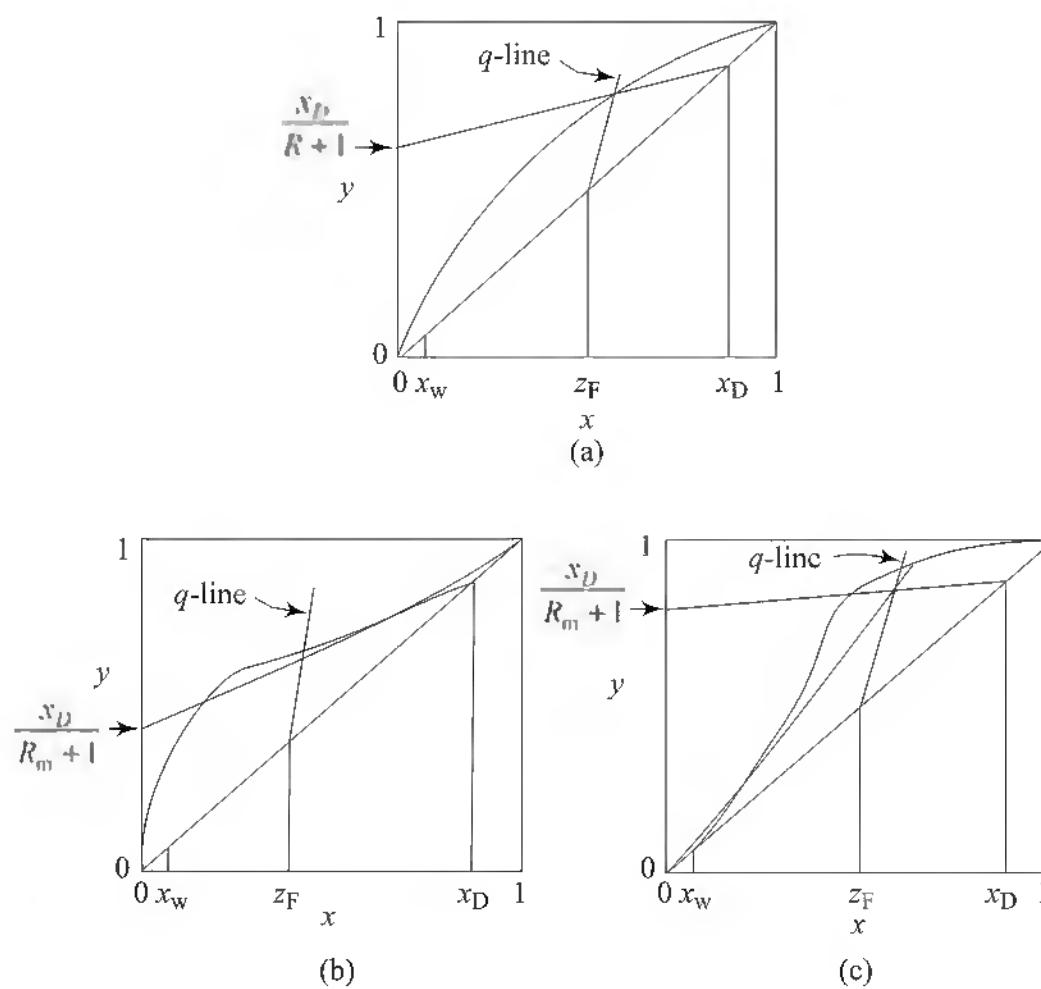


Fig. 8.1 Determination of Minimum Reflux Ratio for Binary Distillation

In case of multicomponent distillation, R_m can be determined by Underwood's method^{1,11}. In this method R_m is determined by solving the following two equations.

$$\sum \frac{\infty_i x_{id}}{\infty_i - \vartheta} = R_m + 1 \quad (8.14)$$

$$\sum \frac{\infty_i x_{if}}{\infty_i - \vartheta} = 1 - q \quad (8.15)$$

where, ∞_i = Average relative volatility of each component i with respect to heavy key

x_{id} = Mole fraction of component i in distillate

x_{if} = Mole fraction of component i in feed

q = Heat required to convert 1 mole of feed from its condition to a saturated vapour divided by latent heat of vaporization.

ϑ = Constant

$$\text{or } q = \frac{H_G - H_F}{H_G - H_L} \quad (8.10)$$

$$\text{Here } \infty_i = (\infty_{i\text{top}} \cdot \infty_{i\text{bottom}})^{1/2} \quad (8.8)$$

Top most temperature of distillation column is dew point temperature of overhead vapour. Bottom most temperature of distillation column is bubble point temperature of residue.

For accuracy purpose, it is suggested that ϑ , must be determined correct upto four decimal places¹², Eq., (8.15) may give many values of ϑ , but correct value of ϑ is that belongs to the interval starting from 1 to the value of ∞_{LK} .

$$\vartheta \in \{\infty_{HK}, \infty_{LK}\} \text{ or } \infty_{LK} > \vartheta > \infty_{HK}$$

where, ∞_{LK} = Relative volatility of light key component with respect to heavy key component.

∞_{HK} = Relative volatility of heavy key component with respect to heavy key component = 1

Example 8.2

Determine the minimum reflux ratio for the binary distillation at standard atmospheric pressure based on the following data.

Feed = 100 kmol/h

Feed mixture: benzene – toluene

Mole fraction of benzene in feed = 0.4

Condition of feed = Saturated liquid

Mole fraction of benzene in distillate required = 0.99

Mole fraction of benzene in Residue required = 0.02

Average relative volatility = 2.25

Solution:

Benzene-toluene mixture behaves as an ideal mixture.

$$R_m = \frac{1}{\infty - 1} = \left[\frac{x_D}{x_F} - \frac{\infty (1 - x_D)}{1 - x_F} \right] \quad (8.9)$$

$$R_m = \frac{1}{2.25 - 1} \left[\frac{0.99}{0.4} - \frac{2.25 (1 - 0.99)}{1 - 0.4} \right]$$

Minimum reflux ratio $R_m = 1.95$

Example 8.3

In the previous example, if the feed is at room temperature (30°C), what is the value of R_m required?

Solution:

To find the value of R_m , first value of q must be determined.

$$q = \frac{H_G - H_F}{H_G - H_L} \quad (8.10)$$

$$\frac{H_G - H_L + (H_L - H_F)}{H_G - H_L} = \frac{\lambda + C_p \Delta t}{\lambda} \quad (8.16)$$

t_F = Temperature of feed = 30°C

t_b = Bubble point temperature of feed

At bubble point:

$$\sum x_i K_i = 1$$

$$\sum x_i \frac{p_{vi}}{p_t} = 1 \text{ (for ideal VLE)}$$

Antoine equations¹³:

$$\text{For benzene, } \ln p_{vB} = 15.9008 - \frac{2788.51}{T - 52.36}$$

$$\text{For toluene, } \ln p_{vT} = 16.0137 - \frac{3096.52}{T - 53.67}$$

For the first trial calculations let $t_B = \sum t_{Bi} x_i$
where t_{Bi} = boiling point of pure component i

Normal boiling point of benzene = 80.1°C

Nomal boiling point of toluene = 110.6°C

For first trial calculations, $t_B = 0.4 \times 80.1 + 0.6 \times 110.6 = 98.4^\circ\text{C}$ (371.4 K)

$$\ln p_{vB} = 15.9008 - \frac{2788.51}{371.4 - 52.36} = 7.16$$

$p_{vB} = 1287.54$ torr, similarly

$p_{vT} = 527.4$ torr

$$\sum x_i p_{vi} = 0.4 \times 1287.54 + 0.6 \times 527.4 = 831.456 \text{ torr} \neq 760 \text{ torr}$$

After trial and error calculations,

at $t_B = 95.3^\circ\text{C}$, $p_{vB} = 1181.72$ torr, $p_{vT} = 479.1$ torr

$$\sum x_i p_{vi} = 0.4 \times 1181.72 + 0.6 \times 479.1 = 760.15 \text{ torr}$$

Bubble point of feed mixture, $t_{BP} = 95.3^\circ\text{C}$

Specific heat must be determined at the average temperature $t_{av} = \frac{95.3 + 30}{2} = 62.65^\circ\text{C}$

Table 8.3 Property Data of Benzene and Toluene

Component	C_L at 62.65 °C, kJ/(kmol · °C)	λ at 95.3°C, kJ/kmol
Benzene	146.96	29 391.3
Toluene	173.33	34 666.7

(Figure 3.11 and Table 3–179, of Ref. 14)

Average molar heat capacity, $C_L = \sum C_{Li} x_i$

$$C_L = 146.96 \times 0.4 + 173.33 \times 0.6 = 162.78 \text{ kJ/(kmol · °C)}$$

Average latent heat

$$\begin{aligned} \lambda &= \sum x_i \lambda_i = 0.4 \times 29 391.3 + 0.6 \times 34 666.7 \\ &= 32 556.54 \text{ kJ/kmol} \end{aligned}$$

$$q = \frac{32\ 556.54 + 162.78 \times (95.3 - 30)}{32\ 556.54}$$

$$= 1.3265$$

R_m can be determined by following equation.

$$\frac{R_m z_F + q x_D}{R_m (1 - z_F) + q (1 - x_D)} = \frac{\infty [x_D (q - 1) + z_F (R_m + 1)]}{(R_m + 1) (1 - z_F) + (q - 1) (1 - x_D)} \quad (8.12)$$

$$\frac{(R_m \times 0.4) + (1.3265 \times 0.99)}{R_m (1 - 0.4) + 1.3265 (1 - 0.99)} = \frac{2.25 [0.99(1.3265 - 1) + 0.4(R_m + 1)]}{(R_m + 1) (1 - 0.4) + (1.3265 - 1) (1 - 0.99)}$$

$$\frac{0.4R_m + 1.313\ 235}{0.6R_m + 0.013\ 265} = \frac{0.7273 + 0.9(R_m + 1)}{0.6(R_m + 1) + 0.003\ 265}$$

After the trial and error calculations, for $R_m = 1.67$, LHS (1.9514) \approx RHS (1.95)
Use of a mathematical software such as Mathcad® or Maple® can be made to solve such an equation.

Minimum reflux ratio, $R_m = 1.67$

Example 8.4

A distillation column is to separate 4750 mol/h of feed composed of 37% *n*-butane, 32% *iso*-pentane, 21% *n*-pentane and 10% *n*-hexane. The column operates at an average pressure of 2 atm and will produce a distillate product containing 95% *n*-butane and 5% *iso*-pentane. The bottom product is allowed to contain no more than 570 mol/h of *n*-butane. Use Underwood's method to determine the minimum reflux for the required separation. Feed is 25% (by mole) vapour. Assume ideal vapour–liquid equilibrium. All compositions are mole %.

Solution:

In this example, distillate composition is given. Hence residue composition can be determined by material balance. Distillate composition indicates that *n*-butane is light key component and *iso*-pentane is heavy key component.

Overall material balance:

$$F = D + W$$

$$4750 = D + W$$

n-butane balance:

$$0.37 \times 4750 = 0.95D + 570$$

$$D = 1250 \text{ mol/h and } W = 3500 \text{ mol/h}$$

Table 8.4 Feed and Product Compositions

Component	Feed		Distillate		Residue	
	mol %	mol/h	mol %	mol/h	mol %	mol/h
<i>n</i> -butane	37	1757.5	95	1187.5	16.3	570.0
<i>iso</i> -pentane	32	1520.0	5	62.5	41.6	1457.5
<i>n</i> -pentane	21	997.5	—	—	28.5	997.5
<i>n</i> -hexane	10	475.0	—	—	13.6	475.0
Total	100	4750.0	100	1250.0	100.0	3500.0

In Underwood's method, to find average relative volatilities, top most and bottom most temperatures of distillation column are required.

Top most temperature = Dew point temperature of overhead vapour
At dew point

$$\sum \frac{y_i p_t}{p_{vi}} = 1 \text{ (for ideal VLE)}$$

$$y_i = x_{iD} \text{ (for total condenser)}$$

$$\left(\frac{0.95}{P_{vnB}} + \frac{0.05}{P_{vip}} \right) \times 2 = 1$$

where, p_{vnB} = Vapour pressure of *n*-butane

p_{vip} = Vapour pressure of *iso*-pentane.

Antoine equations⁹:

n-butane:

$$\ln p_{vnB} = 15.6782 - \frac{2154.9}{T - 34.42}$$

where, T is temperature in K and p_{vnB} is in torr

iso-pentane:

$$\ln p_{vip} = 15.6338 - \frac{2348.67}{T - 40.05}$$

where p_{vip} is in torr.

After a trial and error calculations dew point temperature can be determined.

For $t = 22^\circ\text{C} = 295 \text{ K}$

$$p_{vnB} = 1650 \text{ torr} = 2.17 \text{ atm}$$

$$p_{vip} = 615 \text{ torr} = 0.8 \text{ atm}$$

$$\text{LHS} = \left(\frac{0.95}{2.17} + \frac{0.05}{0.8} \right) \times 2 = 1 = \text{RHS}$$

Dew point temperature of overhead vapour = 22°C .

Bottom most temperature = Bubble point of residue

At bubble point:

$$\sum x_i p_i^{\text{sat}} = p_t \text{ (for ideal VLE)}$$

$$0.163 p_{vnP} + 0.416 p_{vip} + 0.285 p_{vnP} + 0.136 p_{vhH} = 2$$

Antoine equations⁹:

$$\ln p_{vnP} = 15.8333 - \frac{2477.07}{T - 39.94}$$

$$\ln p_{vhH} = 15.8366 - \frac{2697.55}{T - 48.78}$$

where, p_{vnP} = vapour pressure of *n*-pentane, torr

p_{vhH} = vapour pressure of *n*-hexane, torr

T = temperature, K

After a trial and error calculations, final $T = 320\text{ K}$ or $t = 47^\circ\text{C}$

$$p_{vnB} = 3403.3 \text{ torr} = 4.478 \text{ atm}$$

$$p_{viP} = 1399.95 \text{ torr} = 1.842 \text{ atm}$$

$$p_{vnP} = 1084.1 \text{ torr} = 1.426 \text{ atm}$$

$$p_{vnH} = 361.6 \text{ torr} = 0.476 \text{ atm}$$

$$\begin{aligned} \text{LHS} &= 0.163 \times 4.478 + 0.416 \times 1.842 + 0.285 \times 1.426 + 0.136 \times 0.476 \\ &= 1.97 \approx 2 = \text{RHS} \end{aligned}$$

Table 8.5 Relative Volatilities Calculations

Component	$\alpha_{22} = \frac{p_{vi}}{p_{vhK}}$	$\alpha_{47} = \frac{p_{vi}}{p_{vhK}}$	$\alpha_{av} = (\alpha_{22} \times \alpha_{47})^{1/2}$
<i>n</i> -butane (LK)	$\frac{2.17}{0.8} = 2.7125$	$\frac{4.478}{1.842} = 2.43$	2.567
<i>iso</i> -pentane (HK)	$\frac{0.8}{0.8} = 1$	1	1
<i>n</i> -pentane	$\frac{0.6}{0.8} = 0.75$	$\frac{1.426}{1.842} = 0.774$	0.762
<i>n</i> -hexane	$\frac{0.173}{0.8} = 0.216$	$\frac{0.476}{1.842} = 0.258$	0.236

Underwood's equations:

$$\sum \frac{\alpha_i x_{id}}{\alpha_i - \vartheta} = R_m + 1 \quad (8.14)$$

$$\text{and } \sum \frac{\alpha_i z_{if}}{\alpha_i - \vartheta} = 1 - q \quad (8.15)$$

Feed contains 25% vapour

$$H_F = 0.25 \times H_G + 0.75 \times H_L$$

$$q = \frac{H_G - H_F}{H_G - H_L} = \frac{H_G - (0.25H_G + 0.75H_L)}{H_G - H_L} \quad (8.10)$$

$$q = 0.75$$

$$\frac{2.567 \times 0.37}{2.567 - \vartheta} + \frac{1 \times 0.32}{1 - \vartheta} + \frac{0.762 \times 0.21}{0.762 - \vartheta} + \frac{0.236 \times 0.1}{0.236 - \vartheta} = 0.25$$

$$\text{For } \vartheta = 1.6045, f(\vartheta) = 0.25025 = \text{LHS} \approx 0.25 = \text{RHS}$$

Also $\vartheta \in \{1, \dots, 2.567\}$ (check)

$$\sum \frac{\alpha_i x_{id}}{\alpha_i - \vartheta} = R_m + 1 \quad (8.14)$$

$$\frac{2.567 \times 0.95}{2.567 - 1.6045} + \frac{1 \times 0.05}{1 - 1.6045} = R_m + 1$$

$$R_m + 1 = 2.45095$$

$$R_m = 1.45095$$

8.4.5 Step-5: Determination of Optimum Reflux Ratio

Ideally the reflux ratio which should be used for a new design should be optimum or the most economical. Optimum reflux ratio is the value of reflux ratio for which the total cost of distillation is minimum. Value of optimum reflux ratio depends on the type of system or feed mixture, specified separation and operating conditions. Change in desired purity of products changes the value of optimum reflux ratio and also the minimum reflux ratio. Based on cost analysis, optimum reflux ratio is defined as that value of reflux ratio which causes the total variable annual cost (annual fixed charges plus annual operating costs) to be a minimum.

At the minimum reflux ratio, distillation column requires infinite number of trays or infinite packing height and consequently the fixed cost is infinite but minimum operating cost (cost for the heating medium of reboiler, cooling medium for the condenser, power for reflux pump, etc. minimum). As the reflux ratio increases above the minimum, number of trays required or packing height required rapidly decreases but column diameter, sizes of condenser and reboiler, and size of reflux pump increases. Despite the increase in column diameter and increase in sizes of condenser and reboiler, the annual fixed cost for the distillation goes down as the increase in reflux ratio above minimum results in saving in tower height which more than offsets other fixed costs. This is not continued as the reflux ratio increases further. At certain value of reflux ratio the tray requirement or height of packing requirement approaches a near constant value characteristic of the minimum trays or height requirement, while the diameter and sizes of heat exchangers continue to increase and hence at some value of reflux ratio, fixed cost passes through minimum point. Increase in the value of reflux ratio further increases the fixed cost again. Operating cost always increases with increase in reflux ratio. The total annual cost, which is sum of operating and fixed costs, must therefore pass through a minimum at the optimum reflux ratio.

For many hydrocarbon distillations, optimum reflux ratio lie between 1.2 to 1.5 times the minimum reflux ratio. But for the difficult distillation, like distillation of acetic acid – water system, optimum reflux ratios are higher than 1.5. For azeotropic mixtures, reflux ratio is usually high. For the optimum design of distillation column, optimum values of reflux ratio, operating pressure and purity of products must be determined.

8.4.5.1 Determination of Theoretical Stages

To determine the number of theoretical stages or equilibrium stages, required for

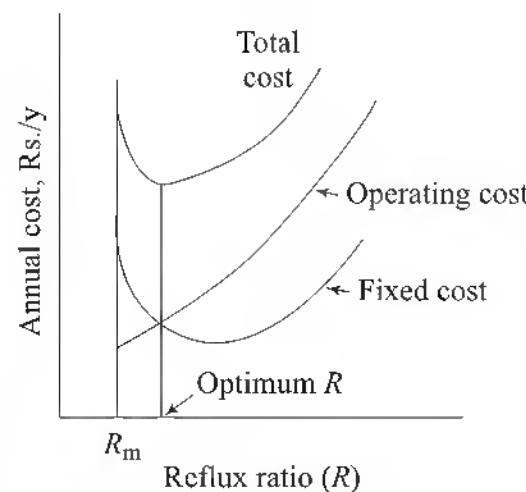


Fig. 8.2 Determination of Optimum Reflux Ratio

desired separation, many methods are available. But they can be broadly divided in two groups.

- (a) Methods valid for binary distillation
- (b) Methods valid for multicomponent distillation

(a) Methods Valid for Binary Distillation: Both short cut methods and detailed methods are available. Short cut methods available for binary distillation are Fensky's equation¹, Smoker's equation⁹, etc. Detailed methods available for binary distillation are McCabe–Thiele method, Ponchon–Savarit method¹, etc. Also to find the number of theoretical stages for high purity products, Kremser equation¹, Robinson and Gilliland equation⁹, etc. are available.

A. McCabe–Thiele Method^{1, 10}

Assumptions made in McCabe–Theile method are discussed in the previous section. McCabe–Thiele method cannot be used for the following cases.

- (i) Cases where heat of solution or heat losses are significantly large. Example: Separation of ammonia from dilute aqueous solution by distillation.
 - (ii) For the cases in which high purity of products are required. In such cases other method is used to support the McCabe–Thiele method. However, for the same cases one can use only McCabe–Thiele method, but more than one graphs are to be drawn at different scales. Stepwise procedure for applying McCabe–Thiele method is as follows.
1. Draw the vapour liquid equilibrium curve from the VLE data available at the operating pressure of distillation column.
 2. Mark the point x_D , z_F , and x_w on 45° diagonal line.

$$3. \text{ Draw the } q\text{-line. The equation of } q\text{-line is } y = \frac{q}{q-1}x - \frac{z_F}{q-1} \quad (8.13)$$

It starts from $x = y = z_F$ point and its slope is $q/(q-1)$, where q is the heat required to convert the feed from its condition to saturated vapour divided by molar latent heat of vaporization.

4. Determine the minimum reflux ratio R_m .
5. Select the value of actual reflux ratio R . Draw the enriching section operating line. The equation of this line is

$$y = \frac{R}{R+1}x + \frac{x_D}{R+1} \quad (8.17)$$

This line starts from $x = y = x_D$ point. Its intercept is $x_D/(R+1)$

6. Draw the stripping or exhausting section operating line. It starts from the point $x = y = x_w$ pass through the point of intersection of enriching section operating line and the q line.
7. Starting from x_D to x_W or from x_W to x_D , carry out the stepwise construction between operating lines and equilibrium curve. Total number of steps is equal to number of theoretical or equilibrium stages, required for the desired separation.

Example 8.5

Acetic acid is to be separated from a process stream containing 80% acetic acid and 20% water (by mass) by continuous distillation at atmospheric pressure. Concentration of water in bottom product (pure acetic acid) should not be greater than 50 ppm. Top product (distillate) contains 80% water and 20% acetic acid (by mass). The feed is liquid at 30°C. Estimate the number of theoretical stages required.

Table 8.6 Vapour–Liquid Equilibrium Data (t-x-y Data) at 101.325 kPa

Temperature, °C	Mole fraction of water in liquid, x	Mole fraction of water in vapour, y
118.3	0	0
110.6	0.1881	0.3063
107.8	0.3084	0.4467
105.2	0.4498	0.5973
104.3	0.5195	0.6580
103.5	0.5824	0.7112
102.8	0.6750	0.7797
102.1	0.7261	0.8239
101.5	0.7951	0.8671
100.8	0.8556	0.9042
100.8	0.8787	0.9186
100.5	0.9134	0.9409
100.2	0.9578	0.9708
100.0	1.0000	1.0000

(Table 13.1 of Ref. 14)

Solution:

Feed contains 20% water and 80% acetic acid by mass. Molar mass of water is 18 and molar mass of acetic acid is 60. Water is the lighter component.

Mole fraction of water in feed.

$$x_F = \frac{\left(\frac{20}{18}\right)}{\left(\frac{20}{18}\right) + \left(\frac{80}{60}\right)} = 0.4545$$

Mole fraction of water in distillate

$$x_D = \frac{\left(\frac{80}{18}\right)}{\left(\frac{80}{18}\right) + \left(\frac{20}{60}\right)} = 0.9302$$

Mole fraction of water in bottom product

$$x_w = \frac{\left(50 \times 10^{-6} / 18\right)}{\left(\frac{50 \times 10^{-6}}{18}\right) + \left(\frac{1 - 50 \times 10^{-6}}{60}\right)} = 1.666 \times 10^{-4}$$

***q*-line:**

Feed is liquid at 30°C temperature. From *t-x-y* data, bubble point of feed can be easily determined. t = bubble point temperature of feed when $x = x_F$. For $x = 0.4545$ corresponding value of $t = 105.14^\circ\text{C}$.

Bubble point of feed = 105.14°C

Latent heat of water at 105.14°C, $\lambda_w = 40\ 392\ \text{kJ/kmol}$

Latent heat of acetic acid at 105.14°C, $\lambda_A = 20\ 096.6\ \text{kJ/kmol}$

$$\text{Specific heat at } \frac{30 + 105.14}{2} = 67.57^\circ\text{C}$$

$$C_{pW} = 75.36\ \text{kJ/(kmol} \cdot {}^\circ\text{C})$$

$$C_{pA} = 131.88\ \text{kJ/(kmol} \cdot {}^\circ\text{C})$$

Latent heat of feed, $\lambda_F = 0.4545 \times 40\ 392 + (1 - 0.4545) \times 20\ 096.6 = 29\ 320.86\ \text{kJ/kmol}$

Mean specific heat of feed,

$$C_{pF} = 0.4545 \times 75.36 + (1 - 0.4545) \times 131.88 = 106.19\ \text{kJ/(kmol} \cdot {}^\circ\text{C})$$

$$q = \frac{H_G - H_F}{H_G - H_L} = \frac{(H_G - H_L) + (H_L - H_F)}{H_G - H_L} = \frac{\lambda_F + C_{pF}(t_{BP} - t_F)}{\lambda_F}$$

$$q = \frac{29\ 320.86 + 106.19(105.14 - 30)}{29\ 320.86}$$

$$q = 1.272$$

Equation of *q*-line:

$$y = \frac{q}{q-1}x - \frac{z_F}{q-1} \quad (8.13)$$

$$y = \frac{1.272}{0.272}x - \frac{0.4545}{0.272}$$

$$y = 4.676\ 47x - 1.67$$

q-line starts from $x = y = z_F$ or $x = y = 0.4545$ point.

To find another point, when $y = 0.6$, $x = 0.4854$ using *q*-line equation.

q-line passes through (0.4854, 0.6) point.

Draw the *q*-line in the graph. From Fig. (8.3), intercept for the operating line at mini-

$$\text{mum reflux, } 0.304 = \frac{x_D}{R_m + 1}$$

$$\frac{0.9302}{R_m + 1} = 0.3$$

$$R_m = 2.1$$

Let

$$R = 2 R_m = 2 \times 2.1 = 4.2$$

$$\frac{x_D}{R+1} = \frac{0.9302}{4.2+1} = 0.1789$$

On stagewise construction starting from $x = y = x_D$, as shown in Fig. (8.3), up to $x = 0.02$, number of theoretical or equilibrium stages required is 16 in which 8th theoretical stage from the top is corresponding to feed tray.

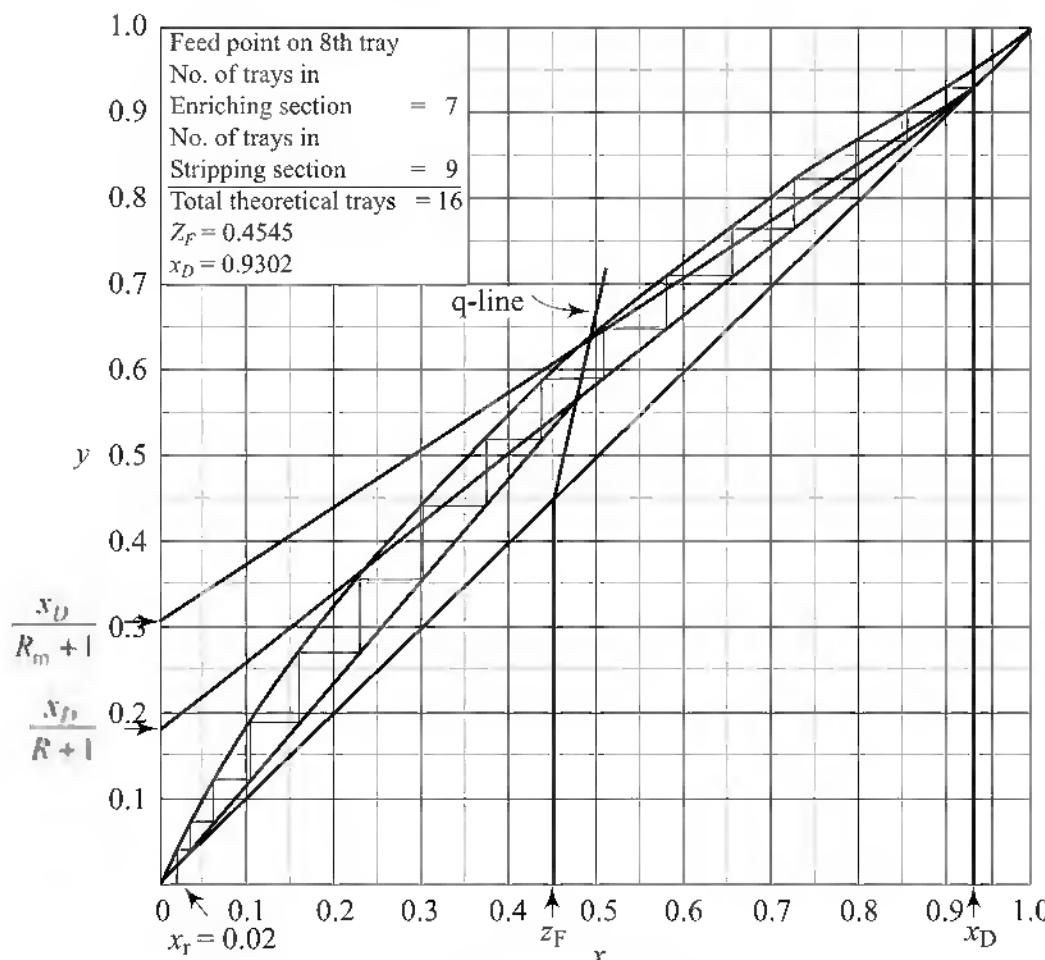


Fig. 8.3 McCabe-Thiele Diagram for Acetic Acid-Water System

Similarly, for the different values of R , number of equilibrium stages, N , required for the same separation are determined and listed in the following table.

Table 8.7 Values of N for Different values of R/R_m

R/R_m	2	2.5	3	3.5	4
N	16	15.0	14	14.0	13.5

On increasing the value of R/R_m above 2, decrease in the value of N is very marginal. Hence, $R = 2 R_m$ is considered to be nearer to the optimum value.

To find the number of equilibrium stages required to increase the purity of bottom product (acetic acid) from $x = 0.02$ to $x_w = 1.666 \times 10^{-4}$, Robinson and Gilliland equation⁹ can be used. For the stripping section, Robinson – Gilliland correlation is

$$N_s = \frac{\log \left[\frac{(K'/S' - 1)(x'_r/x_w - 1)}{1/S'(K' - 1)} + 1 \right]}{\log (K'/S')} + 1 \quad (8.18)$$

where,

N_s = Number of ideal stages required from the reference point x'_r to x_w

x_w = Mole fraction of the more volatile component in the residue (bottom product)

x'_r = Mole fraction of more volatile component at the reference point

S' = slope of the bottom section operating line

K' = equilibrium constant for more volatile component

For the enriching section, Robinson and Gilliland correlation is

$$N_r = \frac{\log \left[\frac{(1-S) + \frac{x_r}{x_d'} (S-K)}{1-K} \right]}{\log \left(\frac{S}{K} \right)} - 1 \quad (8.19)$$

where,

N_r = Number of ideal stages required from reference point x_r to x_d'

x_d' = Mole fraction of least volatile component in distillate

x_r = Mole fraction of least volatile component at reference point

K = Equilibrium constant for the least volatile component

S = Slope of enriching section operating line

Here, in this example, Eq. (8.18) can be used to find the number of theoretical stages required to purify the bottom product (acetic acid) from $x_r' = 0.02$ to $x_w = 1.666 \times 10^{-4}$.

S' = Slope of stripping section operating line = 1.2

K' = Slope of equilibrium line in the portion from 0.02 to 0. [From Fig. (8.3)]
= 2.5

$$N_s = \frac{\log \left\{ \frac{[(2.5/1.2) - 1][(0.02/1.666 \times 10^{-4}) - 1]}{(1/1.2)(2.5 - 1)} + 1 \right\}}{\log (2.5/1.2)} + 1$$

$$N_s = \frac{\log (104.17495)}{\log (2.08333)} + 1 = 7.33$$

As discussed earlier, second graph with different scale can be drawn for finding theoretical stages in the stripping section below $x_r = 0.02$. Actual plotting also gives additional stages of 7.

Total number of theoretical stages required for desired separation = $16 + 7.33 = 23.33$, say 24.

Note: It can be seen that nearly 30% theoretical stages are required to reduce the water content in the bottom product 0.02 mole % to 50 mg/L.

As stated earlier for the separation of pure acetic acid from its dilute aqueous solution containing less than 30% by mass acetic acid, liquid-liquid extraction and azeotropic distillation are more economical than ordinary distillation. Figure 8.4 shows that for separating nearly pure water and pure acetic acid from 25.18% by mass acetic acid solution, $22 + 7 = 29$ theoretical stages required for the value of reflux ratio $R = 3.5823$. Comparison between the separation of 80% acetic acid solution and 25.18% acetetic acid solution is given in following table.

Table 8.8 shows that for the separation of dilute solution about 20% more stages and approximately three times higher condenser duty and reboiler duty are required. Hence, for the separation of dilute aqueous acetic acid solution liquid-liquid extraction or azeotropic distillation is more economical.

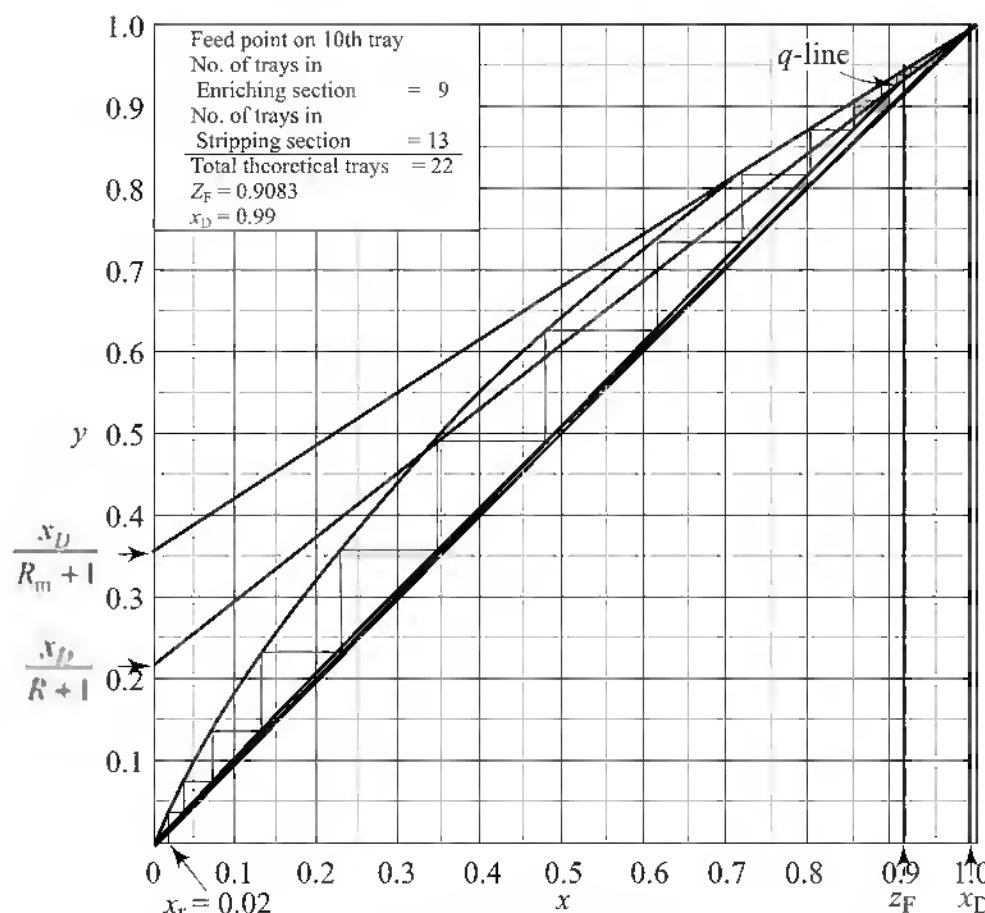


Fig. 8.4 McCabe-Thiele Diagram for Acetic Acid-Water System

Table 8.8 Comparison between Two Cases

	Feed flow rate, kg/h	% by mass acetic acid	R	N	Distillate kg /h	λ_{av} kJ/h	Condenser duty, kW	Reboiler duty, kW
I st	12 000	80	4.2	23	3000	1886.7	8 175.7	9 000
II nd	12 000	25.18	3.5813	29	8978	2256.9	25 785.7	29 000

(Condenser duty, $\phi_C = (R + 1) D \lambda_{av}$)

B. Use of Open Steam

While distilling an aqueous solution, if nonaqueous component is more volatile than water is removed as residue product. In such a case instead of using separate reboiler at bottom, heat required can be provided by admission of steam directly to the bottom of tower. For the same value of reflux ratio and the given extent of separation, use of open steam requires more number of trays or equilibrium stages. However, it is usually cheaper than the separate reboiler system as it avoids fixed cost and cleaning cost of reboiler. It also facilitates the use of lower pressure steam as heating medium but with the use of open steam, condensate cannot be recycled back to boiler feed tank.

Overall material balance,

$$F + S = D + W \quad (8.20)$$

where, S = Molar flow rate of saturated steam entering at bottom, kmol/h

More volatile component balance

$$Fz_F = D x_D + W x_W \quad (8.6)$$

In this case stripping section operating line starts from ($x = x_w$, $y = 0$) point and has a slope equal to \bar{L}/S or W/S .

where, \bar{L} = Molar flow rate of liquid in stripping section, kmol/h
 $= W$, molar flow rate of residue, kmol/h

If superheated steam is used as open steam, then S and \bar{L} are calculated by the following equations.

$$S = S_s \left(1 + \frac{H_s - H_{\text{sat}}}{\lambda M} \right) \quad (8.21)$$

$$\bar{L} = S - S_s + W \quad (8.22)$$

where, S_s = Molar flow rate of superheated steam, kmol/h

H_s = Enthalpy of superheated steam, kJ/mol

H_{sat} = Enthalpy of saturated steam, kJ/mol

λ = Latent heat of vaporization at operating temperature, kJ/kg

M = Molar mass of water = 18.0153 kg/kmol

Example 8.6

Aqueous solution of ethanol contains 6 % by mass ethanol. It is to be concentrated in the distillation column to 30 % ethanol (by mass). Open steam, saturated at 80 kPa g pressure will be used for the distillation column. Feed flow rate is 5000 kg/h and it is saturated liquid at its bubble point. Residue should not contain more than 0.02 mass per cent ethanol. Sparging steam flow is 0.2 kg per kg of feed. Calculate the number of theoretical stages required for the desired separation.

Solution:

Basis: 5000 kg/h feed

$$F = \frac{5000 \times 0.06}{46} + \frac{5000 \times 0.94}{18} = 6.52 + 261.11 = 267.63 \text{ kmol/h}$$

$$S = (0.2 \times 5000)/18 = 55.556 \text{ kmol/h}$$

$$F + S = D + W \quad (8.20)$$

$$D + W = 323.186 \text{ kmol/h}$$

$$z_F = \frac{(0.06/46)}{(0.06/46) + (0.94/18)} = 0.02437$$

$$x_D = \frac{(0.3/46)}{(0.3/46) + (0.7/18)} = 0.1436$$

$$x_W = \frac{0.0002/46}{(0.0002/46) + (0.9998/18)} = 7.827 \times 10^{-5}$$

$$Fz_F = D x_D + W x_W \quad (8.6)$$

$$267.63 \times 0.02437 = 0.1436 D + 7.827 \times 10^{-5} W$$

$$6.522 = 0.1436 D + 7.827 \times 10^{-5} (323.186 - D)$$

$$D = 45.266 \text{ kmol/h}$$

$$W = 277.92 \text{ kmol/h}$$

Feed is saturated liquid at its bubble point.

Hence, $q = 1$

$$\bar{L} = L + Fq = L + F = W$$

$$\bar{G} = G + F(q - 1) = G$$

$$S = \bar{G} = G = 55.556 \text{ kmol/h}$$

$$G = (R + 1) D$$

$$55.556 = (R + 1) 45.266$$

$$R = 0.2273$$

Table 8.9 Equilibrium Data for Ethanol-Water System at 101.3 kPa

x	0.019	0.0721	0.0966	0.1238	0.1661
y	0.17	0.3891	0.4375	0.4704	0.5089

(Table 13.1 of Ref. 14)

Intercept of enriching section

$$\text{operating line} = x_D/(R + 1) = 0.1436/(0.2273 + 1) = 0.117$$

$$\text{Slope of stripping section operating line} = \bar{L}/S = W/S = \frac{277.92}{55.556} = 5$$

Stripping section operating line starts from $(x_w, 0)$ point.

From Fig. 8.5, number of equilibrium stages required is $N = 7$.

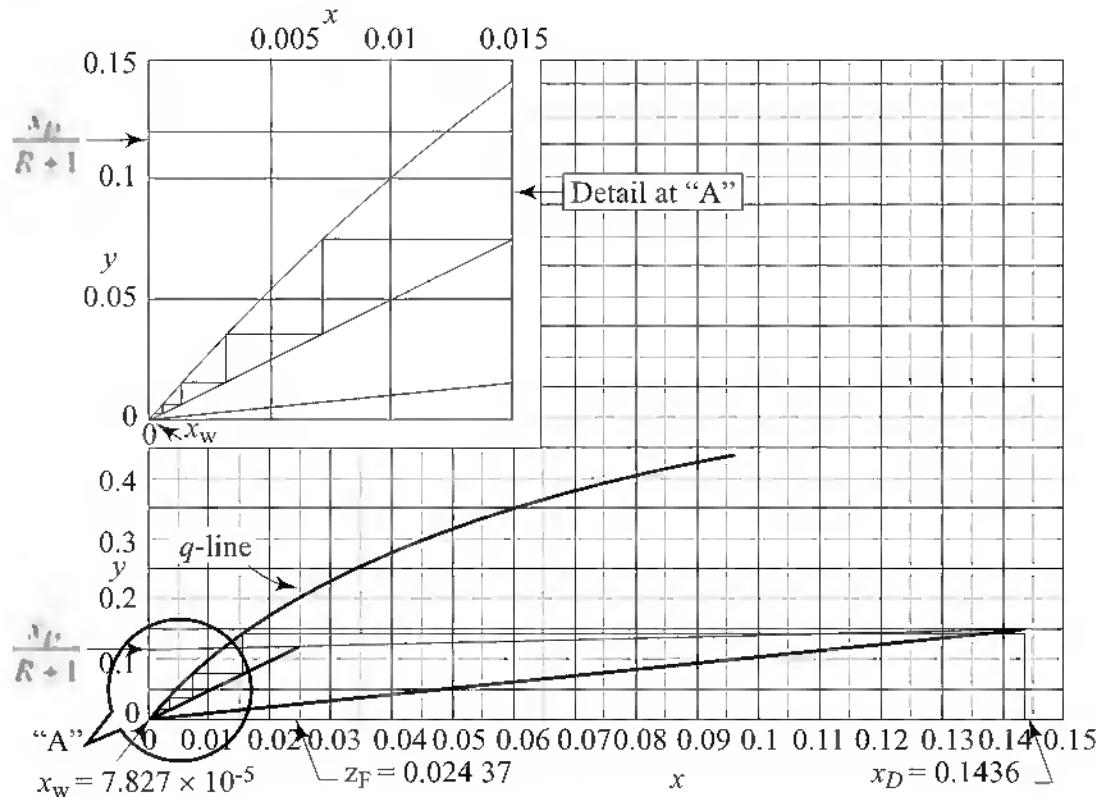


Fig. 8.5 McCabe-Thiele Diagram for Steam Stripping of Ethanol-Water Solution

C. Ponchon-Savarit Method

This method is more rigorous than McCabe-Thile method. This method requires detailed enthalpy data and it is particularly applied to a binary system in which heat of solution is significant. This method is explained by following illustration.

Example 8.7

Determine the minimum reflux ratio and number of theoretical stages required for the distillation column of ammonia–water absorption refrigeration plant based on following data. Also find the heat duty of overhead condenser ϕ_C and reboiler heat duty ϕ_B for the given value of reflux ratio R .

Data: (Example 8.4 of Ref. 13)

(a) Feed:

Feed is strong aqueous (SA) ammonia solution which enters the distillation column at 102.5°C

Enthalpy of feed = 325.2 kJ/kg

Mass % of ammonia in SA = 36.3%

Feed flow rate = 7523.28 kg/h

(b) Distillate: 100% pure ammonia in the form of saturated liquid at 40°C

(c) Residue: Weak aqueous (WA) solution of ammonia, having 29.8 % strength by mass.

(d) Operating pressure of column = 1555.5 kPa a

(e) Cooling water at 32°C is used as cooling medium in overhead condenser and saturated steam at 3 bar a is used as heating medium in generator (reboiler).

(f) H - x - y data (Fig. 5.19, of Ref. 13)

Table 8.10 Enthalpies of Saturated Liquid and Vapour Mixtures

x, y	H_L , kJ/kmol	H_G , kJ/kmol
0	15 480.7	50 310.7
0.15	10 887.3	48 194.8
0.30	7 519.0	45 665.1
0.40	5 808.0	43 985.9
0.45	5 614.9	42 997.0
0.50	4 900.0	42 078.8
0.60	4 523.2	40 024.9
0.70	5 189.7	38 234.3
0.80	6 449.3	36 122.0
0.90	7 181.6	33 334.7
1.00	9 159.6	27 832.4

where,

H_L = Enthalpy of saturated liquid at 1555.5 kPa a pressure, kJ/kmol

H_G = Enthalpy of saturated vapour at 1555.5 kPa a pressure, kJ/kmol

x, y = Mole fraction of ammonia in ammonia-water liquid solution and in ammonia–steam mixture, respectively.

(g) VLE data at $p_t = 1555.5$ kPa a

(Table 3.23 and Table 3.24 of Ref. 14)

Table 8.11 Equilibrium Data for Ammonia-Water System

<i>x</i>	<i>y</i>
0	0
0.30	0.9120
0.35	0.9456
0.40	0.9690
0.45	0.9800
0.50	0.9880
0.60	0.9950
0.75	0.9980
1.00	1.0000

- (h) Actual or operating reflux ratio, $R = 0.3137$ which is nearly equal to 0.08 kg per kg ammonia (as NH_3) in the feed.

Solution:

Material balance

Feed flow rate, $\dot{m}_F = 7523.28 \text{ kg/h}$

$$x_F = \frac{0.363/17}{(0.363/17) + (0.637/18)} = 0.3763$$

Average molar mass of feed

$$\begin{aligned} M_{av} &= \sum M_i x_i \\ M_{av} &= 17(0.3763) + 18(1 - 0.3763) = 17.6237 \text{ kg/kmol} \end{aligned}$$

$$F = \frac{7523.28}{17.6237} = 426.88 \text{ kmol/h}$$

$$x_W = \frac{0.298/17}{(0.298/17) + (0.702/18)} = 0.31$$

$$x_D \approx 1$$

$$F = D + W \text{ or } 426.88 = D + W \quad (\text{a})$$

Ammonia balance:

$$\begin{aligned} Fx_F &= D x_D + W x_W \\ 426.88 \times 0.3763 &= D + 0.31 W \\ D &= 41.0176 \text{ kmol/h}, W = 385.8624 \text{ kmol/h} \end{aligned}$$

Minimum reflux ratio, R_m :

To find R_m , in $H - x, y$ diagram, draw the tie-lines. (Line connecting equilibrium values of x and y is called tie-line.) Extend the tie lines and allow to intersect $x = x_D$ and $x = x_W$ lines. Intersection point with $x = x_D$ is represented as ΔD and with $x = x_W$ as ΔW . Extended tie lines which give the farthest locations of ΔD_m and ΔW_m and are corresponding to minimum reflux ratio. But if x, y equilibrium curve is everywhere concave downward, then a tie line which when extended passes through F corresponds to minimum amount of reflux.

Here x, y equilibrium curve is everywhere concave downward. Hence, the minimum reflux ratio is established by the tie line which when extended passes through F .

Mark the point F in $H-x, y$ diagram. $x_F = 0.3763$ and $H_F = 325.2 \times 17.6237 = 5731.23 \text{ kJ/kmol}$.

Find the tie line which when extended, passes through point F . One method is, draw the two nearest tie lines from the given equilibrium data. Here, these two tie lines are (i) $x = 0.35$ and $y = 0.9456$, and (ii) $x = 0.4$ and $y = 0.969$. Then draw the line having average slope and passes through point F . Extend this tie line and allow it to intersect $x = x_D$ line. Intersection point is ΔD_m . From Fig. 8.4 at ΔD_m , $\varphi_m = 31\ 818 \text{ kJ/kmol}$.

$$R_m = \frac{\varphi_m - H_{G1}}{H_{G1} - H_{L0}}$$

$$R_m = \frac{31\ 818 - 27\ 832.4}{27\ 832.4 - 9159.6} = 0.213\ 44$$

$$R_m = 0.213\ 44$$

Actual R is 0.3137. Thus actual reflux ratio is 1.47 times the minimum reflux ratio.
For $R = 0.3137$

$$R = \frac{\varphi - H_{G1}}{H_{G1} - H_{L0}}$$

$$0.3137 = \frac{\varphi - 27\ 832.4}{27\ 832.4 - 9159.6}$$

$$\varphi = 33\ 690 \text{ kJ/kmol}$$

Mark ΔD on $x = x_D$ line corresponding to the value of φ .

$$FH_F = D\varphi + W\varphi'$$

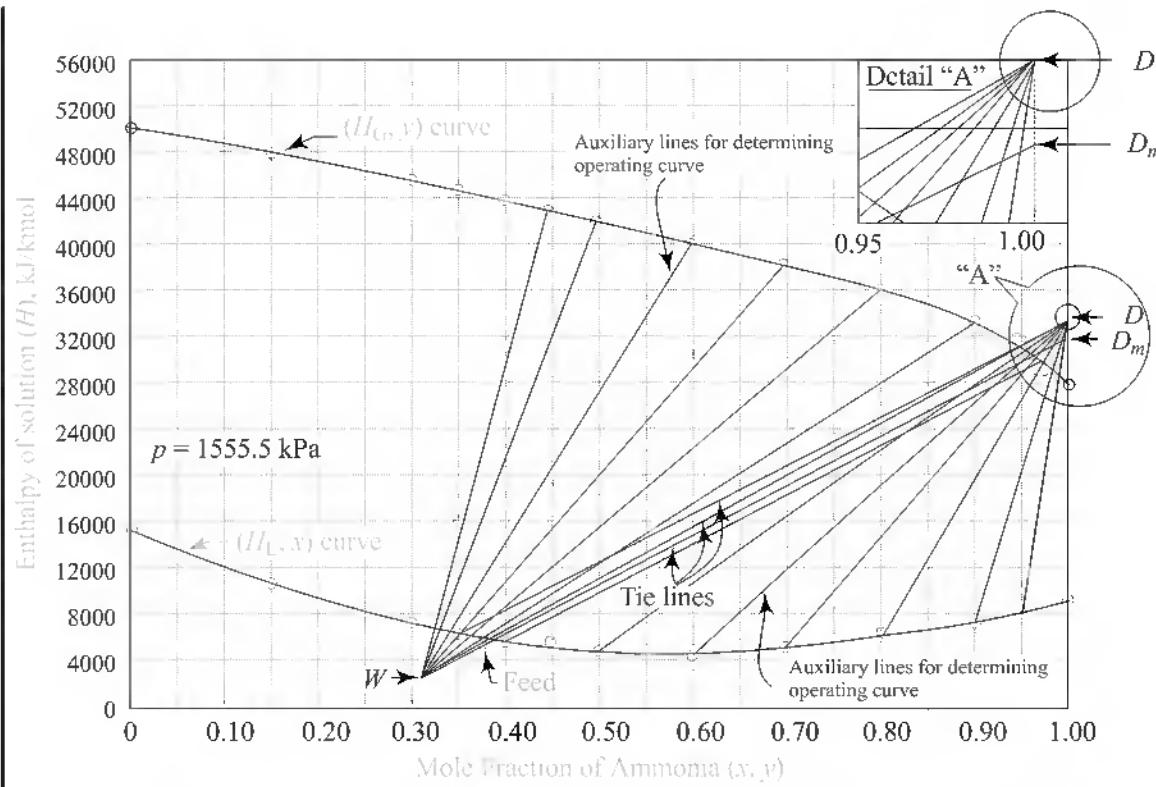
$$426.88 \times 5731.23 = 41.0176 \times 33\ 690 + 385.8624 \varphi'$$

$$\varphi' = 2759.18 \text{ kJ/kmol}$$

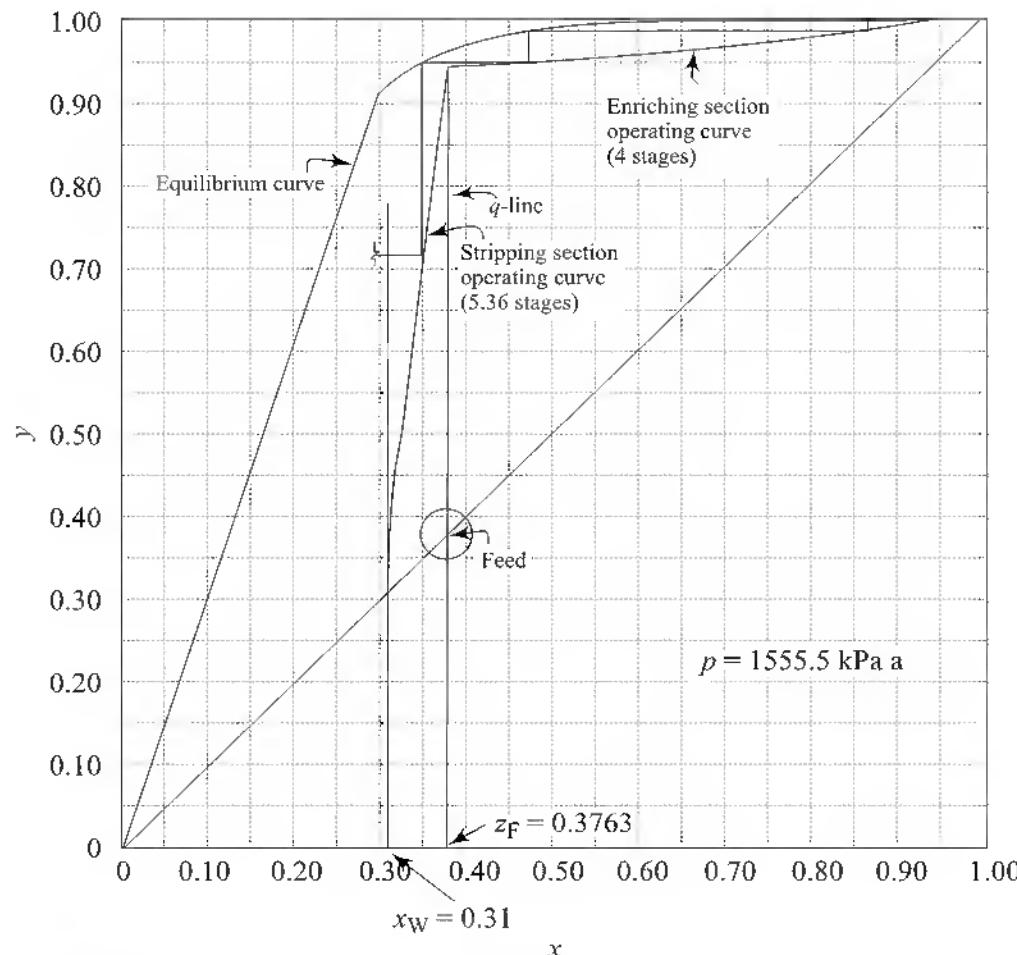
Mark ΔW on $x = x_W$ line corresponding to value of φ' . ΔD , F and ΔW are on the same line. In Fig. (8.6(a)), ΔD at $x_D = 1$, $\varphi = 33\ 690 \text{ kJ/kmol}$ and ΔW at $x_W = 0.31$, $\varphi' = 2759.18 \text{ kJ/kmol}$ are plotted. Auxiliary random lines from the Δ points are drawn and allowed to intersect the saturated vapour and saturated liquid curves at values of y and x , respectively. These (x, y) points are the points of operating curves. Operating curves are plotted in Fig. 8.6(b). Stagewise construction from x_D to x_W between equilibrium curve and operating curve gives number of theoretical stages required for the desired separation.

Table 8.12 x, y Values for Enriching Section Operating Curve and Stripping Section Operating Curve

Enriching		Stripping	
x	y	x	y
1.00	1.000	0.381	0.942
0.95	0.990	0.373	0.900
0.90	0.982	0.360	0.800
0.80	0.970	0.350	0.700
0.70	0.962	0.339	0.600
0.60	0.955	0.329	0.500
0.50	0.949	0.324	0.400
0.381	0.942	0.310	0.310



(a) Enthalpy-Concentration Diagram for Aqueous Ammonia Solution



(b) Theoretical Stage Calculations for Distillation of Aqueous Ammonia Solution

Fig. 8.6 Separation of Ammonia from Aqueous Solution

Here stage wise construction is started from $x_D = 0.99972$ to $x_W = 0.31$. Total number of theoretical stages required (as shown in Fig. 8.6(b)) is 5.36.

$$N = 5.36.$$

$$\varphi = H_D + \frac{\phi_C}{D} = 9159.6 + \frac{\phi_C}{41.0176}$$

$$\phi_C = 1006178.1 \text{ kJ/h} \equiv 279.493 \text{ kW}$$

$$\varphi' = H_W - \frac{\phi_B}{W} = 7347.86 - \frac{\phi_B}{385.8624}$$

$$\phi_B = 1770599.1 \text{ kJ/h} \equiv 491.83 \text{ kW}$$

(b) Methods Valid for Multicomponent Distillation: To determine by number of theoretical stages or equilibrium stages required for desired separation in multicomponent distillation many methods are available. None of them is totally reliable. Hence, for a new case of distillation, it is recommended to use more than one method for the determination of theoretical stages. Also it is better to design the distillation column, first for pilot plant and then modify the design based on the results of pilot plant column rather than designing the column directly for the commercial scale plant. Methods available for multicomponent distillation can be broadly divided into two groups.

- (i) Short cut methods: FUG method, FUE method, Smith–Brinkley method, etc.
- (ii) Rigorous methods: Lewis Matheson method, Thiele–Geddes method, Relaxation methods, Tridiagonal Matrix method, Linear Algebra methods, etc.

A. FUG Method (Fenskey–Underwood–Gilliland's Method):

It is the most widely used short cut method for determining number of equilibrium stages for multicomponent distillation.

In this method first minimum reflux ratio R_m is determined by Underwood's method, which was explained in Sec. 8.4.4.1.

Then Fenskey's equation is used to find the minimum number of theoretical stages required for the desired separation.

Fenskey's equation

$$N_m = \frac{\log \left[\left(\frac{x_{LK}}{x_{HK}} \right)_d \left(\frac{x_{HK}}{x_{LK}} \right)_b \right]}{\log \infty_{LK}} \quad (8.23)$$

where, ∞_{LK} = Average relative volatility of light key with respect to heavy key

$(x_{LK}, x_{HK})_d$ = Mole fraction of light key and heavy key in distillate

$(x_{LK}, x_{HK})_b$ = Mole fraction of light key and heavy key in residue

If there is a wide difference in the values of ∞_{LK} at the top most temperature and at bottom most temperature, then the use of following equation in Fenskey's equation gives lesser value of N_m than actual N_m required,

$$\infty_{LK} = (\infty_{LK, \text{top}} \times \infty_{LK, \text{bottom}})^{1/2} \quad (8.8)$$

In such case it is recommended to find ∞_{LK} by the following equation.

$$\infty_{LK} = (\infty_{LK, \text{top}} \times \infty_{LK, \text{feed}} \times \infty_{LK, \text{bottom}})^{1/3} \quad (8.24)$$

where $\infty_{LK, \text{feed}}$ = relative volatility of light key with respect to heavy key at the bubble point of feed.

After finding the values of R_m and N_m , number of theoretical stages required or number of equilibrium stages required for desired separation can be determined by Gilliland's correlation. It was originally presented in terms of graph. Many attempts are made to represent Gilliland's correlation analytically. One of the analytical equations for Gilliland's correlation is developed by Malokanov¹⁴.

$$f(N) = \frac{N - N_m}{N + 1} = 1 - \exp \left[\left(\frac{1 + 54.4\psi}{11 + 117.2\psi} \right) \left(\frac{\psi - 1}{\psi^{0.5}} \right) \right] \quad (8.25)$$

$$\text{where, } \psi = \frac{R - R_m}{R + 1} \quad (8.26)$$

Example 8.8

Determine the number of theoretical stages required for desired separation by FUG method for the case given in Example 8.4.

Solution:

Minimum reflux ratio determined by Underwood's method in the Example 8.4.

$$R_m = 1.4509$$

Using Fenskey's equation to find the minimum number of theoretical stages required for desired separation, N_m for total reflux.

$$N_m = \frac{\log \left[\left(\frac{x_{LK}}{x_{HK}} \right)_d \left(\frac{x_{HK}}{x_{LK}} \right)_b \right]}{\log \infty_{LK}} \quad (8.23)$$

$$N_m = \frac{\log \left[\left(\frac{0.95}{0.05} \right) \left(\frac{0.416}{0.163} \right) \right]}{\log (2.567)}$$

$$N_m = 4.117$$

Table 8.13 Values of N for Different Values of R

R	ψ	$f(N)$	N
1.7	0.092 26	0.5616	10.67
2.0	0.183 03	0.4752	8.75
2.5	0.299 74	0.3811	7.27
3.0	0.387 30	0.3198	6.52
3.5	0.455 36	0.2761	6.07
4.0	0.509 82	0.2433	5.76

Gilliland's correlation,

$$f(N) = \frac{N - N_m}{N + 1} = 1 - \exp \left[\left(\frac{1 + 54.4\psi}{11 + 117.2\psi} \right) \left(\frac{\psi - 1}{\psi^{0.5}} \right) \right] \quad (8.25)$$

where, $\psi = \frac{R - R_m}{R + 1}$ (8.26)

Refer Table 8.13 for the calculated results.

Above the value of reflux ratio $R = 3$, there is a tapering in values of N . Hence, the optimum reflux ratio is nearer to $R = 3$.

Let actual reflux ratio, $R = 3$

Number of theoretical stages required for the desired separation $N = 6.52$.

Example 8.9

A fatty acid mixture contains 11% palmitic acid, 4% stearic acid, 28.5% oleic acid and rest linoleic acid (by mass). It is to be distilled to separate in pure components in distillation columns operating at 2 torr absolute at top.

Table 8.14 Pressure vs. Boiling Point Data of Fatty Acids

Absolute Pressure		Boiling Point, °C			
kPa	torr	Palmitic acid	Stearic acid	Oleic acid	Linoleic acid
0.133	1	167.4	183.6	177.6	178.5
0.267	2	179.0	195.9	189.5	190.1
0.533	4	192.2	209.2	202.6	202.8
1.07	8	206.1	224.1	217.0	216.9
2.13	16	221.5	240.0	232.9	232.6
4.27	32	238.4	257.1	250.6	250.0
8.53	64	257.1	276.8	270.3	269.7
17.1	128	278.7	299.7	292.5	291.9
34.1	256	303.6	324.8	317.7	317.2
68.3	512	332.6	355.2	346.5	346.5
101.325	760	351.5	376.1	364.9	365.2

(Table 2 of Ref.: 15)

Table 8.15 Chemical Formulas and Molar Masses of Fatty Acids

Fatty acid	Chemical Formula	Molar mass
Palmitic acid	C ₁₆ H ₃₂ O ₂	256.42
Stearic acid	C ₁₈ H ₃₆ O ₂	284.48
Oleic acid	C ₁₈ H ₃₄ O ₂	282.46
Linoleic acid	C ₁₈ H ₃₂ O ₂	280.45

Feed is a saturated liquid. Determine the number of theoretical stages required for the desired separation.

Solution:

Boiling points of oleic acid and linoleic acid are very close. Hence they are near impossible or very difficult to separate by distillation. It is planned to separate the following three products from the feed mixture.

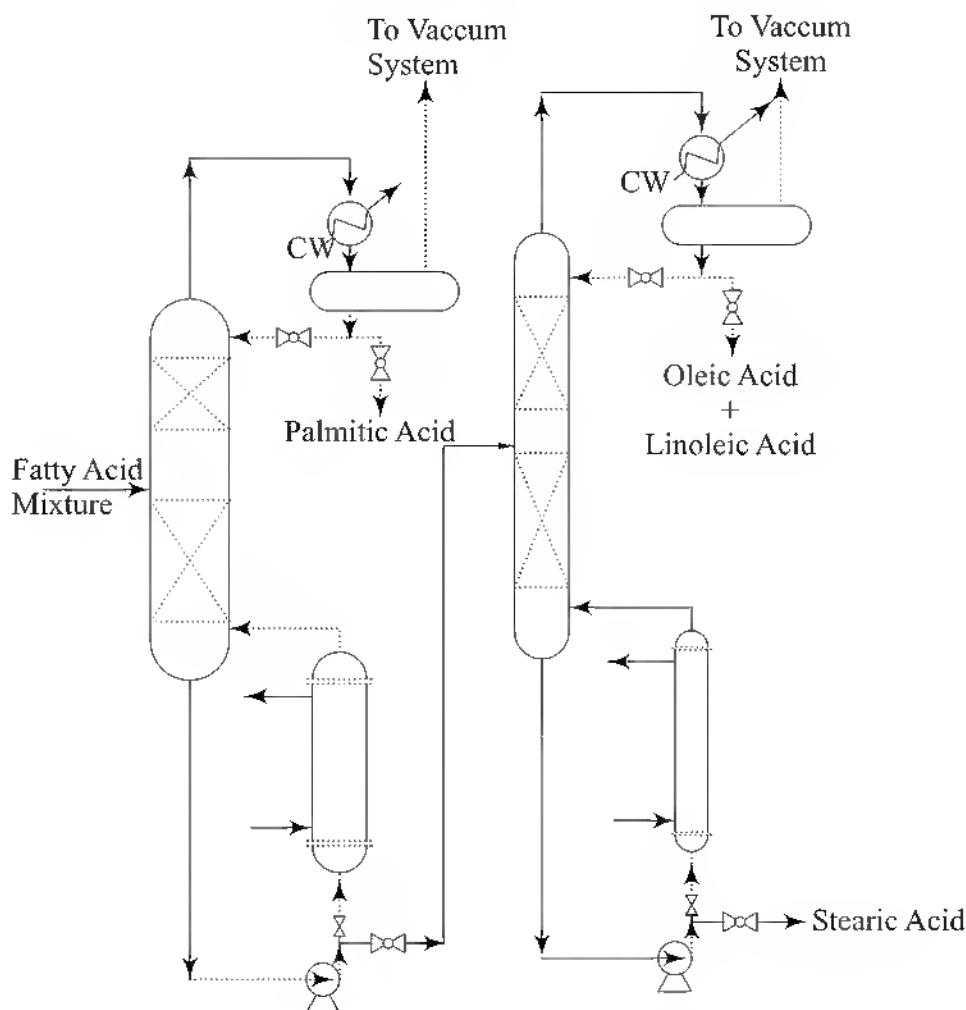


Fig. 8.7(a) Separation of Fatty Acids

<i>Products</i>	<i>Purity in % by mole</i>
1. Palmitic acid	99%
2. Mixture of linoleic and oleic acids	97% (Combinedly)
3. Stearic acid	95%

Two distillation columns are required. First column will separate 99% by mole pure palmitic acid as top product and remaining as bottom product. Second column will give 97% by mole pure (combinedly) mixture of linoleic and oleic acid as the top product and 95% by mole stearic acid as the bottom product. Both columns will be operated under vacuum and operating pressure at the top of both columns will be 2 torr a.

(a) Determination of number of theoretical stages required for first distillation column:

Operating pressure at top: 2 torr a. Top product is nearly pure palmitic acid. At 179°C vapour pressure of palmitic acid is 2 torr (Table 8.14). Hence top temperature of column will be very close to 179°C. Let operating pressure at the base of first column is 30 torr a. (This is decided based on one trial calculation). Bottom most temperature of distillation column is bubble point temperature of residue. To carry out the material balance and to find the feed composition in mole %, let the feed flow rate be 1000 kg/h (basis).

Mass flow rate of feed, $\dot{m}_F = 1000 \text{ kg/h}$

Table 8.16 Feed Composition

Component	mass %	Molar mass	kmol/h	mole %
Palmitic acid	11	256.42	0.428 98	11.94
Stearic acid	4	284.48	0.140 61	3.91
Oleic acid	28.5	282.46	1.008 99	28.08
Linoleic acid	56.5	280.45	2.014 62	56.07
Total	100.00		3.5932	100.00

Table 8.17 Composition of Distillate and Residue of 1st Column

Component	Distillate, D		Bottom, W	
	kmol/h	mole %	kmol/h	mole %
Palmitic acid	0.3971	99	0.031 88	1
Stearic acid	—	—	0.140 61	4.4
Oleic acid	0.002	0.5	1.006 99	31.55
Linoleic acid	0.002	0.5	2.012 62	63.05
Total	0.4011	100.0	3.1921	100.00

Vapour pressures of oleic acid and linoleic acid are nearly same. Hence, in distillation calculations they are considered as one product. For first column palmitic acid is the light key and mixture of oleic acid – linoleic acid is the heavy key.

Component balance around 1st distillation column: Palmitic acid balance.

$$0.1194 F = 0.99 D + 0.01 W$$

Let mole % of palmitic acid in residue = 1%.

$$0.1194 \times 3.5932 = 0.99 D + 0.01 (3.5932 - D)$$

$$D = 0.4011 \text{ kmol/h}, W = 3.1921 \text{ kmol/h}$$

Let mole % of heavy key in distillate = 1%

Resulting material balance is shown in Table 8.17.

At bubble point temperature of residue

$$\sum x_{wi} p_{vi} = p_t = 30 \text{ torr}$$

Trial temperature = 250°C

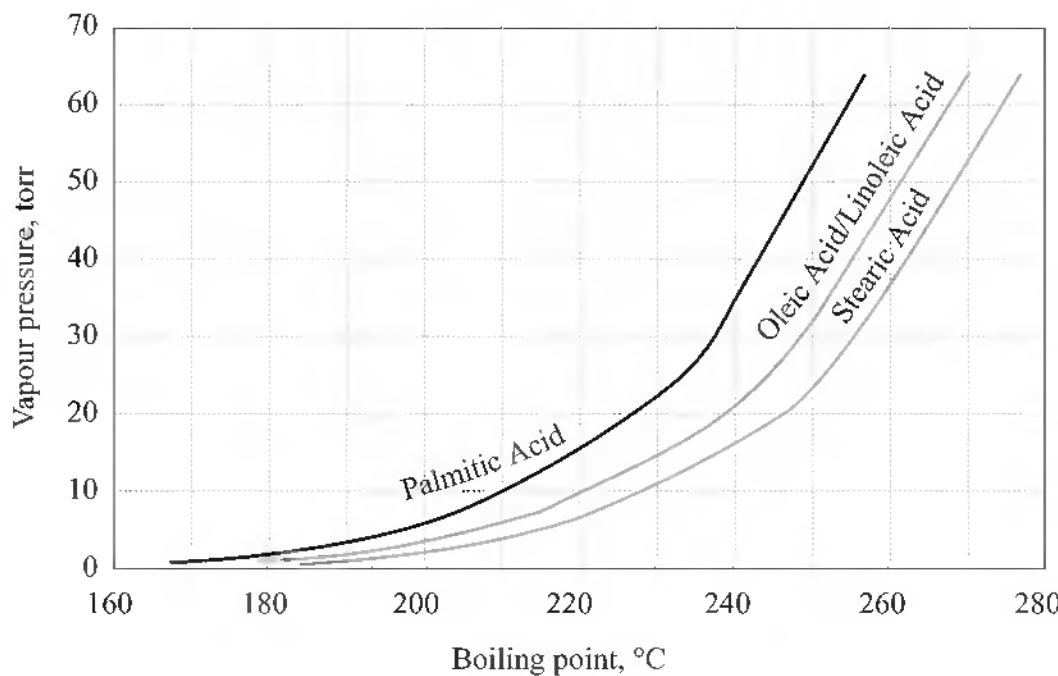
Table 8.18 Vapour Pressures as Read from Fig. 8.7(b)

Component	Vapour pressure torr	
	At 179°C, torr	At 250°C, torr
Palmitic acid	2	51.71
Stearic acid	0.714	23.29
Linoleic acid/Oleic acid	0.903	31.43

Using Fig. 8.7(b) and Table 8.17,

$$0.01 \times 51.71 + 0.044 \times 23.29 + (0.3155 + 0.6305) \times 31.43 = 31.27 \text{ torr} \cong 30 \text{ torr}$$

Hence bottom most temperature of 1st distillation column $\cong 250^\circ\text{C}$

**Fig. 8.7(b)** Vapour Pressures of Fatty Acids based on Table 8.14**Table 8.19** Relative Volatilities Calculations

Component	α_{top}	α_{bottom}	α_{av}
Palmitic acid (LK)	2.2148	1.6452	1.9089
Stearic acid	0.7907	0.741	0.7654
Linoleic acid/Oleic acid (HK)	1	1	1

Here FUG method is used to find the number of theoretical stages required for desired separation.

Minimum number of theoretical stages required by using Fenskey's method, N_m :

$$N_m = \frac{\log \left[\left(\frac{x_{LK}}{x_{HK}} \right)_d \left(\frac{x_{HK}}{x_{LK}} \right)_b \right]}{\log \alpha_{LK}} \quad (8.23)$$

$$N_m = \frac{\log \left[\left(\frac{0.99}{0.01} \right) \left(\frac{0.3155 + 0.6305}{0.01} \right) \right]}{\log (1.9089)}$$

$$N_m = 14.144$$

Underwood's method:

$$\sum \frac{\alpha_i x_{if}}{\alpha_i - \vartheta} = 1 - q \quad (8.15)$$

Feed is saturated liquid. Hence $q = 1$

$$\frac{1.9089 \times 0.1194}{1.9089 - \vartheta} + \frac{0.7654 \times 0.0391}{0.7654 - \vartheta} + \frac{1 \times (0.2808 + 0.5607)}{1 - \vartheta} = 1 - 1 = 0$$

Solving by Mathcad,

For $\vartheta = 1.71932$, $f(\vartheta) = 0.001 \approx 0$

Also, $\vartheta \in \{1, \dots, 1.9089\}$

$$\sum \frac{\infty_i x_{id}}{\infty_i - \vartheta} = R_m + 1$$

$$\frac{1.9089 \times 0.99}{1.9089 - \vartheta} + \frac{1 \times (0.005 + 0.005)}{1 - \vartheta} = R_m + 1$$

$$R_m = 8.9545$$

Gilliland's correlation

$$f(N) = \frac{N - N_m}{N + 1} = 1 - \exp \left[\left(\frac{1 + 54.4\psi}{11 + 117.2\psi} \right) \left(\frac{\psi - 1}{\psi^{0.5}} \right) \right] \quad (8.25)$$

where, $\psi = \frac{R - R_m}{R + 1}$ (8.26)

Table 8.20 Evaluation of Parameters

R	ψ	$f(N)$	N
10	0.095	0.5586	33.31
12	0.234	0.4318	25.65
15	0.378	0.326	21.47
17	0.447	0.2813	20.07

Let, $R = 15$, for which $N = 21.47 \approx 22$

FUG method does not give information about feed tray location. Feed tray location can be determined by using following equation (Kirkbride equation).

$$\log \left(\frac{N_r}{N_s} \right) = 0.206 \log \left[\left(\frac{W}{D} \right) \left(\frac{x_{HK}}{x_{LK}} \right)_f \left(\frac{x_{bLK}}{x_{dHK}} \right)^2 \right] \quad (8.27)$$

where, N_r = Number of stages above the feed

N_s = Number of stages below the feed

$$\log \left(\frac{N_r}{N_s} \right) = 0.206 \log \left[\left(\frac{3.1921}{0.4011} \right) \left(\frac{0.2808 + 0.5607}{0.1194} \right) \left(\frac{0.01}{0.01} \right)^2 \right]$$

$$\frac{N_r}{N_s} = 2.292$$

$$\begin{aligned}N_r + N_s &= 22 \\3.292 N_s &= 22 \\N_s &= 6.683 \cong 7, N_r = 15\end{aligned}$$

Hence, seventh stage from bottom will be the feed stage.

For second distillation column $W = F'$. (As shown in Fig. 8.7(a)), where F' is molar flow rate of feed to second distillation column.

Linoleic – Oleic acids balance around second column:

$$\begin{aligned}F' \times (0.3155 + 0.6305) &= 0.97 D' + 0.05 W' \\3.1921 \times 0.946 &= 0.97 D' + 0.05 (3.1921 - D') \\2.86012 &= 0.92 D' \\D' &= 3.1088 \text{ kmol/h}, W' = 0.0833 \text{ kmol/h}\end{aligned}$$

Table 8.21 Composition of Distillate and Residue of 2nd Column

Component	Distillate D'		Bottom, W'	
	kmol/h	mole %	kmol/h	mole %
Palmitic acid	0.0319	1.026	—	—
Stearic acid	0.0615	1.978	0.0791	95
Oleic acid	1.0056	32.347	0.0014	1.68
Linoleic acid	2.0098	64.649	0.0028	3.36
Total	3.1088	100.000	0.0833	100.00

Operating pressure at the top of second column = 2 torr a.

Top most temperature of distillation column is dew point temperature of overhead vapour.

At dew point temperature

$$\sum \frac{y_i p_t}{p_{vi}} = 1$$

Let top most temperature = 190.1°C (Trial) $y_i = x_{D_i}$

At 190.1°C temperature, from Fig. 8.7(b) vapour pressure of components are as follows.

Table 8.22 Vapour Pressures as Read from Fig. 8.7(b)

Component	Vapour pressure, torr	
	At 190°C, torr	At 268.2°C, torr
Linoleic/oleic acid	2	63.5
Stearic acid	1.64	50

Table 8.23 Relative Volatilities Calculations

Component	α_{top}	α_{bottom}	α_{av}
Linoleic/oleic acid	1.22	1.27	1.245
Stearic acid	1	1	1

For the calculations of number of theoretical stages required for desired separation feed to second column can be approximated as binary system.

$$F' = 3.1921 \frac{\text{kmol}}{\text{h}}, \quad D' = 3.1088 \frac{\text{kmol}}{\text{h}}, \quad W' = 0.0833 \frac{\text{kmol}}{\text{h}},$$

$$Z_F = 0.946, \quad x_D = 0.97, \quad x_W = 0.05$$

Vapour–liquid equilibrium data can be determined by using following equation.

$$y = \frac{\alpha x}{1 + (\alpha - 1)x}$$

Table 8.24 VLE Data for 2nd Column

x	y
0	0
0.2	0.2374
0.4	0.4536
0.6	0.6513
0.8	0.8328
1.0	1.0000

Feed is saturated liquid $q = 1$.

From Fig. (8.7(c))

$$\frac{x_D}{R_m + 1} = 0.4301$$

$$R_m = 1.2553$$

$$R = 3 \times R_m = 3.7659$$

$$\frac{x_D}{R+1} = \frac{0.97}{4.7659} = 0.20353$$

For $R = 3.7659$, number of theoretical stages required for desired separation, $N = 33$.

Feed tray location = 4 from top

B. Rigorous Methods:

Rigorous or detailed methods available for multicomponent distillation for finding or verifying number of theoretical stages required for desired separation are as follows.

- (I) Lewis–Matheson method
- (II) Theile–Geddes method
- (III) Relaxation methods
- (IV) Equation tearing procedures using tridiagonal matrix algorithm.

Among these methods only Lewis–Matheson method calculate the number of theoretical stages. In other methods number of equilibrium stages required above and below the feed point must be specified by the user. In other methods, for the given number of theoretical stages, product compositions are determined.

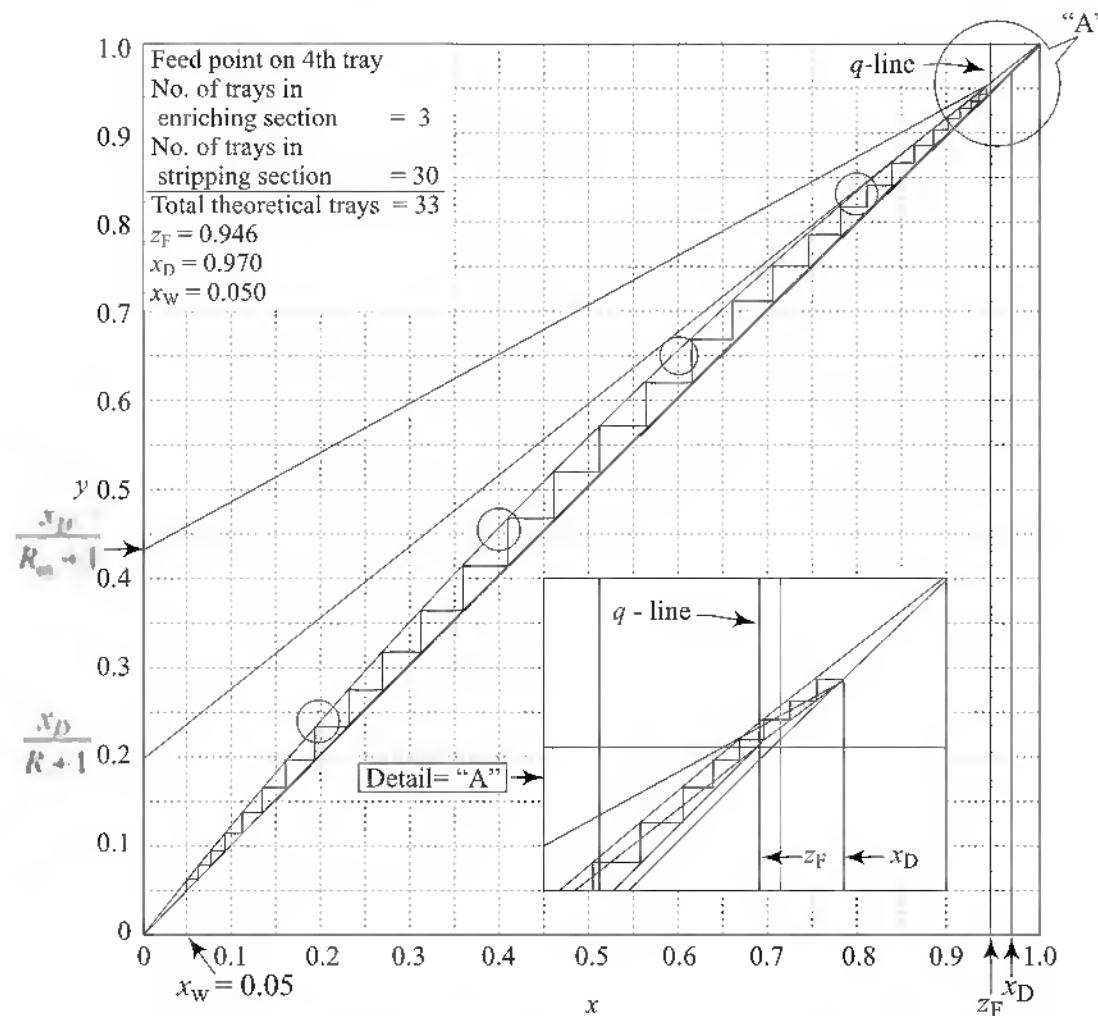


Fig. 8.7(c) McCabe-Thiele Diagram for Second Column

(I) Lewis–Matheson Method¹²

In this method, constant molar overflow is assumed. In other words, in this method it is assumed that for each stage, molar latent heat of vaporization of liquid phase is equal to latent heat of condensation of vapour phase. Hence moles of liquid vaporized over each theoretical stage is equal to moles of vapour condensed on that stage. This implies that, molar flow rates of liquid and vapour in enriching section (L and V) and also in stripping section (\bar{L} and \bar{V}) remain constant. To use this method following information must be specified.

1. Feed flow rate, conditions and compositions
2. One product flow (D or W)
3. Product compositions or distribution of key and non-key components
4. Reflux ratio
5. Column pressure

Stepwise Lewis–Matheson method is as given below:

- (i) Assuming that total condenser is used. $L = RD$ and $V = (R + 1)D$. Determine the dew point temperature of overhead vapour by using equation

$$\sum \frac{y_{Ni} \theta_i p_t}{\gamma_i p_{vi}} = 1 \text{ (For non-ideal system)} \quad (8.28)$$

$$\text{or } \sum \frac{y_{Ni} p_t}{p_{vi}} = 1 \text{ (For ideal vapour - liquid equilibrium)} \quad (8.29)$$

where, $y_{Ni} = x_{Di} = x_{oi}$

y_{Ni} = Mole fraction of component i in vapour phase leaving the N^{th} stage

x_{Di} = Mole fraction of component i in distillate

x_{oi} = Mole fraction of component i in reflux

p_t = Operating pressure at top of distillation column, kPa a

p_{vi} = Vapour pressure of component i at dew point temperature of overhead vapour, kPa a

θ_i = Fugacity coefficient of component i in vapour phase

γ_i = Activity coefficient of component i in liquid phase

Same calculations (dew point temperature calculation) also give the value of x_{Ni}

$$x_{Ni} = \frac{y_{Ni} p_t}{p_{vi}} \text{ (For ideal VLE)} \quad (8.30)$$

where x_{Ni} = mole fraction of component i in liquid phase leaving the N^{th} tray.

Composition of vapour leaving the $(N-1)^{\text{th}}$ tray can be determined by a material balance around the top section as shown in Fig. (8.8)

$$V y_{(N-1)i} = L x_{Ni} + D x_{Di} \quad (8.31)$$

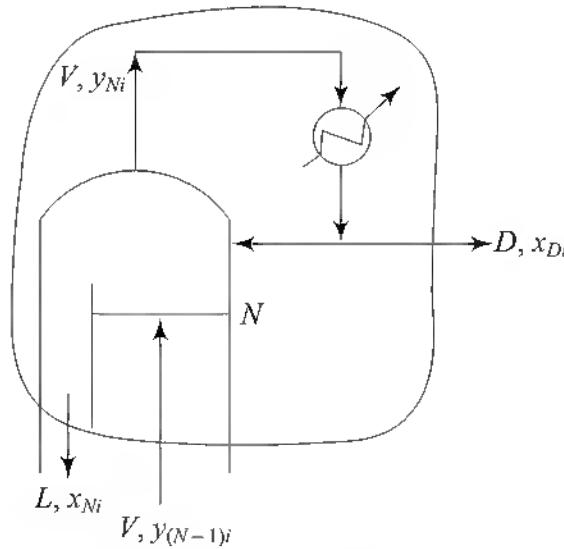


Fig. 8.8 Material Balance of Top Tray

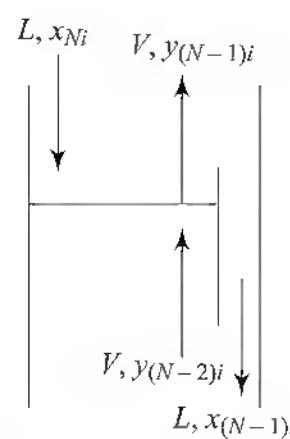


Fig. 8.9 Material Balance for $(N-1)^{\text{th}}$ Tray

By using above equation $y_{(N-1)i}$ are determined. After finding $y_{(N-1)i}$, i.e. composition of vapour leaving the $(N-1)^{\text{th}}$ tray, find the dew point of this vapour.

$$\sum \frac{y_{(N-1)i} p_t}{p_{vi}} = 1 \text{ (for ideal VLE)} \quad (8.32)$$

Same calculations also give the values of $x_{(N-1)i}$, composition of liquid phase leaving the $(N-1)^{\text{th}}$ tray.

Material balance around $(N-1)^{\text{th}}$ tray gives composition of vapour leaving the $(N-2)^{\text{th}}$ tray.

$$V_{(N-2)i} = V_{(N-1)i} + Lx_{(N-1)i} - Lx_{Ni} \quad (8.33)$$

These calculations (i.e. dew point temperature calculations and material balance calculations) are repeated upto the feed tray.

(ii) Feed Tray Identification:

To identify the feed tray following conditions must be satisfied.

If feed is liquid then mole fraction ratio or molar ratio of light key to heavy key in the feed should lie in between the same ratios of the liquid leaving the feed tray and of the liquid leaving the tray, next above the feed tray.

$$\left(\frac{x_{LK}}{x_{HK}} \right)_{\text{Feed tray}} \leq \left(\frac{x_{LK}}{x_{HK}} \right)_{\text{Feed}} \leq \left(\frac{x_{LK}}{x_{HK}} \right)_{\text{Tray above the feed tray}}$$

or

$$\left(\frac{x_{LK}}{x_{HK}} \right)_{N_F} \leq \left(\frac{x_{LK}}{x_{HK}} \right)_{\text{Feed}} \leq \left(\frac{x_{LK}}{x_{HK}} \right)_{N_F+1} \quad (8.34)$$

If the feed is a mixture of liquid and vapour then also the above condition is valid. However, the difference is only liquid phase molar ratio is to be considered for satisfying this condition.

If feed is vapour then condition is

$$\left(\frac{x_{LK}}{x_{HK}} \right)_{\text{Tray beneath feed tray}} \leq \left(\frac{y_{LK}/K_{LK}}{y_{HK}/K_{HK}} \right)_{\text{Feed}} \leq \left(\frac{x_{LK}}{x_{HK}} \right)_{N_F}$$

or

$$\left(\frac{x_{LK}}{x_{HK}} \right)_{N_F-1} \leq \left(\frac{y_{LK}/K_{LK}}{y_{HK}/K_{HK}} \right)_{\text{Feed}} \leq \left(\frac{x_{LK}}{x_{HK}} \right)_{N_F} \quad (8.35)$$

(iii) For Stripping Section:

molar flow rate of liquid, $\bar{L} = L + qF$

molar flow rate of vapour, $\bar{V} = \bar{L} - W$

where, W = Molar flow rate of residue

Calculate the bubble point temperature of residue

$$\sum \frac{x_{wi} p_{vi} \gamma_i}{\theta_i p_t} = 1 \quad (\text{For non-ideal VLE}) \quad (8.28)$$

$$\text{or} \quad \sum \frac{x_{wi} p_{vi}}{p_t} = 1 \quad (\text{For ideal VLE}) \quad (8.30)$$

Same calculations also give the values of y_{wi} composition of vapour leaving the reboiler.

$$y_{wi} = \frac{x_{wi} p_{vi}}{p_t} \quad (8.36)$$

The material balance around the reboiler gives

$$\bar{L}x_{1i} = \bar{V}y_{wi} + Wx_{wi} \quad (8.37)$$

Calculate the bubble point temperature of liquid leaving the 1st tray

$$\sum \frac{x_i p_{vi}}{p_t} = 1 \text{ (For ideal VLE)} \quad (8.38)$$

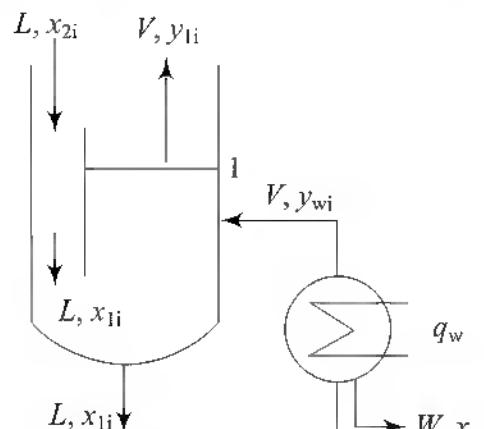


Fig. 8.10 Material Balance Around Reboiler

Same calculations also give y_{1i} , composition of vapour leaving the first tray. Then material balance around 1st tray gives x_{2i} .

$$\bar{L}x_{2i} = \bar{L}x_{1i} + \bar{V}y_{1i} - \bar{V}y_{wi} \quad (8.39)$$

x_{2i} is the composition of liquid leaving the 2nd tray. These iterative calculations are repeated from bottom to feed tray. For feed tray x_{LK}/x_{HK} ratios, one obtained from enriching section calculations and other obtained from stripping section calculations, must be very close.

- (iv) Normally, in the first trial calculations, it happens that on getting close values of the ratios x_{LK}/x_{HK} , one from enriching section and other from stripping section, mole fractions of the components of feed, one obtained from top section and other from bottom section do not match. If they are different then it is called "mismatch".

If composition of liquid phase, leaving the feed tray, obtained from top section calculations and the same from bottom section calculations are different, then to match them one has to change product distribution and product rates. In other words one has to change x_{Di} , x_{Wi} , D and W . For the new values, entire calculations must be repeated until the entire composition of liquid leaving the feed tray calculated from top and bottom closely match.

In the first trial calculations, if composition of liquid leaving the feed tray from top to down calculations and the same from bottom to up calculations are not matching, then Smith suggests the following method to make the change in product composition for 2nd trial calculations¹².

Change in moles of component i in distillate for 2nd trial calculations, Δd_i is calculated by following equation

$$\Delta d_i = \frac{(x_{FB} - x_{FD})_i}{\left(\frac{x_{FD}}{d} \right)_i + \left(\frac{x_{FB}}{b} \right)_i} \quad (8.40)$$

where, x_{FBi} = Mole fraction of component i in the liquid from feed tray as found from the bottom to up calculations

x_{FDi} = Mole fraction of component i in the liquid from feed tray as found from the top to down calculations

d, b = Moles of component i in top and bottom products in last iteration

Then corresponding changes required in bottom product for component i is $\Delta b_i = -\Delta d_i$. For new distribution, entire calculation is repeated. This calculation is continued until compositions of liquid, leaving the feed tray, obtained from top-down calculations and bottom-up calculations are approximately same.

For more exact calculations, enthalpy balance should be done for each tray to find the variation in liquid rate and vapour rate on each tray. But these calculations require detailed and exact enthalpy data. Also, if one considers it, then calculations become more laborious and complicated. Use of computer is then essential.

Example 8.10

A saturated liquid, consisting of phenol and cresols with some xylenols, is fractionated to give a top product of 95.3 mole% phenol. Metacresol is heavy key and phenol is light key component. Total condenser is used. The composition of the top product and of the phenols in the bottoms are given.

- Complete the material balance over the still for a feed rate of 100 kmol/h.
- Calculate the minimum reflux ratio by Underwood's method.
- For $R = 3R_m$, calculate the composition of vapour entering to the top most tray by Lewis-Matheson method.

Table 8.25 Distillation Column Data and Relative Volatilities

Component	∞_{av}	Feed, mole %	Top product, mole %	Bottom product, mole %
Phenol	1.98	35	95.30	5.24
<i>o</i> -Cresol	1.59	15	4.55	?
<i>m</i> -Cresol	1.00	30	0.15	?
Xylenols	0.59	20	—	?
		100	100.00	

Solution:

- (a) Overall material balance

$$\begin{aligned} F &= D + W \\ 100 &= D + W \end{aligned} \quad (1)$$

Phenol balance:

$$F \times 0.35 = D \times 0.953 + W \times 0.0524 \quad (2)$$

From Eq. (1) and (2), $D = 33$ kmol/h, $W = 67$ kmol/h.

Component balance gives the composition of residue.

o-Cresol balance:

$$\begin{aligned} F \times 0.15 &= 0.0455 \times D + x_{W_0} \times W \\ 100 \times 0.15 &= 0.0455 \times 33 + x_{W_0} \times 67 \\ x_{W_0} &= 0.2015 \end{aligned}$$

Similarly, $x_{Wm} = 0.447, x_{Wx} = 0.2985$

where, x_{Wm} = mole fraction of *m*-cresol in Residue

x_{Wx} = mole fraction of xylenol in Residue

(b) R_m by Underwood's method:

$$\sum \frac{\infty_i x_{id}}{\infty_i - \vartheta} = R_m + 1 \quad (8.14)$$

$$\frac{1.98 \times 0.953}{1.98 - \vartheta} + \frac{1.59 \times 0.0455}{1.59 - \vartheta} + \frac{1 \times 0.0015}{1 - \vartheta} = R_m + 1 \quad (3)$$

where ϑ is determined by following equation

$$\sum \frac{\infty_i x_{if}}{\infty_i - \vartheta} = 1 - q \quad (8.15)$$

$$\frac{1.98 \times 0.35}{1.98 - \vartheta} + \frac{1.59 \times 0.15}{1.59 - \vartheta} + \frac{1 \times 0.3}{1 - \vartheta} + \frac{0.59 \times 0.2}{0.59 - \vartheta} = 1 - 1 = 0 \quad (4)$$

Here $q = \frac{H_G - H_F}{H_G - H_L}$ and for saturated liquid $H_F = H_L$

By trial and error calculations, or by Mathcad, determine ϑ .

Also $\vartheta \in \{1, \dots, 1.98\}$.

$\vartheta = 1.22$

On substituting this value of ϑ in Eq. (3) gives

$$R_m = 1.67$$

(c) $R = 3 R_m = 3 \times 1.67 = 5.01$

$$L = RD, V = (R + 1) D$$

$$L = 5.01 \times 33 = 165.33 \text{ kmol/h}$$

$$V = (5.01 + 1) \times 33 = 198.33 \text{ kmol/h}$$

Refer Fig. 8.8.

Component balance around top segment

$$V_{(N-1)i} = Lx_{Ni} + Dx_{Di}$$

Composition of vapour entering to top most tray

$$y_{(N-1)i} = \frac{L}{V} x_{Ni} + \frac{D}{V} x_{Di} \quad (5)$$

At dew point of overhead vapour

$$\sum \frac{y_{Ni} p_t}{p_{vi}} = \Sigma x_{Ni} = 1 \quad (6)$$

But here operating pressure p_t is not specified. So x_{Ni} cannot be determined by Eq. (6). x_{Ni} can be determined based on the data of average relative volatility, ∞_{avi} .

$$\Sigma x_{Ni} = x_{NP} + x_{NO} + x_{Nm} + x_{Nx} = 1$$

where, x_{Ni} = Mole fraction of component *i* in the liquid leaving the *N*th tray

x_{NP} = Mole fraction of phenol in the same

x_{NO} = Mole fraction of *o*-cresol in the same

x_{Nm} = Mole fraction of *m*-cresol in the same

x_{Nx} = Mole fraction of xylenols in the same

$$\frac{x_{N_p}}{x_{N_m}} + \frac{x_{N_o}}{x_{N_m}} + 1 + \frac{x_{N_x}}{x_{N_m}} = \frac{1}{x_{N_m}}$$

Average relative volatility of phenol

$$\begin{aligned} \infty_{avP} &= \frac{y_{N_p}/x_{N_p}}{y_{N_m}/x_{N_m}} \\ \frac{x_{N_p}}{x_{N_m}} &= \frac{y_{N_p}}{y_{N_m} \infty_{av_p}} \end{aligned} \quad (7)$$

$$\begin{aligned} \frac{y_{N_p}}{\infty_{av_p}} &= \frac{x_{N_p}}{x_{N_m}} y_{N_m} \\ \frac{y_{N_p}}{\infty_{av_p}} + \frac{y_{N_o}}{\infty_{av_o}} + \frac{y_{N_m}}{\infty_{av_m}} + \frac{y_{N_x}}{\infty_{av_x}} &= \frac{y_{N_m}}{x_{N_m}} \sum x_{N_i} = \frac{y_{N_m}}{x_{N_m}} \end{aligned}$$

$$\sum \frac{y_{N_i}}{\infty_{av_i}} = \frac{y_{N_m}}{x_{N_m}} \quad (8)$$

From Eq. (7) and (8):

$$x_{NP} = \frac{y_{N_p}/\infty_{av_p}}{\sum \frac{y_{N_i}}{\infty_{av_i}}} = \frac{0.953/1.98}{(0.953/1.98) + (0.0455/1.59) + (0.0015/1)}$$

$$x_{NP} = 0.9411$$

Since total condenser is used, $y_{Ni} = x_{Di}$

Similarly

$$x_{No} = \frac{\left(\frac{0.0455}{1.59}\right)}{\left(\frac{0.953}{1.98}\right) + \left(\frac{0.0455}{1.59}\right) + \left(\frac{0.0015}{1}\right)} = 0.05595$$

$$x_{Nm} = 0.00295$$

On substituting the values of x_{Ni} in Eq. (5)

$$y_{(N-1)i} = \frac{165.33}{198.33} \times x_{Ni} + \frac{33}{198.33} \times x_{Di}$$

$$y_{(N-1)p} = \frac{165.33}{198.33} \times 0.9411 + \frac{33}{198.33} \times 0.953$$

$$y_{(N-1)p} = 0.8336 \times 0.9411 + 0.1664 \times 0.953$$

$$y_{(N-1)p} = 0.943$$

$$y_{(N-1)o} = 0.8336 \times 0.05595 + 0.1664 \times 0.0455 = 0.0542$$

$$y_{(N-1)m} = 0.8336 \times 0.00295 + 0.1664 \times 0.0015 = 0.0027$$

Example 8.11

Find the number of theoretical stages required for the desired separation by Lewis–Matheson method for the following case.

Table 8.26 Distillation Column Details

Component	Feed, mole %	Distillate, mole %	Residue, mole %
<i>n</i> -Butane	37	95.0	16.3
<i>i</i> -Pentane	32	5.0	41.6
<i>n</i> -Pentane	21	—	28.5
<i>n</i> -Hexane	10	—	13.6
Total	100	100.0	100.0

1. $F = 4750 \text{ mol/h}$, $D = 1250 \text{ mol/h}$, $W = 3500 \text{ mol/h}$ and $q = 1$
2. *n*-Butane is light key component and *i*-Pentane is heavy key component.
3. Operating pressure $p_t = 2 \text{ atm a}$
4. Reflux ratio $R = 3$
5. Vapour pressures of components

$$\ln p_v = A - \frac{B}{(T + C)}, \quad p_v \text{ in torr and } T \text{ in K}$$

Table 8.27 Antoine Constants⁹

Component	A	B	C
<i>n</i> – Butane	15.6782	2154.9	– 34.42
<i>i</i> – Pentane	15.6338	2348.67	– 40.05
<i>n</i> – Pentane	15.8333	2477.07	– 39.94
<i>n</i> – Hexane	15.8366	2697.55	– 48.78

Solution:

Lewis–Matheson method

Molar flow rates of liquid and vapour in enriching section

$$L = RD, V = (R + 1)D$$

$$L = 3 \times 1250 = 3750 \text{ mol/h}, V = (3 + 1) \times 1250 = 5000 \text{ mol/h}$$

For total condenser,

$$y_{Ni} = x_{Di} = x_{oi}$$

To find the dew point of over head vapour

$$\sum \frac{y_{Ni} p_t}{p_{vi}} = 1$$

For $T = 295 \text{ K}$ (i.e. $t = 22^\circ\text{C}$), $p_{vB} = 1650 \text{ torr}$, $p_{viP} = 615 \text{ torr}$

$$\frac{0.95 \times (2 \times 760)}{1650} + \frac{0.05 \times (2 \times 760)}{615} \cong 1$$

or $0.875 \times 15 + 0.1236 \cong 1$

Hence dew point temperature of over head vapour, $t_{DP} = 22^\circ\text{C}$.

The same calculation also gives

$$x_{NB} = 0.87515$$

$$x_{NiP} = 0.1236$$

Material balance around the top segment, shown in Fig. 8.8.

$$V y_{(N-1)i} = L x_{Ni} + D x_{Di}$$

$$y_{(N-1)i} = \frac{L}{V} x_{Ni} + \frac{D}{V} x_{Di}$$

$$y_{(N-1)B} = \frac{3750}{5000} \times x_{NB} + \frac{1250}{5000} \times x_{DB}$$

$$y_{(N-1)B} = 0.75 \times 0.87515 + 0.25 \times 0.95 = 0.8939$$

$$y_{(N-1)iP} = 0.75 \times 0.1236 + 0.25 \times 0.05 = 0.1052$$

Find the dew point temperature of vapour leaving the $(N-1)^{\text{th}}$ tray by trial and error calculations.

At

$$t = 24^{\circ}\text{C} \text{ or } T = 297 \text{ K}$$

$$p_{viB} = 1757.3 \text{ torr}, \quad p_{viP} = 660.65 \text{ torr}$$

$$\sum \frac{y_{(N-1)} p_t}{p_{vi}} = \frac{0.8911 \times (2 \times 760)}{1757.3} + \frac{0.1050 \times (2 \times 760)}{660.65} = 1.012$$

Dew point temperature of vapour leaving the $(N-1)^{\text{th}}$ tray is 24°C .

The same calculations also give

$$x_{(N-1)B} = 0.77 \text{ and } x_{(N-1)iP} = 0.24$$

Here $\Sigma x_{(N-1)i} = 1.01$. To make this total = 1.000 normalized values of $x_{(N-1)i}$ are determined as follows.

$$x_{(N-1)B} = \frac{0.77}{0.77 + 0.24} = 0.7624$$

$$x_{(N-1)iP} = \frac{0.24}{0.77 + 0.24} = 0.2376$$

Check whether the $(N-1)^{\text{th}}$ tray is feed tray or not.

$$\frac{x_{(N-1)B}}{x_{(N-1)iP}} = \frac{0.7624}{0.2376} = 3.2 \triangleq \frac{37 \text{ (moles of } n\text{C}_4 \text{ in feed)}}{32 \text{ (moles of } i\text{C}_5 \text{ in feed)}} (= 1.15625)$$

Hence $(N-1)^{\text{th}}$ tray is not feed tray.

Component balance around $(N-1)^{\text{th}}$ tray

Refer Fig. 8.9.

$$V y_{(N-2)i} = L x_{(N-1)i} + V y_{(N-1)i} - L x_{Ni}$$

$$y_{(N-2)i} = \frac{L}{V} x_{(N-1)i} + y_{(N-1)i} - \frac{L}{V} x_{Ni}$$

$$y_{(N-2)B} = 0.75 \times 0.7624 + 0.8911 - 0.75 \times 0.87515$$

$$y_{(N-2)B} = 0.8065, \quad y_{(N-2)iP} = 1 - y_{(N-2)B} = 0.1935$$

Find the dew point temperature of vapour leaving the $(N - 2)^{\text{th}}$ tray.

At, $T = 301 \text{ K}$ or $t = 28^\circ \text{ C}$, $p_{vB} = 1987.6 \text{ torr}$, $p_{viP} = 760 \text{ torr}$

$$\sum \left(\frac{y_{(N-2)i} p_t}{p_{vi}} \right) = \frac{0.8065 \times (2 \times 760)}{1987.6} + \frac{0.1935 \times (2 \times 760)}{760}$$

$$= 0.6168 + 0.387 = 1$$

The same calculations also give

$$x_{(N-2)B} = 0.6168 \text{ and } x_{(N-2)IP} = 0.387$$

Normalized values of $x_{(N-2)i}$

$$x_{(N-2)B} = 0.6182, \quad x_{(N-2)IP} = 0.3818$$

Check whether $(N - 2)^{\text{th}}$ tray is feed tray or not.

$$\frac{x_{(N-2)B}}{x_{(N-2)IP}} = \frac{0.6182}{0.3818} = 1.62 < \frac{37}{32} (= 1.156)$$

$(N - 2)^{\text{th}}$ tray is not the feed tray.

Component balance around $(N - 2)^{\text{th}}$ tray

$$V y_{(N-3)i} = L x_{(N-2)i} + V y_{(N-2)i} - L x_{(N-1)i}$$

$$y_{(N-3)i} = \frac{L}{V} x_{(N-2)i} + y_{(N-2)i} - \frac{L}{V} x_{(N-1)i}$$

$$y_{(N-3)B} = 0.75 \times 0.6182 + 0.8065 - 0.75 \times 0.7624 = 0.6984$$

$$y_{(N-3)IP} = 0.3016$$

Find the dew point of vapour, leaving the $(N - 3)^{\text{th}}$ tray.

At $T = 305 \text{ K}$ or $t = 32^\circ \text{ C}$, $p_{vB} = 2239.9 \text{ torr}$, $p_{viP} = 870.63 \text{ torr}$.

$$\sum \frac{y_{(N-3)i} p_t}{p_{vi}} = \frac{0.6984 \times 1520}{2239.9} + \frac{0.3016 \times 1520}{870.63} = 1.00$$

Dew point temperature of vapour leaving the $(N - 3)^{\text{th}}$ tray, $t_{DP} = 32^\circ \text{ C}$

The same calculations also give

$$x_{(N-3)B} = 0.47 \text{ and } x_{(N-3)IP} = 0.53$$

Check $(N - 3)^{\text{th}}$ tray is feed tray or not.

$$\frac{x_{(N-3)B}}{x_{(N-3)IP}} = \frac{0.47}{0.53} = 0.887 < 1.156 \left(= \frac{37}{32} \right)$$

$(N - 3)^{\text{th}}$ tray or 4^{th} tray from the top is the feed tray.

Molar flow rate of liquid and vapour in stripping or exhausting section:

$$\bar{L} = L + Fq = 3750 + 4750 = 8500 \text{ kmol/h}$$

$$\bar{V} = \bar{L} - W = 8500 - 3500 = 5000 \text{ kmol/h}$$

Correct mole fraction of liquid leaving the feed tray

$$x_{BF} \bar{L} = x_B L + x'_B F$$

$$x_{BF} = 0.47 \times \frac{3750}{8500} + 0.37 \times \frac{4750}{8500} = 0.414$$

(where x'_B is mole fraction of *n*-Butane in feed)

$$x_{iPF} = 0.53 \times \frac{3750}{8500} + 0.32 \times \frac{4750}{8500} = 0.413$$

$$x_{nPF} = \frac{0.21 \times 4750}{8500} = 0.1173$$

$$x_{nHF} = \frac{0.1 \times 4750}{8500} = 0.056$$

Composition of the liquid, leaving the feed tray obtained from top to down calculations.

$$x_{BF} = 0.412, \quad x_{iPF} = 0.413, \quad x_{nPF} = 0.1173, \quad x_{nHF} = 0.056$$

$$\frac{x_{BF}}{x_{iPF}} \cong 1 < 1.156$$

Bottom to up calculations:

Find the bubble point of residue via trial and error calculations:

$$\sum \frac{x_{wi} p_{vi}}{p_t} = \frac{0.163 \times p_{vB}}{1520} + \frac{0.416 \times p_{viP}}{1520} + \frac{0.285 \times p_{viP}}{1520} + \frac{0.136 \times p_{viH}}{1520}$$

At

$$t = 48^\circ\text{C} \text{ or } T = 321\text{K}$$

$$\begin{aligned} \sum \frac{x_{wi} p_{pvi}}{p_t} &= \frac{0.163 \times 3494 + 0.416 \times 1442.4 + 0.285 \times 1118.8 + 0.136 \times 375}{1520} \\ &= 1.01 \cong 1 \end{aligned}$$

Hence bubble point temperature, $t_{BP} = 48^\circ\text{C}$

The same calculations also give the vapour composition, leaving the reboiler

$$y_{wB} = \frac{x_{wB} p_{vB}}{p_t} = 0.375$$

$$y_{wiP} = 0.395, \quad y_{wnP} = 0.2098, \quad y_{wnH} = 0.033$$

Component balance around reboiler gives (Ref. Fig. 8.10)

$$\begin{aligned} \bar{L}x_{1i} &= \bar{V}y_{wi} + Wx_{wi} \\ x_{1i} &= \frac{\bar{V}}{\bar{L}}y_{wi} + \frac{W}{\bar{L}}x_{wi} = \frac{5000}{8500}y_{wi} + \frac{3500}{8500}x_{wi} \\ &= 0.5882y_{wi} + 0.4118x_{wi} \end{aligned}$$

Composition of liquid, leaving the 1st tray

$$x_{1B} = 0.5882 \times 0.375 + 0.4118 \times 0.163 = 0.2877$$

$$x_{1iP} = 0.5882 \times 0.395 + 0.4118 \times 0.416 = 0.4036$$

$$x_{1nP} = 0.5882 \times 0.2098 + 0.4118 \times 0.285 = 0.24$$

$$x_{1nH} = 0.5882 \times 0.033 + 0.4118 \times 0.136 = 0.075$$

$$\frac{x_{1B}}{x_{1iP}} = \frac{0.2877}{0.4036} = 0.7128, \quad \frac{x_{BF}}{x_{iPF}} = 1.0036$$

Hence $\frac{x_{1B}}{x_{1iP}} < \frac{x_{BF}}{x_{iPF}}$ and 1st tray is not the feed tray.

Find the bubble point of liquid leaving the 1st tray by trial and error calculations.

At $t = 41^\circ\text{C}$ or $T = 314 \text{ K}$

$$p_{vB} = 2894.5 \text{ torr}, \quad p_{viP} = 1164.97 \text{ torr}, \quad p_{vnP} = 893.26 \text{ torr}$$

$$p_{vnH} = 288.76 \text{ torr, (by using Antoine equations)}$$

$$\sum \frac{x_{1i} p_{vi}}{p_t} = \frac{0.2877 \times 2894.5}{1520} + \frac{0.4036 \times 1164.97}{1520} + \frac{0.24 \times 893.26}{1520}$$

$$+ \frac{0.075 \times 288.76}{1520} = 1.012 = 1$$

Bubble point of liquid leaving the 1st tray, $t_{BP} = 41^\circ\text{C}$. Same calculations also give the composition of vapour, leaving the 1st tray.

$$y_{1B} = \frac{x_{1B} p_{vB}}{p_t} = \frac{0.2877 \times 2894.5}{1520} = 0.548$$

$$y_{1iP} = 0.309, \quad y_{1nP} = 0.141, \quad y_{1nH} = 0.014$$

Normalised values of y_{1i}

$$y_{1B} = \frac{0.548}{\Sigma y_{1i}} = \frac{0.548}{1.012} = 0.5415$$

$$y_{1iP} = \frac{0.309}{1.012} = 0.305$$

$$y_{1nP} = \frac{0.141}{1.012} = 0.139$$

$$y_{1nH} = \frac{0.014}{1.012} = 0.0138$$

Component balance around the 1st tray gives

$$\bar{L}x_{2i} = \bar{V}y_{1i} + \bar{L}x_{1i} - \bar{V}y_{wi}$$

$$x_{2i} = \frac{\bar{V}}{\bar{L}} y_{1i} + x_{1i} - \frac{\bar{V}}{\bar{L}} y_{wi}$$

$$x_{2B} = \frac{5000}{8500} \times 0.5415 + 0.2877 - \frac{5000}{8500} \times 0.375 = 0.3856$$

$$x_{2iP} = \frac{5000}{8500} \times 0.305 + 0.4036 - \frac{5000}{8500} \times 0.395 = 0.3506$$

$$x_{2nP} = \frac{5000}{8500} \times 0.139 + 0.24 - \frac{5000}{8500} \times 0.2098 = 0.1984$$

$$x_{2nH} = \frac{5000}{8500} \times 0.0138 + 0.075 - \frac{5000}{8500} \times 0.033 = 0.0637$$

$$\frac{x_{2B}}{x_{2iP}} = \frac{0.3856}{0.3506} = 1.1 \cong \frac{x_{BF}}{x_{iPF}} (= 1.025)$$

2^{nd} tray from bottom can be called the feed tray. Ideally values of this ratio $\left(\frac{x_{BF}}{x_{iPF}} \right)$ obtained from bottom to up calculations should be same as that obtained from top to down calculation. Here composition of liquid leaving the feed tray obtained from top to down calculations and the same obtained from bottom to up calculations are not matching.

Table 8.28 Summary of First Trial Calculations of Feed Tray Liquid

Component	Composition based on top-down calculation	Composition based on bottom-up calculation
<i>n</i> -Butane	0.412	0.3856
<i>i</i> -Pentane	0.413	0.3506
<i>n</i> -Pentane	0.1173	0.1984
<i>n</i> -Hexane	0.056	0.0637

To match this composition and to get the exact value of number of theoretical stages required for the desired separation of key components, second trial calculations are required. Based on 1^{st} trial calculations number of theoretical stages required for the desired separation of key components is 6. (Reboiler, 1^{st} , $2^{\text{nd}} = N - 3 = \text{Feed tray}, N - 2, N - 1, N$)

How to start the second trial calculations?

To start the second trial calculations, product composition x_{Di} and x_{Wi} (hence, *D* and *W* also) must be changed. Based on the Smith's suggestion following equation can be used to make the change in product composition for the 2^{nd} trial calculations.

Change in moles of component in distillate.

$$\Delta d_i = \frac{(x_{FB} - x_{FD})_i}{\left(\frac{x_{FD}}{d} \right)_i + \left(\frac{x_{FB}}{b} \right)_i} \quad (8.40)$$

For *n*-Butane or for nC_4

$$\Delta d_{nC_4} = \frac{0.3856 - 0.414}{\left(\frac{0.412}{1250 \times 0.95} \right) + \left(\frac{0.3856}{0.163 \times 3500} \right)} = -27.72 \text{ mol/h}$$

nC_4 in distillate for the 2^{nd} trial calculation

$$= 1250 \times 0.95 - 27.72 = 1159.78 \text{ mol/h}$$

nC_4 in residue = $0.163 \times 3500 + 27.72 = 598.22 \text{ mol/h}$

For *iso*-Pentane or iC_5 :

$$\Delta d_{iC_5} = \frac{0.3506 - 0.413}{\left(\frac{0.413}{1250 \times 0.05} \right) + \left(\frac{0.3506}{3500 \times 0.416} \right)} = -9.1 \text{ mol/h}$$

iC_5 in distillate = $1250 \times 0.05 - 9.1 = 53.4 \text{ mol/h}$

iC_5 in residue = $3500 \times 0.416 + 9.1 = 1465.1 \text{ mol/h}$

For *n*-Pentane or *nC*₅:

$$\Delta d_{nC_5} = \frac{0.1984 - 0.1173}{\left(\frac{0.1173}{d_{nC_5}}\right) + \left(\frac{0.1984}{3500 \times 0.285}\right)}$$

In 1st trial calculation $d_{nC_5} = 0$. But to use above equation one cannot put $d_{nC_5} = 0$. Let $d_{nC_5} = 1$ mol/h (negligible value)

$$\Delta d_{nC_5} = 0.6913$$

$$\begin{aligned} n_{C_5} \text{ in distillate} &= 1 + 0.6913 = 1.7 \text{ mol/h} \\ n_{C_5} \text{ in residue} &= 3500 \times 0.285 - 1.7 = 995.8 \text{ mol/h} \end{aligned}$$

For *n*-Hexane or *nC*₆:

$$\Delta d_{nC_6} = \frac{0.0637 - 0.056}{\left(\frac{0.056}{d_{nC_6}}\right) + \left(\frac{0.0637}{3500 \times 0.136}\right)}$$

Let $d_{nC_6} = 1$ mol/h (negligible)

$$d_{nC_6} = 0.137 \text{ mol/h}$$

$$n_{C_6} \text{ in distillate} = 1.137 \text{ mol/h}$$

$$n_{C_6} \text{ in residue} = 3500 \times 0.137 - 1.137 = 474.86 \text{ mol/h}$$

Table 8.29 Product Composition for Second Trial Calculations

Component	D		W	
	mol/h	mole fraction	mol/h	mole fraction
<i>nc</i> ₄	1159.78	0.954	598.22	0.169
<i>ic</i> ₅	53.4	0.044	1465.1	0.415
<i>nc</i> ₅	1.7	0.001	995.8	0.282
<i>nC</i> ₆	1.137	0.001	474.86	0.134
Total	1216.02	1.00	3533.98	1.00

Based on this new product distribution one can start the second trial calculations. These trial calculations are repeated until the composition of liquid leaving the feed tray obtained from top-down calculations match with the same obtained from bottom-up calculations. Results obtained at the end of 1st trial calculations are matching with the same given by FUG method.

(II) Thiele Geddes Method¹:

Original method of Thiele and Geddes was developed for manual calculation in 1933. There after computer programs are developed for this method to reduce the labour of calculations. This method is a rating method in which distribution of components between distillate and bottoms is predicted for a specified number of stages.

In this method following variables are to be specified as input data.

1. Number of equilibrium stages above and below the feed.
2. Column pressure

3. Temperatures (stage wise)
4. Reflux flow rate
5. Distillate rate
6. Feed flow rate, feed condition and feed composition
7. Liquid and vapour flow rates, leaving each stage

Calculation procedure is as follows.

Material balance for any component around the condenser is

$$G_1 y_1 = L_o x_o + D z_D$$

$$\frac{G_1 y_1}{D z_D} = \frac{L_o x_o}{D z_D} + 1$$

For a total condenser, $y_1 = x_o = z_D = x_D$ and $\frac{L_o}{D} = R$

$$\frac{G_1 y_1}{D z_D} = \frac{L_o}{D} + 1 = R + 1 = A_o + 1$$

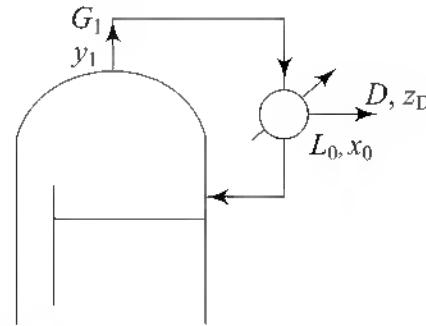


Fig. 8.11 Material Balance Around Overhead Condenser

where $A_o = \frac{L_o}{D} = R$ (for total condenser)

For a partial condenser, $z_D = y_D = K_o x_o$

where, K_o = equilibrium constant for partial condenser

$$\frac{G_1 y_1}{D z_D} = \frac{L_o x_o}{D z_D} + 1 = \frac{L_o}{D K_o} + 1 = \frac{R}{K_o} + 1 = A_o + 1$$

where $A_o = \frac{R}{K_o}$ (for partial condenser)

Component balance upto tray 1 gives

$$G_2 y_2 = L_1 x_1 + D z_D$$

$$\frac{G_2 y_2}{D z_D} = \frac{L_1 x_1}{D z_D} + 1 \quad (8.41)$$

for Tray 1, absorption factor

$$A_1 = \frac{L_1}{K_1 G_1} = \frac{L_1 x_1}{G_1 y_1} \quad (8.42)$$

$$L_1 x_1 = G_1 y_1 A_1$$

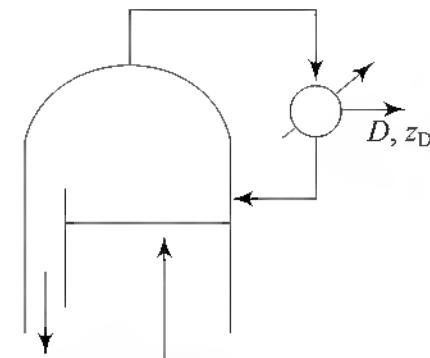


Fig. 8.12 Material Balance Around Top Section up to First Tray

From Eq. (8.41) and Eq. (8.42)

$$\frac{G_2 y_2}{D z_D} = \frac{G_1 y_1 A_1}{D z_D} + 1 = (A_o + 1) A_1 + 1 = A_1 A_o + A_1 + 1$$

Similarly for the vapour coming from any n^{th} tray

$$\begin{aligned} \frac{G_n y_n}{D z_D} &= \frac{A_{n-1} G_{n-1} y_{n-1}}{D z_D} = A_0 A_1 A_2 \dots A_{n-1} \\ &\quad + A_1 A_2 \dots A_{n-1} + \dots + A_{n-1} + 1 \end{aligned} \quad (8.43)$$

For the vapour coming from feed tray

$$\frac{G_F y_F}{Dz_D} = A_0 A_1 A_2 \dots A_{F-1} + A_1 A_2 \dots A_{F-1} + \dots + A_{F-1} + 1 \quad (8.44)$$

For stripping or exhausting section:

Component balance around reboiler

$$\begin{aligned}\bar{L}_N x_N &= \bar{G}_{N+1} y_{N+1} + Wx_w \\ \frac{\bar{L}_N x_N}{Wx_w} &= \frac{\bar{G}_{N+1} y_{N+1}}{Wx_w} + 1 \\ \frac{\bar{L}_N x_N}{Wx_w} &= S_w + 1\end{aligned}\quad (8.45)$$

where S_w is stripping factor of reboiler.

For kettle type reboiler stripping factor

$$S_w = \frac{\bar{G}_{N+1} y_{N+1}}{Wx_w} = \frac{\bar{G}_{N+1} K_b}{W} \quad (8.46)$$

(Since for kettle type reboiler $y_{N+1} = K_b x_w$)

where, K_b = equilibrium constant for kettle type reboiler

For thermosyphon reboiler $y_{N+1} \approx x_w$

Stripping factor for thermosyphon reboiler

$$S_w = \frac{\bar{G}_{N+1}}{W} \quad (8.47)$$

Component balance around bottom section up to the last tray.

$$\begin{aligned}\bar{L}_{N-1} x_{N-1} &= \bar{G}_N y_N + Wx_w \\ \frac{\bar{L}_{N-1} x_{N-1}}{Wx_w} &= \frac{\bar{G}_N y_N}{Wx_w} + 1\end{aligned}\quad (8.48)$$

Stripping factor for N^{th} tray

$$S_N = \frac{\bar{G}_N K_N}{\bar{L}_N} = \frac{\bar{G}_N y_N}{\bar{L}_N x_N}$$

where, K_N = Equilibrium constant for N^{th} tray

$$\bar{G}_N y_N = \bar{L}_N x_N S_N \quad (8.49)$$

From the Eqs (8.44) and (8.48)

$$\frac{\bar{L}_{N-1} x_{N-1}}{Wx_w} = \frac{\bar{L}_N x_N}{Wx_w} S_N + 1 \quad (8.50)$$

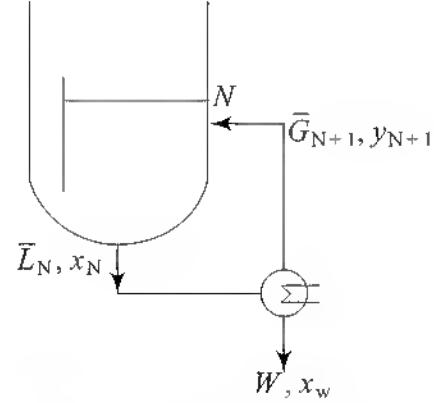


Fig. 8.13 Material Balance Around Reboiler

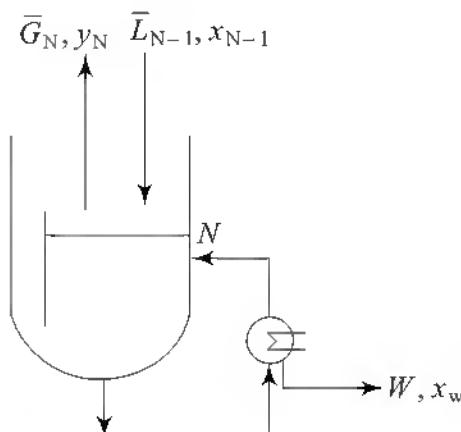


Fig. 8.14 Material Balance Around of Bottom Section up to Final Tray

From Eqs (8.47) and (8.50)

$$\frac{\bar{L}_{N-1} x_{N-1}}{Wx_W} = (S_w + 1) S_N + 1 = S_w S_N + S_N + 1 \quad (8.51)$$

For any m^{th} tray of stripping section

$$\begin{aligned} \frac{\bar{L}_m x_m}{Wx_W} &= S_{m+1} \cdot S_{m+2} \dots S_{N-1} \cdot S_N \cdot S_w + S_{m+1} \cdot S_{m+2} \dots \\ &S_{N-1} S_N + S_{m+1} \cdot S_{m+2} \dots S_{N-1} + \dots + S_{m+1} + 1 \end{aligned} \quad (8.52)$$

For the liquid leaving the feed tray

$$\begin{aligned} \frac{\bar{L}_F x_F}{Wx_W} &= S_{F+1} \cdot S_{F+2} \dots S_N \cdot S_w + S_{F+1} \cdot S_{F+2} \dots S_N \\ &+ S_{F+1} \dots S_{N-1} + \dots + S_{F+1} + 1 \end{aligned} \quad (8.53)$$

Absorption factor for feed tray

$$A_F = \frac{\bar{L}_F}{K_F G_F} = \frac{\bar{L}_F x_F}{G_F y_F} \quad (8.54)$$

where, K_F = Equilibrium constant for feed tray

$$\frac{Wx_W}{Dz_D} = \frac{G_F y_F / Dz_D}{\bar{L}_F x_F / Wx_W} \frac{\bar{L}_F x_F}{G_F y_F} = \frac{(G_F y_F / Dz_D)}{(\bar{L}_F x_F / Wx_W)} \cdot A_F \quad (8.55)$$

Symbol z_D is mole fraction of distillate which can be liquid, vapour or liquid-vapour mixture.

In calculations, $G_F y_F / Dz_D$ ratio is determined by Eq. (8.44). $(\bar{L}_F x_F) / (Wx_W)$ ratio is determined by Eq. (8.53).

Absorption factor for feed tray is determined by Eq. (8.54). The values are substituted in Eq. (8.55) and the ratio Wx_W / Dz_D is determined.

Overall component balance

$$FZ_F = Wx_W + Dz_D \quad (8.56)$$

$$\frac{FZ_F}{Dz_D} = \frac{Wx_W}{Dz_D} + 1$$

$$Dz_D = \frac{FZ_F}{\frac{Wx_W}{Dz_D} + 1} \quad (8.57)$$

Equation (8.57) gives the value of Dz_D , then Eq. (8.56) gives Wx_W . Thus finally one can get the product composition.

This product composition is correct, if all input data are correct. Normally in the first trial calculations temperatures at the trays are assumed to calculate the equilibrium constants; K_s . To verify whether assumed values of temperatures are correct or not, find y_n by following equation.

$$y_n = \frac{G_n y_n}{Dz_D} \times \frac{Dz_D}{G_n} \quad (8.58)$$

where, $\frac{G_n y_n}{Dz_D}$ is determined by Eq. (8.43).

For any tray n , y_n 's for all components must be determined by Eq. (8.58). Then check whether $\sum y_{ni} = 1$ or not.

If $\sum y_{ni} = 1$, it means assumed value of temperature for tray n is correct. If $\sum y_{ni} \neq 1$, adjust the values of y_{ni} and find new dew point temperature. Same calculation should be done for all the trays.

Similarly in stripping section for each tray find

$$x_m = \frac{\bar{L}_m x_m}{Wx_W} \times \frac{Wx_w}{\bar{L}_m} \quad (8.59)$$

and check whether $\sum x_{mi} = 1$ or not.

Here $\frac{\bar{L}_m x_m}{Wx_W}$ is determined by Eq. (8.52).

If $\sum x_{mi} \neq 1$, then adjust the values of x_i and find new bubble point.

For new temperatures, carry out the material balance and enthalpy balance simultaneously and find the new values of molar liquid flow rates and molar vapour flow rates (new values of L 's, G 's, \bar{L} 's, \bar{G} 's). Then repeat the calculations.

Example 8.12

Find the product composition by Thiele – Geddes method based on the following data.

Table 8.30 Feed Composition

(i)	Component	mole %
	<i>n</i> -Butane	37
	<i>i</i> -Pentane	32
	<i>n</i> -Pentane	21
	<i>n</i> -Hexane	10

n-Butane is light key component and *i*-Pentane is heavy key component.

- (ii) Feed flow rate $F = 4750$ mol/h. Feed is saturated liquid at its bubble point.
- (iii) Reflux ratio, $R = 3$
- (iv) $D = 1250$ mol/h, $W = 3500$ mol/h
- (v) Operating pressure, $p_t = 2$ atm a
- (vi) Assume constant molar overflow. Total condenser is used.

Table 8.31 Equilibrium Constants Data (K_i s)

Tray number	Temperature	K_{C_4}	K_{iC_5}	K_{nC_5}	K_{C_6}
1	22°C	1.086	0.4046	0.3000	0.0870
2	24°C	1.156	0.4346	0.3230	0.0950
3	28°C	1.308	0.5000	0.3747	0.1125
4 = feed	32°C	1.474	0.5730	0.4324	0.1330
5	41°C	1.904	0.7660	0.5877	0.1900
Reboiler	48°C	2.300	0.9500	0.7360	0.2500

(Read from Fig. 6.20)

Solution:

Molar flow rates of liquid and vapour in enriching section.

$$L = R D = 3 \times 1250 = 3750 \text{ mol/h}$$

$$G = (R + 1) D = 4 \times 1250 = 5000 \text{ mol/h}$$

$$\frac{Wx_W}{Dx_D} = \frac{G_F y_F / Dx_D}{L_F x_F / Wx_W} \times A_F$$

$$\frac{G_F y_F}{Dx_D} = A_0 A_1 A_2 \dots A_{F-1} + \dots + A_{F-1} + 1 \quad (8.44)$$

Here 4th tray is feed tray.

$$\frac{G_F y_F}{Dx_D} = A_0 A_1 A_2 A_3 + A_1 A_2 A_3 + A_2 A_3 + A_3 + 1$$

for n-Butane

$$A_0 = R = 3$$

$$A_1 = \frac{L_1}{K_1 G} = \frac{L}{K_{C_4} G} = \frac{3750}{1.086 \times 5000} = 0.6906$$

$$A_2 = \frac{L}{K_{C_4} G} = \frac{3750}{1.156 \times 5000} = 0.6488$$

$$A_3 = \frac{3750}{1.308 \times 5000} = 0.5734$$

$$\begin{aligned} \frac{G_F y_F}{Dx_D} &= 3 \times 0.6906 \times 0.6488 \times 0.5734 + 0.6906 \times 0.6488 \times 0.5734 \\ &\quad + 0.6488 \times 0.5734 + 0.5734 + 1 = 2.973 \end{aligned}$$

$$A_F = \frac{\bar{L}_F}{K_F G_F}$$

$$\bar{L} = L + Fq = 3750 + 4750 \times 1 = 8500 \text{ mol/h}$$

$K_F = K_{C_4}$ at feed tray temperature (equilibrium constant for n-Butane)

$$K_F = 1.474$$

$$A_F = \frac{8500}{1.474 \times 5000} = 1.1533$$

$$\frac{\bar{L}_F x_F}{Wx_W} = S_{F+1} \cdot S_{F+2} \dots S_N \cdot S_W + S_{F+1} \dots S_N + \dots S_{F+1} + 1 \quad (8.53)$$

$$\bar{G} = G + F(q - 1) \text{ or } \bar{G} = \bar{L} - W = 8500 - 3500 = G = 5000 \text{ mol/h}$$

$$\frac{\bar{L}_F x_F}{Wx_W} = S_5 S_w + S_5 + 1$$

$$S_5 = \frac{K_5 \bar{G}}{\bar{L}} = \frac{1.904 \times 5000}{8500} = 1.12$$

$$S_W = \frac{K_W \bar{G}}{\bar{L}} = \frac{2.3 \times 5000}{8500} = 1.353$$

$$\frac{\bar{L}_F x_F}{Wx_W} = 1.12 \times 1.353 + 1.12 + 1 = 3.6354$$

$$\frac{Wx_W}{Dx_D} = \frac{(G_F y_F / Dx_D)}{(\bar{L}_F x_F / Wx_W)} \times A_F = \frac{2.973}{3.6354} \times 1.1533 = 0.94316 \quad (8.55)$$

$$Dz_D = \frac{Fz_F}{\frac{Wx_W}{Dz_D} + 1} = \frac{4750 \times 0.37}{0.94316 + 1} = 904.4546 \text{ mol/h} \quad (8.57)$$

$$Wx_w = 853.05 \text{ mol/h}$$

$$z_D = 0.72356, x_w = 0.244$$

For *iso*-Pentane:

$$A_0 = R = 3, \quad A_1 = \frac{L}{K_1 G} = \frac{3750}{K_1 \times 5000}$$

$$A_1 = \frac{0.75}{K_{iC_5} \text{ at } 22^\circ\text{C}} = \frac{0.75}{0.4046} = 1.8537$$

$$A_2 = \frac{L}{K_2 G} = \frac{0.75}{0.4346} = 1.7257$$

$$A_3 = \frac{L}{K_3 G} = \frac{0.75}{0.5} = 1.5$$

$$\frac{G_F y_F}{Dx_D} = 3 \times 1.8537 \times 1.7257 \times 1.5 + 1.8537 \times 1.7257 \times 1.5 + 1.7257 \times 1.5 + 1.5 + 1$$

$$= 24.282$$

Absorption factor for feed tray

$$A_F = \frac{\bar{L}}{K_F G} = \frac{8500}{0.573 \times 5000} = 2.967$$

$$\frac{\bar{L}_F x_F}{Wx_W} = S_5 S_W + S_5 + 1$$

$$S_5 = \frac{K_5 \bar{G}}{\bar{L}} = \frac{0.766 \times 5000}{8500} = 0.45$$

$$S_W = \frac{K_W \bar{G}}{\bar{L}} = \frac{0.95 \times 5000}{8500} = 0.56$$

$$\frac{\bar{L}_F x_F}{Wx_W} = 0.45 \times 0.56 + 0.45 + 1 = 1.702$$

$$\frac{Wx_W}{Dx_D} = \frac{24.282}{1.702} \times 2.967 = 42.33$$

$$Dx_D = \frac{4750 \times 0.32}{(42.33 + 1)} = 35.08 \text{ mol/h}$$

$$Wx_w = 1485 \text{ mol/h}$$

$$x_D = 0.028, x_w = 0.4243$$

For *i*-pentane, similarly one can calculate, $x_D = 0.005\ 58$, $x_w = 0.283$ and for *n*-hexane $x_D \approx 0$ and $x_w = 0.1357$

Table 8.32 Resulting Product Compositions

Component	x_D	$x_{D\text{nor}}$	x_w	$x_{w\text{nor}}$
<i>n</i> -Butane	0.723 56	0.9556	0.2440	0.2244
<i>i</i> -Pentane	0.028 00	0.0370	0.4243	0.3900
<i>n</i> -Pentane	0.005 58	0.0070	0.2830	0.2600
<i>n</i> -Hexane	—	—	0.1357	0.1248
Total	0.757 14	1.0000	1.0870	1.0000

$x_{D\text{nor}}$ = Normalized values of x_{Di}

$x_{w\text{nor}}$ = Normalized values of x_{wi}

(III) Equation-Tearing Procedures Using Tridiagonal Matrix Algorithm¹⁴:

This method require the help of a computer. This method is more general and flexible, compared to Lewis – Matheson method and Thiele – Geddes method.

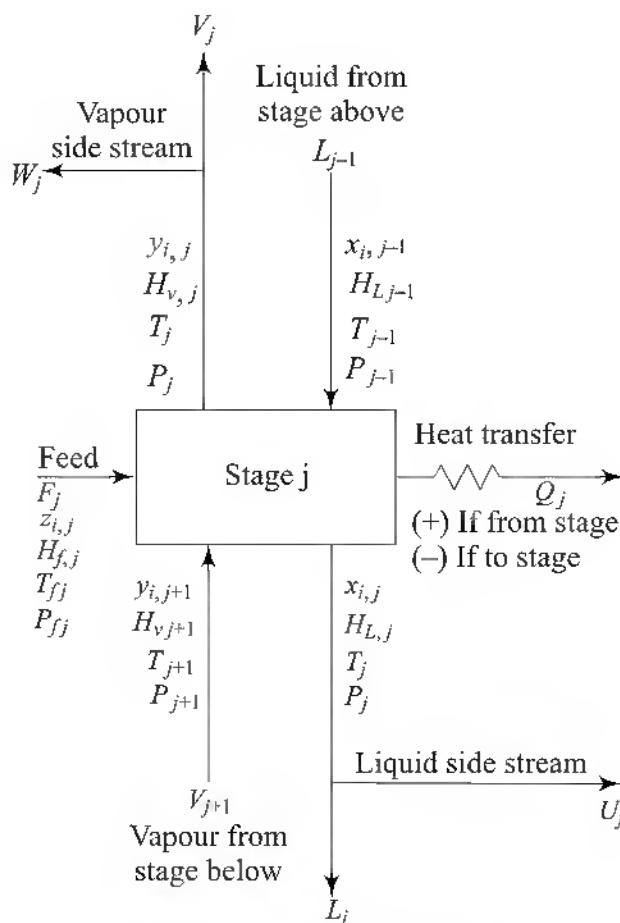
Consider a general continuous flow steady state, multicomponent multistage distillation operation. Assume that phase equilibrium between an existing vapour phase and existing liquid phase is achieved at each stage, that no chemical reaction takes place, and that neither of the existing phase entrains the other phase. Consider the general equilibrium stage *j* as shown in Fig. 8.15.

Here F_j represent molar flow rate of feed to a stage *j* at temperature T_{Fj} and pressure p_{Fj} and with overall composition in mole fractions z_{ij} . If stage *j* is not feed stage then in calculation $F_j = 0$. Heat is transferred from stage *j* (+ ve) or to stage *j* (- ve) at rate ϕ_j . If there is no external heat exchanger, attached with stage *j*, then $\phi_j = 0$. Equilibrium vapour and liquid phases leave the stage *j* at temperature T_j and pressure p_j and with mole fraction y_{ij} and x_{ij} respectively. The vapour may be partially withdrawn from the column as side stream at a molar flow rate W_j and the remainder V_j is sent to adjacent stage *j* – 1 above. Similarly existing liquid may be split into a side stream at a molar flow rate of U_j , with the remainder L_j , sent to adjacent stage *j* as follows:

For each stage *j* following independent equations can be written.

1. Component material balance equations (M)

$$L_{j-1} x_{i, j-1} + V_{j+1} y_{i, j+1} + F_j z_{ij} - (L_j + U_j) x_{ij} - (V_j + W_j) y_{ij} = 0 \quad (8.60)$$

**Fig. 8.15 General Equilibrium Stage**

- Phase equilibrium equation (E)

$$y_{ij} - K_{ij} x_{ij} = 0 \quad (8.61)$$

- Mole fraction summation equation (S)

$$\begin{aligned} \sum y_{ij} - 1.0 &= 0 \\ \sum x_{ij} - 1.0 &= 0 \end{aligned} \quad (8.62)$$

- Energy balance or enthalpy balance equation (H)

$$L_{j-1} H_{Lj-1} + V_{j+1} H_{v,j+1} + F_j H_{Fj} - (L_j + U_j) H_{Lj} - (V_j + W_j) H_{vj} - \phi_j = 0 \quad (8.63)$$

These equations are known as MESH equations. For evaluating different variables namely; flows, compositions, temperatures and pressures, number of MESH equations are necessary. For this purpose, degree of freedom dictates that for any stage j total $2C + 3$ MESH ($= C$ No. of material balance equations + C No. of phase equilibrium equation + 2 No. of mole fraction summation equations + 1 No. of energy balance equations) equations (where, C = number of components) can be written. For total N number of equilibrium stages, N ($2C + 3$) MESH equations can be written. Total number of variables are $N(3C + 10) + 1$. Difference between total number of variables and number of equations is $N(C + 7) + 1$. Hence $N(C + 7) + 1$ variables must be specified.

On combining MESH equations to eliminate y_i 's and L_j 's, following equations are obtained.

$$A_j x_{ij-1} + B_{ij} x_{ij} + C_{ij} x_{i,j+1} = D_{ij}$$

$$\text{where, } A_j = V_j + \sum_{m=1}^{j-1} (F_m - W_m - U_m) - V_i \quad 2 \leq j \leq N$$

$$B_{ij} = - \left[V_{j+1} + \sum_{m=1}^j (F_m - W_m - U_m) - V_1 + U_j + (V_j + W_j) K_{ij} \right] \quad 1 \leq j \leq N$$

$$C_{ij} = V_{j+1} K_{i,j+1} \quad 1 \leq j \leq N-1$$

$$D_{ij} = - F_j Z_{ij} \quad 1 \leq j \leq N$$

Equation (8.63) is a general representation of total NC equations. Each set of N equations is a tridiagonal matrix equation. For example, if $N = 5$, for each component following equations can be written in tridiagonal matrix form.

$$\begin{bmatrix} B_1 & C_1 & 0 & 0 & 0 \\ A_2 & B_2 & C_2 & 0 & 0 \\ 0 & A_3 & B_3 & C_3 & 0 \\ 0 & 0 & A_4 & B_4 & C_4 \\ 0 & 0 & 0 & A_5 & B_5 \end{bmatrix} \begin{bmatrix} x_1 \\ x_2 \\ x_3 \\ x_4 \\ x_5 \end{bmatrix} = \begin{bmatrix} D_1 \\ D_2 \\ D_3 \\ D_4 \\ D_5 \end{bmatrix} \quad (8.64)$$

Each set of above equations give the mole fractions of one component in the liquids, leaving all the trays (equilibrium stages). Substitution of x_{ij} values in the phase equilibrium equations give the values of y_{ij} . Like that all unknown variables are determined. For solving such equations, a mathematical software (such as Mathcad) is useful.

8.4.6 Step 6: Selection of Type of Tower

Criteria of selection between tray tower and packed tower are discussed in Sec. 8.3 of this chapter.

Selection of Tray Type: Different types of trays used in tray tower are (i) sieve tray, (ii) bubble cap tray, (iii) valve tray, (iv) Linde trays and other modified sieve trays.

Various factors that must be considered for the selection of tray type are
 (a) cost (b) capacity (c) operating range, (d) efficiency and (e) pressure drop

(a) **Cost:** Bubble cap tray is the costliest type of tray. With mild steel as a material of construction, the ratios of cost

Bubble cap: Valve: Sieve trays $\approx 3: 1.5: 1$ (general guideline)

In other words, cost of bubble cap tray is three times the cost of sieve tray and double the cost of valve tray with mild steel as material. With different materials these ratios are somewhat different.

(b) **Capacity:** If tower diameter required for given flow rates is more it means capacity of tray is less. The ranking for capacity is in order of modified sieve tray, sieve tray, valve tray and bubble cap tray. In other words for the given flow rates, tower diameter required is maximum with bubble cap trays and minimum with modified sieve tray. However, the difference in the values of tower diameters is not large.

(c) **Operating range:** It is an important factor for the selection of tray. Operating range of tray means range of liquid and vapour flow rates over which

tray works satisfactorily. Bubble cap tray provides the maximum operating range while sieve tray provides the minimum operating range. Operating process plants are designed and operated with some maximum capacity and also for some minimum capacity. Hence, distillation columns are operated in the range of some maximum feed flow rate to minimum feed flow rate. The ratio of maximum flow rate to minimum flow rate is called turn-down ratio. Some process plants are frequently operated at very high turn-down ratio to meet the product's demand and to avoid the shut down and start-up conditions as during shut down and start-up periods, operating plant provides very poor efficiency. For higher turndown ratio, bubble cap trays are preferred. Valve trays also offer more flexibility or higher turn down ratio than the sieve trays. However, in actual plant operation, valves get stuck-up or fly-away if operating conditions are frequently changed. In such a case, valve trays loose efficiency.

- (d) **Efficiency:** This factor is not generally considered for the selection of tray type. Efficiency of trays are almost same, if they are operated at design flow rates, or with minimum turn down. However, bubble cap columns give good efficiency over a wider range of turn down in general.
- (e) **Pressure drop:** Pressure drop over the tray and for entire column is important for vacuum distillation for which a packed tower is the first choice. But for the low vacuum (upto 700 torr) operation, tray tower can be selected to get other advantages. For example, to get the better vapour – liquid contact in large diameter column or to facilitate the side draws. Among the trays, sieve tray provides the lowest pressure drop, followed by valve tray, while bubble cap provides the highest pressure drop.

Sieve trays and modified sieve trays are the cheapest and are satisfactory for the majority of the applications. Nowadays, majority of chemical plants are having automatic and precise control system and hence flow rates are not much fluctuating. However, to meet the fluctuation in the demand of product, some plants are intentionally operated with different flow rates or capacities. To handle the higher turn down ratio, valve trays or bubble cap trays are considered. Valve trays are cheaper than bubble cap trays but they are not suitable for very low vapour rate. Bubble cap tray provides higher efficiency even with low vapour flow rate.

8.4.7 Step 7: Tower Diameter and Pressure Drop

Determine the tower diameter and pressure drop. Method to find the tower diameter and pressure drop for the packed tower type absorber is explained in Sec. 9.3.1 of Chapter 9. Same method and same Fig. 9.3 can also be used for the distillation in packed tower.

8.4.7.1 Determining the Tower Diameter of Sieve Tray Tower

Flooding in sieve tray tower fixes the upper limit of vapour velocity through the tower. Velocity of vapour at which flooding in sieve tray tower occurs is called flooding velocity, v_F . Tower diameter of sieve tray column is decided based on

the value of v_F . Actual velocity or design velocity through distillation column should be in the range of 70 to 90% of flooding velocity. Ideally it should be in between 80 to 85%.

Flooding is an inoperative condition. Flooding phenomena or flooding velocity are defined differently for different equipments. For example, in case of knock back condenser, flooding velocity is the velocity of vapour at which entrainment of liquid droplets in vapour just starts or the velocity of vapour at which % entrainment of liquid droplets in vapour becomes greater than zero. While in distillation column entrainment of liquid droplets in vapour up to 10% is normally accepted.

Flooding in tray tower of distillation column can occur or observed in either of the following ways¹⁶:

- (i) **Jet Flooding:** In distillation operation froth of liquid–vapour mixture forms on each tray from which nearly clear vapour is separated and rises upward to meet the liquid on the next above tray. When froth of liquid–vapour mixture touches the next above tray it is called jet flooding. Actually the vapour flows through perforations of tray forms a free flowing jet after leaving the orifice. Liquid droplets are entrained in these free flowing jets. These free flowing jets combinedly form the froth. When this froth touches the next tray above, it is called jet flooding.
- (ii) **Downcomer Flooding:** In distillation column, liquid flows in downward direction by gravitational force but it flows against the pressure. When liquid flows from one tray to next below tray, it flows from lower pressure to higher pressure. Hence, to compensate that, it elevates certain level inside the downcomer. When the liquid level in downcomer on any tray rises above the weir, it is called downcomer flooding.

Nowadays, it is possible to find tray or trays which restrict the capacity of existing tower or which decide the maximum capacity of existing tower. To find these trays of existing tower, one can intentionally operate the tower with maximum flow rates or even in flooding condition for a short period of time. During that time photograph of each section of tower can be taken with γ -rays. In these photographs one can clearly see the froth zone above the trays and liquid level inside the downcomers. Based on that one can find a tray or more than one tray which decide the maximum capacity of tower. If these sieve trays, which restrict the capacity of sieve tray tower, are replaced by modified or high capacity sieve trays, then it is possible to increase the capacity of existing tower (may be up to 20%).

Flooding velocity through sieve tray tower can be determined by following equation.

$$v_F = C_f \left(\frac{\sigma}{0.02} \right)^{0.2} \left(\frac{\rho_L - \rho_V}{\rho_V} \right)^{0.5} \quad (8.65)$$

where, v_F = Flooding vapour velocity based on net cross-sectional area, m/s
 C_f = Flooding constant which can be obtained from Fig. 8.16.
 σ = Surface tension of liquid, N/m

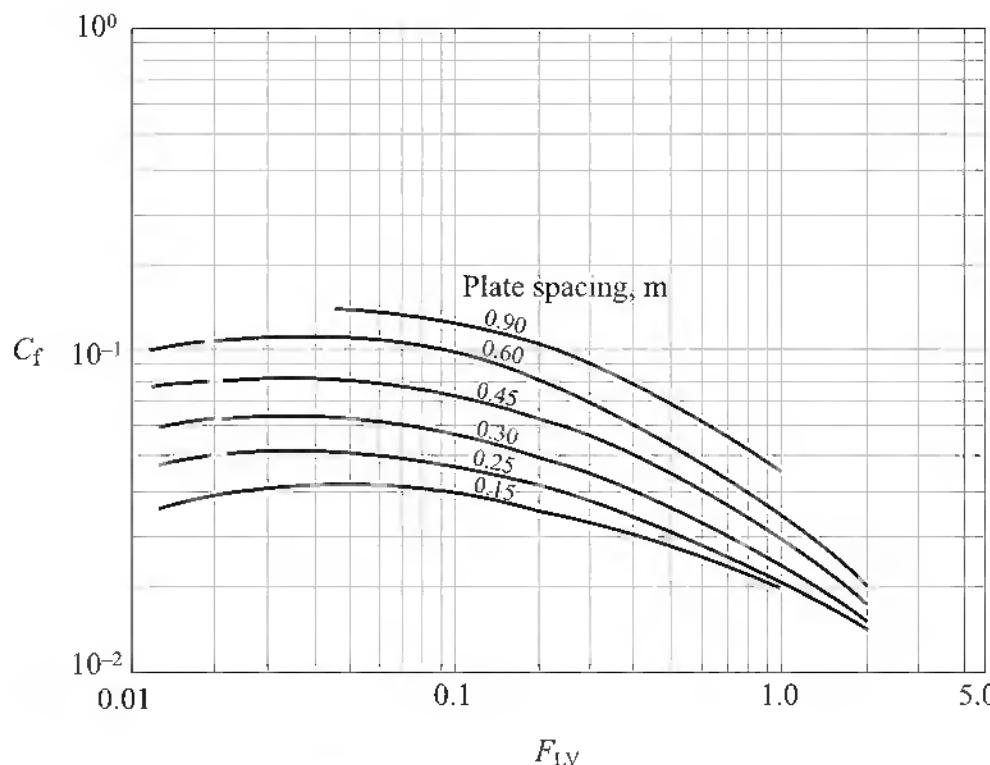


Fig. 8.16 Flooding Velocity (Sieve Plates)⁹
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ρ_v = Density of vapour, kg/m³

ρ_L = Density of liquid kg/m³

$$F_{LV} = \text{liquid-vapour flow factor} = \frac{L_W}{V_W} \left(\frac{\rho_v}{\rho_L} \right)^{0.5} \quad (8.66)$$

L_W = mass flow rate of liquid, kg/s

V_W = mass flow rate of vapour, kg/s

Figure (8.16) gives flooding velocity with $\pm 10\%$ error, subject to the following restrictions:

1. System is non foaming.
2. Weir height is less than 15% of plate spacing.
3. Sieve plate perforations are 13 mm or less in diameter.
4. Value of C_f obtained from Fig. (8.16) is valid only if $A_h/A_a \geq 0.1$. If $A_h/A_a < 0.1$ then C_f Value obtained from Fig. 8.16. must be corrected by multiplying it with following correction factor.

A_h/A_a	Correction Factor
0.08	0.9
0.06	0.8

where, A_h = Total area of all active holes

A_a = Active or bubbling area of tray

A_n = Net area available for vapour-liquid disengagement

For a single pass tray $A_n = A_c - A_d$ and $A_a = A_c - 2A_d$,

where, A_c = Total column cross-sectional area

A_d = Cross-sectional area of down comer. Down comer area

A_d ranges from 8 to 12 % of A_c

8.4.7.2 Selection of Liquid Flow Pattern

After finding the tower diameter, liquid flow pattern over sieve tray is decided. Common liquid flow patterns of cross flow sieve tray are shown in Fig. (8.17). In most of the cases single pass (cross flow; Fig. 8.17(a)) pattern is selected. Liquid flow pattern depends on the liquid flow rate and tower diameter. Following table can be used for the selection of liquid flow pattern.

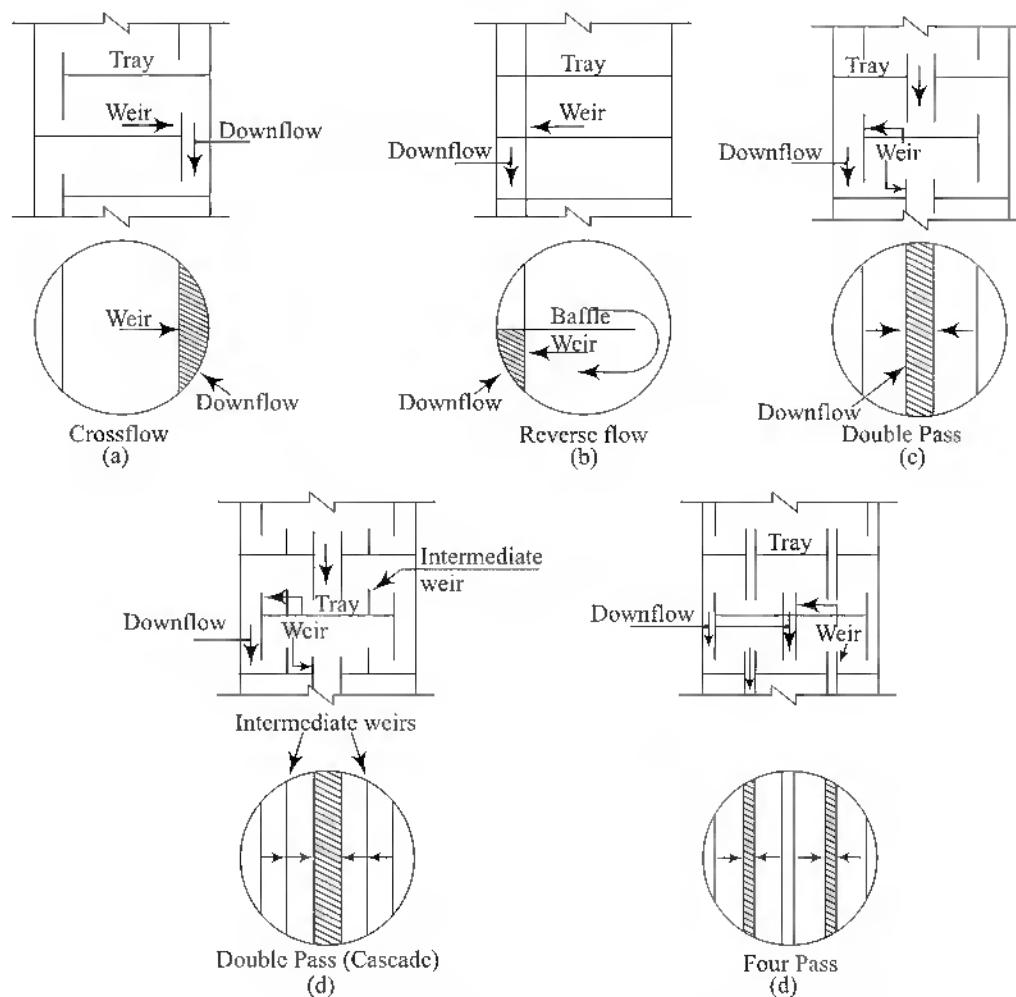


Fig. 8.17 Common Liquid Flow Patterns in Cross Flow Plates¹⁴.
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Table 8.33 Selection of Liquid Flow Pattern

Column diameter, m	Range of liquid flow rate, m ³ /s			
	Reverse flow	Cross flow (single pass)	Double pass	Cascade double pass
0.9	$0 - 1.90 \times 10^{-3}$	$1.90 \times 10^{-3} - 0.0126$		
1.2	$0 - 2.50 \times 10^{-3}$	$2.50 \times 10^{-3} - 0.0190$		
1.8	$0 - 3.15 \times 10^{-3}$	$3.15 \times 10^{-3} - 0.0250$	$0.025 - 0.0440$	
2.4	$0 - 3.15 \times 10^{-3}$	$3.15 \times 10^{-3} - 0.0315$	$0.0315 - 0.050$	
3	$0 - 3.15 \times 10^{-3}$	$3.15 \times 10^{-3} - 0.0315$	$0.0315 - 0.057$	$0.057 - 0.0880$
3.66	$0 - 3.15 \times 10^{-3}$	$3.15 \times 10^{-3} - 0.0315$	$0.0315 - 0.063$	$0.063 - 0.1000$
4.6	$0 - 3.15 \times 10^{-3}$	$3.15 \times 10^{-3} - 0.0315$	$0.0315 - 0.070$	$0.070 - 0.1136$
6	$0 - 3.15 \times 10^{-3}$	$3.15 \times 10^{-3} - 0.0315$	$0.0315 - 0.070$	$0.070 - 0.1260$

Tower diameter also depends on tray spacing. Value of flooding constant depends on the value of tray spacing (Fig. 8.16). Tray spacing ranges from 0.15 m to 1 m. At design stage, if the tray spacing is increased, required tower diameter decreases, tray efficiency increases and hence actual number of trays required for desired separation decreases but overall height for tower increases. Ideally one should find the optimum tray spacing or the tray spacing for which total cost of tower is minimum. If column diameter is less than 1 m, tray spacing is kept 0.2 to 0.3 m. For columns above 1 m diameter, tray spacing of 0.3 to 0.6 m is used. For the first trial calculations one can start with tray spacing equal to 0.3 m but if column diameter is calculated to be more than 1 m then for the second trial calculations, tray spacing can be taken equal to 0.4 to 0.5 m. Even with smaller tray spacing of 0.15 to 0.3 m, larger spacing is needed between certain plates to accommodate nozzles for feed, side streams, for manholes, etc.

8.4.7.3 Checking of Liquid Entrainment

As a rough guide, the % of liquid entrainment should be less than 10%

$$\% \text{ liquid entrainment} = \frac{\text{kg of liquid droplets entrained}}{\text{kg of gross liquid flow}} \times 100 \quad (8.67)$$

or % liquid entrainment = 100 Ψ

where, Ψ = Fractional liquid entrainment

$$\Psi = \frac{\text{kg of liquid entrained}}{\text{kg of gross liquid flow}} = \frac{\text{Moles of liquid entrained}}{\text{Moles of gross liquid flow}}$$

Value of Ψ as a function of liquid-vapour factor (F_{LV}) with % of flood as a parameter is given by Fair¹⁷. The same is reproduced in Fig. (8.18). Ψ should be less than 0.1, to get higher tray efficiency. Optimum value of ψ can be greater than 0.1.

$$\% \text{ of flood} = \frac{\text{Actual vapour velocity based on net area}}{\text{Flooding velocity}} \times 100 \quad (8.68)$$

$$= \frac{v_n}{v_f} \times 100$$

8.4.7.4 Weir

The height of weir decides the height of liquid over the tray. In most cases, weirs are straight and rectangular plates. With bubble cap trays, to compensate the fluctuations in the flow rate, V-notch type weirs are used. A high weir provides higher plate efficiency but at the expense of pressure drop. For a distillation column operating above atmospheric pressure, weir height is kept in between 40 mm to 90 mm; while 40 mm to 50 mm is more common. For vacuum operation, to control the tray pressure drop, weir height is reduced and it is kept in between 6 to 12 mm. In old designs, inlet weir was also used, but in recent designs inlet weir is not recommended as it results in hydraulic jump.

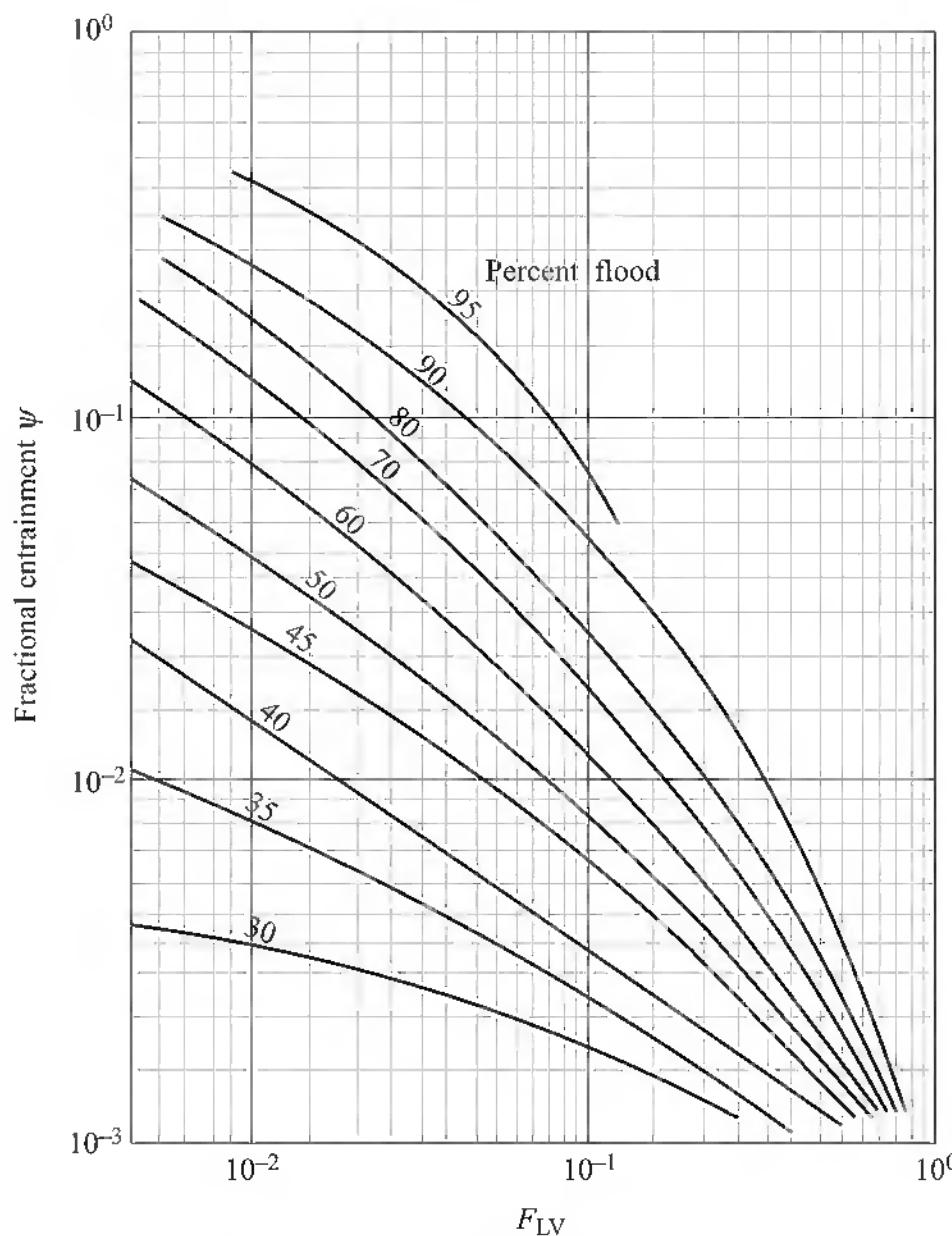


Fig. 8.18 Entrainment Correlation for Sieve Plates¹⁴
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Weir length fixes area of segmental downcomer. Weir length is normally kept in between 0.6 to 0.85 times column diameter. For the first trial calculations, weir length can be taken as $0.77D_i$ and equivalent down comer area is 12%. Decrease in weir length increases the net area of tray and decreases the tower diameter, but it decreases the down comer area, increases the pressure drop in down comer and also decreases the volume of down comer. It also elevates the liquid level in down comer. Increase in weir length increases the tower diameter. Hence, balanced weir length and weir height are required. Relation between weir length and down comer area is given in Table 8.34.

Clear liquid depth over the tray is equal to height of the weir, h_w , plus the depth of the crest of liquid over the weir, h_{ow} . The height of the liquid crest over the weir can be estimated by using Francis formula (Ref.: 9).

Table 8.34 Relationship of Weir Length and Down Comer Area

Weir length <i>ID of tower</i> = $\frac{l_w}{D_i}$	Down comer area A_d × 100 Column area A_c
0.600	5.25
0.650	6.80
0.700	8.80
0.705	9.00
0.715	9.50
0.727	10.00
0.745	11.00
0.770	12.00
0.780	13.00
0.800	14.00
0.815	15.00
0.826	16.00
0.840	17.00
0.850	18.00

$$h_{ow} = 750 \left(\frac{L_m}{\rho_L l_w} \right)^{\frac{2}{3}} \quad (8.69)$$

where,
 h_{ow} = Weir crest of liquid, mm
 L_m = Mass flow rate of liquid, kg/s
 l_w = Weir length, m
 ρ_L = Liquid density, kg/m³

8.4.7.5 Checking for Weeping

At a very low value of vapour velocity liquid rain down through the perforations. This phenomena is known as weeping. It is again an inoperative condition. Minimum vapour velocity required to avoid the weeping is given by Eduljee's equation.

$$v_{h \min} = \frac{K - 0.9(25.4 - d_h)}{\sqrt{\rho_v}} \quad (8.70)$$

where,
 $v_{h \min}$ = Minimum vapour velocity through the holes, m/s
 d_h = Hole diameter, mm
 ρ_v = Density of vapour, kg/m³
 K = Constant, a function of depth of clear liquid on the plate, which can be obtained from Fig. (8.19).

8.4.7.6 Total Pressure Drop in Sieve Tray Tower

Total pressure drop in sieve tray tower, Δp_T is given by the following equation.

$$\Delta p_T = \text{Pressure drop in inlet nozzle} + \text{Pressure drop/tray} \times \text{No. of trays} + \text{Pressure drop in outlet nozzle}$$

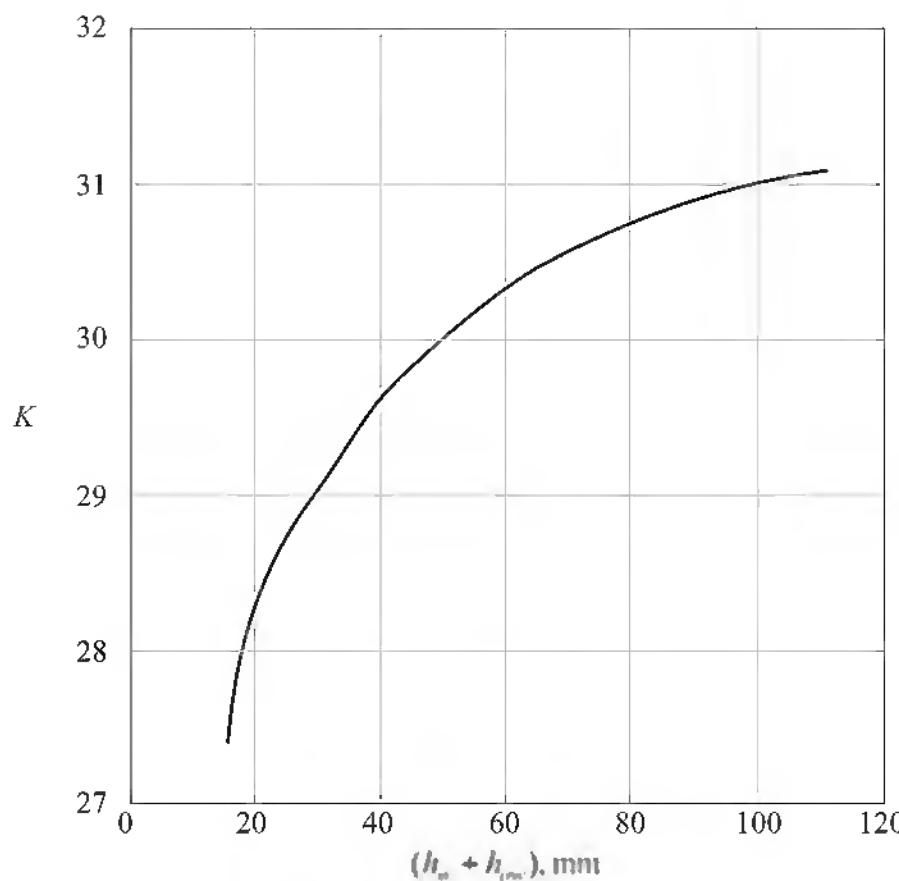


Fig. 8.19 Weep-Point Correlation (Eduljee, 1959)⁹
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Hence major pressure drop in vapour is provided by trays.

$$\Delta p_T \approx N \times \Delta p_t \quad (8.71)$$

where, N = Total number of sieve trays of tower

Δp_t = Tray pressure drop, Pa

Tray pressure drop in Pa or N/m^2 can be determined from the tray pressure drop in mm of liquid by following equation.

$$\Delta p_t = 9.8 \times 10^{-3} h_t \rho_L \quad (8.72)$$

where, Δp_t = Tray pressure drop, N/m^2 or Pa

h_t = Tray pressure drop, mm of liquid column (LC)

ρ_L = Density of liquid, kg/m^3

To find the tray pressure drop simple additive model is used.

$$h_t = h_d + (h_w + h_{ow}) + h_r \quad (8.73)$$

where h_d = Pressure drop through dry plate, mm LC

h_w = Height of weir, mm

h_{ow} = Height of liquid crest, mm

h_r = Residual pressure drop, mm LC

Tray pressure drop (h_t) is the sum of pressure drop which occurs as vapour rises through the perforations of dry plate (h_d), pressure drop which occurs as vapour rises through the pool of clear liquid ($h_w + h_{ow}$) and pressure drop which occurs in vapour–liquid disengagement space (h_r). Due to the high vapour velocity, froth formation takes place over the tray. Some liquid particles are entrained in vapour phase. Hence, some additional energy loss is taking place in carrying

the liquid particles. These liquid particles are disengaged from the vapour after travelling a certain distance. This additional energy loss is counted by residual pressure drop, h_r .

Pressure drop through dry plate is given by following equation.

$$h_d = 51 \left(\frac{v_h}{C_o} \right)^2 \frac{\rho_v}{\rho_L} \quad (8.74)$$

where, h_d = Dry plate pressure drop, mm LC

v_h = Velocity of gas through the holes, m/s

$$= \frac{\text{Volumetric flow rate of gas, } m^3/\text{s}}{\text{Total hole area, } m^2} = \frac{Q_v}{A_h} \quad (8.75)$$

ρ_v, ρ_L = Densities of vapour phase and liquid phases, respectively, kg/m^3

C_o = Orifice coefficient which is a function of ratio of plate thickness to hole diameter and % perforated area of plate. It can be determined by using Fig. 8.20.

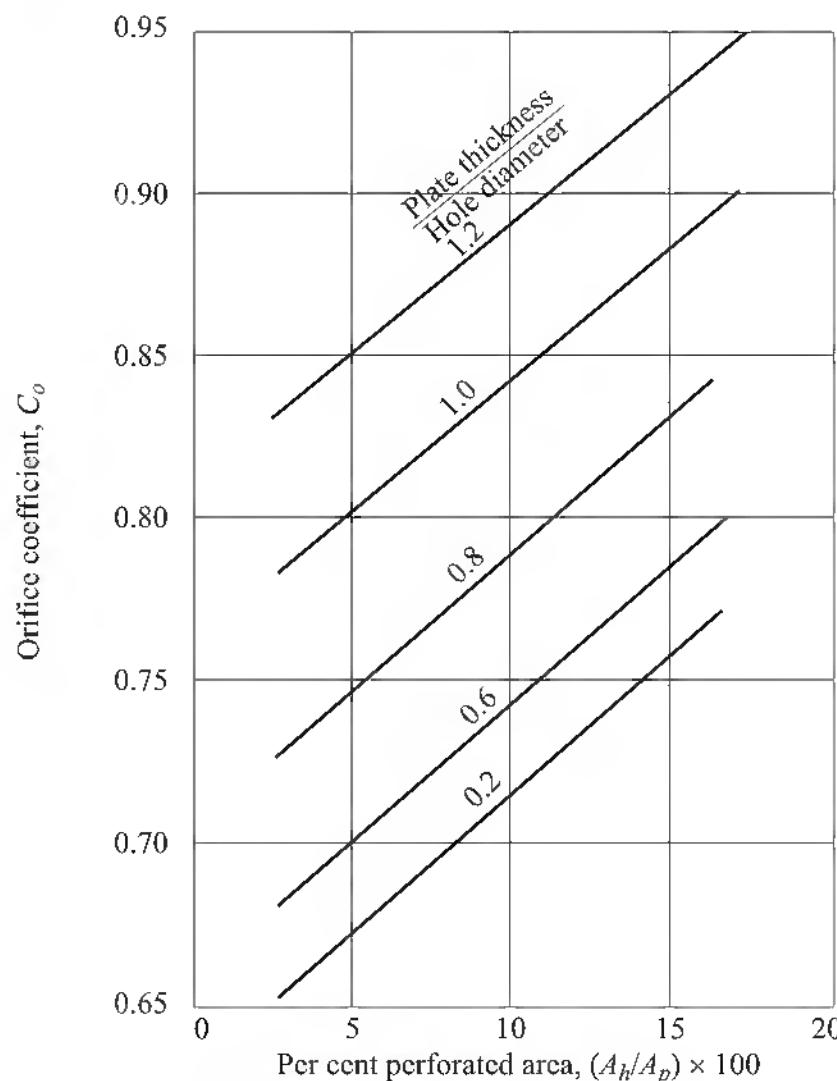


Fig. 8.20 Discharge Coefficient (Sieve Plates)¹⁴
(Reproduced with the permission of McGraw-Hill Education, USA)

$$\% \text{ Perforated area} = \frac{A_h}{A_a} \times 100 \quad (8.76)$$

where, A_h = Total area of all active holes of tray, m^2
 A_a = Active area of tray = $A_c - 2 A_d$, m^2
 A_c = Inside area of column, m^2
 A_d = Downcomer area of tray, m^2

Residual pressure drop is calculated by the following equation.

$$h_r = \frac{12.5 \times 10^3}{\rho_L} \quad (8.77)$$

where, h_r = Residual pressure drop, mm LC
 ρ_L = Density of liquid, kg/m^3

8.4.7.7 Design of Downcomer

There are two functions of a downcomer:

- (a) It provides the passage for the flow of liquid.
- (b) Foamy or frothy liquid enters into downcomer. Hence, downcomer also acts as a vapour-liquid separator. To facilitate the vapour-liquid disengagement, certain minimum residence time (at least 3 seconds) is required in the downcomer.

Following types of downcomers are used:

- (i) **Straight segmental downcomer:** It is the simplest and cheapest and is preferred for most applications. In this type, downcomer channel is formed by a flat plate. The portion of the flat plate which extends down below the plate from the outlet weir is called apron. With this type of downcomer, apron is straight and vertical.
- (ii) **Inclined segmental downcomer:** With this type of segmental downcomer, apron is inclined towards the wall. It is preferred with foamy liquid, with which vapour-liquid disengagement is difficult. Inclined or sloppy apron provides more resistance to flow and makes the phase separation easier. It also increases tray area available for perforations. But with this type, liquid back-up in downcomer is higher compared to straight segmental downcomer.
- (iii) **Circular downcomers or pipes:** They are used for small liquid flow rates in a few cases.

Liquid is falling from tray to tray by passing through the downcomer. Due to the pressure drop across the tray, liquid is flowing from lower pressure to higher pressure. To overcome the pressure difference liquid elevates its level in the downcomer. This is called liquid back-up in the downcomer. Also some pressure drop occurs when liquid flows through the downcomer area. Downcomer pressure drop increases the liquid back-up further. Head loss or pressure drop in the downcomer is estimated by following equation.

$$h_{dc} = 166 \left[\frac{L_{md}}{\rho_L A_m} \right]^2 \quad (8.78)$$

where,
 h_{dc} = Downcomer pressure drop, mm LC
 L_{md} = Liquid flow rate in down comer, kg/s
 ρ_L = Density of liquid, kg/m³
 $A_m = A_d$ or A_{ap} whichever is smaller, m²
 A_d = Downcomer area, m²
 A_{ap} = Clearance area under downcomer apron, m²
 $A_{ap} = h_{ap} l_w / 10^6$ (8.79)

where,
 h_{ap} = Height of the bottom edge of the apron above the plate, mm
 l_w = Length of weir, mm
 $h_{ap} = h_w - (5 \text{ to } 10) \text{ mm}$ (8.80)

where, h_w = Height of weir, mm

Liquid back-up in downcomer in terms of clear liquid is given by following equation.

$$\begin{aligned} h_b &= (h_w + h_{ow}) + h_t + h_{dc} \\ &= \text{Downcomer back-up measured from plate surface, mm (i.e. elevation of clear liquid in downcomer)} \end{aligned} \quad (8.81)$$

Due to the formation of froth and foam, actual height of foamy or frothy liquid in downcomer is quite great, nearly double than h_b .

Density of froth or “aerated” liquid is in between 0.4 to 0.7 times that of the clear liquid. For design purpose, it is taken as 0.5 times the density of liquid. Hence, to avoid the downcomer flooding.

$$h_b \geq \frac{I_t + h_w}{2} \quad (8.82)$$

where, h_b = Liquid back-up in the downcomer measured from plate surface, mm
 I_t = Tray spacing, mm
 h_w = Height of weir, mm

Certain minimum residence time must be allowed in the downcomer to facilitate the disengagement of vapour and liquid phases. Residence time of at least 3 seconds is recommended. Residence time in downcomer is given by following equation.

$$\theta_r = \frac{A_d \cdot h_{bc} \cdot \rho_L}{L_{md}} \quad (8.83)$$

where, A_d = Downcomer area, m²
 h_{bc} = Liquid back-up in downcomer in terms of clear liquid, m
 ρ_L = Density of liquid, kg/m³
 L_{md} = Liquid flow rate in downcomer, kg/s

8.4.8 Step 8: Tray Efficiency and HETP

8.4.8.1 HETP

For the distillation in packed tower, height of packed bed (Z), required for the desired separation, is determined by following equation.

$$Z = N_t \times \text{HETP}$$

where, N_t = Number of theoretical stage required for the desired separation

HETP = Height equivalent to a theoretical plate.

The height of an equivalent equilibrium stage or theoretical plate (HETP) is the height of packing that gives the same separation as an equilibrium stage. HETP for the given type and size of random packing is nearly constant and is nearly independent of the system physical properties. HETP values available in literature are valid only for good liquid distribution and for the reasonable pressure drop.

HETP values for various packing can be obtained from the Table 8.35.

Table 8.35 HETP for Various Packings

Sr. No.	Packing types	Packing size, mm	HETP, m
1.	Pall ring, random (for $\Delta P > 17$ mm WC per m of packing)	25	0.40 – 0.50
		38	0.60 – 0.75
		50	0.75 – 1.00
2.	Berl saddle and Intalox saddle, random (for $\Delta P > 29$ mm WC per m of packing)	25	0.40 – 0.50
		38	0.60 – 0.75
3.	Raschig rings	25 to 50 mm	1.0 m
	Structured packings *		< 0.5 m

*HETP of a structured packing depends on a factor known as F_v which is defined as

$$F_v = v_g \sqrt{\rho_V} \quad (8.84)$$

where, v_g = Superficial vapour velocity, m/s

ρ_V = Vapour density, kg/m³

HETP equal to around 0.5 m is reported for F factor, not exceeding 3, beyond which pressure drop increases steeply.

8.4.8.2 Tray Efficiency

Like VLE data, in case of tray efficiency also, it is better to predict the tray efficiency based on actual data on the same system or similar system of commercial scale plant or of pilot plant. There is no entirely satisfactory method available for finding the tray efficiency. Various correlations are available for finding tray efficiency, but they are used only if the reliable actual data on the same or similar system are not available. Overall efficiency of tray tower is given by following equation:

$$\eta_{oc} = \frac{N_t}{N_a} \quad (8.85)$$

where, N_t = Number of theoretical stages required for the desired separation.

N_a = Actual number of trays required for the desired separation.

Tray efficiency depends on (i) system composition and properties, (ii) rate of throughput and (iii) geometry of tray tower (geometry of tray, tray spacing, etc.).

Two methods are given here to find the tray efficiency; (i) Van Winkle's method and (ii) AIChE method. If actual data for predicting tray efficiency for the given system are not available, then it can be predicted approximately by using these two methods. For the safe design calculation, it is suggested that if the value of tray efficiency obtained based on any of these methods is more than 0.5 then consider the value of overall efficiency as 0.5.

(i) Van Winkle's Correlation or Method¹⁸:

Van Winkle's correlation can be used to predict the tray efficiency of a binary system of distillation. This correlation can be used for both bubble-cap and sieve trays.

$$\eta = 0.07 D_g^{0.14} Sc_L^{0.25} Re^{0.08} \quad (8.86)$$

where, D_g = Surface tension number = $\sigma_L / (\mu_L \cdot u_v)$ (8.87)

σ_L = Liquid surface tension, N/m

μ_L = Liquid viscosity, N · s/m² or kg/(m · s)

u = Superficial vapour velocity, m/s

$$Sc_L = \text{Liquid Schmidt number} = \mu_L / (\rho_L \cdot D_{LK}) \quad (8.88)$$

D_{LK} = Liquid diffusivity of light key component, m²/s

$$Re = \text{Reynolds number} = \frac{h_w u_v \rho_v \times 10^{-3}}{\mu_L (F_A)} \quad (8.89)$$

where, h_w = Weir height, mm

ρ_v = Vapour density, kg/m³

F_A = Fractional area = A_h/A_c

A_h = Area of holes of tray, m²

A_c = Inside area of column, m²

(ii) AIChE Method⁴:

The point efficiency $\dot{\eta}$ is related to number of transfer units by following equation.

$$\frac{1}{\ln(l - \dot{\eta})} = - \left[\frac{1}{N_G} + \frac{mV}{L} \times \frac{1}{N_L} \right] \quad (8.90)$$

where, $\dot{\eta}$ = Point efficiency

N_G = Number of gas phase transfer unit

m = Slope of equilibrium curve at the point in consideration

V, L = Molar flow rates of vapour and liquid phases respectively at the point in consideration

N_L = Number of liquid phase transfer unit

The number of gas phase transfer unit is given by following equation.

$$N_G = \frac{(0.776 + 4.57 \times 10^{-3} h_w - 0.24 F_v + 105 L_p)}{\left(\frac{\mu_v}{\rho_v D_v} \right)^{0.5}} \quad (8.91)$$

where, h_w = Weir height, mm

$$F_v = v_a \sqrt{\rho_v}$$

v_a = Vapour velocity based on active tray area, m/s

$$= \frac{Q_v}{A_a} = \frac{\text{Volumetric flow rate of vapour}}{\text{Active tray area}}$$

L_p = Volumetric liquid flow rate across the plate divided by average width of plate, $\text{m}^3/(\text{s} \cdot \text{m})$

$$\text{Average width of plate} = \frac{A_a}{Z_L} \quad (8.92)$$

Z_L = Length of liquid path across the tray, m

μ_v = Viscosity of vapour, $\text{N} \cdot \text{s}/\text{m}^2$

ρ_v = Density of vapour, kg/m^3

D_v = Vapour diffusivity, m^2/s

The number of liquid phase transfer unit is given by following equation

$$N_L = (4.13 \times 10^8 D_L)^{0.5} (0.21 F_v + 0.15) \theta_L \quad (8.93)$$

where, D_L = Liquid diffusivity, m^2/s

θ_L = Liquid contact time, s

Liquid contact time θ_L can be determined by following equation.

$$\theta_L = \frac{Z_c Z_L}{L_p} \quad (8.94)$$

where, Z_L = Length of liquid path from inlet downcomer to outlet weir, m

Z_c = Liquid hold-up on the plate, m^3 per m^2 active area

For sieve trays,

$$Z_c = 0.006 + 0.73 \times 10^{-3} h_w - 0.24 \times 10^{-3} F_v h_w + 1.22 L_p \quad (8.95)$$

Tray efficiency η is equal to the point efficiency $\dot{\eta}$, if the liquid on tray is perfectly mixed. For the real tray, this may not be true.

Tray efficiency η can be determined from point efficiency by using Fig. (8.21).

In this figure, graph of $\frac{\eta}{\dot{\eta}}$ vs. $\frac{mV}{L} \dot{\eta}$ is drawn for different values of Peclet number. For a tray, Peclet number is given by following equation.

$$Pe = \frac{Z_L^2}{D_e \theta_L} \quad (8.96)$$

where, D_e = Eddy diffusivity, m^2/s

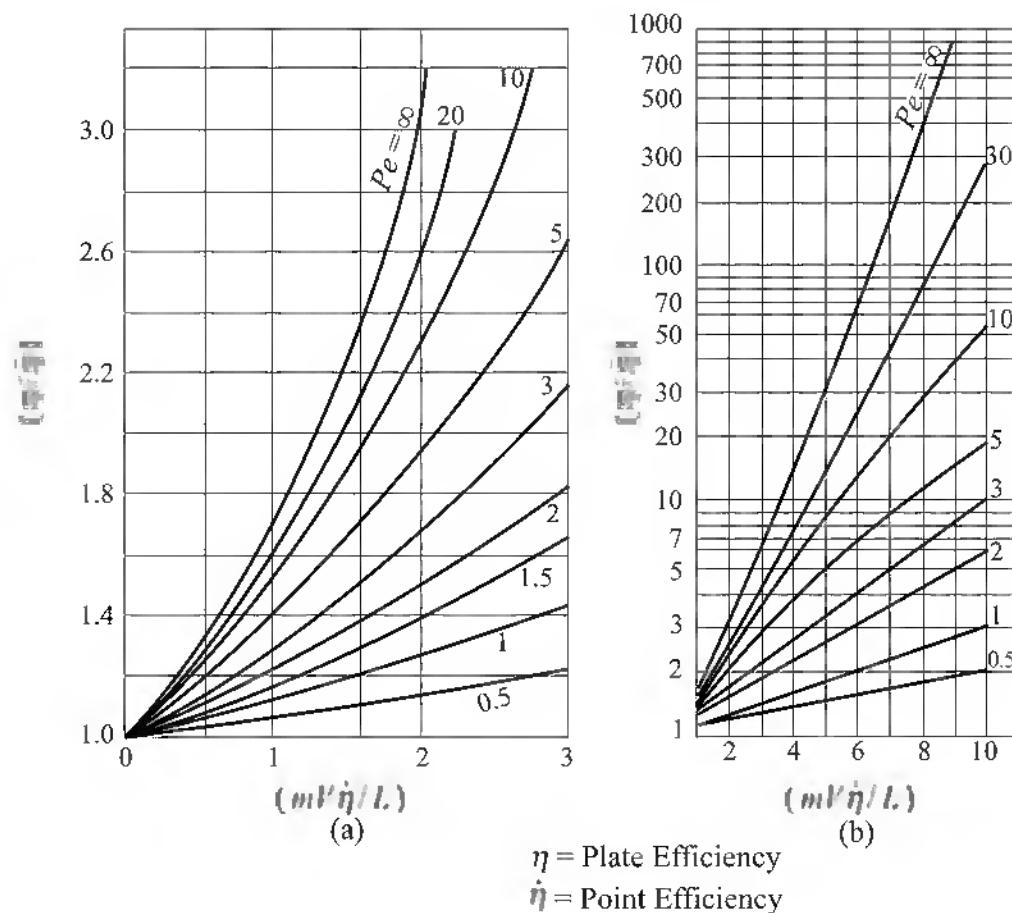


Fig. 8.21 Relationship between Plate and Point Efficiencies¹⁴
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For sieve trays, the Eddy diffusivity can be estimated by the following equation.

$$D_e = (0.0038 + 0.017 v_a + 3.86 L_p + 0.18 \times 10^{-3} h_w)^2 \quad (8.97)$$

In actual operation liquid droplets get entrained in vapour phase. This reduces the value of tray efficiency η . Actual tray efficiency η_a considering the liquid entrainment can be calculated by following equation.

$$\eta_a = \frac{\eta}{1 + \eta \left(\frac{\Psi}{1 - \Psi} \right)} \quad (8.98)$$

where, Ψ = Fractional liquid entrainment.

$$= \frac{\text{Liquid entrained}}{\text{Gross liquid flow}}$$

In Sec. 8.4.7, various parameters relating to the process design of a distillation tower were discussed in general. However, detailed design of the tower internals are well covered in the book by Billet¹⁹. For preparation of the specifications of a tray tower, useful guidelines can be derived from the article by Mukherjee²⁰.

Example 8.13

Design a sieve tray tower for the distillation of acetic acid–water system, specified in Example 8.5. Maximum feed flow rate is 12 000 kg/h and minimum feed flow rate is 8400 kg/h (70% of the maximum).

Solution:

Data obtained from Example 8.5:

- (i) Feed composition: 80% acetic acid and 20% water (by mass) or
54.55% acetic acid and 45.45% water (by mole)
- (ii) Mole fractions of water: In distillate $x_D = 0.9302$, In residue $x_w = 1.666 \times 10^{-4}$
- (iii) $q = 1.272$
- (iv) Reflux ratio, $R = 4.2$
- (v) Number of theoretical stages required for desired separation, $N = 25$

Flow rates:

Average molar mass of feed,

$$M_{av} = \sum x_i M_i = 0.5455 \times 60 + 0.4545 \times 18 = 40.911 \text{ kg/kmol}$$

$$\text{Molar flow rate of feed, } F = \frac{12\,000}{40.911} = 293.32 \text{ kmol/h}$$

$$F = D + W$$

$$Fx_F = Dx_D + Wx_W$$

$$D + W = 293.22$$

$$0.9302 D + 1.666 \times 10^{-4} W = 0.4545 \times 293.32 = 133.314$$

$$0.9302 D + 1.666 \times 10^{-4} (293.3 - D) = 133.314$$

$$D = 143.29 \text{ kmol/h}$$

$$W = 293.32 - 143.29 = 150.03 \text{ kmol/h}$$

Molar flow rates of vapour and liquid at the top in enriching section:

$$L = RD = 4.2 \times 143.29 = 601.818 \text{ kmol/h}$$

$$V = (R + 1) D = 5.2 \times 143.29 = 745.108 \text{ kmol/h}$$

Molar flow rates of vapour and liquid in stripping section:

$$\bar{L} = L + Fq = 601.818 + 293.32 \times 1.272 = 974.92 \text{ kmol/h}$$

$$\bar{V} = F(q - 1) + V = 293.32 \times (1.272 - 1) + 745.108 = 824.89 \text{ kmol/h}$$

Tower diameter calculation:**(a) Tower diameter required at top:**

Operating pressure at top of column = 1 atm = 101.325 kPa

$$V = 745.108 \text{ kmol/h}$$

$$L = 601.818 \text{ kmol/h}$$

Here total condenser is used, hence composition of vapour leaving the top most tray and composition of liquid entering to the top most tray (reflux) are equal.

$$\frac{L_w}{V_w} = \frac{L}{V} = \frac{601.818}{745.108} = 0.8077$$

where, L_w , V_w = Mass flow rates of liquid and vapour at the top, kg/s

Density of vapour, ρ_v :

$$\rho_v = \frac{pM_{av}}{RT}$$

Average molar mass of vapour at top

$$M_{av} = \sum x_i M_i = (1 - 0.9302) \times 60 + 0.9302 \times 18 = 20.9316 \text{ kg/kmol}$$

$$\rho_v = \frac{20.9316}{(t_T + 273)} \times \frac{273}{22.414}$$

To find the temperature of vapour at top, in $t - x - y$ data, put $y = x_D = 0.9302$ and find the corresponding value of temperature from Table (8.6).

$$y = x_D = 0.9302 \quad t = t_T = 100.64^\circ\text{C}$$

$$\rho_v = 0.6823 \text{ kg/m}^3$$

Density of liquid at top, ρ_L :

$$\rho_L = \frac{1}{\sum \frac{w_i}{\rho_i}}$$

Density of pure water at 100.64°C , $\rho_w = 958 \text{ kg/m}^3$

Density of pure acetic acid at 100.64°C , $\rho_{AA} = 1010 \text{ kg/m}^3$

Mass fraction of water in distillate = 0.8

Mass fraction of acetic acid in distillate = 0.2

$$\rho_L = \frac{1}{\left(\frac{0.8}{958}\right) + \left(\frac{0.2}{1010}\right)} = 967.97 \text{ kg/m}^3$$

Liquid-vapour flow factor at top,

$$F_{LV} = \frac{L_W}{V_W} \left(\frac{\rho_v}{\rho_L} \right)^{0.5} \quad (8.66)$$

$$F_{LV} = 0.8077 \left(\frac{0.6823}{967.97} \right)^{0.5} = 0.021444$$

For the first trial calculations, tray spacing = 0.3 m (assumed)

From Fig. 8.16,

$$C_f = 0.0635$$

Flooding velocity,

$$v_F = C_f \left(\frac{\sigma}{0.02} \right)^{0.2} \left(\frac{\rho_L - \rho_v}{\rho_v} \right)^{0.5} \quad (8.65)$$

where, v_F = Flooding velocity, m/s

σ = Surface tension of liquid, N/m

$$\sigma = \sum \sigma_i x_i = (1 - 0.9302) \sigma_{AA} + 0.9302 \sigma_w$$

where, σ_w = Surface tension of pure water, N/m = 58×10^{-3} N/m

σ_{AA} = Surface tension of pure acetic acid, N/m

σ_{AA} at 100.64°C

$$\sigma_{AA}^{1/4} = [P](\rho'_L - \rho'_V) \quad (8.99)$$

σ_{AA} = surface tension of acetic acid, dyn/cm
 ρ'_L = density of liquid acetic acid, mol/cm³

$$\rho'_L = \frac{1010 \times 10^{-3}}{60} = \frac{1.01}{60} = 0.016833 \frac{\text{mol}}{\text{cm}^3}$$

ρ'_V = density of acetic acid vapour, mol/cm³

$$\rho'_V = \frac{1}{(273 + 100.64)} \times \frac{T_s}{p_s \cdot V_s}$$

$$\rho'_V = \frac{1}{(273 + 100.64)} \times \frac{273}{22414} = 3.26 \times 10^{-5} \text{ mol/cm}^3$$

[P] = Parachor can be calculated from Table 3–343, of Ref. 14.

[P] = 55.5 (Contribution of CH₃ –) + 73.8 (Contribution of – COOH)

[P] = 129.3

$$\sigma_{AA}^{1/4} = 129.3 (0.016833 - 3.26 \times 10^{-5}) = 2.1723$$

$$\sigma_{AA} = 22.268 \text{ dyn/cm} \equiv 22.268 \times 10^{-3} \text{ N/m}$$

$$\sigma = (1 - 0.9302) \times 22.268 \times 10^{-3} + 0.9302 \times 58 \times 10^{-3}$$

$$\sigma = 55.5 \times 10^{-3} \text{ N/m}$$

$$v_F = 0.0635 \left(\frac{55.5 \times 10^{-3}}{0.02} \right)^{0.2} \left(\frac{967.97 - 0.6823}{0.6823} \right)^{0.5}$$

$$v_F = 2.93 \text{ m/s}$$

Let actual vapour velocity through tower, $v = 0.85 v_F$

$$v = 2.49 \text{ m/s}$$

Volumetric flow rate of vapour at top

$$\dot{Q} = \frac{VM_{av}}{\rho_v} = \frac{745.108 \times 20.9316}{0.6823} = 22858.4 \text{ m}^3/\text{h} \equiv 6.3496 \text{ m}^3/\text{s}$$

Net area required at top

$$A_n = \frac{\dot{Q}}{v} = \frac{6.3496}{2.49} = 2.55 \text{ m}^2$$

Let downcomer area, $A_d = 0.12 A_c$

$$A_n = A_c - A_d = A_c - 0.12 A_c = 0.88 A_c$$

where, A_c = Inside cross sectional area of tower

$$0.88 A_c = 2.55$$

$$A_c = \frac{2.55}{0.88} = 2.8977 \text{ m}^2$$

Inside diameter of column required at top

$$D_i = \sqrt{\frac{4 \times A_c}{\pi}} = \sqrt{\frac{4 \times 2.8977}{\pi}} = 1.92 \text{ m say } 1.95 \text{ m at the top}$$

(b) Tower diameter required at bottom:

Operating pressure at the base of column = Operating pressure at top + Δp_T

where, Δp_T = Total pressure drop in sieve tray tower

$$h_t = 120 \text{ mm WC}$$

$$\Delta p_T \equiv \text{Actual no. of trays} \times \rho_w g h_t$$

Assuming tray efficiency = 0.5

$$\text{Actual number of trays, } N_a = \frac{N}{0.5} = \frac{25}{0.5} = 50$$

$$\Delta p_T = 50 \times 1000 \times 9.81 \times 120 \times 10^{-3} = 58860 \text{ Pa} \equiv 58.86 \text{ kPa (assumed)}$$

Operating pressure at base

$$p_t = p_t + \Delta p_T = 101.325 + 58.86 = 160.185 \text{ kPa} \equiv 1.58 \text{ atm}$$

Bottom product is nearly pure acetic acid. Antoine equation¹³ for acetic acid

$$\ln p_v = 15.8667 - \frac{4097.86}{T - 27.4937}$$

When $p_v = p'_v$, $T = T_b$ = Base temperature of column

$$\ln (160.185) = 15.8667 - \frac{4097.86}{T - 27.4937}$$

$$T = 407.26 \text{ K} \equiv 134.26^\circ\text{C (Estimated)}$$

Molar flow rates of vapour and liquid at bottom

$$\bar{V} = 804.89 \text{ kmol/h}, \bar{L} = 974.92 \text{ kmol/h}$$

At the base of column,

$$\frac{\bar{L}_w}{\bar{V}_w} \equiv \frac{\bar{L}}{\bar{V}} = \frac{974.92}{824.89} = 1.182$$

where \bar{L}_w, \bar{V}_w = Mass flow rates of vapour and liquid at bottom, kg/s

Density of vapour at bottom

$$\rho_v' = \frac{p_t' M}{R T_b} = \frac{160.185 \times 60}{(273 + 134.260)} \times \frac{273}{101.325 \times 22.414}$$

$$\rho_v' = 2.8368 \text{ kg/m}^3$$

Density of liquid (nearly pure acetic acid) at bottom

$$\begin{aligned} \rho_L &= \text{density of acetic acid at } 134.26^\circ\text{C and at } 160.185 \text{ kPa} \\ &= 1000 \text{ kg/m}^3 \text{ (assumed)} \end{aligned}$$

Liquid-vapour flow factor at bottom

$$F_{LV} = \frac{\bar{L}_w}{\bar{V}_w} \left(\frac{\rho_v'}{\rho_L} \right)^{0.5}$$

$$F_{LV} = 1.182 \left(\frac{2.8368}{1000} \right)^{0.5} = 0.06296$$

For tray spacing = 0.3 m, from Fig. 8.16

$$C_f = 0.0616$$

Flooding velocity

$$v_F = C_f \left(\frac{\sigma'}{0.02} \right)^{0.2} \left(\frac{\rho_L - \rho_V}{\rho_V} \right)^{0.5} \quad (8.65)$$

Surface tension of pure acetic at 134.26°C temperature

$$\sigma_{AA}^{1/4} = [\varphi](\rho_L'' - \rho_V'')$$

$$\rho_L'' = 1000 \times 10^{-3} \times \frac{1}{60} = 0.01667 \text{ mol/cm}^3$$

$$\rho_V'' = 2.8368 \times \frac{10^{-3}}{60} = 4.728 \times 10^{-5} \text{ mol/cm}^3$$

$$\sigma_{AA}^{1/4} = 129.3 (0.01667 - 4.728 \times 10^{-5}) = 2.1493$$

$$\sigma_{AA} = 21.34 \text{ dyn/cm} \equiv 21.34 \times 10^{-3} \text{ N/m}$$

$$v_F = 0.0616 \left(\frac{21.34 \times 10^{-3}}{0.02} \right)^{0.2} \left(\frac{1000 - 2.8368}{2.8368} \right)^{0.5}$$

$$v_F = 1.17 \text{ m/s}$$

Actual vapour velocity, $v = 0.85 \times 1.17 = 0.9945 \text{ m/s}$

Volumetric flow rate of vapour at bottom

$$\dot{Q} = \frac{\bar{V}M_{av}}{\rho_V''} = \frac{(824.89 \times 60)/3600}{2.8368} = 4.8464 \text{ m}^3/\text{s}$$

Net area required at bottom

$$A_n = \frac{4.8464}{0.9945} = 4.8732 \text{ m}^2$$

Inside area of column

$$A_c = \frac{4.8732}{0.88} = 5.538 \text{ m}^2$$

Inside diameter of column required at base

$$D_i = \sqrt{\frac{4 \times A_c}{\pi}} = \sqrt{\frac{4 \times 5.538}{\pi}} = 2.655 \text{ m}$$

Up to 1 m diameter 0.3 m tray spacing is sufficient. Here, tower diameters are greater than 1 m. Hence, for the 2nd trial calculations, increase the tray spacing to 0.45 m.

For tray spacing = 0.45 m

(a) Tower diameter required at top:

For $F_{LV} = 0.02144$ and tray spacing = 0.45 m

From Fig. 8.16, $C_f = 0.0814$

$$v_F = \frac{0.0814}{0.0635} \times 2.93 = 3.756 \text{ m/s}$$

Actual vapour velocity, $v = 0.85 v_F = 3.1926 \text{ m/s}$

Net area required

$$A_n = \frac{6.3496}{3.1926} = 1.989 \text{ m}^2$$

$$A_c = \frac{A_n}{0.88} = 2.26 \text{ m}^2$$

Inside diameter at top

$$D_i = \sqrt{\frac{4 \times 2.26}{\pi}} = 1.7 \text{ m}$$

(b) Tower diameter required at bottom:

For $F_{LV} = 0.06296$ and tray spacing = 0.45 m

From Fig. 8.16, $C_f = 0.0794$

$$v_F = \frac{0.0794}{0.0616} \times 1.17 = 1.5 \text{ m/s}$$

Actual vapour velocity, $v = 0.85$ $v_F = 1.275 \text{ m/s}$

$$\text{Net area required, } A_n = \frac{4.8464}{1.275} = 3.8 \text{ m}^2$$

Inside diameter of column required at bottom

$$A_c = \frac{A_n}{0.88} = 4.32 \text{ m}^2$$

Inside diameter of column required at bottom

$$D_i = \sqrt{\frac{4 \times 4.32}{\pi}} = 2.345 \text{ m say } 2.36 \text{ m}$$

Provide different diameters in two different sections.

Table 8.36 Summary of Calculations

Enriching Section	Stripping Section
$D_i = 1.7 \text{ m}$	$D_i = 2.36 \text{ m}$
$A_c = 2.27 \text{ m}^2$	$A_c = 4.374 \text{ m}^2$
$A_n = 1.998 \text{ m}^2$	$A_n = 3.85 \text{ m}^2$
Downcomer area, $A_d = 0.2724 \text{ m}^2$	$A_d = 0.525 \text{ m}^2$
Active area, $A_a = 1.7252 \text{ m}^2$	$A_a = 3.3242 \text{ m}^2$
Hole area, $A_h = 0.17252 \text{ m}^2$	$A_h = 0.33242 \text{ m}^2$

For the 1st trial, hole area A_h is taken as 10% of active area, A_a .

Volumetric flow rate of liquids:

$$\text{In enriching section, } \dot{Q}_L = \frac{LM_{av}}{\rho_L} = \frac{601.818 \times 20.9316}{967.97}$$

$$= 13.0138 \text{ m}^3/\text{h} \equiv 3.6 \times 10^{-3} \text{ m}^3/\text{s}$$

In stripping section

$$\dot{Q}_L = \frac{\bar{L}M_{av}}{\rho_L} = \frac{974.92 \times 60}{1000} = 54.495 \text{ m}^3/\text{h} \equiv 16.25 \times 10^{-3} \text{ m}^3/\text{s}$$

Referring Table 8.33, single pass tray can be used for both sections.

Check for weeping:

Minimum vapour velocity through holes to avoid the weeping is given by following equation.

$$v_{h\ min} = \frac{K - 0.9(25.4 - d_h)}{\sqrt{\rho_v}} \quad (8.70)$$

where, K = constant can be obtained from Fig. 8.19 is a function of $(h_w + h_{ow})$

Take

Weir height, $h_w = 50 \text{ mm}$ (For both sections)

Hole diameter, $d_h = 5 \text{ mm}$ (For both sections)

Plate thickness, $t = 5 \text{ mm}$ (For both sections)

(a) For enriching section:

Height of liquid crest over the weir

$$h_{ow} = 750 \left(\frac{L_m}{\rho_L l_w} \right)^{2/3} \quad (8.69)$$

For the checking of weeping conditions minimum value of h_{ow} at 70% turn down must be determined. Minimum value of

$$L_W = 0.7 LM_{av} = 0.7 \times 601.818 \times 20.9316$$

$$L_W = 8817.9 \text{ kg/h} \equiv 2.45 \text{ kg/s (minimum)}$$

$$\rho_L = 967.97 \text{ kg/m}^3$$

Referring Table 8.34, for $A_d/A_c = 0.12$, $l_w/D_i = 0.77$

$$l_w = \text{Length of weir} = 0.77 D_i$$

$$l_w = 0.77 \times 1.7 = 1.309 \text{ m}$$

$$\text{Minimum } h_{ow} = 750 \left(\frac{2.45}{967.97 \times 1.309} \right)^{2/3} = 11.64 \text{ mm}$$

At minimum rate, $h_w + h_{ow} = 50 + 11.64 = 61.64 \text{ mm}$

From Fig. 8.19, $K = 30.38$

$$v_{h\ min} = \frac{30.38 - 0.9(25.4 - 5)}{(0.6823)^{0.5}} = 14.552 \text{ m/s}$$

Actual vapour velocity through holes at minimum vapour flow rate,

$$v_{ha} = \frac{0.7 \dot{Q}_v}{A_h} = \frac{0.7 \times 6.3496}{0.17252} = 25.76 \text{ m/s}$$

Hence, $v_{ha} > v_{h\ min}$

Thus in the enriching section, minimum operating rate is well above weep point.

(b) For stripping section:

Minimum value of $\bar{L}_w = 0.7 \times \bar{L}_w = 0.7 \times 974.92 \times 60$

Minimum $\bar{L}_w = 40946.64 \text{ kg/h} \equiv 11.374 \text{ kg/s}$

$$\rho_L = 1000 \text{ kg/m}^3$$

$$l_w = 0.77 \times 2.36 = 1.8172 \text{ m}$$

$$\text{Minimum } h_{ow} = 750 \left(\frac{11.374}{1000 \times 1.8172} \right)^{2/3} = 25.47 \text{ mm}$$

At minimum rate, $h_w + h_{ow} = 50 + 25.47 = 75.47 \text{ mm}$

From Fig. 8.19, $K = 30.69$

$$v_{h \min} = \frac{30.69 - 0.9(25.4 - 5)}{(2.8368)^{0.5}} = 7.32 \text{ m/s}$$

Actual vapour velocity through holes at minimum vapour flow rate

$$v_{ha} = \frac{0.7 \times \dot{Q}_v}{A_h} = \frac{0.7 \times 4.8464}{0.33242} = 10.2054 \text{ m/s}$$

Hence, $v_{ha} > v_{h \ min}$

In stripping section also, minimum operating rate is above the weep point.

Tray pressure drop:

(a) Tray pressure drop for enriching section

Dry plate pressure drop

$$h_d = 51 \left(\frac{v_h}{C_o} \right)^2 \frac{\rho_v}{\rho_L} \quad (8.74)$$

$$v_h = \frac{\dot{Q}_v}{A_h} = \frac{6.3496}{0.17252} = 36.8 \text{ m/s (maximum)}$$

$$\rho_v = 0.6823 \text{ kg/m}^3, \rho_L = 967.97 \text{ kg/m}^3$$

From Fig. 8.20, for $\frac{\text{Plate thickness}}{\text{Hole diameter}} = 1$ and $\frac{A_h}{A_p} \approx \frac{A_h}{A_a} = 0.1, C_o = 0.8422$

(A_p = Perforated area is slightly less than active area, A_a)

$$h_d = 51 \left(\frac{36.8}{0.8422} \right)^2 \times \frac{0.6823}{967.97} = 68.64 \text{ mm LC}$$

$$h_w = 50 \text{ mm LC}$$

Maximum height of liquid crest over the weir

$$\text{Maximum } h_{ow} = 750 \left(\frac{L_w}{\rho_L l_w} \right)^{2/3} = 750 \left(\frac{2.45/0.7}{967.97 \times 1.309} \right)^{2/3} = 14.765 \text{ mm LC}$$

Residual pressure drop,

$$h_r = \frac{12.5 \times 10^3}{\rho_L} \quad (8.77)$$

$$h_r = \frac{12.5 \times 10^3}{967.97} = 12.9136 \text{ mm LC}$$

Total tray pressure drop

$$h_t = h_d + (h_w + h_{ow}) + h_r \quad (8.73)$$

$$h_t = 68.64 + (50 + 14.765) + 12.9136 = 146.32 \text{ mm LC}$$

Note: This is equivalent to 142 mm WC. Tray pressure drop was assumed equal to 120 mm WC to estimate the base pressure. The calculations can be repeated with a revised value of tray pressure drop but small changes in physical properties will have little effect on the tray design. However, correct estimate of base temperature is required and is important for the reboiler design.

(b) Tray pressure drop for stripping section:

Dry plate pressure drop, h_d :

$$v_h = \frac{4.8464}{0.33242} = 14.58 \text{ m/s}$$

$$\rho_v = 2.8368 \text{ kg/m}^3, \quad \rho_L = 1000 \text{ kg/m}^3, \quad C_o = 0.8422 \text{ (From Fig. 8.20)}$$

$$h_d = 51 \left(\frac{14.58}{0.8422} \right)^2 \times \frac{2.8368}{1000}$$

$$h_d = 43.36 \text{ mm LC}$$

$$h_w = 50 \text{ mm LC}$$

Maximum height of liquid crest over the weir

$$h_{ow} = 750 \left(\frac{11.374/0.7}{1000 \times 1.8172} \right)^{2/3} = 32.31 \text{ mm LC}$$

Residual pressure drop

$$h_r = \frac{12.5 \times 10^3}{1000} = 12.5 \text{ mm LC}$$

Total tray pressure drop

$$h_t = 43.36 + (50 + 32.31) + 12.5 = 138.17 \text{ mm: say 140 mm LC}$$

Checking of downcomer design:

Types of downcomer: Straight and segmental downcomer area, $A_d = 0.12 A_c$ (For both sections)

(a) For enriching section:

Pressure drop in downcomer is given by following equation.

$$h_{dc} = 166 \left[\frac{L_{md}}{\rho_L A_m} \right]^2 \quad (8.78)$$

L_{md} = Liquid flow rate through downcomer, kg/s

$$L_{md} = LM_{av} = 601.818 \times 20.9316 \times \frac{1}{3600}$$

$$= 3.5 \text{ kg/s}$$

$$\rho_L = 967.97 \text{ kg/m}^3$$

$$A_m = A_d \text{ or } A_{ap}, \text{ whichever is smaller, m}^2$$

$$A_d = 0.2724 \text{ m}^2$$

$$A_{ap} = h_{ap} \times l_w$$

$$\begin{aligned}
 l_w &= 1.309, \\
 h_{ap} &= h_w - 10 \\
 &= 50 - 10 = 40 \text{ mm} \equiv 0.04 \text{ m} \\
 A_{ap} &= 0.04 \times 1.309 = 0.05236 \text{ m}^2 \\
 A_{ap} &\ll A_d. \text{ Therefore, take } A_m = A_{ap}. \\
 A_m &= 0.05236 \text{ m}^2
 \end{aligned}$$

$$h_{dc} = 166 \left(\frac{3.5}{967.97 \times 0.05236} \right)^2$$

$$h_{dc} = 0.79 \text{ mm, say 1 mm}$$

Liquid back-up in downcomer

$$\begin{aligned}
 h_b &= h_w + h_{ow} + h_t + h_{dc} \\
 h_b &= 50 + 14.765 + 144.25 + 1 = 210 \text{ mm}
 \end{aligned} \tag{8.81}$$

$$\frac{l_t + h_w}{2} = \frac{450 + 50}{2} = 250 \text{ mm}$$

$$h_b = 210 < \frac{l_t + h_w}{2}$$

Downcomer area and tray spacing are acceptable.

Check residence time:

$$\theta_r = \frac{A_d h_{bc} \rho_L}{L_{md}} \tag{8.83}$$

$$\theta_r = \frac{0.2724 \times 0.210 \times 967.97}{3.5}$$

$$(h_{bc} = h_b = 210 \text{ mm})$$

$$\theta_r = 15.82 \text{ s} > 3 \text{ s}$$

Hence, it is satisfactory.

(b) For stripping section:

Pressure drop in downcomer

$$h_{dc} = 166 \left(\frac{11.374/0.7}{1000 \times A_m} \right)^2$$

Downcomer area, $A_d = 0.525 \text{ m}^2$

$$A_{ap} = h_{ap} \cdot l_w$$

$$h_{ap} = 50 - 10 = 40 \text{ mm}, l_w = 1.8172 \text{ m}$$

$$A_{ap} = 0.04 \times 1.8172 = 0.0727 \text{ m}^2$$

$$A_m = 0.0727 \text{ m}^2$$

$$h_{dc} = 8.29 \text{ mm}$$

Liquid back-up in downcomer

$$h_b = h_w + h_{ow} + h_t + h_{dc}$$

$$h_b = 50 + 32.31 + 140 + 8.29 = 230.6 \text{ mm}$$

$$\frac{l_t + h_w}{2} = \frac{450 + 50}{2} = 250 \text{ mm}$$

$$h_b = 230.6 < \frac{l_t + h_w}{2}$$

Here, downcomer area and tray spacing are acceptable.

Residence time in downcomer

$$t_r = \frac{A_d h_b \rho_L}{L_{md}} = \frac{0.525 \times 0.2306 \times 1000}{(11.374/0.7)}$$

$$t_r = 7.45 \text{ s} > 3 \text{ s}$$

Hence, it is satisfactory.

Checking of entrainment:

(a) For enriching section:

Vapour velocity based on net area, $v_n = \frac{\dot{Q}}{A_n}$

$$v_n = \frac{6.3496}{1.998} = 3.178 \text{ m/s}$$

$$\% \text{ flooding} = \frac{v_n}{v_f} \times 100 = \frac{3.178}{3.756} \times 100 = 85\%$$

$$F_{LV} = 0.021444, \quad \Psi = 0.21 \text{ (from Fig. 8.18)}$$

% entrainment = 21 % > 10 %

If Ψ is less, then higher tray efficiency is obtained. But to decrease the value of Ψ , tower diameter must be increased further. Ideally, optimum value of Ψ must be determined for the given case. Optimum value of Ψ can be greater than 0.1 or 10%.

(b) For stripping section:

% Flooding = 85 %, $F_{LV} = 0.06296$

From Fig. 8.18 $\Psi = 0.084$

% entrainment = 8.4 % < 10 %

It will provide higher tray efficiency.

Tray efficiency by AIChE method:

(a) For enriching section:

Physical properties of liquid and vapour for the same tray are

$$\rho_L = 967.97 \text{ kg/m}^3, \sigma_L = 55.5 \times 10^{-3} \text{ N/m}$$

μ_L = Viscosity of reflux or distillate (water-acetic acid solution at 100.64°C)

$$\ln \mu_L = w_1 \ln \mu_1 + w_2 \ln \mu_2$$

$w_1 = 0.8$ = mass fraction of water

$w_2 = 0.2$ = mass fraction of acetic acid

μ_w = Viscosity of pure water at 100.64°C = 0.28 mPa · s or cP

μ_{AA} = Viscosity of pure acetic acid at 100.64°C = 0.48 mPa · s or cP

(Fig. 3-43 of Ref. 14)

$$\mu_L = 0.312 \text{ mPa · s or cP} = 0.312 \times 10^{-3} (\text{N} \cdot \text{s})/\text{m}^2$$

Diffusivity of acetic acid in water at 100.64°C

$$D_L = 1.24 \times 10^{-5} \text{ cm}^2/\text{s} \text{ at } 25^\circ\text{C}$$

(Table 3.319 of Ref. 14)

To find D_L at 100.64°C,

$$\frac{D_L \mu}{T} = \text{constant}$$

where, μ = Viscosity of solvent (water)

$$\mu_w \text{ at } 25^\circ\text{C} = 0.95 \text{ mPa}\cdot\text{s or cP}$$

$$D_L = \frac{1.24 \times 10^{-5} \times 0.95}{(273 + 25)} \times \frac{(273 + 100.64)}{0.28}$$

$$D_L = 5.275 \times 10^{-5} \text{ cm}^2/\text{s} \equiv 5.275 \times 10^{-9} \text{ m}^2/\text{s}$$

For vapour:

$$\rho_v = 0.6823 \text{ kg/m}^3$$

μ_v = Viscosity of vapour mixture

$$\mu_v = \sum \frac{y_i \mu_i}{\sum y_{ij} \Theta_{ij}}$$

$$\Theta_{ij} = \frac{\left[1 + \left(\frac{\mu_i}{\mu_j} \right)^{1/2} \left(\frac{M_j}{M_i} \right)^{1/4} \right]^2}{\left[8 \left(1 + \frac{M_i}{M_j} \right) \right]^{1/2}}$$

and

$$\Theta_{ij} = \left(\frac{\mu_j}{\mu_i} \right) \left(\frac{M_i}{M_j} \right) \Theta_{ij}$$

(Eq. (3-87) to (3-89) of Ref. 14)

Viscosity of acetic acid vapour at 100.64°C

$$\mu_A = 1000 \times 10^{-7} \text{ poise} = 0.01 \text{ cP or mPa}\cdot\text{s} \text{ (} i \text{ for acetic acid)}$$

Viscosity of water vapour at 100.64°C

$$\mu_w = 1250 \times 10^{-7} \text{ poise} = 1250 \times 10^{-5} \text{ cP or mPa}\cdot\text{s} \text{ (} j \text{ for water)}$$

$$\Theta_{ij} = \frac{\left[1 + \left(\frac{1000}{1250} \right)^{1/2} \left(\frac{18}{60} \right)^{1/4} \right]^2}{\left[8 \left(1 + \frac{60}{18} \right) \right]^{1/2}} = 0.4691$$

$$\Theta_{ij} = \left(\frac{1250}{1000} \right) \left(\frac{60}{18} \right) \times 0.4691 = 1.9546$$

$$y_i = 1 - 0.9302 = 0.0698, \quad y_j = 0.9302$$

$$\mu_v = \frac{(0.0698 \times 0.01) + (0.9302 \times 0.0125)}{(0.9302 \times 0.4691) + (0.0698 \times 1.9546)}$$

$$\mu_v = 0.0215 \text{ cP or mPa}\cdot\text{s} \equiv 0.0215 \times 10^{-3} (\text{N}\cdot\text{s})/\text{m}^2$$

D_v = Diffusivity of acetic acid vapour in water vapour.

$$D_v = \frac{10^{-3} T^{1.75} [(M_A + M_w)/(M_A M_w)]^{1/2}}{p[(\Sigma V)_A^{1/3} + (\Sigma V)_w^{1/3}]^2}$$

where, D_v is in cm^2/s . T in K, p is operating pressure in atmosphere, M_A and M_w are molar masses and ΣV is atomic diffusion volume.

(Equation 3–133 of Ref. 14. ΣV can be determined by using Table–342 of the same reference)

For acetic acid (CH_3COOH)

$$(\Sigma V)_A = 2 \times 16.5 + 4 \times 1.98 + 2 \times 5.48 = 51.88$$

For water (H_2O),

$$(\Sigma V)_w = 2 \times 1.98 + 5.48 \times 1 = 9.44$$

$$T = 273 + 100.64 = 373.64 \text{ K}$$

$$D_v = \frac{10^{-3} \times (373.64)^{1.75} [(60+18)/(60 \times 18)]^{1/2}}{1 \times [51.88^{1/3} + 9.44^{1/3}]^2}$$

$$D_v = 0.25 \text{ cm}^2/\text{s} = 0.25 \times 10^{-4} \text{ m}^2/\text{s}$$

Number of gas phase transfer units is calculated by following equation

$$N_G = \frac{0.776 + 4.57 \times 10^{-3} h_w - 0.24 F_v + 105 L_p}{\left(\frac{\mu_v}{\rho_v D_v} \right)^{0.5}} \quad (8.91)$$

h_w = weir height = 50 mm

$$F_v = v_a \sqrt{\rho_v} \quad (8.84)$$

$$v_a = \text{Vapour velocity based on active tray area} = \frac{\dot{Q}_{v \min}}{A_a} = \frac{0.7 \dot{Q}_v}{A_a}$$

$$= \frac{0.7 \times 6.3496}{1.7252} = 2.5764 \text{ m/s}$$

$$F_v = 2.5764 \sqrt{0.6823} = 2.128$$

$$L_p = \frac{\text{Volumetric flow rate of liquid}}{\text{Average width of plate}}$$

$$L_p = \frac{0.7 \times \dot{Q}_L}{(A_a / Z_L)} = \frac{0.7 \times 3.6 \times 10^{-3}}{1.7252 / Z_L}$$

where,

Z_L = Length of liquid path
= Horizontal distance between weir and downcomer apron

Using Mathematical Table 1.19 a of Ref. 14.

$$\frac{l_w}{D/2} = 2 \frac{l_w}{D} = 2 \times 0.77 = 1.54$$

Chord h is given by

$$\frac{h}{R} = 0.362$$

$$h = \frac{0.362 \times D_i}{2} = 0.181 D_i$$

$$Z_L = D_i - 2h = D_i - 2 \times 0.181 D_i = D_i (1 - 2 \times 0.181)$$

$$Z_L = 0.638 \times D_i = 0.638 \times 1.7 = 1.0846 \text{ m}$$

$$L_p = 1.5843 \times 10^{-3} \text{ m}^2/\text{s}$$

$$\frac{\mu_v}{\rho_v D_v} = \frac{0.0215 \times 10^{-3}}{0.6823 \times 0.25 \times 10^{-4}} = 1.26$$

$$N_G = \frac{(0.776 + 4.57 \times 10^{-3} \times 50 - 0.24 \times 2.128 + 105 \times 1.5843 \times 10^{-3})}{\sqrt{1.26}}$$

$$N_G = 0.588$$

Liquid hold-up on the plate, m^3 per m^2 active area, Z_c is given by equation

$$\begin{aligned} Z_c &= 0.006 + 0.73 \times 10^{-3} h_w - 0.24 \times 10^{-3} F_v h_w + 1.22 L_p \\ &= 0.006 + (0.73 \times 10^{-3} \times 50) - (0.24 \times 10^{-3} \times 2.128 \times 50) \\ &\quad + (1.22 \times 1.5846 \times 10^{-3}) = 0.0189 \text{ m}^3/\text{m}^2 \end{aligned} \quad (8.95)$$

Liquid contact time

$$\theta_L = \frac{Z_c Z_L}{L_p} \quad (8.94)$$

$$\theta_L = \frac{0.0189 \times 1.0846}{1.5843 \times 10^{-3}} = 12.94 \text{ s}$$

Number of liquid phase transfer unit

$$\begin{aligned} N_L &= (4.13 \times 10^8 D_L)^{0.5} (0.21 F_v + 0.15) \theta_L \\ &= (4.13 \times 10^8 \times 5.275 \times 10^{-9})^{0.5} (0.21 \times 2.128 + 0.15) \times 12.94 = 11.4 \end{aligned} \quad (8.93)$$

Eddy diffusivity D_e for sieve tray can be estimated by following equation.

$$\begin{aligned} D_e &= (0.0038 + 0.017 v_a + 3.86 L_p + 0.18 \times 10^{-3} h_w)^2 \\ &= (0.0038 + 0.017 \times 2.5764 + 3.86 \times 1.5843 \times 10^{-3} + 0.18 \times 10^{-3} \times 50)^2 \\ &= 0.00393 \text{ m}^2/\text{s} \end{aligned}$$

$$\text{Peclet number } Pe = \frac{Z_L^2}{D_e \theta_L} = \frac{1.0846^2}{0.00393 \times 12.94} = 23.13 \quad (8.96)$$

Point efficiency, $\dot{\eta}$

$$\frac{1}{\ln(1-\dot{\eta})} = - \left(\frac{1}{N_G} + \frac{mV}{L} \times \frac{1}{N_L} \right) \quad (8.90)$$

Slope of equilibrium curve of top tray

$$m = \frac{y}{x} = \left[\left(\frac{0.9703}{0.9578} \right) \times \left(\frac{0.9409}{0.9134} \right) \left(\frac{0.9186}{0.8787} \right) \right]^{1/3}$$

(Value of y and x are obtained from vapour-liquid equilibrium data, given in Example 8.5)

$$m = (1.0136 \times 1.0301 \times 1.0454)^{1/3} = 1.0296$$

$$\frac{mV}{L} = \frac{1.0296 \times 745.108}{601.818} = 1.2747$$

Note: At 70 % turndown, individual values of V and L will change, but value of ratio V/L will remain constant.

$$\frac{1}{\ln(1-\dot{\eta})} = -\left(\frac{1}{0.588} + 1.2747 \times \frac{1}{11.4} \right)$$

$$\dot{\eta} = 0.424$$

$$\frac{mV}{L} \dot{\eta} = 1.2747 \times 0.424 = 0.54 \text{ and } Pe = 23.13$$

From Fig. (8.21)

$$\frac{\eta}{\dot{\eta}} = 1.297$$

Efficiency of sieve tray $\eta = 1.297 \times 0.424 = 0.55$

Actual tray efficiency η_a , considering liquid entrainment

$$\eta_a = \frac{\eta}{1 + \eta \left(\frac{\Psi}{1 - \Psi} \right)} \quad (8.98)$$

$$\eta_a = \frac{0.55}{1 + 0.55 \left(\frac{0.21}{1 - 0.21} \right)} = 0.4798$$

(b) For stripping section:

To find the tray efficiency of the bottom most tray of stripping section, physical properties of liquid and vapour of the same tray are required.

Liquid:

$$\rho_L'' = 1000 \text{ kg/m}^3$$

$$\sigma_L = 21.334 \times 10^{-3} \text{ N/m}$$

Viscosity of acetic acid liquid at 134.26°C

$$\mu_L = 0.34 \text{ mPa}\cdot\text{s} \text{ or } cP = 0.34 \times 10^{-3} \text{ kg/(m} \cdot \text{s)}$$

Diffusivity D_L at 134.26°C

$$D_L = \frac{1.24 \times 10^{-5} \times 0.95}{(273 + 25)} \times \frac{(273 + 134.26)}{0.28}$$

$$= 5.75 \times 10^{-5} \text{ cm}^2/\text{s} \equiv 5.75 \times 10^{-9} \text{ m}^2/\text{s}$$

Vapour:

$$\rho_v'' = 2.8368 \text{ kg/m}^3$$

$$\mu_v = 0.009 \text{ mPa}\cdot\text{s or cP}$$

$$D_v = \frac{10^{-3} \times (407.26)^{1.75} [(60+18)/(60 \times 18)]^{1/2}}{(1.58)[51.88^{1/3} + 9.44^{1/3}]^2}$$

$$= 0.1839 \text{ cm}^2/\text{s} \equiv 0.1839 \times 10^{-4} \text{ m}^2/\text{s}$$

Number of gas phase transfer unit:

Vapour velocity based on active area

$$v_a = \frac{0.7 \times \dot{Q}_v}{A_a} = \frac{0.7 \times 4.8464}{3.3242} = 1.02 \text{ m/s}$$

$$F_v = 1.02 \sqrt{2.8368} = 1.718$$

$$L_p = \frac{0.7 \times \dot{Q}_L}{(A_a / Z_L)} = \frac{0.7 \times 16.25 \times 10^{-3}}{3.3242 / Z_L}$$

$$Z_L = 0.638 D_i = 0.638 \times 2.36 = 1.5 \text{ m}$$

$$L_p = 5.1328 \times 10^{-3} \text{ m}^2/\text{s}$$

$$\frac{\mu_v}{\rho_v'' D_v} = \frac{0.009 \times 10^{-3}}{2.8368 \times 0.1839 \times 10^{-4}} = 0.1725$$

$$N_G = \frac{0.776 + (4.57 \times 10^{-3} \times 50) - (0.24 \times 1.718) + (105 \times 5.1328 \times 10^{-3})}{\sqrt{0.1725}}$$

$$= 2.7234$$

Liquid hold-up on the plate,

$$Z_c = 0.006 + (0.73 \times 10^{-3} \times 50) - (0.24 \times 10^{-3} \times 1.718 \times 50)$$

$$+ (1.22 \times 5.1328 \times 10^{-3}) = 0.028 \text{ m}^3/\text{m}^2$$

Liquid contact time

$$\theta_L = \frac{Z_c Z_L}{L_p} = \frac{0.028 \times 1.5}{5.1328 \times 10^{-3}} = 8.18 \text{ s} > 3 \text{ s}$$

Number of liquid phase transfer units,

$$N_L = (4.13 \times 10^8 \times 5.75 \times 10^{-9})^{0.5} (0.21 \times 1.718 + 0.15) \times 8.18 = 6.44$$

Point efficiency, $\dot{\eta}$

$$\frac{1}{\ln(1-\dot{\eta})} = -\left(\frac{1}{N_G} + m \frac{\bar{V}}{L} \frac{1}{N_L}\right) = -\left(\frac{1}{2.7234} + 2.5 \times \frac{824.89}{974.92} \times \frac{1}{6.44}\right) \quad (8.90)$$

$$\dot{\eta} = 0.7625$$

$$\frac{m\bar{V}}{L} \cdot \dot{\eta} = 2.5 \times 0.7625 \times \frac{824.89}{974.92} = 1.613$$

Eddy diffusivity

$$\begin{aligned} D_e &= (0.0038 + 0.017 v_a + 3.86 L_p + 0.18 \times 10^{-3} h_w)^2 \quad (8.97) \\ &= (0.0038 + 0.017 \times 1.02 + 3.86 \times 5.1328 \times 10^{-3} + 0.18 \times 10^{-3} \times 50)^2 \\ &= 0.0025 \text{ m}^2/\text{s} \end{aligned}$$

Peclet number

$$Pe = \frac{Z_L^2}{D_e \theta_L} = \frac{1.5^2}{0.0025 \times 8.18} = 110 \quad (8.96)$$

From Fig. 8.21,

$$\begin{aligned} \frac{\eta}{\dot{\eta}} &= 2.46 \\ \eta &= 2.46 \times 0.7625 = 1.876 \end{aligned}$$

Efficiency cannot be greater than 1.

Hence $\eta = 1$

Actual tray efficiency η_a considering liquid entrainment

$$\eta_a = \frac{\eta}{1 + \eta \left(\frac{\Psi}{1 - \Psi} \right)} = \frac{1}{1 + \left(\frac{0.084}{1 - 0.084} \right)} = 0.916$$

Tray efficiency by Van Winkle's correlation:

For the top most tray

$$\eta = 0.07 D_g^{0.14} Sc_L^{0.25} Re^{0.08} \quad (8.86)$$

where, $D_g = \text{Surface tension number} = \frac{\sigma_L}{u_L u_v}$ (8.87)

$$\sigma_L = 55.5 \times 10^{-3} \text{ N/m}, \mu_L = 0.312 \times 10^{-3} (\text{N} \cdot \text{s})/\text{m}^2$$

$$u_v = \text{Superficial vapour velocity} = \frac{6.3496}{\pi/4 D_i^2} = \frac{6.3496}{(\pi/4)(1.7)^2} = 2.797 \text{ m/s}$$

$$D_g = \frac{55.5 \times 10^{-3}}{0.312 \times 10^{-3} \times 2.797} = 63.6$$

$$F_A = \text{Fractional area} = \frac{A_h}{A_c} = 0.07718$$

$$\text{Liquid Schmidt number, } Sc_L = \frac{\mu_L}{\rho_L D_{LK}} \quad (8.88)$$

$$\rho_L = 967.97 \text{ kg/m}^3, D_{LK} = 5.275 \times 10^{-9} \text{ m}^2/\text{s}$$

$$Sc_L = \frac{0.312 \times 10^{-3}}{967.97 \times 5.275 \times 10^{-9}} = 61.1$$

Reynolds number,

$$Re = \frac{h_w u_v \rho_v}{\mu_L (F_A)} \times 10^{-3} = \frac{0.05 \times 2.797 \times 0.6823}{0.312 \times 10^{-3} \times 0.07718} = 3962.6 \quad (8.89)$$

$$\begin{aligned}\eta &= 0.07 (63.6)^{0.14} (61.1)^{0.25} (3962.6)^{0.08} \\ \eta &= 0.679,\end{aligned}$$

$$\eta_a = \frac{0.679}{1 + 0.679 \left(\frac{0.21}{1 - 0.21} \right)} = 0.5752$$

For the bottom most tray:

Surface tension number

$$D_g = \frac{\sigma_L}{\mu_L u_v} \quad (8.87)$$

$$\sigma_L = 21.334 \times 10^{-3} \text{ N/m}, \mu_L = 0.34 \times 10^{-3} \text{ kg/(m} \cdot \text{s)}$$

$$u_v = \text{Superficial vapour velocity} = \frac{4.8464}{(\pi/4)(2.36)^2} = 1.108 \text{ m/s}$$

$$D_g = \frac{21.334 \times 10^{-3}}{0.34 \times 10^{-3} \times 1.108} = 56.63$$

$$\rho_L = 1000 \text{ kg/m}^3, D_L = 5.75 \times 10^{-9} \text{ m}^2/\text{s}$$

$$Sc = \frac{\mu_L}{\rho_L D_L} = \frac{0.34 \times 10^{-3}}{1000 \times 5.75 \times 10^{-9}} = 59.13$$

$$F_A = \frac{A_h}{A_c} = \frac{0.33242}{4.374} = 0.076$$

$$\begin{aligned}Re &= \frac{h_w u_v \rho_v \times 10^{-3}}{\mu_L (F_A)} = \frac{0.05 \times 1.108 \times 2.8368}{0.34 \times 10^{-3} (0.076)} \\ &= 6082\end{aligned}$$

$$\eta = 0.07 (56.63)^{0.14} (59.31)^{0.25} (6082)^{0.08}$$

$$\eta = 0.6863$$

$$\eta_a = \frac{\eta}{1 + \eta \left(\frac{\Psi}{1 - \Psi} \right)} = \frac{0.6863}{1 + 0.6863 \left(\frac{0.084}{1 - 0.084} \right)} = 0.6457$$

Table 8.37 Resulting Data of Tray Efficiency

Method	Tray efficiency	
	Top most tray	Bottom most tray
AICHE Method	0.4798	0.916
Van Winkle's Method	0.5752	0.6457

Let tray efficiency for enriching section = 0.45 and
tray efficiency for stripping section = 0.5

$$\text{Actual number of trays in enriching section} = \frac{7}{0.45} \approx 16$$

$$\text{Actual number of trays in stripping section} = 18/0.5 = 36$$

Actual number of trays = $18 + 36 = 54$, assume 55.

Feed nozzles can be provided at 32nd, 36th and 40nd trays. More number of feed nozzles are provided to facilitate the flexibility in the operation. For example, in actual operation if the feed is introduced on 36th tray, purity of distillate could be more than desired but bottom product (in this case it is pure acetic acid) may not be of desired purity. In such a case to get desired result, feed can be introduced on 32nd tray.

Pressure drop:

Pressure drop in the top most tray = 146.32 mm LC

Pressure drop in the bottom most tray = 140 mm LC

Assume tray pressure drop for the entire column = 145 mm LC

$$\rho_L \text{ (at top)} = 967.97 \text{ kg/m}^3, \rho_L \text{ (at bottom)} = 1000 \text{ kg/m}^3$$

$$\Delta p_T = 55 \times 145 = 7975 \text{ mm LC}$$

$$\Delta p_T = 7975 \times 10^{-3} \times 1000 \times 9.81 = 78235 \text{ N/m}^2 \equiv 78.2 \text{ kPa}$$

8.5 BATCH DISTILLATION

It is a very common unit operation used in the manufacturing of chemicals which are produced in batch processes. Batch distillation is carried out with or without using a rectification column (tray tower or packed tower). Thus, it is divided in two types; (i) simple distillation and (ii) batch distillation with rectification.

8.5.1 Simple Distillation

This type of batch distillation consists of a heated vessel (coiled vessel or jacketed vessel or kettle type reboiler), a condenser and one or more receiving tanks. Feed mixture is charged into the vessel and brought to boiling. Vapours are condensed and condensate is collected in the receiver. No reflux is returned (Fig. 8.22). Simple batch distillation provides only one theoretical plate of separation. In some batch reactors of small plants, at the end of reactions, volatile components

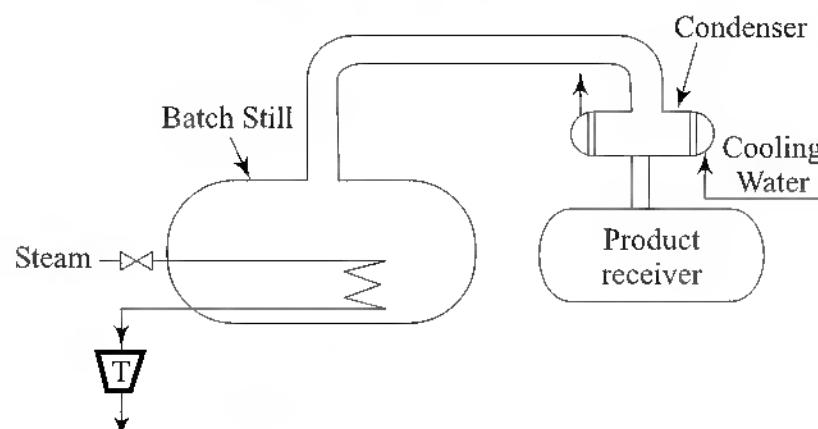


Fig. 8.22 Simple Batch Distillation

are separated from high boiling liquid solution by simple distillation. Following equation is applicable to the simple batch distillation.

$$\ln \left(\frac{F}{W} \right) = \ln \left(\frac{M_i}{M_F} \right) = \int_{x_B}^{x_F} \frac{dx}{y - x} \quad (8.99)$$

where,
 $M_i = F$ = Moles of liquid in initial condition
 $M_F = W$ = Moles of liquid in final condition
 x_F = Mole fraction of more volatile component in liquid phase in initial conditions
 x_B = Mole fraction of more volatile component in liquid phase in final conditions
 y = Mole fraction of more volatile component in vapour phase

If the liquid mixture is a binary mixture for which relative volatility α is constant, then above Eq. (8.99) can be converted into following equation.

$$\ln \left(\frac{M_F}{M_i} \right) = \left[\frac{1}{(\alpha - 1)} \right] \ln \left[\frac{x_B (1 - x_F)}{x_F (1 - x_B)} \right] + \ln \left[\frac{1 - x_F}{1 - x_B} \right] \quad (8.100)$$

For the binary system and for simple batch distillation following equation is also applicable.

$$\ln \left(\frac{M_{Af}}{M_{Ai}} \right) = \alpha \left(\frac{M_{Bf}}{M_{Bi}} \right) \quad (8.101)$$

where,
 M_{Af} = Moles of component A in the pot after distillation
 M_{Ai} = Moles of component A in the pot before distillation
 M_{Bi}, M_{Bf} = Moles of component B in the pot before and after distillation respectively.
 A, B = Components of binary system

Example 8.14

1000 kg of feed, containing 30% by mass ethylene glycol and remaining water, is charged to a batch still. Simple batch distillation is carried out at 30.4 kPa absolute pressure to get the residue which must contain 95% by mass ethylene glycol. Find the amount of residue (final product).

Table 8.38 Vapour – liquid Equilibrium Data of Ethylene Glycol–Water System at 30.4 kPa ^a¹⁴

Temperature, °C	x	y
69.5	1.00	1.000
76.1	0.77	0.998
78.9	0.69	0.997

(Contd.)

Table 8.38 (Contd.)

83.1	0.60	0.990
89.6	0.46	0.980
103.1	0.27	0.940
118.4	0.15	0.870
128.0	0.10	0.780
134.7	0.07	0.700
145.0	0.03	0.530
160.7	0.00	0.000

Solution:

For simple batch distillation following equation is applicable.

$$\ln\left(\frac{F}{W}\right) = \ln\left(\frac{M_i}{M_F}\right) = \int_{x_B}^{x_F} \frac{dx}{y-x} \quad (8.99)$$

$F = M_i$ = moles of liquid feed

Feed contains 300 kg ethylene glycol and 700 kg water.

Molar mass of water = 18

Molar mass of ethylene glycol = 62

$$F = M_i = \frac{300}{62} + \frac{700}{18} = 43.7276 \text{ kmol}$$

Mole fraction of water in feed

$$x_F = \frac{700/18}{43.7276} = 0.8893$$

Mole fraction of water in residue

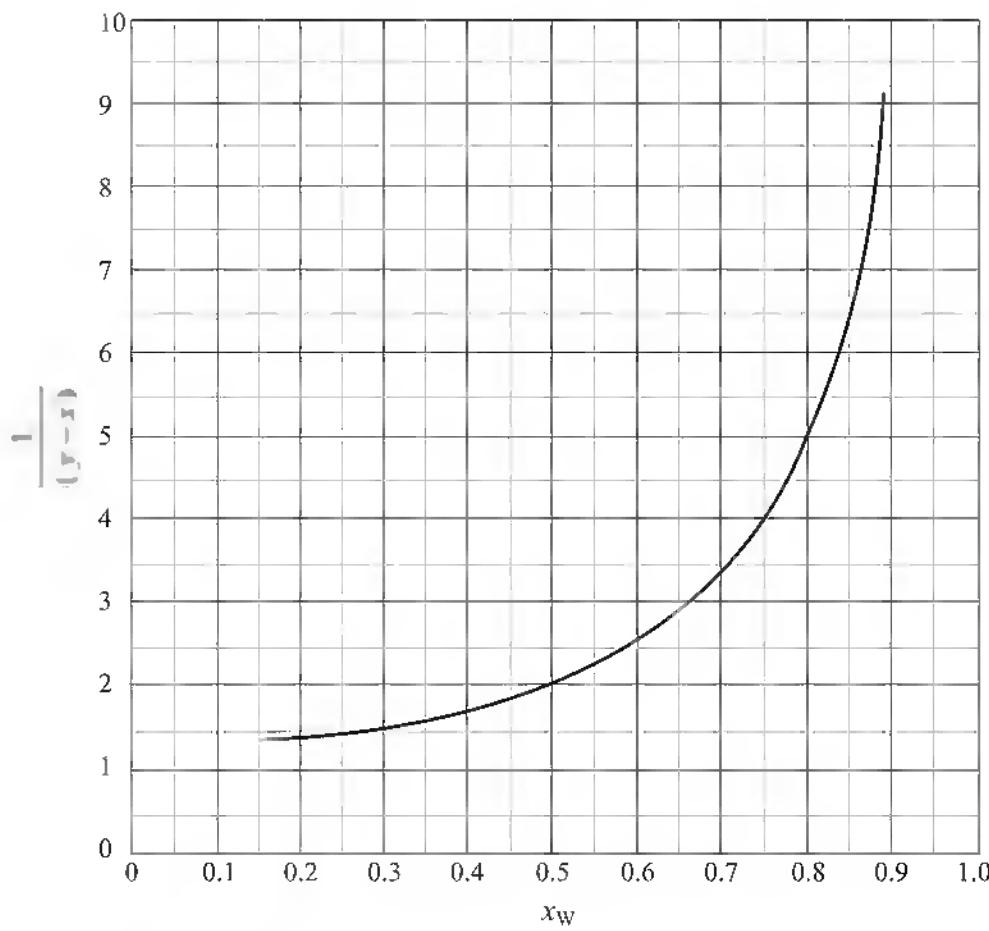
$$x_B = \frac{5/18}{(5/18)+(95/62)} = 0.15347$$

$$\ln\left(\frac{43.7276}{M_F}\right) = \int_{0.15347}^{0.8893} \frac{dx}{y-x}$$

To solve this integration, following table is prepared based on the given VLE data as well as based on the equilibrium curve drawn.

Table 8.39 Reference Points of Fig. 8.23

x	y	$y - x$	$\frac{1}{y - x}$
0.153 47	0.875	0.721 53	1.3859
0.270 00	0.940	0.670 00	1.4925
0.350 00	0.970	0.620 00	1.6129
0.460 00	0.980	0.520 00	1.9230
0.600 00	0.990	0.390 00	2.5641
0.690 00	0.997	0.307 00	3.2573
0.770 00	0.998	0.228 00	4.3900
0.889 30	0.999	0.109 70	9.1158

**Fig. 8.23 Evaluation of Integral for Batch Distillation**

Draw the graph of $\frac{1}{y-x}$ vs x (Fig. 8.23)

$$\int_{0.15347}^{0.8893} \frac{dx}{y-x} = \text{area under curve from } x_B \text{ to } x_F = 2.0643 \text{ (integral value)}$$

$$\ln \left(\frac{43.7276}{M_F} \right) = 2.0643$$

$$B = M_F = 5.5493 \text{ kmol}$$

$$\begin{aligned} \% \text{ recovery of ethylene glycol in final product } (M_f) &= \frac{(1-x_B) M_F}{(1-x_F) M_i} \times 100 \\ &= \frac{(1-0.15347) 5.5493}{(1-0.8893) 43.7276} \times 100 \\ &= 97.05 \% \end{aligned}$$

8.5.2 Batch Distillation with Rectification

Simple batch distillation is a single stage separation hence it does not give good separation unless the relative volatility is very high. In many cases, a rectification

column which may be a tray tower or a packed tower with reflux is used to improve the performance of batch distillation. If the column is not too large, then it is mounted on top of the still or heated vessel (Fig. 8.24).

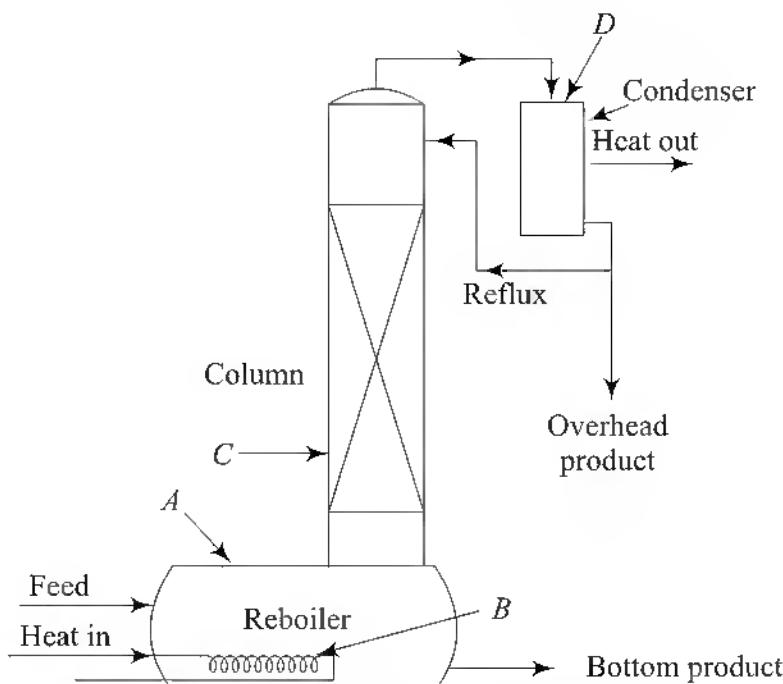


Fig. 8.24 Batch Distillation with Rectification Column

This type of batch distillation consists of a still (heated vessel or reboiler), a rectification column, a condenser, some means of splitting off a portion of the condensed vapour (distillate) as reflux and one or more top product receivers. In operation, a batch of liquid feed is charged to the reboiler or still. Then first, batch distillation is carried out under total reflux. After achieving steady state, portion of the overhead condensate is continuously withdrawn in accordance with the established reflux policy. To get the constant overhead composition, the amount of reflux returned to the column must be continuously increased throughout the run. An alternate method of running a batch distillation is to fix the reflux ratio constant and let the overhead product purity vary with time, stopping the distillation when the amount of product or the average concentration in the total product reaches a certain value.

8.5.2.1 Process Design of Binary Batch Rectification with Constant Overhead Composition¹⁴

Since compositions within column are continuously changing during the batch distillation, rigorous calculation methods are extremely complex. For a binary system, approximate but useful method is based on McCabe–Thiele graphical method. In addition to the assumptions of McCabe–Thiele method (negligible heat of solution and equimolar overflow on the trays), this approximate method assumes negligible hold-up of liquid on the trays in the column and in the condenser. Stepwise procedure of this approximate method is as follows.

- (i) Determine the minimum reflux ratio by using following equation.

$$R_{\min} = \frac{(L/V)_{\min}}{1 - (L/V)_{\min}} \quad (8.102)$$

where, $\left(\frac{L}{V}\right)_{\min}$ = Minimum internal reflux ratio

$(L/V)_{\min}$ is determined by using following equation.

$$\left(\frac{L}{V}\right)_{\min} = \frac{y_D - y_{pi}}{x_D - x_{pi}} \quad (8.103)$$

where, $x_D = y_D$ = Mole fraction of more volatile component in distillate, if total condenser is used.

For partial condenser,

y_D = Mole fraction of more volatile component in distillate vapour

x_D = Mole fraction of more volatile component in reflux

$x_{pi} = x_F$ = Mole fraction of more volatile component in liquid feed

y_{pi} = Value of y which is in equilibrium with x_F which can be determined from the vapour–liquid equilibrium curve or data or equation. x_{pi} and y_{pi} defined above are valid only for normal equilibrium curve, as shown in Fig. 8.25.

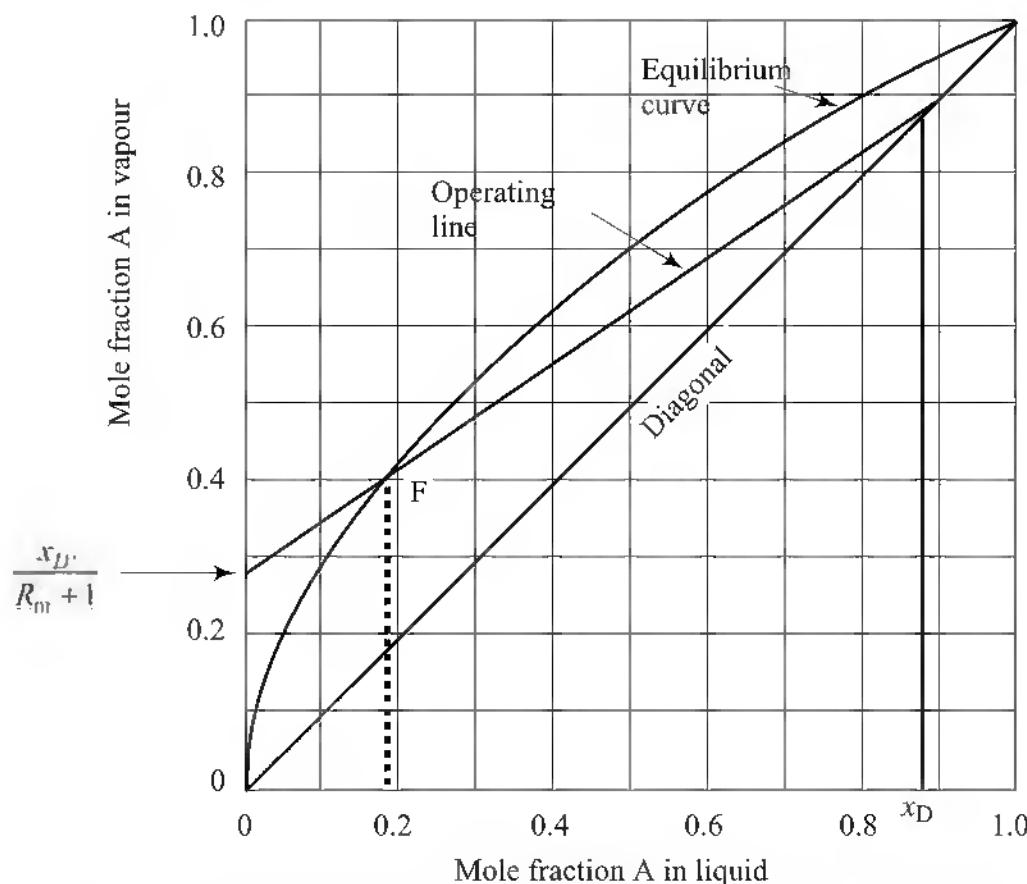


Fig. 8.25 Determination of Minimum Reflux for Equilibrium Curve without Inflection

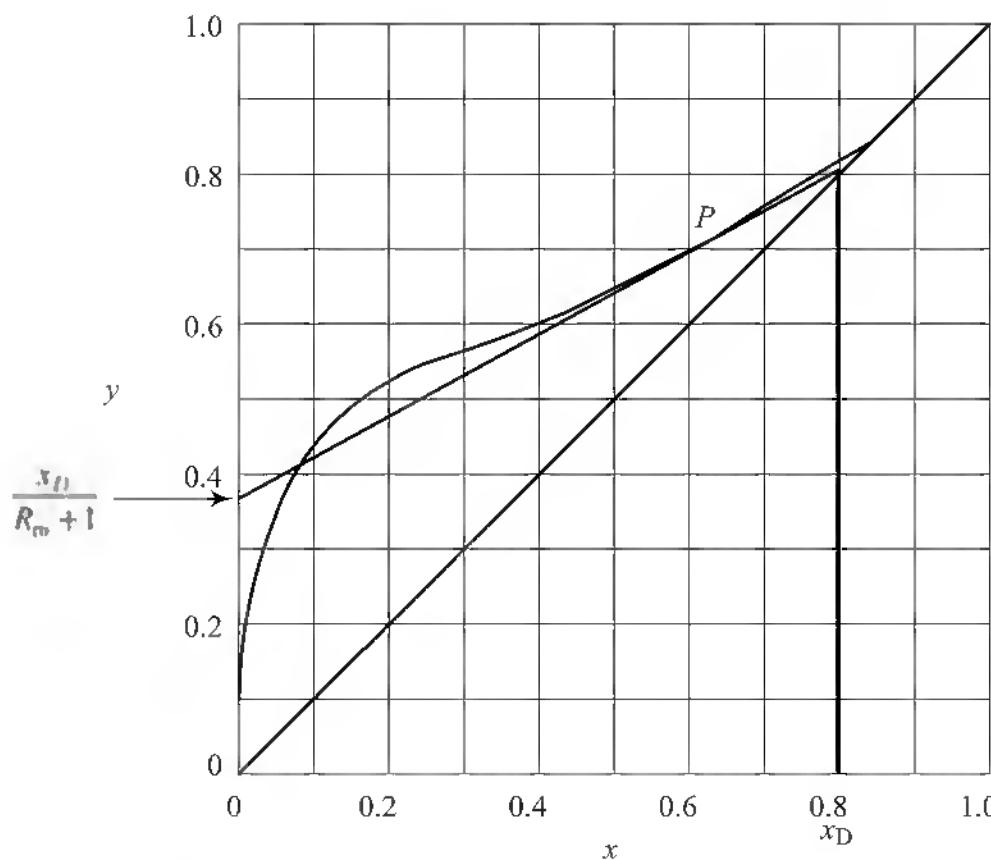


Fig. 8.26 Determination of Minimum Reflux for Equilibrium Curve with Inflection

But for the equilibrium curve with inflection as shown in Fig. 8.26, x_{pi} and y_{pi} are equilibrium compositions corresponding to the inflection point. "P".

- (ii) Actual reflux ratio is 1.5 to 10 times minimum. Decide the actual reflux ratio R for starting of batch distillation. Determine the value of internal reflux ratio L/V required for starting condition.
- (iii) Draw the operating line for starting of batch distillation. Slope of operating line is L/V (for starting condition). Determine the number of theoretical stages required for the desired separation at the start of batch distillation. To find this, start the stair case construction from x_D to x_F in between equilibrium curve and operating line.
- (iv) Select the slightly higher value of R and L/V than the same of starting condition. For this new value of L/V draw the operating line from x_D . Draw the same number of steps (or equilibrium stages) from x_D which was obtained for starting condition. Find the composition of liquid in reboiler (x_{wi}) corresponding to new value of R and L/V .
- (v) Select again slightly greater value of R and L/V . Draw the operating line for the new value of L/V . Find the composition of liquid in reboiler or still (residue), x_{wi} , by drawing the same number of steps from x_D which was required for starting condition. Continue these calculations until, new value of x_{wi} obtained, is equal to x_B , where x_B is equal to mole fraction of more volatile component required in bottom product.

- (vi) Time required for batch distillation rectification and with constant overhead composition can be calculated by Bogart equation¹⁴.

$$\theta = \frac{F(x_D - x_F)}{V} \int_{x_B}^{x_F} \frac{dx_w}{(1 - L/V)(x_D - x_w)^2} \quad (8.104)$$

where, θ = Total time required in batch distillation with rectification, s

F = Total moles of feed charged, kmol

V = Molar flow rate of vapour phase, kmol/s

L = Molar flow rate of liquid phase, kmol/s

x_D = Mole fraction of more volatile component in distillate

x_B = Mole fraction of more volatile component in bottom product or final residue.

x_W = Mole fraction of more volatile component in liquid inside the still or reboiler

x_F = Mole fraction of more volatile component in feed

Above equation can also be written as

$$\theta = \frac{F(x_D - x_F)}{V} \int_{x_{wi}}^{x_F} \frac{dx_w}{(1 - L/V)(x_D - x_w)^2} \quad (8.105)$$

where, x_{wi} = Intermediate composition (mole fraction of more volatile component) of liquid in still or reboiler.

By using Eq. (8.105), for the different values of θ , L/V and R can be determined. Based on these data, the graph of R vs θ can be developed. This graph or curve is also useful in actual operation, giving information about how to increase the value of reflux ratio R with time to maintain the constant composition of overhead (top) product.

- (vii) The quantity of distillate at any time θ can be determined by using the following equation,

$$F - W_i = \frac{F(x_F - x_{wi})}{x_D - x_{wi}} \quad (8.106)$$

where, W_i = Moles of liquid in still at any time θ , kmol

At the end of distillation, quantity of material distilled

$$D = F - B = \frac{F(x_F - x_B)}{(x_D - x_B)} \quad (8.107)$$

where, B = Moles of final bottom product, kmol

- (viii) Decide the material of construction of packed tower or tray tower.

- (ix) Select the type of tower; tray tower or packed tower

- (x) Determine the tower diameter and pressure drop per unit height or tray.

- (xi) Determine the tray efficiency of tray tower or HETP in case of packed tower.

- (xii) Determine the actual number of trays required in tray tower or height of packing required in packed tower.
- (xiii) Determine the actual total pressure drop.
- (xiv) Design the condenser, reboiler, etc.

Example 8.15

1000 kg of feed, containing 40% by mass methanol and balance water, is to be separated in batch distillation with a rectification column. Top product must contain 99.9 % by mass of methanol. Composition of distillate should remain constant throughout the batch distillation. Desired composition of residue is 95% by mass water. (a) Design the rectification column (b) Determine the operating (actual) reflux ratio vs time data.

Operating pressure in rectification column is atmospheric.

Table 8.40 Equilibrium Data for Methanol-Water System at 101.3 kPa

Temperature, °C	x	y
100.0	0.0	0.0
96.4	0.02	0.134
93.5	0.04	0.230
91.2	0.06	0.304
89.3	0.08	0.365
87.7	0.100	0.418
84.4	0.150	0.517
81.7	0.200	0.579
78.0	0.300	0.665
75.3	0.400	0.729
73.1	0.500	0.779
71.2	0.600	0.825
69.3	0.700	0.870
67.5	0.800	0.915
66.0	0.900	0.958
65.0	0.950	0.979
64.5	1.000	1.000

(Table 13-1 of Ref. 14)

Solution:

Basis: 1000 kg feed consisting 40% methanol (by mass)

$$\text{Feed, } F = \frac{0.4 \times 1000}{32} + \frac{0.6 \times 1000}{18} = 45.833 \text{ kmol}$$

Mole fraction of methanol in feed,

$$x_F = \frac{(400/32)}{45.833} = 0.2727$$

Mole fraction of methanol in distillate,

$$x_D = \frac{(99.9/32)}{(99.9/32) + (0.1/18)} = 0.9982$$

Mole fraction of methanol in residue,

$$\begin{aligned}x_B &= \frac{(5/32)}{(5/32)+(95/18)} = 0.02875 \\D &= F - B = \frac{F(x_F - x_B)}{(x_D - x_B)} \\&= \frac{45.833(0.2727 - 0.02875)}{(0.9982 - 0.02875)} \\&= 11.5333 \text{ kmol} \\B &= 45.833 - 11.5333 = 34.2997 \text{ kmol}\end{aligned}\quad (8.107)$$

Draw the equilibrium curve and find the minimum reflux ratio, R_m .

$$R_m = \frac{(L/V)_{\min}}{1 - (L/V)_{\min}} \quad (8.102)$$

$$\text{where, } (L/V)_{\min} = \frac{y_D - y_{pi}}{x_D - x_{pi}} \quad (8.103)$$

Figure 8.27 shows equilibrium curve for methanol – water system at 101.325 kPa.

$$x_D = y_D = 0.9982$$

$$x_{pi} = x_F = 0.2727$$

Corresponding equilibrium value of y

$$y_{pi} = y_{(x_F)} = 0.6415$$

$$(L/V)_{\min} = \frac{0.9982 - 0.6415}{0.9982 - 0.2727} = 0.49166$$

$$R_m = \frac{0.49166}{1 - 0.49166} = 0.9672$$

Let the reflux ration R at the starting of batch distillation (at $\theta = 0$) is equal to $2R_m$.

$$R_1 = 2 \times 0.9672 = 1.9344$$

Slope of operating line

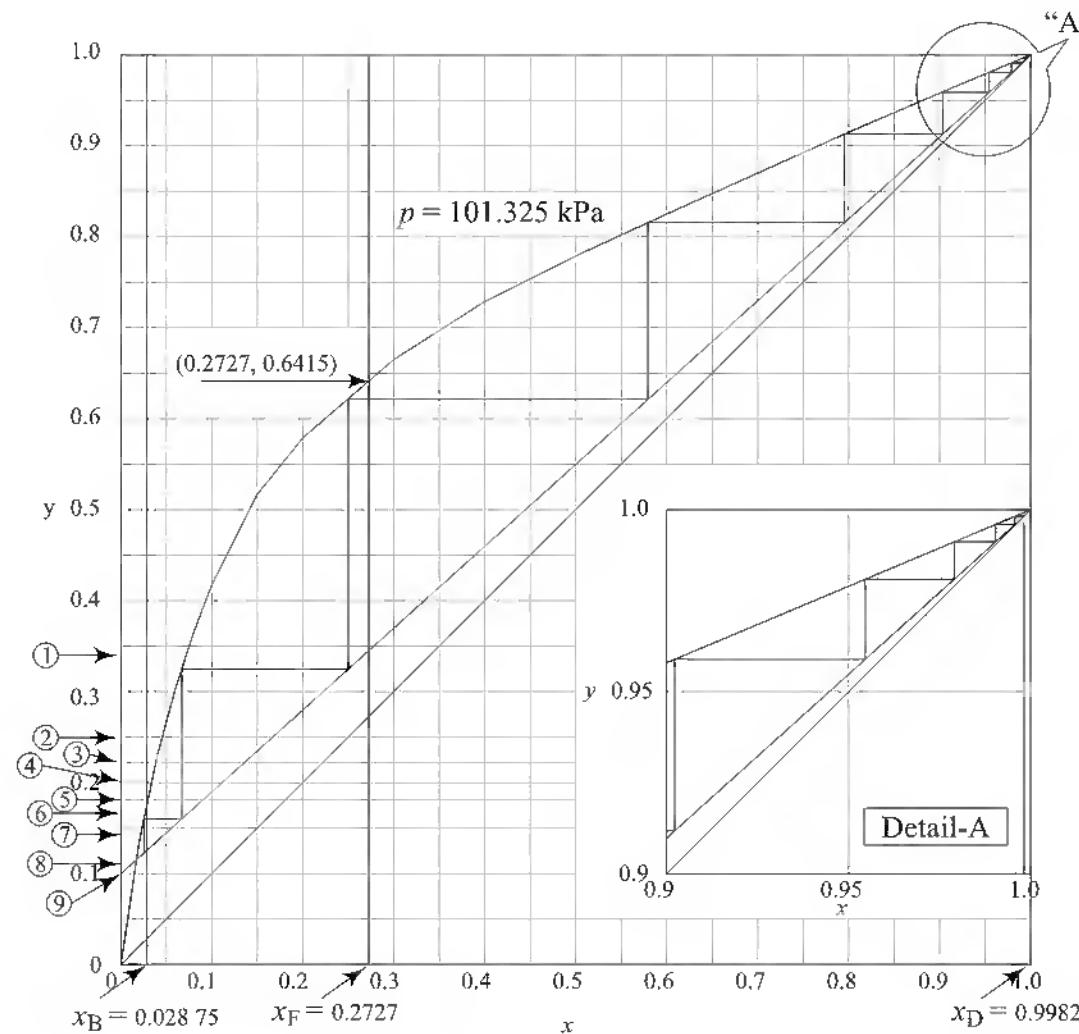
$$L/V = \frac{R_1}{R_1 + 1} = 0.6592$$

Intercept of this operating line

$$x_D/(R_1 + 1) = 0.9982/2.9344 = 0.34$$

Figure 8.27 shows that for $R_1 = 1.9344$ it is not possible to get the bottom composition of $x_B = 0.02875$. Hence it is planned to increase the reflux ratio gradually from $R_1 = 1.9344$ to $R_9 = 9$.

For reflux ratio $R_9 = 9$ and intercept $\frac{x_D}{R_9 + 1} = 0.1$, approximately 10 number of theoretical stages required to get $x_B = 0.02875$ as shown in Fig. 8.27. Based on this the value of number of theoretical stages $N = 10$ is fixed for all values of reflux ratio. For each value of reflux ratio, value of bottom composition x_{wi} is determined for $N = 10$.



Point on y-axis	R	$\frac{x_D}{R+1}$	x_B
①	1.9344	0.3400	0.6354
②	3.0	0.24955	0.2296
③	3.5	0.2218	0.1427
④	4.0	0.2000	0.1036
⑤	4.5	0.1815	0.0774
⑥	5.0	0.1664	0.0616
⑦	6.0	0.1426	0.0447
⑧	8.0	0.1109	0.0294
⑨	9.0	0.1000	0.0288

Fig. 8.27 McCabe-Thiele Diagram for Batch Rectification of Methanol-Water Solution.

Time required for batch distillation and with constant overhead composition is given by Bogart equation

$$\theta = \frac{F(x_D - x_F)}{V} \int_{x_B}^{x_F} \frac{dx_w}{(1 - L/V)(x_D - x_w)^2} \quad (8.104)$$

Table 8.41 Batch Rectification Data

R	L/V	$\frac{x_D}{R+1}$	x_w	$\frac{1}{(1-L/V)(x_D-x_w)^2}$
$R_1 = 1.9344$	0.6592	0.34	0.6354	—
$R_2 = 3$	0.75	0.24955	0.2727	7.6 (At $\theta = 0$)
$R_2 = 3$	0.75	0.24955	0.2296	6.77
$R_3 = 3.5$	0.7778	0.2218	0.1427	6.15
$R_4 = 4$	0.8	0.2	0.1036	6.25
$R_5 = 4.5$	0.8182	0.1815	0.0774	6.49
$R_6 = 5$	0.8333	0.1664	0.0616	6.84
$R_7 = 6$	0.8571	0.1426	0.0447	7.7
$R_8 = 8$	0.8889	0.1109	0.0294	9.59
$R_9 = 9$	0.9	0.1	0.02875	10.64

Note: Table 8.41 shows that for $R_1 = 1.9344$ and $N = 10$, $x_w = 0.6354 > x_F$. It means that for $R_1 = 1.9344$, more than 10 number of equilibrium stages are required to get the desired separation. In actual operation for $R = 1.9344$ and $N = 10$, distillate composition obtained will be less than x_D even at the starting of batch distillation. It is calculated to be 0.9917 from the graph (not shown in Fig. 8.27). For $R_2 = 3$ and $N = 10$, $x_w = 0.2296 < x_F$. Hence it is decided to keep $R = 3$ at the starting of batch distillation.

Total time required

$$\theta = \frac{45.833(0.9982 - 0.2727)}{V} \int_{0.02875}^{0.2727} \frac{dx_w}{(1-L/V)(x_D-x_w)^2}$$

Draw the graph of $\frac{1}{(1-L/V)(x_D-x_w)^2}$ vs x_w and find the area under the curve.

From Fig. 8.28, area under the curve from $x_w = 0.02875$ to $x_w = 0.2727$ is equal to 1.63747.

$$\theta = \frac{45.833(0.9982 - 0.2727)}{V} \times 1.63747$$

$$V\theta = 54.449 \text{ kmol}$$

If the total time of batch distillation is fixed at $\theta = 6 \text{ h.}$, then $V = \frac{54.449}{6}$

$$V = 9.0748 \text{ kmol/h}$$

Total time for batch distillation in any batch process is fixed based on the time required by the equipment which is governed by the batch process. In most of the cases time required by the reaction in batch reactor decides the time of batch process and time of other separation operations like batch distillation.

Total distillation time, $\theta = 6 \text{ h}$

Here reflux ratio R at the starting of batch distillation is 3. In operation R can be kept equal to 3 from $\theta = 0$ to $\theta = \theta_1$.

$$\theta_1 = \frac{\text{Area of segment from } x_w = x_F \text{ to } x_w = 0.2296}{\text{Total area under the curve}} \times \theta$$

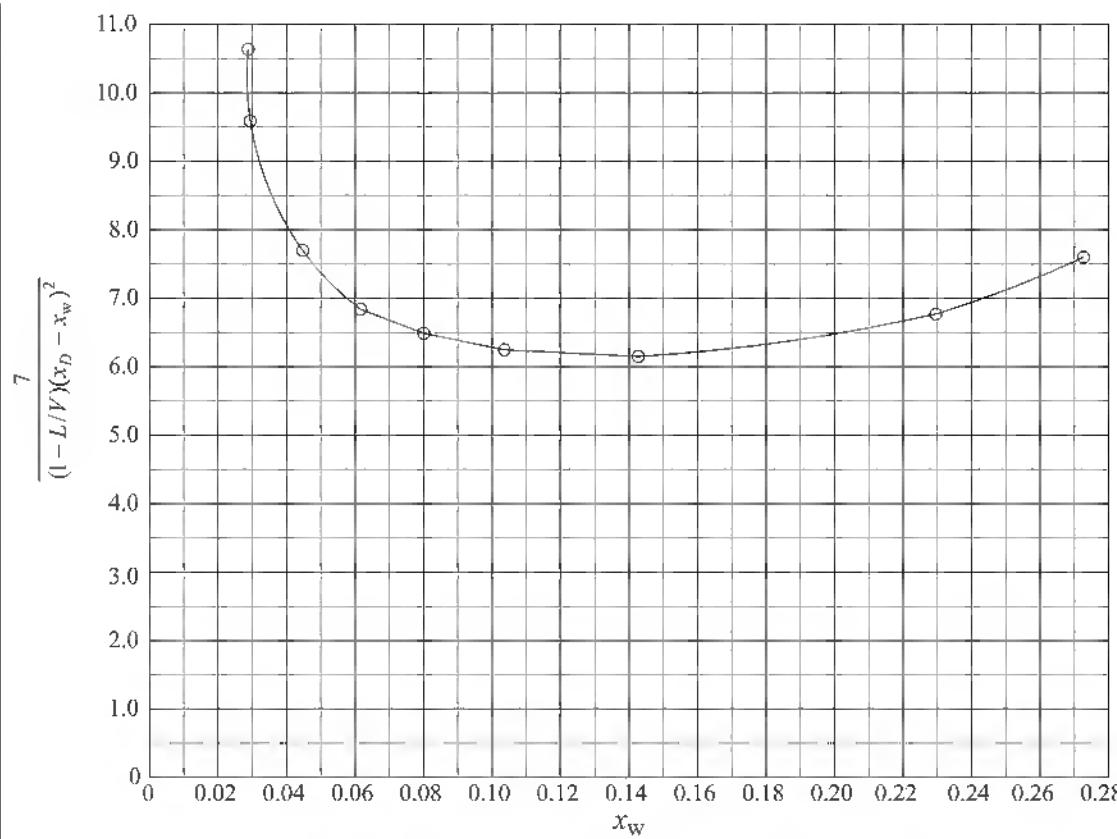


Fig. 8.28 Evaluation of Integral for Batch Distillation

$$\theta_1 = \frac{0.3062}{1.63747} \times 6 = 1.122 \text{ h}$$

For $R_3 = 3.5$, area of segment from $x_w = 0.2296$ to $x_w = 0.1427$ is 0.558 89
(determined from Fig. 8.28)

Hence, $\theta_2 = \frac{0.55889}{1.63747} \times 6 = 2.048 \text{ h}$

Similarly,

For $R_4 = 4$, $\theta_3 = \frac{0.24243}{1.63747} \times 6 = 0.8883 \text{ h}$

For $R_5 = 4.5$, $\theta_4 = \frac{0.15034}{1.63747} \times 6 = 0.5509 \text{ h}$

For $R_6 = 5$, $\theta_5 = \frac{0.12241}{1.63747} \times 6 = 0.4485 \text{ h}$

For $R_7 = 6$, $\theta_6 = \frac{0.12194}{1.63747} \times 6 = 0.4468 \text{ h}$

For $R_8 = 8$, $\theta_8 = \frac{0.12901}{1.63747} \times 6 = 0.473 \text{ h}$

For $R_9 = 9, \theta_9 = \frac{0.00613}{1.63747} \times 6 = 0.0225 \text{ h}$

Table 8.42 Reflux Ratio vs Time

R	θ			x_w	
3	0	to	1.122	0.2727	to 0.2296
3.5	1.122	to	3.17	0.2296	to 0.1427
4	3.17	to	4.0583	0.1427	to 0.1036
4.5	4.0583	to	4.6092	0.1036	to 0.0774
5	4.6092	to	5.0577	0.0774	to 0.0616
6	5.0577	to	5.5045	0.0616	to 0.0447
8	5.5045	to	5.9775	0.0447	to 0.0294
9	5.9775	to	6	0.0294	to 0.02875

Design of distillation column:

Type of tower = Packed tower (small capacity)

Type of packing = Stainless Steel Pall rings

Size of packing = 16 mm

% void space = 92%

Packing factor $F_p = 230 \text{ m}^{-1}$ (Table 9.2)

Tower diameter required at bottom:

$$F_{LG} = \frac{L_w}{G_w} \left(\frac{\rho_G}{\rho_L} \right)^{\frac{1}{2}} \quad (9.15)$$

Molar flow rate of vapour, $V = 9.0748 \text{ kmol/h}$

$$V = L + D \text{ and } L/D = R$$

Maximum value of actual or operating reflux ratio in the entire operation is 9.

$$L_{\max} = R_{\max} D = 9 D$$

$$V = L + D = 9D + D = 10 D$$

$$D = \frac{V}{10} = 0.90748 \text{ kmol/h}$$

$$L = 8.1673 \text{ kmol/h}$$

Composition and average molar mass of vapour and liquid phases at bottom are changing with time. Average molar mass is maximum at the starting of distillation.

At the starting of distillation

$$M_{av} = 0.2727 \times 32 + (1 - 0.2727) \times 18 = 21.8178 \text{ kg/kmol}$$

$$\frac{L_w}{G_w} = \frac{L \times M_{av} / \left(\frac{\pi}{4} \right) D^2}{V \times M_{av} / \left(\frac{\pi}{4} \right) D^2} = \frac{L}{V} = 0.9$$

Bubble point temperature at $x = 0.2727$ is 79°C (from $t-xy$ data)

$$\rho_G = \frac{pM_{av}}{RT} = \frac{101.325 \times 21.8178}{8.314 \times (273 + 79)} = 0.7554 \text{ kg/m}^3$$

$$\rho_L = \frac{1}{\sum \frac{w_i}{\rho_{Li}}} = 1 / \left(\frac{0.4}{753} + \frac{0.6}{972.42} \right)$$

$$\rho_L = 870.9 \text{ kg/m}^3$$

$$F_{LG} = 0.9 \left(\frac{0.7554}{870.9} \right)^{\frac{1}{2}} = 0.0265$$

From Fig. 9.3, $K_F = 0.2$

Let actual velocity of vapour through packed tower = 70 % of flooding velocity

$$\sqrt{\frac{K}{K_F}} \times 1000 = 70$$

$$K = (0.70)^2 K_F = 0.098$$

Corresponding $\Delta p = 83.3 \text{ mm WC/m}$ of packing (From Fig. 9.3)

$$G_w = \left(\frac{K \rho_G \rho_L g}{F_p \Psi \mu_L^{0.2}} \right)^{\frac{1}{2}} \quad (9.18)$$

$$K = 0.098, \quad \rho_G = 0.7554 \text{ kg/m}^3, \quad \rho_L = 870.9 \text{ kg/m}^3 \\ F_p = 230 \text{ m}^{-1}$$

$$\Psi = \frac{\text{Density of water}}{\text{Density of liquid}} = \frac{972.42}{870.9}$$

$$\Psi = 1.1166$$

μ_L = viscosity of liquid solution

Viscosity of 40% methanol solution (by mass) at 79° C,

$$\mu_L = 0.45 \text{ cP} = 0.45 \text{ mPa} \cdot \text{s} \\ (\text{Fig. 3.43, of Ref. 14})$$

$$G_w = \left(\frac{0.098 \times 0.7554 \times 870.9 \times 9.81}{230 \times 1.1166 \times 0.45^{0.2}} \right)^{\frac{1}{2}} \\ = 1.7 \text{ kg/(m}^2 \cdot \text{s)}$$

Mass flow rate of vapour, $\dot{m}_v = 9.0748 \times 21.8178 = 198 \text{ kg/h} \equiv 0.055 \text{ kg/s}$

$$\text{Area required at bottom} = \frac{0.055}{1.7} = 0.03235 \text{ m}^2$$

$$A = 0.03235 = \left(\frac{\pi}{4}\right)D_i^2$$

$$D_i = 0.203 \text{ m}$$

Tower diameter required at top

$$F_{LG} = \frac{L_w}{G_w} \left(\frac{\rho_G}{\rho_L} \right)^{\frac{1}{2}} \quad (9.15)$$

$$\frac{L_w}{G_w} = \frac{L}{V} = 0.9$$

$$M_{av} = 0.9982 \times 32 + (1 - 0.9982) \times 18 = 31.9748 \text{ kg/kmol}$$

$$\rho_G = \frac{pM_{av}}{RT} = \frac{101.325 \times 31.9748}{8.314 \times (273 + 64.5)} = 1.1546 \text{ kg/m}^3$$

Top temperature is the dew point temperature of overhead vapour. It can be determined from $t-x-y$ data. Find the value of t for $y = x_D = 0.9982$

$$\rho_L = \text{Density of methanol at } 64.5^\circ\text{C} = 760 \text{ kg/m}^3$$

$$F_{LG} = 0.9 \left(\frac{1.1546}{760} \right)^{\frac{1}{2}} = 0.035$$

From Fig. 9.3, $K_F = 0.18$

Let actual velocity of vapour is equal to 70% of flooding velocity.

$$K = (0.7)^2 K_F = 0.0882$$

Corresponding $\Delta p = 90 \text{ mm WC/m of packing}$

$$G_w = \left(\frac{K \rho_G \rho_L g}{F_p \Psi \mu_L^{0.2}} \right)^{\frac{1}{2}} \quad (9.18)$$

$$\Psi = \frac{\text{Density of water}}{\text{Density of liquid}} = \frac{980.827}{760} = 1.29$$

(Density of water is from Table 3.28, of Ref. 14)

$$\mu_L = \text{Viscosity of methanol at } 64.5^\circ\text{C} = 0.34 \text{ cP} = 0.34 \text{ mPa} \cdot \text{s}$$

$$G_w = \left(\frac{0.0882 \times 1.1546 \times 760 \times 9.81}{230 \times 1.29 \times 0.34^{0.2}} \right)^{\frac{1}{2}}$$

$$G_w = 1.7819 \text{ kg/(m}^2 \cdot \text{s)}$$

Mass flow rate of vapour, $\dot{m}_v = 9.0748 \times 31.9748 = 290.165 \text{ kg/h} \equiv 0.0806 \text{ kg/s}$

$$\text{Area required at top} = \frac{0.0806}{1.7819} = 0.04523 \text{ m}^2$$

$$D_i = \sqrt{\frac{4 \times 0.04523}{\pi}} = 0.24 \text{ m}$$

Take $D_i = 0.24 \text{ m}$ for the entire tower.

HETP (height equivalent theoretical plate) for Pall rings = 0.5 m

Total height of packing = HETP $\times N = 0.5 \times 10 = 5 \text{ m}$

Let the total height of packing = 5.1 m

Number of packing sections = 3 each with 1.7 m packed height

Type of liquid distributor = Pipe type

Number of holes required in pipe type reflux distributor,

$$n_h = \frac{(\pi/4) D_i^2 \times 10^4}{194}$$

For each 30 in² (194 cm²) area, one distribution point is required.

$$n_h = \frac{(\pi/4) (0.24)^2 \times 10^4}{194} = 2.33$$

Let $n_h = 4$

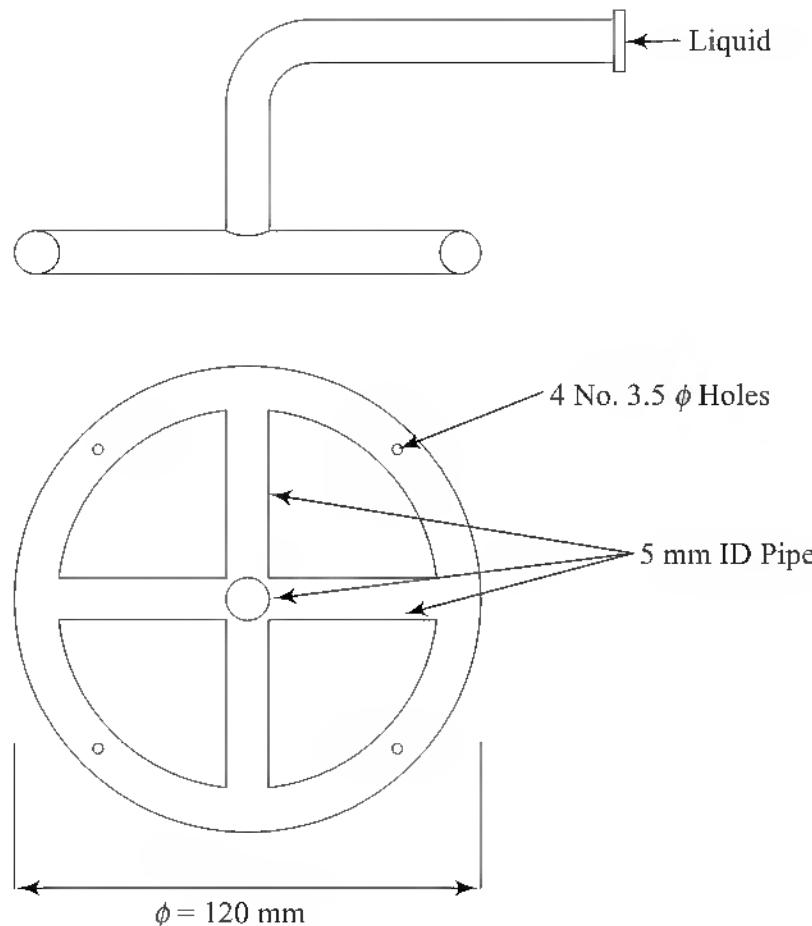


Fig. 8.29 Pipe Type Liquid Distributor

Let the velocity of methanol through pipe = 2 m/s

Volumetric flow rate of reflux

$$= \frac{LM_{av}}{\rho_L \times 3600} = \frac{(0.9 \times 9.0748) \times 31.9748}{760 \times 3600}$$

$$= 9.545 \times 10^{-5} \text{ m}^3/\text{s}$$

Inside diameter of pipe:

$$d_i = \sqrt{\frac{9.545 \times 10^{-5} \times 4}{2 \times \pi}} = 0.00779 \text{ m}$$

Let $d_i = 8 \text{ mm}$

Let the velocity of solvent through holes = 3 m/s

Let d_h = diameter of hole, mm

$$4 \times \frac{\pi}{4} d_h^2 = \frac{9.545 \times 10^{-5}}{3} = 3.18167 \times 10^{-5}$$

$$d_h = 0.0032 \text{ m}$$

Let $d_h = 3.5 \text{ mm}$

Packing Support:

Packing support should be selected such that flow area provided by packing support for the flow of gas should be greater than flow area provided by packing material.

% void space for 16 mm size SS Pall rings = 92 % (Table 9.2)

Hence with Pall rings, any gas-injection type packing support must be used. Let the type of packing support is cap type packing support.

Actual outer diameter of packing support is greater than 240 mm as some portion of packing support is sandwiched between two flanges.

Let h = Height of slot in riser, mm

a = Width of slot in riser, mm

d_r = Diameter of riser, mm

n = Number of risers, mm

D_i = Inside diameter of column, mm

Let $d_r = \frac{240}{6} = 40 \text{ mm}$

Number of risers, $n = 9$

Let n_s = number of slots per riser

Total area of slots of risers = $n_s n h a$ where, distance between two successive slots is a mm.

Let $a = 5 \text{ mm}$

$$n_s = \frac{\pi d_i}{2a} = \frac{\pi \times 40}{2 \times 5} = 12.566$$

Let $n_s = 12$

Hence, total area of slots of risers = $12 \times 9 \times h \times 5 \geq 0.92 \times \frac{\pi}{4} (240)^2$

$$h \geq 77 \text{ mm}$$

Let $h = 80 \text{ mm}$

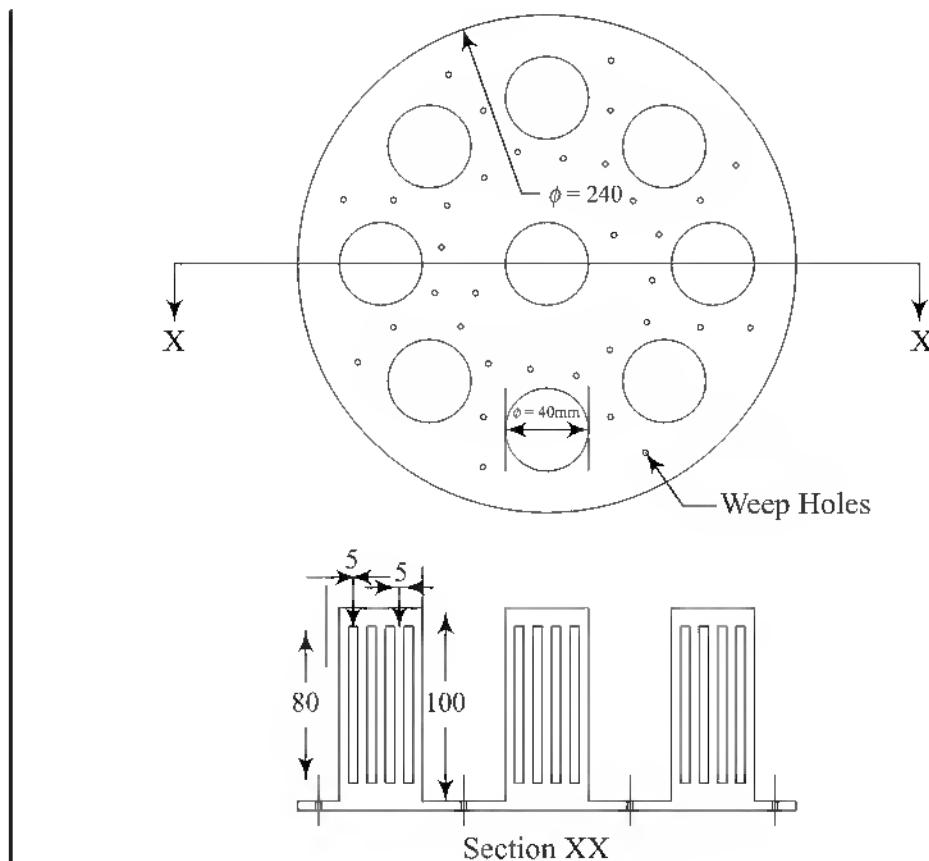


Fig. 8.30 Cap Type Packing Support

8.6 SHORT PATH DISTILLATION^{21,22}

Short path distillation is very high vacuum distillation. Operating pressure at distillation surface in short path distillation unit (SPDU) is as low as 10^{-6} bar ($1 \mu\text{bar}$). Short path distillation provides the distillation at the minimum possible temperature which is desirable for heat sensitive products.

In distillation, maximum boiling point is of the bottom product. If the normal boiling point of any product of distillation is higher than the decomposition temperature or cracking temperature, distillation cannot be carried out at atmospheric pressure. Reduction in absolute pressure results in the reduction in distillation temperature or boiling temperature. In lower pressure range, more reduction in boiling temperature of pure component is observed for a small reduction in operating pressure. For example, boiling point of stearic acid is reduced by 0.2°C when operating pressure is reduced from 251 mbar to 250 mbar. But, its boiling point is reduced by 3.8°C when operating pressure is reduced from 5 mbar to 4 mbar. In both cases, reduction in operating pressure is 1 mbar but in lower pressure range reduction in boiling point is quite higher. Hence, reduction of pressure in lower pressure range is important as it creates considerable reduction in boiling point. For a food grade product (example: orange juice) and for a highly heat sensitive product (example: vitamin E, i.e. α -tocopherol) reduction in boiling point means better recovery. Maximum recovery is obtained at the minimum boiling temperature.

Decreases in pressure results in the increase of vapour volume per unit mass and in turn it results in increase in vapour velocity. In the lower pressure range, even low vapour flow rate or non-condensable flow rate results in relatively higher value of pressure drop.

It will be interesting to note change in properties of ice and water vapour from Table 8.43 at low, high and ultra high vacuum.

Table 8.43 Properties of Water, Ice and Water Vapour at Low Temperatures²³

<i>T</i> K	<i>t</i> °C	Melting point		Saturation vapour pressure, <i>p</i>		Density of ice, ρ_s kg/m ³	Specific volume of saturated vapour, <i>V</i> m ³ /kg	Specific enthalpy, kJ/kg		
		Pa	bar	Ice	Sublimation			Vapour		
273.15	0	611.65	0.611×10^{-2}	916.708	205.99	-333.4	2834.3	2500.9		
270	-3.15	470.06	0.470×10^{-2}	917.170	264.96	-340.0	2835.1	2495.1		
265	-8.15	305.92	0.306×10^{-2}	917.886	399.64	-350.2	2836.2	2486.0		
260	-13.15	195.81	0.196×10^{-2}	918.594	612.65	-360.3	2837.1	2476.8		
255	-18.15	123.15	0.123×10^{-2}	919.295	955.48	-370.2	2837.8	2467.6		
250	-23.15	76.02	0.760×10^{-3}	919.980	1517.5	-380.0	2838.3	2458.3		
245	-28.15	46.01	0.460×10^{-3}	920.658	2457.3	-389.5	2838.6	2449.1		
240	-33.15	27.27	0.273×10^{-3}	921.319	4061.4	-398.9	2838.7	2439.8		
235	-38.15	15.81	0.158×10^{-3}	921.973	6860.7	-408.0	2838.6	2430.6		
230	-43.15	8.95	0.895×10^{-4}	922.611	11861	-417.0	2838.4	2421.4		
225	-48.15	4.94	0.494×10^{-4}	923.233	21021	-425.9	2838.0	2412.1		
220	-53.15	2.65	0.265×10^{-4}	923.839	38249	-434.5	2837.3	2402.8		
215	-58.15	1.39	0.139×10^{-4}	924.437	71588	-443.0	2836.6	2393.6		
210	-63.15	0.702	0.702×10^{-5}	925.018	1.3809×10^5	-451.3	2835.6	2384.3		
205	-68.15	0.344	0.344×10^{-5}	925.583	2.7514×10^5	-459.4	2834.4	2375.0		
200	-73.15	0.1626	1.626×10^{-6}	926.132	5.6759×10^5	-467.3	2833.1	2365.8		
195	-78.15	0.7404	0.740×10^{-6}	926.655	1.2155×10^6	-475.1	2831.6	2356.5		

Reference State:

Specific enthalpy of saturated liquid water at 273.16 K (0.01°C) = 0 kJ/kg

Specific volume of saturated water vapour at 0.1 bar (boiling point = 45.83 °C), 1.232×10^{-3} bar (or 1.232 mbar) and 1.626×10^{-6} bar (or 1.626 µbar) is $14.675 \text{ m}^3/\text{kg}$, $955.48 \text{ m}^3/\text{kg}$ and $5.6759 \times 10^5 \text{ m}^3/\text{kg}$, respectively. Thus several fold increase in vapour volume will have to be handled at ultra high vacuum. This will call for big size pipes (ducts) for transporting vapours at ultra high vacuum so that low pressure drops are achieved.

Hence, in all vacuum distillation columns, minimum possible value of absolute pressure at vaporization surface of boiling liquid in reboiler is decided by pressure drop of vapour flowing from vaporization surface of liquid in reboiler to the heat transfer surface of external condenser. In conventional packed tower type vacuum distillation column, vapour travel a long path from reboiler to external condenser. Also, in conventional design, no extra care is generally taken to reduce the flow of non-condensables.

To break this physical barrier of pressure drop of vapour and non-condensables travelling very long path from vaporizing surface to external condenser, in short path distillation, an internal condenser is used and because of the same, vapour travels through a very short distance, in order of few centimetres, from vaporization surface of liquid of bottom product to the surface of internal condenser. Because of the shortening of the path length of vapour and non-condensables, this new unit is known as short path distillation unit (SPDU).

There is a confusion in between the words "Molecular Distillation" and "Short Path Distillation". In the latter, if the distance travelled by vapour molecules from the vaporization surface to the surface of internal condenser is less than mean free path of molecules between two successive collisions, then short path distillation is called molecular distillation. If in this path length, there is no collision between vapour molecules then pressure drop in vapour flow is the minimum. Decrease in pressure increases the mean free path of molecules between two successive collisions. For commercial applications, it is not necessary to achieve the condition of molecular distillation for each application, hence only a few short path distillation units are operated as molecular distillation.

8.6.1 Design and Working of Short Path Distillation Unit

A short path distillation unit consists of a vertically mounted double walled vacuum chamber with a central inner condenser and roller wiper system.

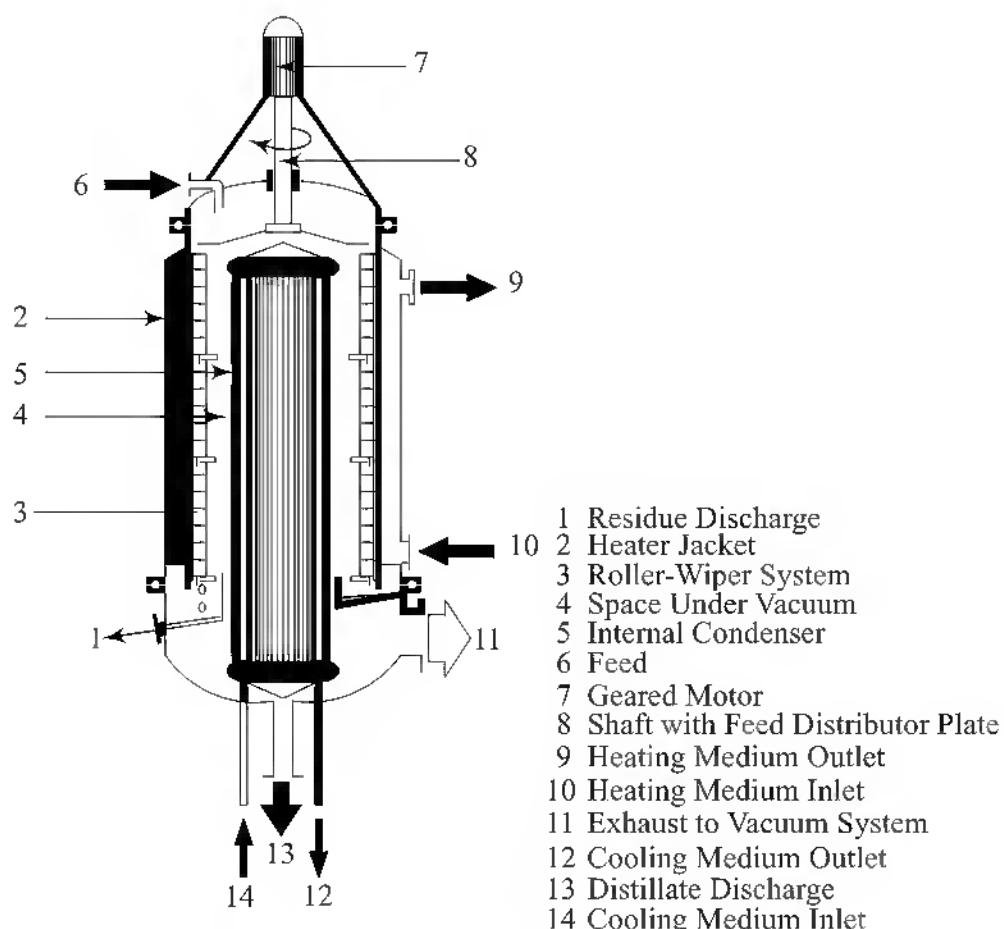


Fig. 8.31 Typical Short Path Distillation Unit

Feed is continuously fed on a rotating distributor plate. At the bottom of rotating distribution plate, 3 to 12 guiding rods are mounted. These guiding rods contain the rollers which are passed over the rods. Distribution plate with rods and rollers is rotated by an electric motor. This entire rotating system is known as Roller-Wiper system. Feed material is thrown by rotating distributor to wall or to heat transfer surface. Then material flows down by gravity over the heat transfer surface. Rollers do not touch the heat transfer surface but they create the pressure and turbulence in liquid film. Rollers are rotated about the central axis of the unit and are also rotated about their own axis. Friction between liquid film and rollers create the automatic rotation in rollers about their own axis. This roller wiper system creates effectively mixed, uniform and thin film over the heating surface. Heating is provided by thermic fluid, saturated steam or hot water. Liquid film is partially vaporized. Vapour travel through the shortest path from liquid film surface to the surface of inner central condenser. Cooling medium may be cooling water, chilled water, oil or brine.

Residue and condensate (distillate) both are falling down by gravity. Partition is provided to avoid the possibility of the mixing of residue and distillate.

Vacuum nozzle located at the side bottom of vessel, through which noncondensables and some vapours are drawn out by vacuum system. Vapours are condensed in a cold trap. Vacuum measuring instrument is also provided in the bottom part.

Unit is kept in the perfect vertical position and hence residence time of liquid on the heating surface is in the order of few seconds. The distillation takes place in "single pass" or in "once through". In this operation, in the short residence time and the lowest, vaporization temperature reduces the possibility of thermal degradation of product. Thus SPDUs provide the most gentle conditions for the distillation of heat sensitive products.

Sometimes it is advisable to have a degassing stage in front of short path distillation unit, to remove the dissolved noncondensable gases like oxygen, nitrogen, etc. In addition to this maximum tightness of the system, the prevention of gaseous cracked products and the lack of stripping gas reduce size of vacuum system required and also reduces the operating cost of the vacuum system.

Standard short path distillation units with heating surface area range from 0.01 m^2 to 42 m^2 , heating temperature up to 450°C and product throughput range from a few g/h to several t/h are available.

8.6.2 Applications of SPDUs^{21,22}

The use of short path distillation to modify the traditional processes or for the development of a new process requires special knowledge because many classical rules and laws of traditional vacuum distillation cannot apply to short path distillation. Recognized suppliers of short path distillation unit provide this knowledge to customers by their engineering services. They provide the facility of a trial run for the new system in their pilot units.

Short path distillation finds its application in pharmaceutical industries, fine chemicals industry, polymer industries, food industries, oil industries and refineries. Some important applications are:

(a) Separation of Vitamin E: The short path distillation is applied for synthetically produced vitamin – E as well as for natural vitamin – E. The synthesized vitamin – E exists in the final stage as acetic acid ester (as vitamin E – acetate). About 100 kg crude vitamin – E acetate contains 78.75 kg pure vitamin – E acetate, 13.75 kg residue and 7.5 kg low boiling impurities (typical data). In two stage short path distillation, pure vitamin E acetate is obtained as light yellow pure oil.

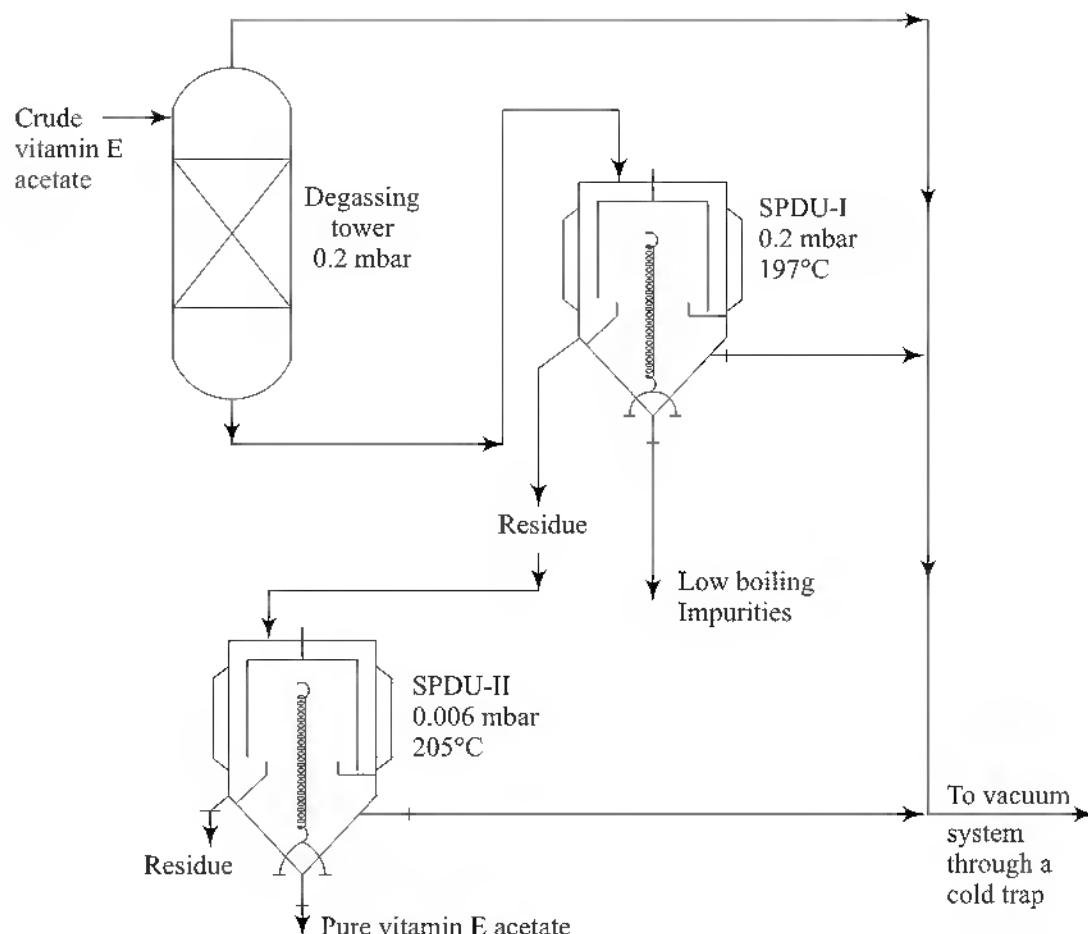
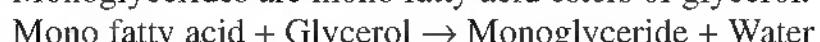


Fig. 8.32 Distillation of Vitamin-E Acetate

Deodorizer distillate of an edible oil refinery is a source of natural vitamin – E. Its concentration varies from 3 to 10% (by mass) in the distillate of soybean oil, depending on the operating conditions of the deodorizer. Concentration upto 40% (by mass) can be carried out in SPDUs.

(b) Separation of Monoglyceride:

Monoglycerides are mono fatty acid esters of glycerol.



Monoglycerides are used as emulsifiers in many of food products. The modern food product technology demands the concentration of monoglyceride more than 90%. It is a heat sensitive product. More than 90% concentration of monoglycerides is achieved by two stage short path distillation.

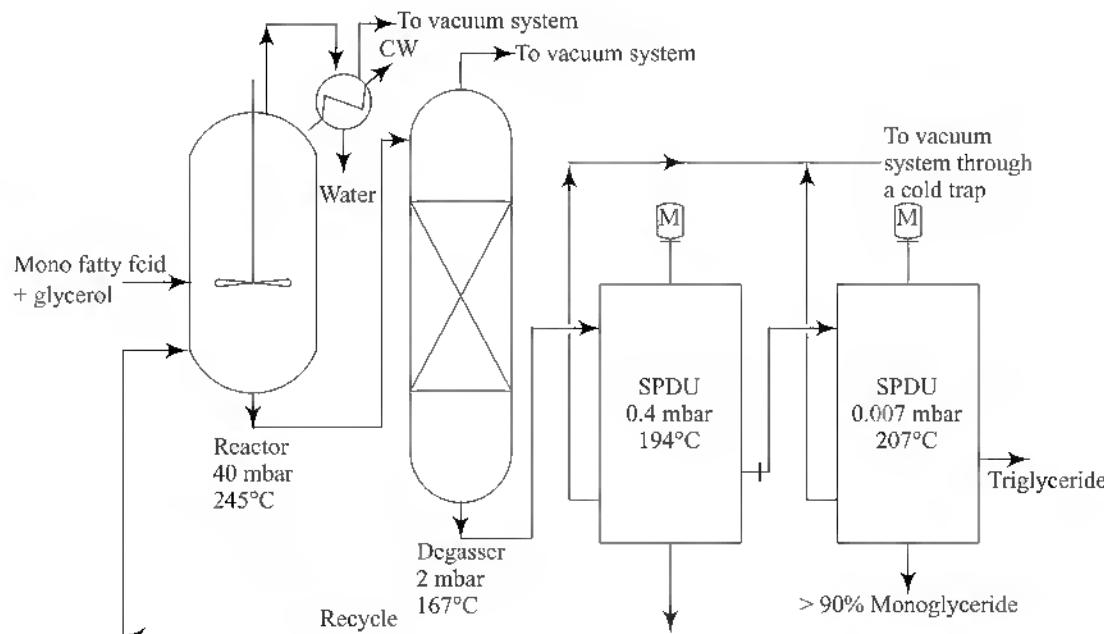


Fig. 8.33 Manufacturing of Monoglyceride

(c) Carotene: It is a pro-vitamin which is used as a supplement in many food products. Red palmoil ester contains about 0.06% carotene. Three stage short path distillation plant is required to increase the concentration upto 40%.

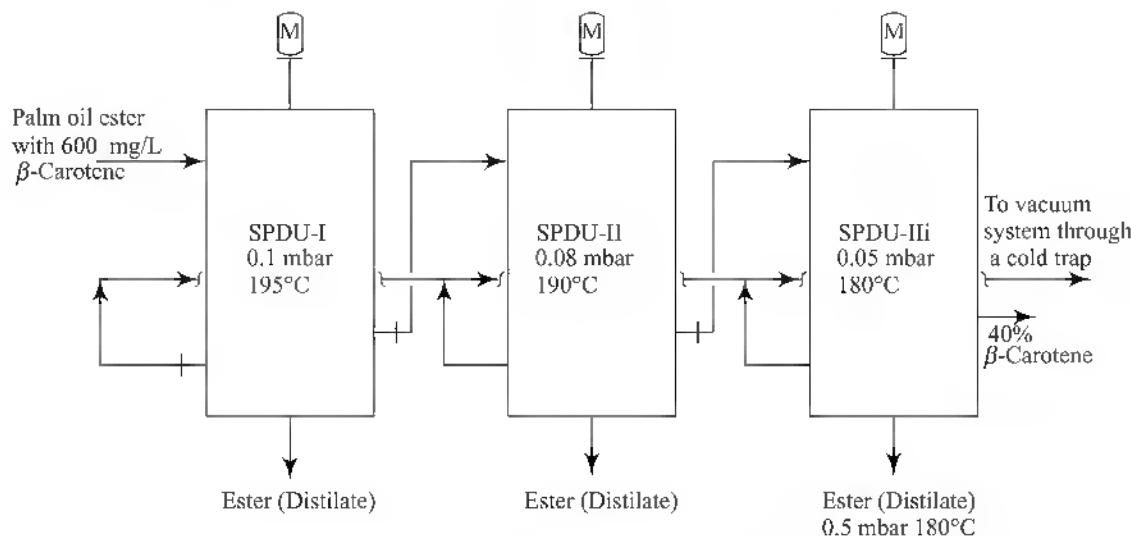


Fig. 8.34 Manufacturing of Cartone

(d) Dimeric Fatty Acids: They are basic materials for polyester or alkyd resins. They are produced by dimerisation of oleic acid. After reaction a mixture of monomer, dimer and oligomer fatty acids are formed. The fractionation of this mixture takes place in two or three stage short path distillation plant.

(e) To recover more middle distillate from the residuum, coming from vacuum distillation unit of refinery, currently vacuum distillation is carried out in conventional distillation equipment in which operating pressure at the base of column or in reboiler is decided by pressure drop in the column. The same operating pressure also decides the certain boiling range of residuum. Currently,

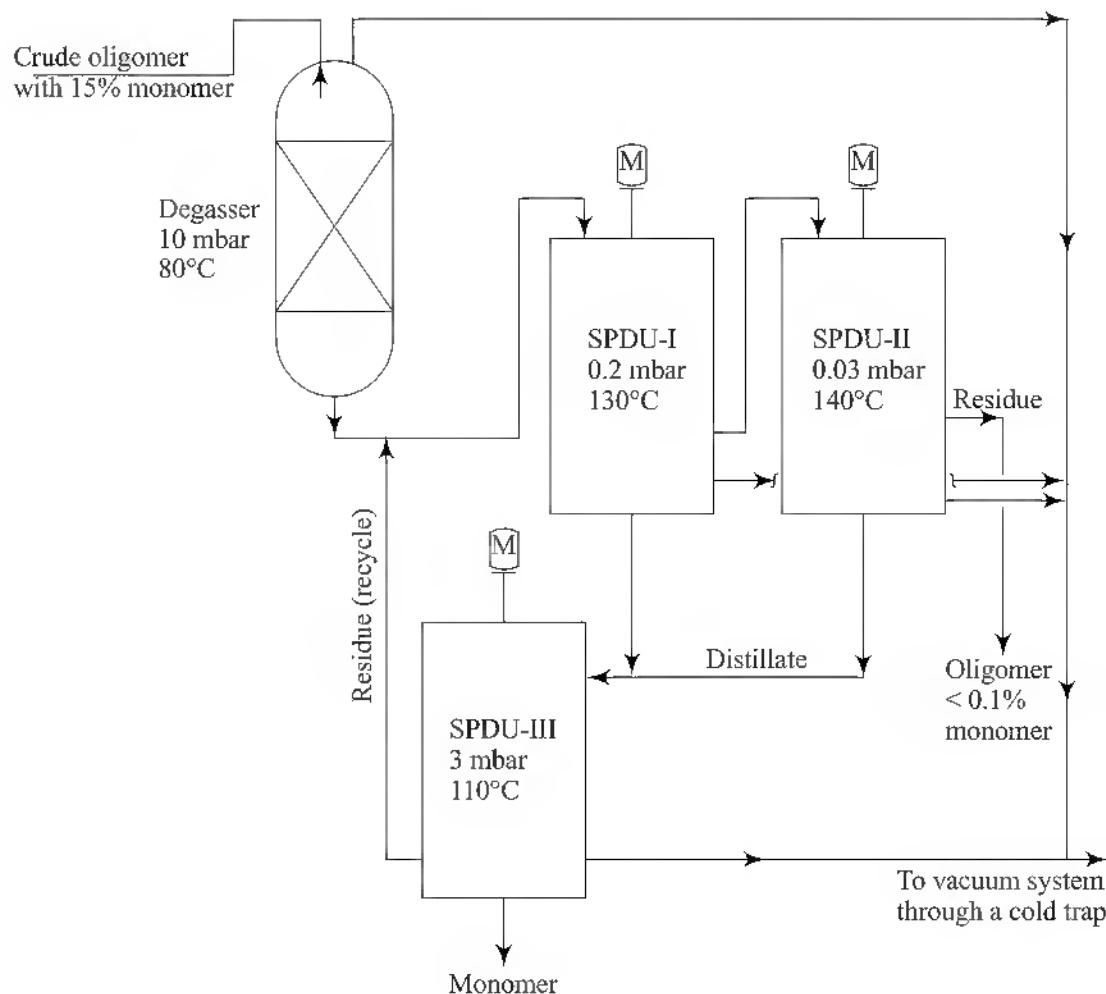


Fig. 8.35 Manufacturing of Dimeric Fatty Acids

considerable amount of middle distillate remains in the residuum. With conventional vacuum distillation unit, one can increase the % recovery of middle distillate by increasing base temperature but the same cannot be permitted because increase in the base temperature above the certain limiting value results in considerable thermal cracking of the residuum. In short path distillation the same residuum can be boiled or distilled at much lower temperature range. Reduction in operating pressure in SPDU creates the sizeable reduction in boiling temperatures. Hence with SPDU, it is possible to recover the middle distillate further without causing thermal cracking.

(f) Oleoresin: Oleoresin is a red colour natural pigment separated from paprika. It is used as a natural colour additive in food products. The oleoresin is extracted by hexane. From the extracted product, which contains 96% oleoresins and 4% hexane (by mass), hexane can be completely removed from oleoresin (down to 10 ppm) by two stage short path distillation.

(g) Deacidification of Ricebran Oil and Palm Oil: Ricebran oil and palm oil, are preferred to be used as cooking mediums because of their high nutritional values. But because of considerable high content of free fatty acids, deacidification by conventional distillation is difficult. With short path distillation, these oils can be gently deacidified to the concentrations of less than 0.1% without loss of nutritional values.

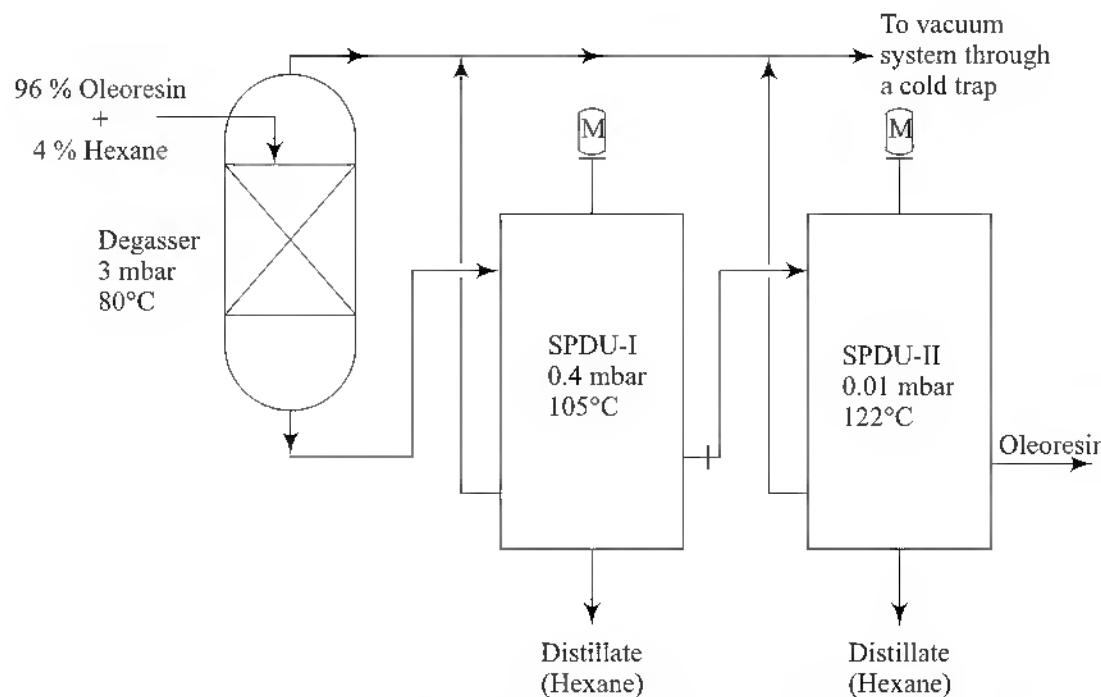


Fig. 8.36 Separation of Oleoresin

(h) Separation of Monomers from Prepolymers: Polyurethanes are polymers with different compositions and wide range of characteristics. They are produced by polyadduct type polycondensation reaction from toluene diisocyanate and polyols. In the first step, so called prepolymers are formed. These prepolymers still contain a certain amount of non-reacted diisocyanate monomers. It is necessary to reduce monomer content before selling to end manufacturers because these monomers are toxic in nature. It can be reduced by short path distillation.

8.6.3 Economics

It is a general impression that cost of short path distillation unit is quite high. The reason for this is historical. The first industrial short path distillation plants were operated with very low throughputs to maintain the very low value of operating pressure at distillation surface. Vacuum technology was not advanced at that time and this was the limiting factor for the throughput. Also in initial stages there were high over dimensioning of the unit. These all reasons created the general impression that short path distillation is very expensive operation and the distillation cost can only be borne by very expensive products. But now, reality is different.

Improved vacuum technology, considerably higher throughput quantities, increased availability of units from different manufacturers and low personnel requirements have led to a substantial reduction in cost of distillation. Results have shown that on increasing product throughput from 150 kg/h to 3600 kg/h, total cost of short path distillation in Rs./kg of product reduces by more than 85%.

Short path distillation unit requires only one week shut down time per year. Hence it requires relatively less maintenance.

8.7 REACTIVE AND CATALYTIC DISTILLATION^{24,25}

In most of the chemical plants one can find the reaction followed by separation. Usually reaction and separation are performed one by one. First reactants are allowed to react in the reactor, then in a separate equipment product mixture is separated from unconverted reactants and/or from byproducts and inert. It is rarely seen that both reaction and separation combined and carried out simultaneously in one equipment.

However, by combining reaction and separation of product in one equipment one can get two advantages.

1. Removal of the product from the reaction mass at the reaction conditions increases equilibrium conversion of reactant or increases the extent of reaction. As per LeChatelier's law, for the reaction in equilibrium, if any change in any process variable like pressure, temperature or composition of reaction mass is made, reaction or process is moved in such a direction which will nullify the effect of the change in the variable. In reactive distillation, change in the composition of reaction mass is made by continuously removing the product from the reacting mass. Hence, to nullify the effect of this change, reaction proceeds in the forward direction and produces more and more products and so equilibrium conversion is improved.
2. Separate equipment for the separation is not required. Also separate piping, instruments, etc. are not required.

There are four commonly used techniques to combine reaction and separation.

- (i) Reactive distillation (Reaction + Distillation)
- (ii) Membrane reaction (Reaction + Membrane separation)
- (iii) Extractive reaction (Reaction + Extraction)
- (iv) Reaction with absorption (Reaction + Absorption)

In reactive distillation, reaction and distillation both are carried out simultaneously in one equipment. If reactive distillation is carried out in presence of heterogeneous solid catalyst, then it is called catalytic distillation.

The concept of reactive distillation is not new. This technique was first applied in 1920 to esterification process using homogeneous liquid phase catalyst. In manufacturing of ethyl acetate, reaction between ethanol and acetic acid and separation of crude ethyl acetate from reaction mass by distillation were carried out simultaneously in one equipment. Ethanol was used as excess reactant. Overhead vapour from reactive distillation column is a ternary azeotrope having composition 82.16% ethylacetate, 8.4% ethanol and 9% water (by mass). On condensation heteroazeotrope vapour is converted in to two layers, ester rich layer and ethanol–water solution layer. Part of ester rich layer is taken out as top product (distillate) and remaining is recycled back to column as reflux. Sulphuric acid is added in feed as catalyst. Waste water (water + sulphuric acid) is continuously taken out as bottom product. Alcohol – water stream is taken out as an intermediate product.

In manufacturing of butyl acetate over head vapour is a ternary hetroazeotrope of butyl acetate, butanol and water. On condensation it is converted into two immiscible liquid layers.

Table 8.44 Ternary Azeotrope Separation at 90°C

Layer	% by mass	% Ester	% Butanol	% Water
Upper	75	80.5	13.4	6.1
Lower	25	1.92	1.69	97.39

(Table 12.7 of Ref. 26)

Lower aqueous layer is continuously taken out from decanter for recovery of butanol (separately) while upper layer or ester rich layer is recycled back to column as a source of reflux. Bottom product is crude butyl acetate. It is sent for further purification. Thus in both cases, concept of reactive distillation, invented in 1920, was applied. But after that for a long period of time very little work was done in this direction. Almost after 50 years, an important research work was done by Sermewald in 1971²⁷, that applying reactive distillation with heterogeneous catalyst is far more useful than homogeneous catalyst. Nowadays, this technique is being successfully applied to some processes like synthesis of methyl tertiary butyl ether (MTBE), synthesis of ethyl tertiary butyl ether (ETBE), selective hydrogenation of butadiene of C₄ stream, synthesis of ethyl propionate, methyl acetate, etc.

If solid catalyst is used, then the same catalyst can also act as packing material of packed tower. In that case solid catalyst plays double role to enhance the rate of chemical reaction as well as to enhance the rate of mass transfer for distillation. This special type of reactive distillation which uses solid catalyst is known as catalytic distillation.

Even though reactive distillation has two major advantages over conventional reactor followed by distillation (as mentioned earlier in this chapter), it finds its place only in few processes. Reason is, it also has one major disadvantage.

Disadvantage:

In majority of the processes which involve reaction followed by distillation, optimum conditions for reaction and optimum conditions for distillation are different. Hence to carry out both simultaneously in one equipment, one has to compromise in reaction or distillation. In most of the cases, the compromise is in the distillation. In a few cases like in the process of MTBE, optimum conditions for the reaction and the same for distillation are more or less same. Hence one can apply the concept of catalytic distillation in the process of MTBE. But in many cases this combination is not economically feasible. For example: in the manufacturing process of acetic acid by carbonylation of methanol, first reaction is carried out in presence of homogeneous phase catalyst at 50 atm pressure and at 180°C temperature. After the reaction, reactants, promoters and catalyst are separated from acetic acid by distillation at atmospheric or near to atmospheric pressure. Hence in this case one cannot apply the concept of reactive distillation because optimum conditions for the reaction and for the distillation are very much different.

8.7.1 Comparison of Reactive Distillation with Conventional Technology

Conventional technology involves two steps.

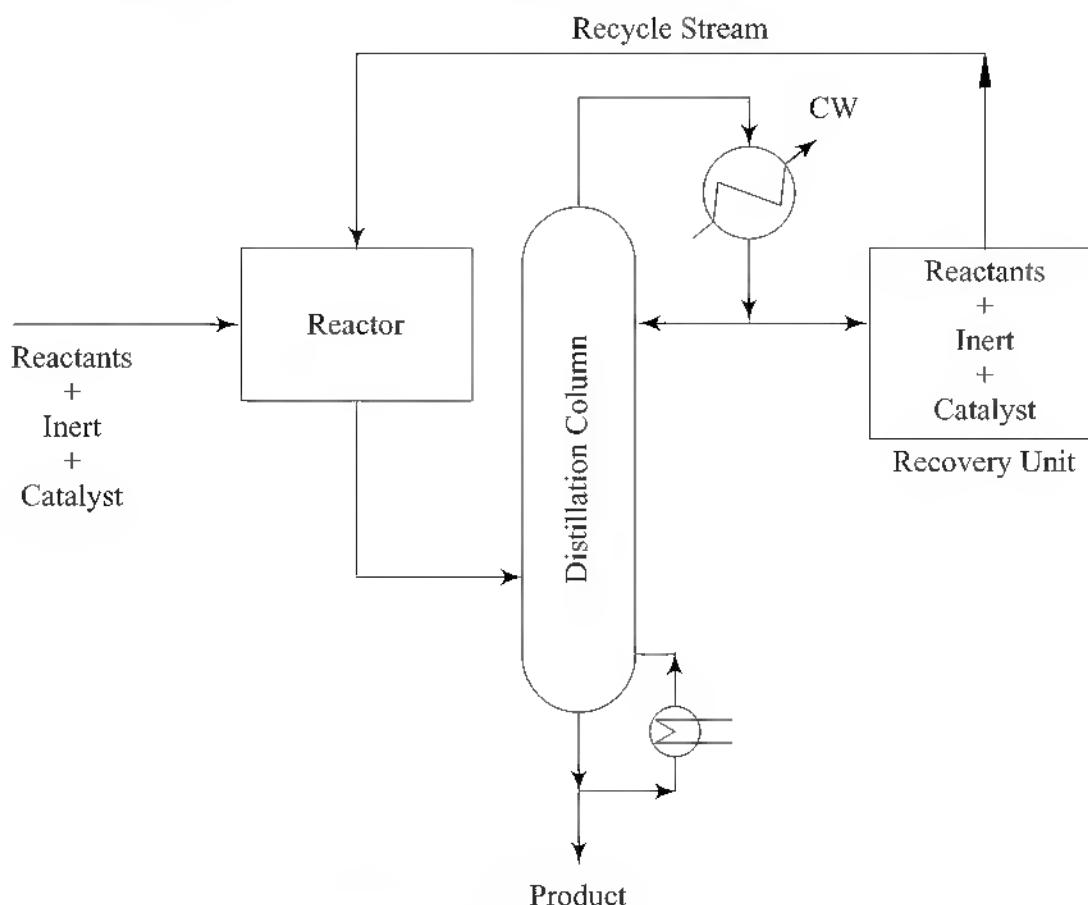


Fig. 8.37 Typical Conventional Process

In the conventional process first step is the reaction step. In this step reactants are brought into contact with solid or liquid catalyst at appropriate temperature and pressure.

In second step stream from the reactor is sent to separation section, where desired product is separated from unconverted reactants, catalyst, byproducts and/or inert. In most of the cases this separation is done by distillation.

When concept of reactive distillation (or catalytic distillation, if catalyst is in solid phase) is applied to such type of processes, flow sheet is changed. Typical flow sheet of catalytic distillation column is shown in Fig. 8.38.

In middle section of catalytic distillation column, reaction and distillation take place simultaneously while upper and lower sections represent enriching and stripping sections of distillation or they are utilized only for separation.

The advantages of reactive distillation can be described best by considering its advantages over reaction step and distillation step of conventional process.

(a) Advantages over Reaction Step:

- (i) For equilibrium limiting reaction, continuous removal of one of the products at process conditions increases equilibrium conversion and it converts

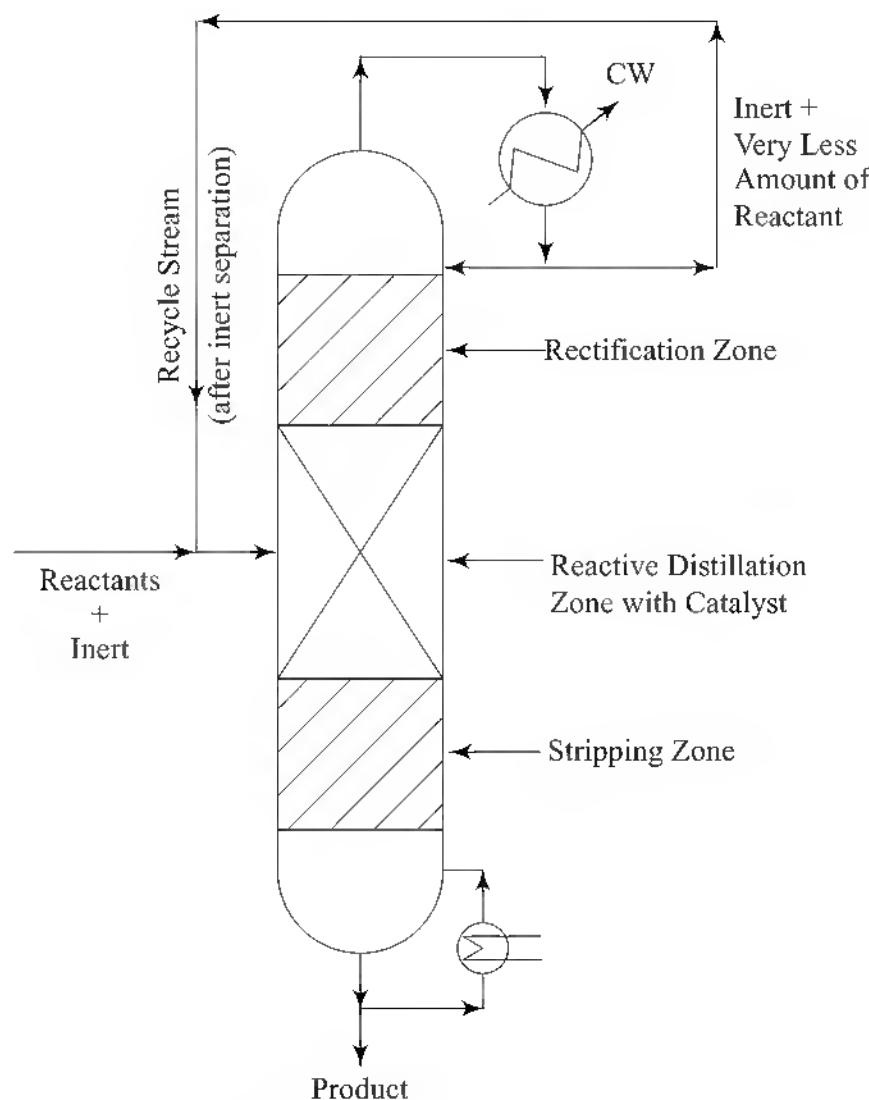


Fig. 8.38 Typical Flow Sheet of Catalytic Distillation

reversible reaction into irreversible reaction. Thus reactive distillation provides high conversion compared to conventional reaction step.

- (ii) For exothermic or endothermic reaction, separate cooling or heating system is required for reactor in conventional process. In reactive distillation, separate heating or cooling system is not required for reaction zone. In reactive distillation, heat of reaction is utilized in distillation either for condensation or for boiling.

(b) Advantages Over Distillation Step:

(b₁) Advantages Related to Reduction in Capital Cost

- (i) In conventional technology, separate equipment is required for distillation. Reactive distillation eliminates use of major process equipment and associated pump, piping and instrumentation.
- (ii) Heat of reaction reduces either condensation duty or reboiling duty. Hence it either reduces condenser size or reboiler size.
- (iii) In reactive distillation very less amount of unconverted reactants are to be separated from products compared to distillation in conventional technology. Hence reactive distillation requires lesser number of stages for same value

of reflux or less amount of reflux compared to distillation of conventional technology or it requires lesser size of distillation column compared to the same required for the distillation in conventional technology for the given production rate. Less amount of reflux also decreases sizes of condenser and reboiler.

(b₂) Advantages Related to Reduction in Operating Cost

- (i) Heat of reaction reduces either condensation duty or reboiling heat duty. Hence, it reduces either load of cooling system or the same of heating system. Consequently, it reduces operating cost.
- (ii) Operating cost required for recycling of reactants is reduced considerably.
- (iii) In reactive distillation, amount of distillate is reduced considerably. Hence, for the same reflux ratio, lesser amount of reflux is required in reactive distillation compared to distillation column of conventional technology. It decreases condenser heat duty, reboiler heat duty and power required for pumping the reflux.

8.7.2 Different Ways by Which Reactive Distillation Can be Applied

There are three different ways by which Reactive Distillation can be applied.

- (i) Reactive Distillation alone
- (ii) Reactive Distillation plus Non-reactive Distillation.
- (iii) Reaction alone plus Reactive Distillation plus Non-reactive Distillation.

(i) Reactive Distillation Alone:

In this case, throughout the whole column, reaction and distillation take place simultaneously. At one of the ends, purified product is obtained. This is possible only for the systems for which pure product point lies within reaction space.

(ii) Reactive Distillation plus Nonreactive Distillation:

There are many cases for which pure product point does not lie within reaction space. For such cases, nonreactive distillation section (either rectifying or stripping or both) is also required to achieve pure product and pure reactants.

(iii) Reaction alone plus Reactive Distillation Plus Non Reactive Distillation (Optional):

If the reaction space required is considerably greater than distillation space, then this method is applied.

Sometimes it is not recommended to shift entire reaction space to reactive distillation unit. The reason may be one of the followings.

- (a) If the optimum reaction conditions and optimum distillation conditions are different, then it is better to improve only equilibrium conversion in reactive distillation, while major part of reaction (up to equilibrium conversion) can be carried out separately in another reactor.
- (b) If the existing plant of conventional technology have more number of reactors in series than one may shift only last or a few of the last reactors to reactive distillation unit.

8.7.3 Various Contact Devices Used for Catalytic Distillation

In catalytic distillation, contact devices are required to improve mass transfer rate and to improve catalyst contact. Various types of contact devices used for catalytic distillation are as follows.

- Tray column in which bed of catalyst is placed either in downcomers or above the trays.
- Packed tower using random packing elements which contain the catalyst (such as special coating) or which may be made of the catalyst.
- Packed tower using ordered packing elements which contain catalyst particles. In orderly packing element, catalyst particles are sandwiched between two supporting materials. Two types of ordered packings are used.
 - Bale packing
 - Structured packing

1. Bale Packing:

In bale packing, catalyst particles are enclosed within several pockets in a fiber glass cloth belt.

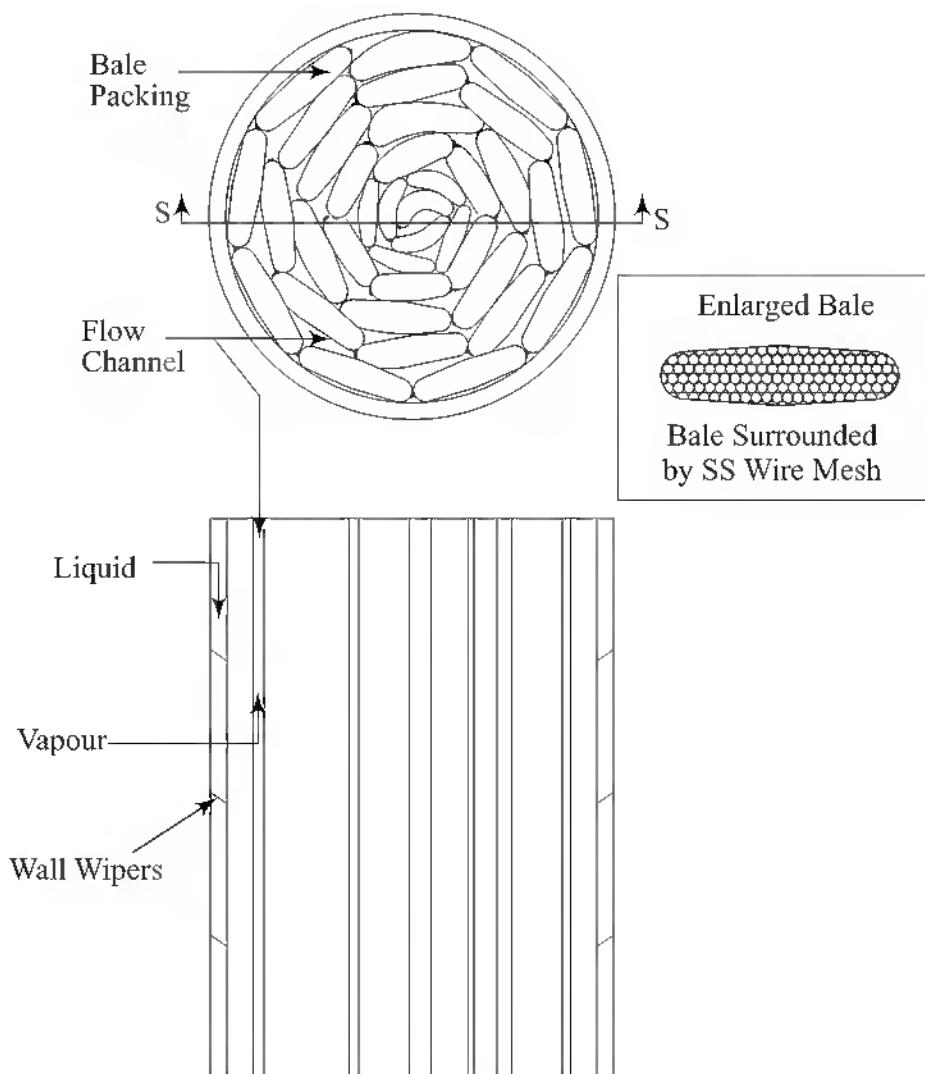


Fig. 8.39 Bale Packing

The pockets are normally 2.5 to 5 cm wide with 0.625 to 1.25 cm web between them. Open ends of pockets are sewn closed. Belts are rolled up with alternating layers of stainless steel mesh. Stainless steel mesh increases structural strength of cloth belt.

Bales constructed for commercial use are roughly cylindrical shape with 20 to 35 cm diameter and 50 cm height. Several bales then packed tightly into a column such that pockets remain vertical. Flow channels facilitate the contact between vapour and liquid. Ideally, contact between liquid reactants and catalyst is taking place in pockets and vapour–liquid contact takes place in flow channel. HETP as well as pressure drop relations for bale packings could be obtained from the supplier.

2. Structured packing:

KATMAX structural packing is developed by Koch Engineering Company, USA. From outside this packing looks like a brick with corrugated surface. In such type of packing solid catalyst is held in a screen envelope.

The catalyst is sandwiched between the layers of screen. It is easily accessed by the liquid. Between two layers of catalyst (surrounded by screen), gaps are provided, known as flow channels which facilitate the flow of vapour. Inside the flow channel vapour makes the contact with liquid film.

8.7.4 Industrial Applications of Reactive Distillation

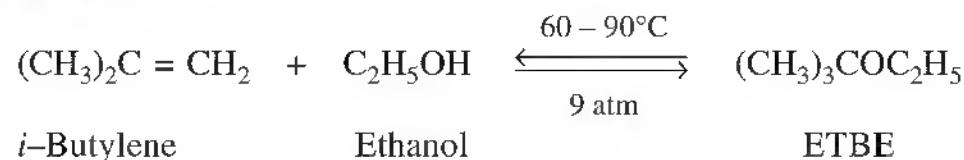
- (a) Manufacturing of ethylacetate from ethanol
- (b) Manufacturing of methylacetate from methanol
- (c) Manufacturing of butylacetate from butanol
- (d) Manufacturing of MTBE (methyl tertiary butyl ether) and ETBE (ethyl tertiary butyl ether)
- (f) Selective hydrogenation of butadine of C₄ stream
- (g) Selective hydrogenation of pentadine of C₅ stream
- (h) Selective hydrogenation of hexadine of C₆ stream
- (i) Manufacturing of benzyl chloride from toluene
- (j) In benzene separation of gasoline.
- (k) Manufacture of ethyl propionate from ethanol
- (l) Manufacture of amino resins

Among these applications, manufacturing of ETBE by catalytic distillation is discussed in this chapter.

8.7.5 Manufacturing of ETBE

Ethyl tertiary butyl ether is used as octane number improver, like MTBE. ETBE has higher octane rating and lower volatility compared to MTBE. However, oxygen content of ETBE is less compared to MTBE, hence larger volumes of ETBE is required. Also, cost of ETBE is higher than the same of MTBE.

Main Relation:



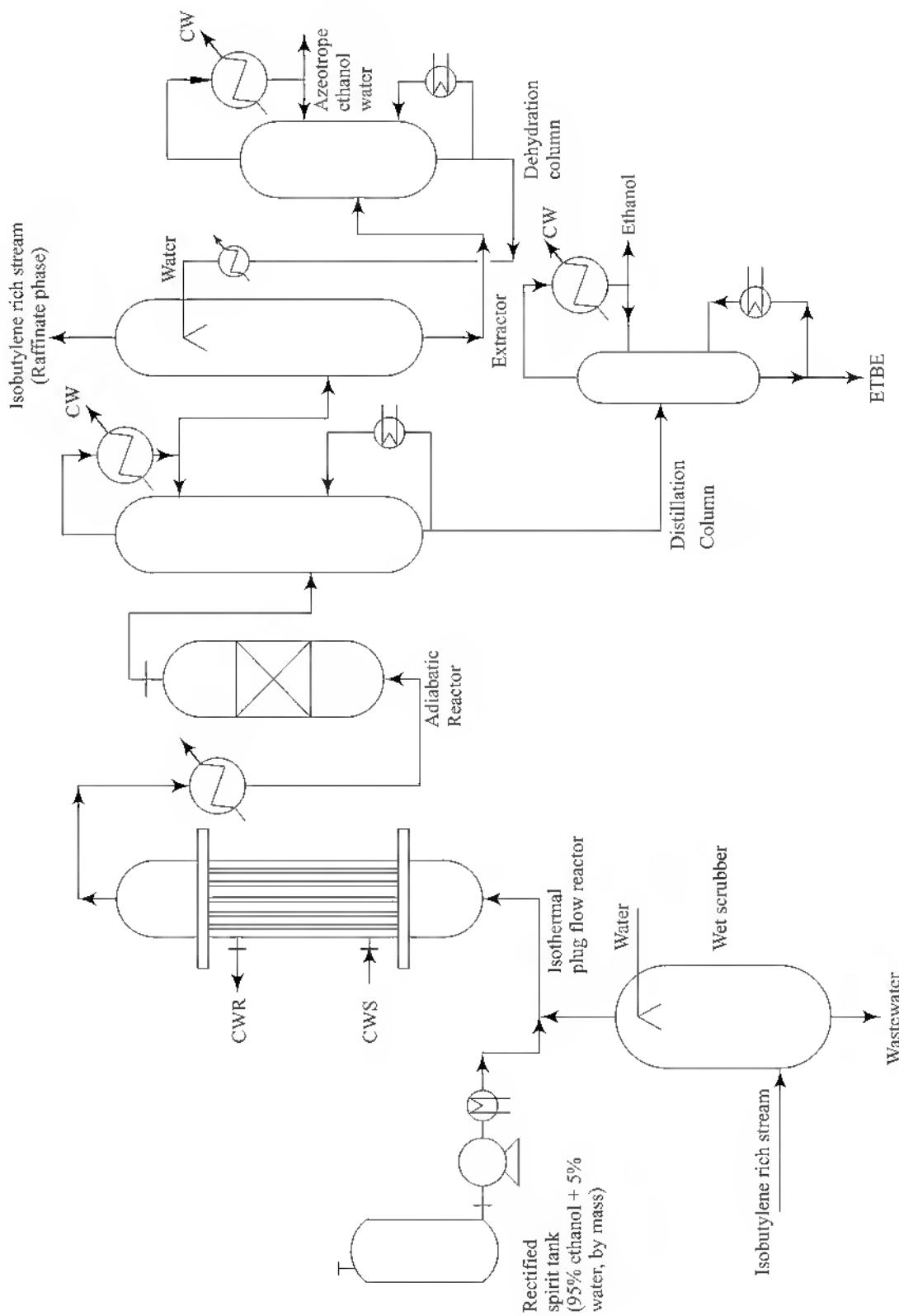
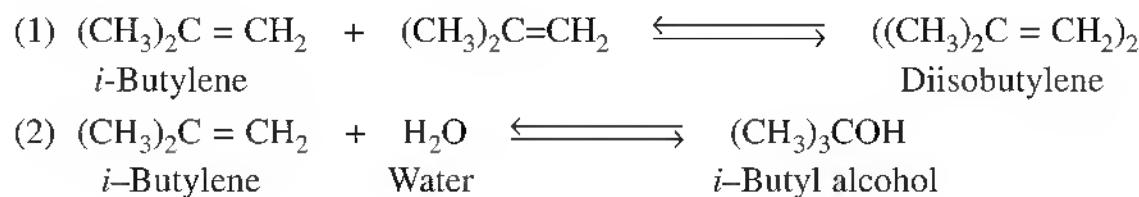


Fig. 8.40 Conventional Process of ETBE

Side reactions:



8.7.5.1 Conventional Process

Isobutylene rich stream is water washed in wet scrubber to remove trace contaminants that can deactivate the catalyst. Then this stream is mixed with rectified spirit and mixture is sent to isothermal, shell and tube type plug flow reactor. Ethanol is used as excess reactant. Second reactor is operated adiabatically. Product from second reactor is sent to distillation column. In first distillation column, mixture of ethanol, water and isobutylene is separated from ETBE. Ethanol cannot be separated from the distillate of first column by common distillation because ethanol forms azeotrope with isobutylene as well as with water.

Further addition of water as solvent separates ethanol and water mixture from isobutylene in liquid-liquid extractor. Isobutylene rich stream is coming out from the top of extractor as light product or as raffinate. Ethanol – water stream or extract stream leaves from the bottom of the extractor. From the extract phase, azeotrope (95.6% ethanol and 4.4% water by mass) is separated by distillation and it is recycled back to the reactor.

Bottom product of first distillation column is ETBE–ethanol mixture from which ethanol is separated from ETBE by distillation.

8.7.5.2 Reactive Distillation Process²⁸

In reactive (catalytic) distillation technology only equilibrium conversion is increased in reactive distillation unit. Up to almost 80% conversion of isobutylene is achieved in a (conventional) separate isothermal shell and tube heat exchanger type plug flow reactor. Hence, feed to reactive distillation column is rich in ETBE but still contains ethanol and isobutylene. Instead of rectified spirit, absolute alcohol is used as raw material with isobutylene.

Overhead product from reactive distillation column is non-reactive hydrocarbons (*n*-butane, *n*-butylene, etc.), isobutylene and ethanol, respectively.

Reactive Distillation column has total ten packing sections. First and second packing sections use simple structured packing, act as rectification or enriching section. Third, fourth and fifth packing sections use ordered packing and act as reactive distillation zones. Sixth to tenth packing sections used simple structural packing which act as stripping section.

Typical data of this process are as follows.

- (a) Feed composition: 29.1% ETBE, 9.1% ethanol,
73% isobutylene, 54.5% *n*-butylene (by mole)
(Feed to reactive distillation unit)
 - (b) Isobutylene conversion: 97.4%
 - (c) Operating pressure = 900 kPa a
 - (d) Reflux ratio = 5.0
 - (e) Bottom composition = 96.5% ETBE, 2.8% ethanol
0.7% butylene, 0.04% diisobutylene (by mole)

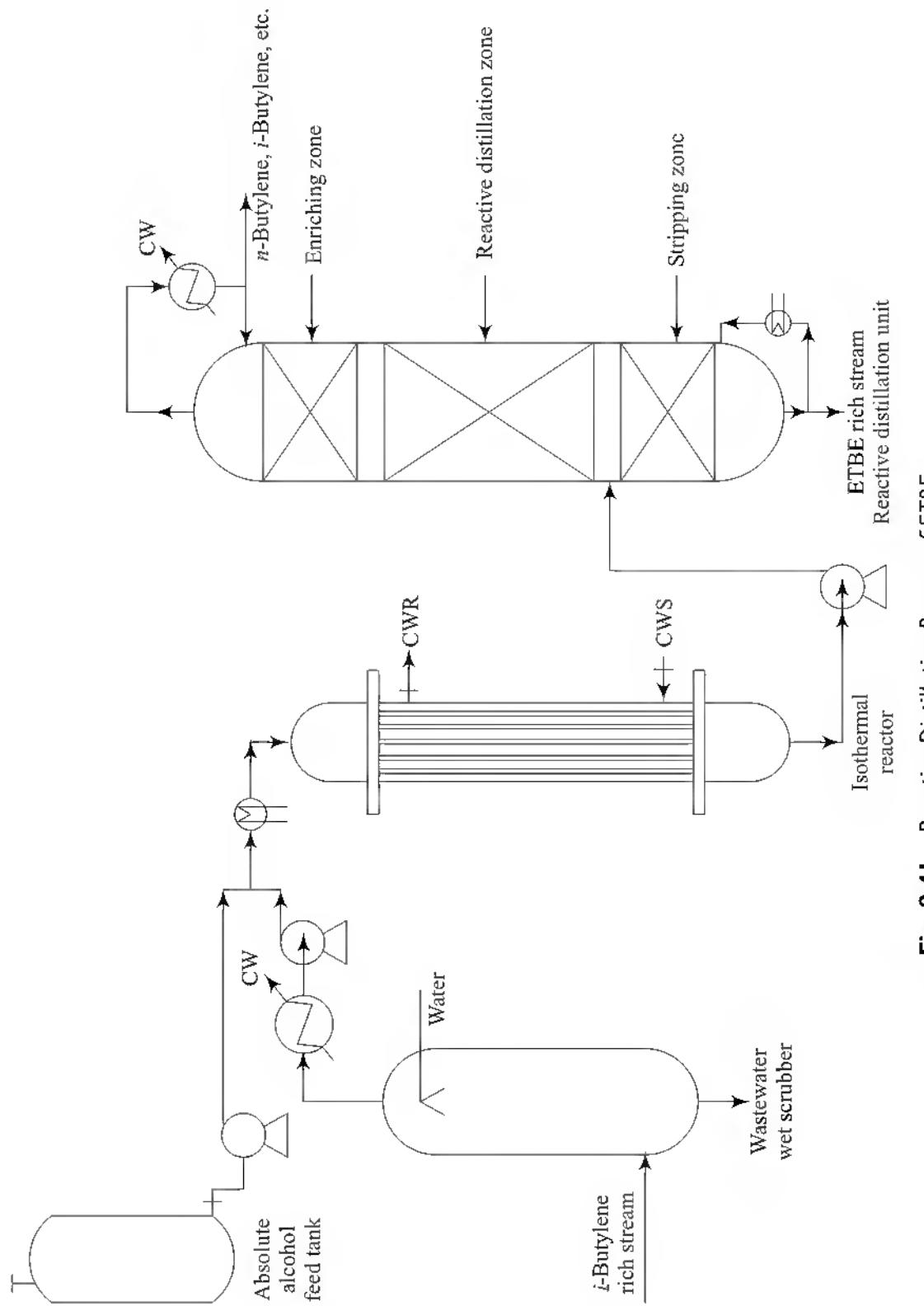


Fig. 8.41 Reactive Distillation Process of ETBE

- (f) Distillate composition = 96.5% *n*-butylene, 1.7% isobutylene, 0.7% ethanol (by mole)

8.7.7 Effect of Various Parameters on Reactive Distillation of ETBE

(i) Effect of Feed Composition: Increase in isobutylene percentage in feed (isobutylene rich stream) results in

- (a) Decrease in energy cost per kg of ETBE produced. As lesser energy is “wasted” in heating and cooling of inert compounds.
- (b) Decrease in size and fixed cost of reactive distillation unit and associated equipments for the same production rate of ETBE.
- (c) Increase in the reactant concentrations in reaction zone, increases reaction rate as well as equilibrium conversion.

$$f(X_e) = K \propto \left(\frac{p_t}{n_t} \right)^{\Delta n} \quad (8.108)$$

In this reaction Δn = change in moles during reaction = 2 – 1 = 1. Hence decrease in total number of moles of reaction system increases the value of equilibrium conversion X_e .

- (d) Increase the temperature of reaction zone due to decrease in stabilizing effect of inert components. Reaction is exothermic.

(ii) Effects of Excess Ethanol: Excess of ethanol is required to suppress rate of side reactions and to increase yield of ETBE. But, increase in excess of ethanol decreases the purity of ETBE as a bottom product of reactive distillation. It has been found that 4% to 7% excess ethanol is sufficient to produce high yield of ETBE without affecting the purity of ETBE as a bottom product from the reactive distillation unit.

(iii) Effect of Column Pressure: In conventional distillation, operating pressure is decided based on use of cooling water or cheaper cooling medium in overhead condenser. Sometimes, to avoid the thermal cracking of product or to alter the vapour–liquid equilibrium or to facilitate the use of cheaper heating medium, etc. conventional distillation is carried out in vacuum.

However, in reactive distillation, choice of operating pressure is more complicated. In reactive distillation pressure affects equilibrium conversion of reaction.

In ETBE, synthesis moles are decreasing during reaction, hence higher pressure is favourable as it increases equilibrium conversion. But, this is not always true, as increase in pressure increases temperature of reaction zone of reactive distillation unit. Since ETBE synthesis is exothermic reaction, increase in temperature decreases equilibrium conversion. It is found that best conversion is achieved at 400 to 500 kPa a. But considering distillation aspect, operating pressure is kept 700 to 900 kPa a.

(iv) Effect of Reactive Distillation Stages: Increase in length of reactor zone or increase in number of stages of reactive distillation zone improves the conversion, but at the same time decomposition of product starts in the lower stages, if the number of stages increased above the optimum number of stages.

(v) Effect of Separation Stages: Ideally the rectification or enriching section of reactive distillation column for ETBE synthesis should (a) remove light inerts

from reaction zone, (b) prevent loss of ether or alcohol in distillate and (c) recycle unreacted reactants to the reaction zone. In actual practice this is almost impossible to achieve as favourable conditions for the reaction cannot be changed. However, the loss of ETBE in distillate can be minimized without rejecting isobutylene from the column. More enriching stages are required to prevent loss of ethanol with distillate.

Ideally stripping section should:

- (a) Remove ETBE from the reaction zone to maintain favourable reaction conditions.
- (b) Purify the ether (ETBE) product.
- (c) Prevent loss of reactive isobutylene with ether (ETBE) product.
- (d) Minimize ethanol loss with the ether product.

These functions are easily achievable at temperature and pressure of reaction conditions.

(vi) Effect of Reflux Ratio:

Effect of increasing reflux ratio are

- (a) The concentration of reactants in the distillate is reduced.
- (b) The reaction zone temperature reduces which is favourable for exothermic reaction to increase equilibrium conversion.
- (c) The concentration of ether in the reaction zone reduces. It reduces catalytic cracking of ether (ETBE).

All these three effects increase the conversion. However, higher reflux ratio increases tower diameter, condenser and reboiler sizes and energy requirements.

(vii) Effects of Feed Point:

In ETBE synthesis, optimum feed point for reactive distillation column is just below the reactive distillation zone. In reaction zone, ETBE can decompose or it can react by cracking reaction in presence of catalyst. So to avoid the decomposition of ETBE in the feed (i.e. ETBE rich stream), it is fed just at the bottom of reaction zone. Feeding too far below the reaction zone reduces the stripping potential and increases energy requirement for the same height of stripping section.

(viii) Effects of Reboiler Duty:

At some optimum value of reboiler duty, higher conversion (more than 95%) and higher purity (~ 90%) is achieved. If reboiler duty is too high or too low, conversion and product purity both are reduced.

Manufacture of ETBE was discussed as a case study of reactive distillation so that a process engineer can learn to analyse a system before actual process design is taken up.

8.7.8 Manufacture of Ethyl Propionate

Ethyl propionate can be synthesized²⁹ by esterification of ethyl alcohol with propionic acid in a reaction distillation unit using cation exchange resin as catalyst.

8.8 AZEOTROPES AND SEPARATION THEREOF

In the previous sections, distillation of liquid mixtures was dealt which are amenable to ordinary fractional distillation. In these cases, VLE plot or an equilibrium curve of a binary is on one (upper) side of the diagonal line. However,

when the equilibrium curve crosses the diagonal line, an azeotrope occurs. Thus at a given pressure, a mixture of specific composition boils at a constant temperature which produces vapour of the same composition. When this happens, the mixture cannot be separated and it is known as azeotropic mixture. When the vapour of the azeotropic mixture are condensed, liquid mixture so obtained decides the type of azeotrope. If the condensed liquid phase is homogeneous, it is called a homogeneous azeotrope. Ethanol-water forms homogenous azeotrope at 101.325 kPa and 78.1°C having 95.6% ethanol (by mass). Figures 8.42 (a) and (b) and Figs 8.43 (a) and (b) refer to homogeneous azeotropes.

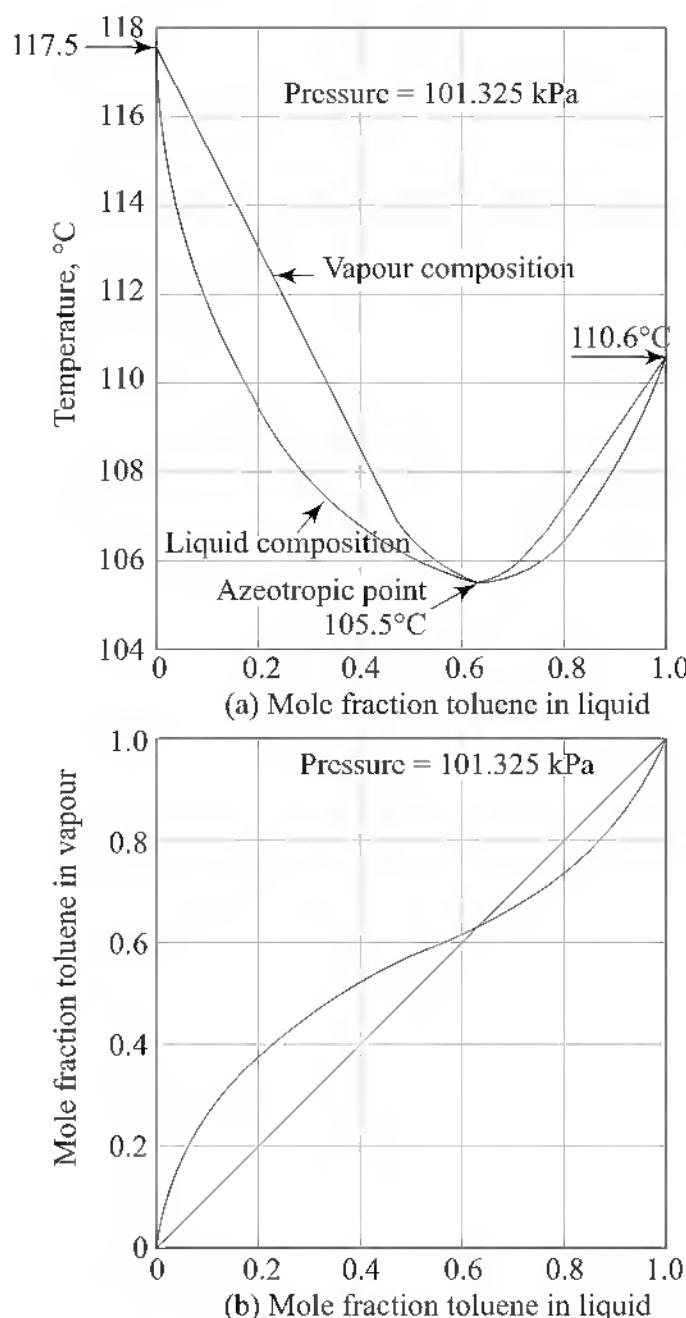


Fig. 8.42 (a) *t-x-y* Diagram for Toluene-n-Butanol System, (b) Homogeneous Minimum Boiling Azeotrope of Toluene-n-Butanol System³⁰
(Reproduced with the permission of Chemical Engineering by Access Intelligence, USA)

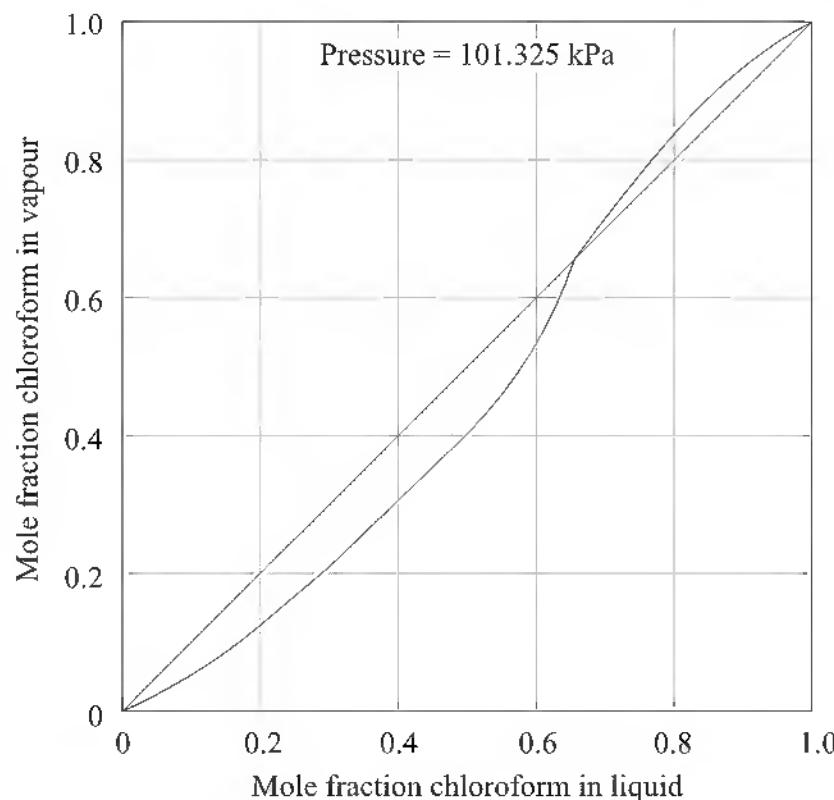
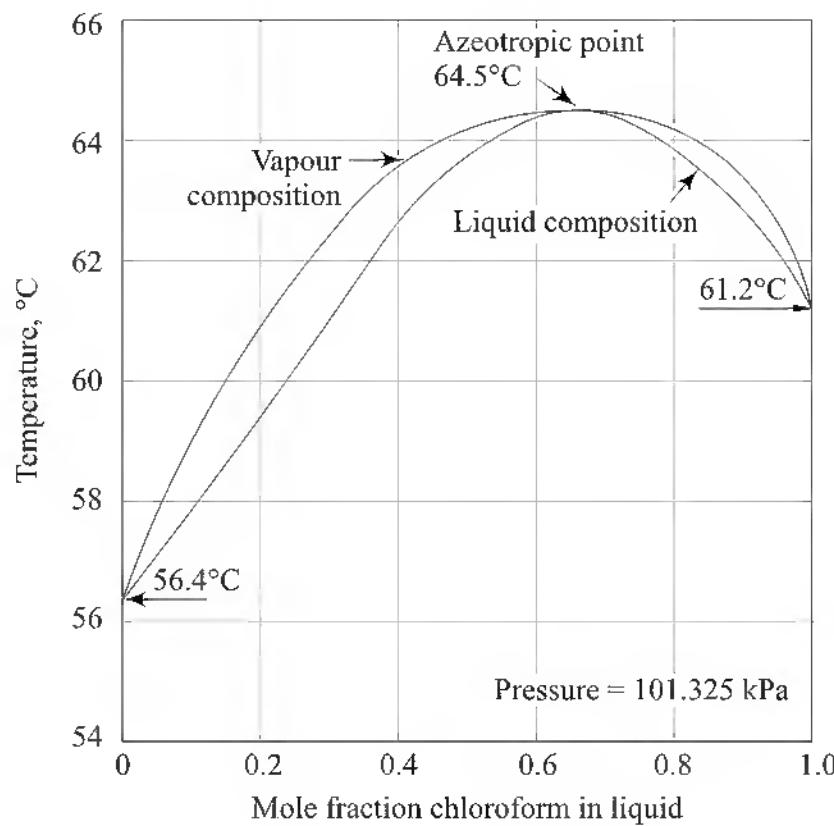


Fig. 8.43 (a) *t-x-y Diagram for Chloroform-Acetone System, (b) Homogeneous Maximum Boiling Azeotrope of Chloroform-Acetone System*³⁰
(Reproduced with the permission of Chemical Engineering by Access Intelligence, USA)

A heterogeneous azeotrope is one for which the overall liquid mixture forms two liquid phases. Mixture of *n*-butanol–water forms an azeotrope at 101.325

kPa and 92.2°C having 57.5% (by mass) or 24.8% (by mole) *n*-butanol in the overall liquid mixture. It separates in two layers. At 40°C, top layer contains 78.51% *n*-butanol while the bottom layer contains 6.60% *n*-butanol (by mass). Refluxing a specific layer provides a method to separate the components of the mixture. Figures 8.44 (a) and (b) refer to heterogeneous azeotrope.

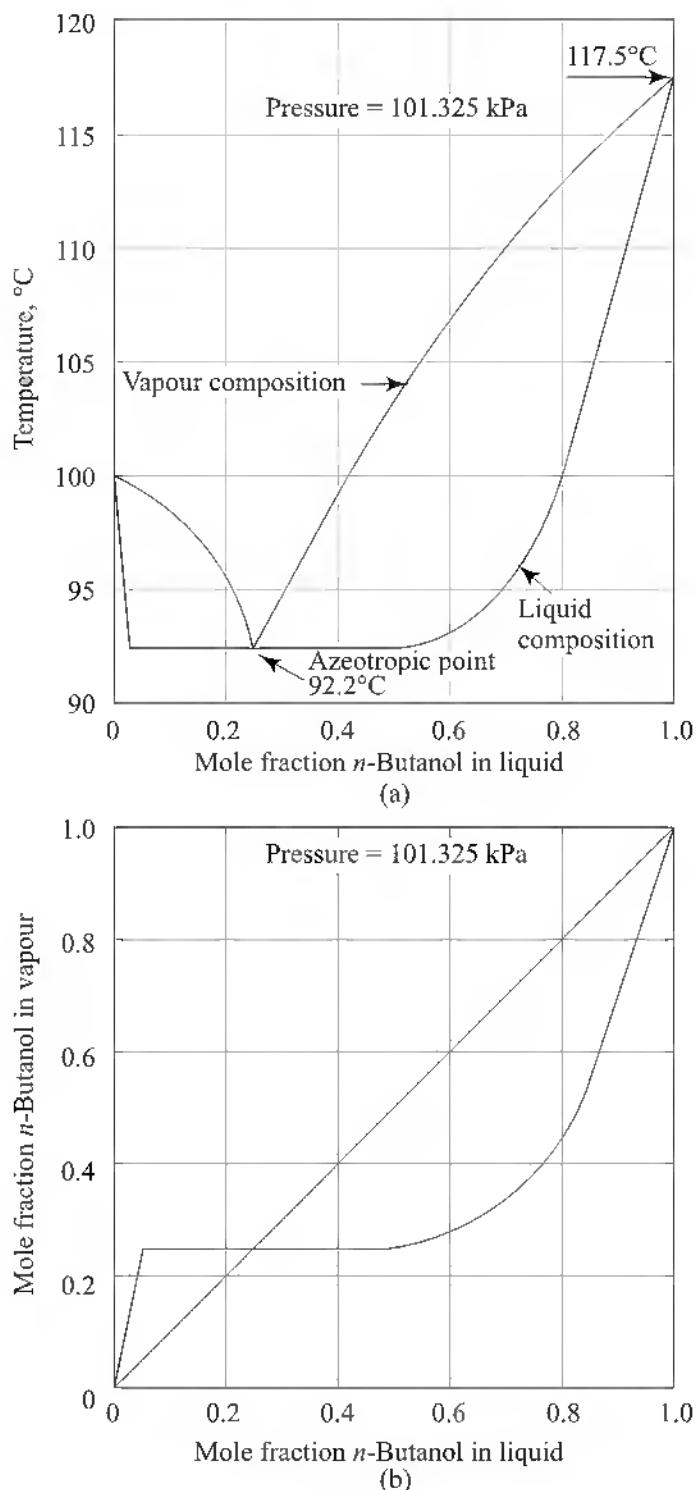


Fig. 8.44 (a) *t-x-y* Diagram for *n*-Butanol-Water System, (b) Heterogeneous Minimum Boiling Azeotrope of *n*-Butanol-Water System³⁰
(Reproduced with the permission of Chemical Engineering by Access Intelligence, USA)

The temperature at which an azeotrope is distilled is important. A minimum boiling azeotrope is one for which the boiling temperature is less than that of pure components. Figures 8.42(a) and (b) and Figures 8.44 (a) and (b) are $t-x-y$ and $y-x$ diagrams of minimum boiling azeotropes. For example, at 101.325 kPa, *n*-butanol and water boil at 117.7°C and 100°C, respectively but their azeotrope boils at 92.2°C. In case of maximum boiling azeotrope, the boiling point of the azeotrope is higher than that of the pure component. Chloroform and acetone boil at 61.2°C and 56.4°C, respectively at 101.325 kPa. The mixture of these two compounds forms a maximum boiling azeotrope at 64.5°C, containing 65.5% (by mole) chloroform. Figures 8.43(a) and (b) are $t-x-y$ and $y-x$ diagrams of the system, comprising chloroform and acetone. Maximum boiling azeotropes are usually homogeneous. About 90% of the known azeotropes are of the minimum variety.

Azeotropic data are well tabulated in literature. Perry's Chemical Engineers' Handbook tabulate data for binary and ternary azeotropes of common interest. Extensive 3-volume compilation by J. Gmehling (Ref. 8) cover practically all known azeotropes.

Property of some organic compounds to form azeotropes with water is advantageously used in many chemical reactions. Manufacture of amino resins involve etherification/esterification reactions. Water is a product of reaction. One of the reactants is *n*-butanol which forms a heterogenous azeotrope with water. While a batch of reaction is in progress, water is removed from the mass as an azeotrope. The vapours of azeotropic composition is condensed which separates into two liquid layers. Top *n*-butanol rich layer is recycled to the reactor and bottom aqueous layer is removed. This way one of the products of reaction is removed from the reaction mass, thereby reaction proceeds in forward direction.

In manufacture of alkyd resins also, water is a product of reaction. Toluene or xylene is added to the reaction mass and water is removed from the mass in form of a heterogeneous azeotrope. On condensation, two layers separate. Water is sparingly soluble in the hydrocarbon. Top hydrocarbon layer is recycled to the reactor.

In all esterification reactions, water is a byproduct. It is a common practice to remove water in the form of an azeotrope from top in a specially designed distillation column.

Removal of water in the form of an azeotrope has also expedited other operations, such as nitration of benzene and toluene. In this case, water formed in the nitration of the hydrocarbon is removed continuously, utilizing hydrocarbon itself as an entrainer. Contrary to semi-containuous operation, mixed acid, consisting of nitric acid and sulphuric acid, is used for nitration in a batch reactor. This batch process suffers from a drawback of disposal of spent acid or recovery of acids from the spent acid is a problem. Bottom still acts as a reaction vessel in which nitric acid and excess hydrocarbon are initially charged. Nitrated hydrocarbon has higher boiling point and thus remain in the still. Azeotrope of hydrocarbon and water goes to the bottom of the dehydrating column. Optimum temperatures and concentrations can readily be maintained in the system with simple

controls. Parameters, normally required to be controlled are feed rates of nitric acid and hydrocarbon, rate of supply of heat to respective heating units and rate of withdrawal of water at the top of the dehydrating column. Product is thus formed in single pass with almost 100% yield on both; nitric acid and hydrocarbon.

Similar azeotropic process is continuous production of acetamide. Continuous streams of ammonia and acetic acid are fed to a dehydrating column. Ammonium acetate, so formed is continuously dehydrated in azeotropic distillation system to give water and acetamide. Water, formed in both the reactions, goes overhead with the help of an entrainer. Acetamide with excess acetic acid flow out from the bottom of the column. This mixture is sent to second distillation column from which pure acetamide is withdrawn from the bottom. Acetic acid vapour from the column are recycled back to reaction cum azeotropic distillation column.

In general, following are the methods, used for separating azeotropic mixtures.

- (a) As mentioned earlier, azeotrope is dependent on operating pressure in most cases. If pressure is altered, it may be possible to avoid an azeotrope thereby fractional distillation can be carried out. For example, if distillation of ethanol–water is carried out below 70 torr, no azeotrope exists. However, this method is limited by many factors in actual practice. However, a few azeotropes do not change composition with pressure. For example, *n*-hexane forms an azeotrope with ethanol³⁰, containing 21.0% ethanol (by mass). It boils at 58.7°C at 101.325 kPa. However, the composition of this azeotropic mixture is unchanged over the pressure range of 20 torr to 10 000 torr.
- (b) A popular method is to add specially chosen chemical to shift the azeotropic point to a more favourable position. When this chemical (also known as entrainer) appears in appreciable amounts at the top of the column, the operation is called azeotropic distillation. When the additional component (called solvent) appears mostly at the bottom of the column, the operation is called extractive distillation.
- (c) In distillation using ionic salt, the salt dissociates in the liquid mixture and alters the relative volatilities sufficiently that the separation becomes possible like fractional distillation.
- (d) Pressure-swing distillation is yet another method where a series of columns, operating at different pressures, are used to separate binary azeotropes which change appreciably in composition over a moderate pressure range or where a separating agent which forms a pressure-sensitive azeotrope is added.
- (e) Use other techniques like pervaporation or adsorption for separating a component (such as water) from the azeotrope. These techniques are discussed in Sec. 8.8.3.
- (f) Reactive distillation can be considered where the separating agent reacts preferentially and reversibly with one of the azeotropic constituents. The reaction product is then distilled from the non-reacting components. Dilute alcohol solutions or dilute aqueous acetic acid solution can be treated by this method to form esters in reactive distillation units.

8.8.1 Azeotropic Distillation

Heterogeneous azeotropic distillation is a widely practiced process for dehydration of number of compounds such as acetic acid, chloroform, ethanol, acetonitrile, etc. The technique involves addition of a third component in a homogeneous azeotrope, called an entrainer to form a minimum boiling ternary azeotrope, carrying water overhead and leaves near dry (pure) product in the bottom. The overhead vapours are condensed to two liquid phases; entrainer rich phase is refluxed while the aqueous phase is decanted.

Dehydration of ethanol-water mixture is carried out with the help of benzene as an entrainer. Ethanol and water forms azeotrope having 95.6% ethanol (by mass) at 78.15°C and 101.325 kPa. When benzene is added to the azeotrope (about 0.427 kg/kg azeotrope) it comes as ternary azeotrope at the top containing 74.1% benzene and 18.5% ethanol (by mass). Bottom product from the column is pure dry ethanol. Top azeotrope at 65°C is condensed which forms two layers. Top layer contains 90.9% benzene by mass (and practically no water). It is refluxed to the column. Bottom aqueous layer contains 53% ethanol and 11% benzene (by mass). Since this layer contains appreciable content of benzene, it can be distilled in another column to recover it in the form of ternary azeotrope. Water is removed from the bottom of the third column. Thus three columns are normally required for this distillation.

Process design of an azeotropic distillation system for separation of ethanol-water using trichloroethylene is given by Coates³⁰ and using benzene is given by Norman³¹.

Another entrainer for ethanol dehydration is *n*-pentane which is eco-friendly. The tower is operated at near 3.45 bar a. Total condenser operates at 3 bar a and a ternary azeotrope, containing 82.64% *n*-pentane and 12.33% ethanol (by mole) is formed. Condensate separates in two layers, top layer contains 87.9% *n*-pentane and 11.6% ethanol (by mole) and is refluxed to the column. Bottom aqueous layer contains 23.8% ethanol (by mole) and practically no *n*-pentane. This layer is distilled in second column to recover ethanol as azeotrope with water as top product. In this case only two columns are required.

Another example of separation of homogeneous azeotrope is that of *iso*-propanol–water. It forms azeotrope at 80.4°C and 101.325 kPa. For their separation, cyclohexane or benzene is used as entrainer. Cho and Jeon³² have given process design of heterogeneous azeotropic distillation system for dehydration of *iso*-propyl alcohol using benzene as the entrainer.

When the binary azeotrope is heterogeneous, separation of the components are possible without addition of an entrainer. Design of *n*-butanol–water distillation system is given by McKetta and Cunningham³³ in detail.

8.8.2 Extractive Distillation

Extractive distillation can be defined as distillation in the presence of a miscible, high boiling relatively non-volatile component (called the solvent) that forms no azeotropes with other components of the mixture. Solvent is continuously added near the top of the column so that an appreciable amount is present in the liquid

phase throughout the column. The component having the greater volatility (not necessarily the lowest boiling point component) is taken overhead as a relatively pure distillate. The other component leaves with the solvent via the column bottoms. The solvent is then separated from the bottoms in second distillation column. Selection of an extractive distillation solvent is the most important step in developing a successful extractive distillation sequence. Important selection criteria are given below.

- (i) It should enhance the relative volatility of the key component significantly.
- (ii) Required solvent quantity to be added to the azeotropic mixture should not be excessive.
- (iii) It should be soluble in the feed components.
- (iv) It should be easily separable from the bottom product.
- (v) It should be relatively inexpensive and easily available.
- (vi) It should be stable at the operating conditions of both the columns.
- (vii) It should be non-reactive with the feed components.
- (viii) It should have low latent heat of vaporization.
- (ix) It should be non-corrosive and non-toxic.
- (x) It should not form immiscible liquid mixtures at any point in the column.

Perrys' Chemical Engineers' Handbook serves as a good reference for the solvent selection.

Cyclohexane is manufactured from benzene. The mixture from the reactor comes out with the desired product (cyclohexane) and also a significant amount of unreacted benzene which is to be recycled back to the reactor for economic reasons. Product mixture contains nearly 45% cyclohexane and balance benzene (by mole). It is desired to operate the distillation column at 150 kPa a as dictated by the process flow sheet. At this pressure, cyclohexane and benzene boil at 94.3°C and 93.5°C, respectively. Binary homogeneous azeotrope at this pressure is formed at 91°C, containing 45.5% cyclohexane (by mole) which is nearly the same composition of reactor exit product mix. In this case propylene glycol is selected as a solvent as benzene is highly soluble in the solvent. Further the solvent has high boiling point (200.4°C) at 150 kPa and no new azeotropes are formed. Distillate from the column is 99.3% pure cyclohexane. Bottoms from the first column is fed to second column from which distillate is 85% pure benzene which is recycled back to the reactor. Bottoms from the second column is nearly pure solvent which is recycled to the first column.

Apart from organic solvents, inorganic salts such as potassium and sodium acetates, calcium chloride, calcium nitrate, etc. can also be used to break the azeotropes.

Another well known example of extractive distillation is dehydration of nitric acid–water azeotrope (containing 64.1% HNO₃ by mass at 101.325 kPa and 122°C). In this case sulphuric acid is used as solvent.

Apart from above examples, extractive distillation finds many applications in the petroleum refinery. Pyrolysis gasoline byproduct from naphtha steam cracking contains a wealth of aromatics. Separation of aromatics, such as xylenes, is carried out by extractive distillation. Similarly, extractive distillation can be used

to selectively extract the sulphur species from cracked gasoline to very low levels without loosing its octane value.

8.8.3 Pressure Swing Distillation

Although known from good olden days, only now pressure swing distillation technique is gaining popularity for separation of a binary azeotrope over heterogeneous azeotropic distillation and extractive distillation. The chief reason for not finding many industrial applications till date was the lack of experimental VLE data for binary systems at different pressures. Two main advantages of pressure swing distillation are; energy saving by heat integration of the system and no need to add an entrainer or a solvent for the separation.

The technique is based on the principle that the composition of almost all azeotropes varies with the operating pressure. In industrially important water-solvent mixtures, the water content of the azeotrope increases with increasing pressure. This variation provides a technique for separation of the components. A shift in composition of azeotropic mixture with respect to pressure can be well understood by studying Figures 8.45 (a) and (b).

Acetonitrile—ACN (also called methyl cyanide; formula: C_2H_3N) forms a minimum boiling azeotrope with water at **1 atm and 77°C** containing 83.5% by mass or **69.0% by mole** ACN. This azeotrope composition shifts to **60.0% by mole** ACN at **3.02 bar a** which boils at **112.5°C**. Using these data a two-column system can be designed to operate at two different pressures to separate ACN and water from the mixture. Feed with lower concentration than 69.0 mole % ACN can be fed to the first column operating at 1 atm. Distillate from the column with near azeotropic composition is the feed to the second column operating at 3.02 bar a. Bottom products from the first and second column are water and ACN, respectively. Distillate from the second column is recycled to the first column along with the fresh feed.

An important point to note is that in heterogeneous azeotropic distillation, bottom product from the first column is the solvent (low boiling component) while in pressure swing distillation, bottom product is water (high boiling component).

In pressure swing distillation since both the columns operate at different pressures, their heat exchangers can be readily thermally integrated. This will result in substantial savings in energy for distillation. However, such an integration results in complexity of the system which requires adequate process control instrumentation. Also hazards of handling flammable (and toxic) solvents at high pressure must be given due considerations while designing such a system.

Both batch and continuous systems are developed. Jen-Uwe Repke et al^{35, 36} have developed mathematical models and process control systems for ACN-water mixture.

Knapp and Doherty³⁷ have listed more than 35 pressure sensitive binaries which can be separated by pressure swing distillation technique.

Ethanol-water is also a pressure sensitive azeotrope but its separation by pressure swing distillation method is considered an uneconomical proposition. For

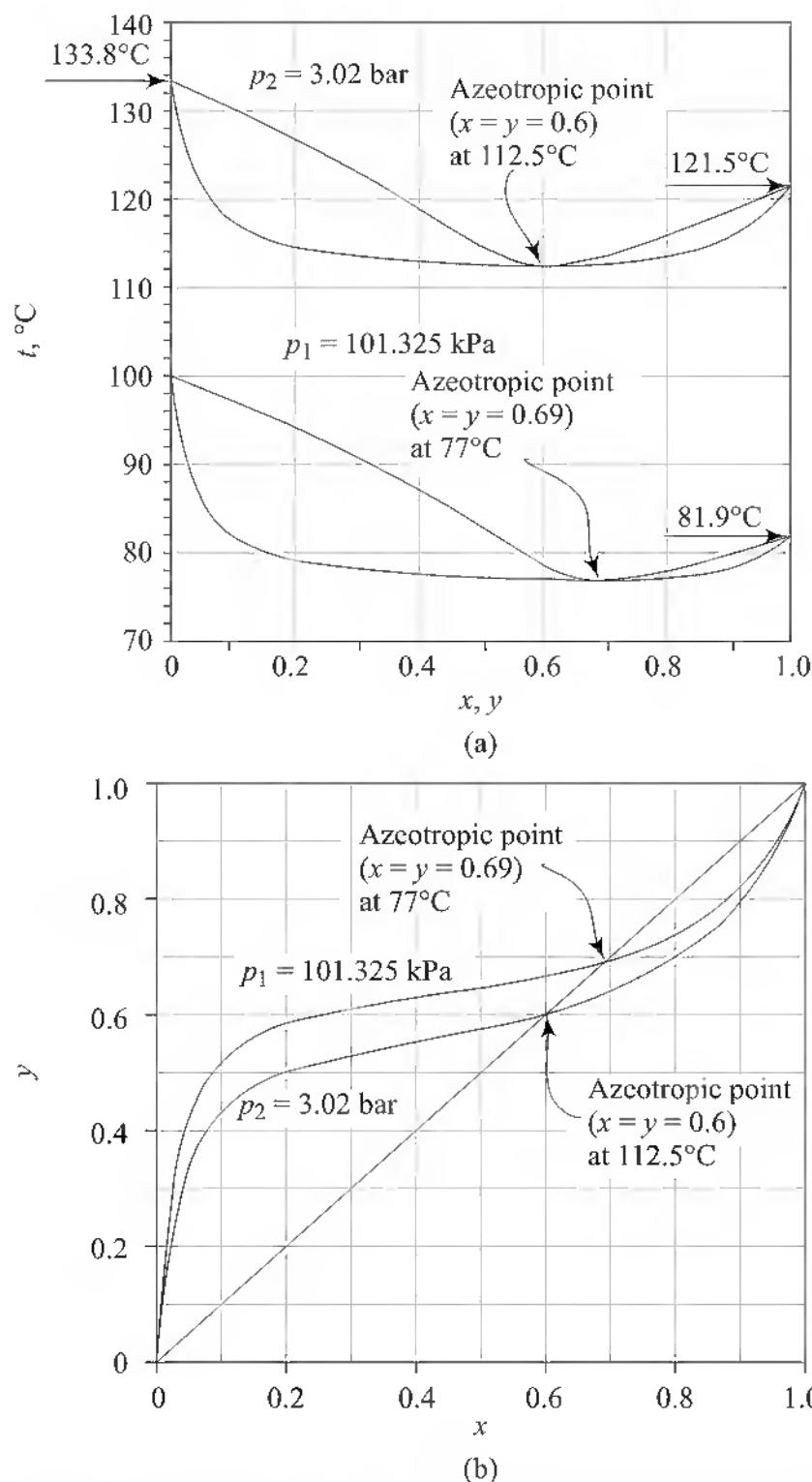


Fig. 8.45 (a) *t-x-y* Diagrams for Acetonitrile-Water System, (b) Homogeneous Minimum Boiling Azeotropes Acetonitrile-Water System³⁴
(Reproduced with the Permission of Dr. Repke)

separation of this azeotrope, Knapp and Doherty³⁷ have suggested to add small amount of acetone in the dilute alcohol stream from a fermentor. The entrainer does not form a ternary azeotrope at atmospheric pressure but forms a ternary azeotrope at high pressure (say at 10 atm). On this basis a scheme is presented to operate first column at 1 atm and second column at 10 atm. Resulting pressure sequence yields dry ethanol from the bottom of the second (HP) column. Also

thermal integration of both the columns result in nearly 53% energy saving over the conventional heterogeneous azeotropic distillation sequence.

Exercise 8.12 is yet another example of separation of tetrahydrofuran (THF) from aqueous THF solution using pressure swing distillation technique.

Example 8.16

Isopropyl alcohol (IPA) forms a minimum boiling azeotrope with water at 80.4°C and 101.325 kPa, having a composition of 31.47 mole % water. Vapour pressure of IPA can be calculated using Antoine equation¹³.

$$\log p_v = 6.8651 - \frac{1359.50}{(T - 75.65)}$$

where, p_v = Vapour pressure, kPa
 T = Saturation temperature, K

- (a) A feed consisting of 50 mass % IPA under saturated liquid conditions is to be distilled to give 67.5 mole % IPA as distillate (near azeotrope) and 0.1 mole % (0.0033 mass %) IPA in the bottoms. Using McCabe-Thiele technique and for $R = 3$, find the number of theoretical stages, required for the separation at 1 atmosphere and locate the feed introduction stage.
- (b) Make tray-to-tray calculations for the desired separation using van Laar constants.

Solution:

Azeotrope contains 0.3147 mole fraction (0.121 mass fraction) water. To find the non-ideal behaviour of the IPA-water system, use of van Laar equation can be made. Assuming vapour fugacity (i.e. vapour escaping tendency) to be unity at low pressure,

$$p \cdot y_i = x_i \gamma_i p_{vi} \quad (I)$$

where, p = Total pressure of the system = 1 atm = 760 torr
 y_i = Mole fraction of i th component in vapour
 x_i = Mole fraction of i th component in liquid
 γ_i = Activity coefficient of i th component
 p_{vi} = Vapour pressure of the i th component at the boiling point, torr

If x_1 and x_2 are mole fractions of the two components in a binary liquid mixture,

$$\ln \gamma_1 = A \left[\frac{x_2}{\left(\frac{A}{B} \right) x_1 + x_2} \right]^2 \quad (8.109)$$

$$\ln \gamma_2 = B \left[\frac{x_1}{\left(\frac{B}{A} \right) x_2 + x_1} \right]^2 \quad (8.110)$$

Equations (8.109) and (8.110) are called van Laar equations and A and B are called van Laar constants. Let 1 refer to IPA, the more volatile component and 2 refer to water. Boiling point of the azeotrope, $t = 80.4^\circ\text{C}$ or $T = 353.55 \text{ K}$.

$$\log p_{v1} = 6.8651 - \frac{1359.50}{(353.55 - 75.65)} = 1.97305$$

$$p_{v1} = 93.984 \text{ kPa} \text{ (vapour pressure of IPA at } 80.4^\circ\text{C}) = 704.94 \text{ torr}$$

From steam tables, at $t = 80.4^\circ\text{C}$, $p_{v2} = 48.14 \text{ kPa} = 361.08 \text{ torr}$
At the azeotropic point,

$$\gamma_1 = 760/704.94 = 1.0781$$

$$\gamma_2 = 760/361.08 = 2.1048$$

$$\ln(1.0781) = A \left[\frac{0.3147}{\left(\frac{A}{B}\right)0.6853 + 0.3147} \right]^2$$

$$\ln(2.1048) = B \left[\frac{0.6853}{\left(\frac{B}{A}\right)0.3147 + 0.6853} \right]^2$$

Solving the equations by Mathcad,

$$A = 2.31187 \text{ and } B = 1.10777$$

These constants are themselves functions of the liquid compositions but their variations can be ignored for all practical purposes (for low heats of mixing) at a given total pressure and varying temperature conditions. Using these constants, van Laar equations can be used to develop the vapour-liquid equilibrium curve.

Consider $x_1 = 0.6$ (of IPA) and $x_2 = 0.4$ (of water) and calculate activity coefficients.

$$\ln \gamma_1 = 0.13551 \text{ or } \gamma_1 = 1.14512$$

$$\ln \gamma_2 = 0.63631 \text{ or } \gamma_2 = 1.88949$$

Assume boiling point of the mixture to be 81°C (or 354.15 K),

$$p_{v1} = 96.292 \text{ kPa} = 722.25 \text{ torr}$$

$$p_{v2} = 49.311 \text{ kPa} = 369.86 \text{ torr}$$

$$\begin{aligned} p &= \sum x_i \cdot \gamma_i \cdot p_{vi} \\ &= (0.6 \times 722.25 \times 1.14512) + (0.4 \times 369.86 \times 1.88949) \\ &= 496.24 + 279.54 = 775.78 \text{ torr} \\ &\neq 760 \text{ torr} \end{aligned}$$

Since total pressure does not equal to 760 torr, assume new temperature equal to 80.5°C (353.65 K).

$$p_{v1} = 94.365 \text{ kPa} = 707.8 \text{ torr}$$

$$p_{v2} = 48.336 \text{ kPa} = 362.55 \text{ torr}$$

$$\begin{aligned} p &= (0.6 \times 707.8 \times 1.14512) + (0.4 \times 362.55 \times 1.88949) \\ &= 486.31 + 274.0 = 760.31 \\ &\approx 760 \text{ torr} \end{aligned}$$

Mole fraction of IPA in vapour (corresponding to x_1), $y_1 = x_1 \cdot \gamma_1 \cdot p_{v1}$
 $= 0.6 \times 707.8 \times 1.14512 / 760$
 $= 0.64$

In this manner, x - y data can be generated for the entire range which is tabulated in Table 8.45.

Table 8.45 Vapour-Liquid Equilibrium of IPA-Water at 1 atm

Mole fraction of IPA		Boiling point (<i>t</i>), °C
in Liquid <i>x</i>	in Vapour <i>y</i>	
0	0	100.0
0.02	0.2495	92.65
0.05	0.3993	87.4
0.075	0.4561	85.15
0.1	0.489	83.85
0.2	0.5371	82.0
0.3	0.5545	81.5
0.4	0.573	81.3
0.5	0.6	80.8
0.6	0.64	80.5
0.6853	0.6853	80.4 (azeotrope)
0.8	0.7682	80.6
0.9	0.87	81.15
0.95	0.93	81.65
1.0	1.0	82.5

Figure 8.46 is the plot of the vapour-liquid equilibrium of IPA-water system.

Feed (z_F) is marked at $x_1 = 0.2306$ (equivalent to 0.5 mass fraction) of IPA. Desired purities of distillate and bottoms are marked at $x_D = 0.675$ and $x_W = 0.001$. *q*-line is vertical as the feed is saturated liquid.

$$\frac{x_D}{(R_m + 1)} = 0.3537$$

$$R_m = 0.9084$$

In case of azeotropic distillation, other methods are available in literature³⁷ for calculation of minimum reflux ratio which are more reliable. Also actual reflux ratio is kept much higher than the minimum.

For $R = 3$

$$\frac{x_D}{(R + 1)} = \frac{0.675}{(3 + 1)} = 0.1688$$

For $R = 3$, from, Fig. 8.46, total 10 theoretical stages are required for the separation and feed will be introduced at 3rd stage from bottom.

(b) Tray-to tray Calculations:

Azeotropic distillation column design of IPA-water system can be carried out by numerical method; i.e. tray-to-tray calculations. Average latent heats of vaporization of IPA and water are 40 093 and 41 081 kJ/kmol, respectively. Activity coefficients for each tray liquid mixture will be calculated using same van Laar constants, used for developing Table 8.45.

Basis: 100 kmol/h of feed consisting of 0.2306 mole fraction IPA.

With distillate, containing 0.675 mole fraction IPA and bottoms, containing 0.001 mole fraction IPA, material balance yields

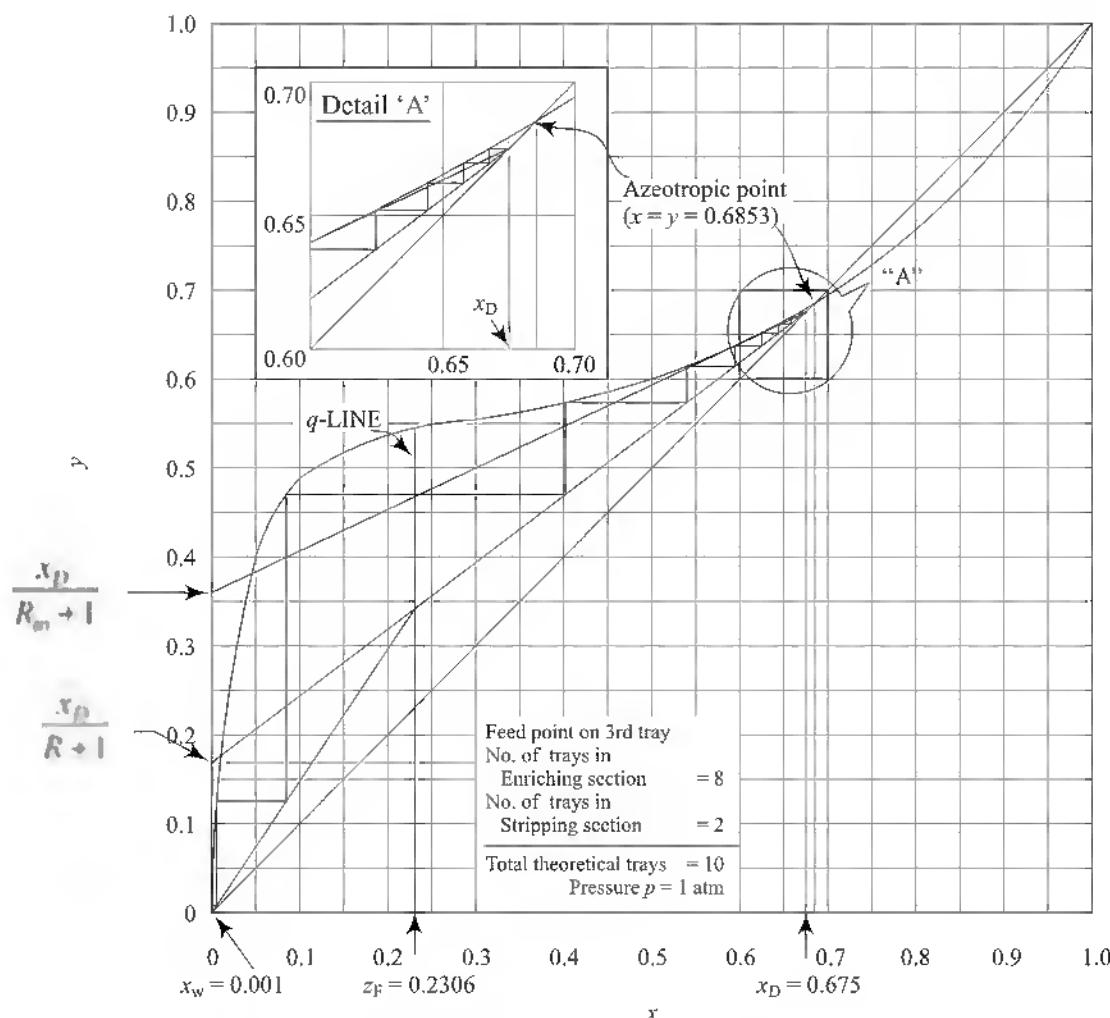


Fig. 8.46 Azeotropic Distillation of Aqueous Isopropyl Alcohol

$$D = 34.065 \text{ kmol/h and}$$

$$W = 65.935 \text{ kmol/h}$$

$$\text{Reflux } R = 3D = 3 \times 65.935 = 102.195 \text{ kmol/h}$$

$$\begin{aligned} \text{Liquid flow in column } \bar{L} &= R + D \\ &= 102.195 + 34.065 \\ &= 136.26 \text{ kmol/h} \end{aligned}$$

Vapour from the bottom most tray (No. 1),

$$\begin{aligned} \bar{V}_1 &= \bar{L} - W \\ &= 136.26 - 65.935 \\ &= 70.325 \text{ kmol/h} \end{aligned}$$

$$\text{For } x_1 = 0.001 \text{ (IPA) and } x_2 = 0.999 \text{ (water)}$$

$$\gamma_1 = 9.996\ 57 \text{ and } \gamma_2 = 1.000\ 00$$

At $t_1 = 99.46^\circ\text{C}$, $y_1 = 0.0191$ and $y_2 = 0.9809$ (Refer Table 8.46)

Latent heat of vapour leaving Tray-1

$$\begin{aligned} \lambda_1 &= 0.0191 \times 40\ 093 + 0.9809 \times 41\ 081 \\ &= 41\ 062.3 \text{ kJ/kmol} \end{aligned}$$

Table 8.46 Material Balance of Tray-1 (from Bottom)

Component	Bottom product, W kmol/h	Vapour, \bar{V}_1		Liquid flow from Tray-2, L_2 kmol/h	
		mol. fr.	kmol/h	kmol/h	mol. fr.
IPA	0.066	0.0191	1.343	1.409	0.0103
Water	65.869	0.9809	68.982	134.851	0.9897
Total	65.965	1.0000	70.325	136.260	1.0000

With $x_1 = 0.0103$ and $x_2 = 0.9897$

$$\gamma_1 = 9.157\ 97 \text{ and } \gamma_2 = 1.000\ 50$$

At $t_2 = 95.54^\circ\text{C}$, $y_1 = 0.1565$ and $y_2 = 0.8435$

$$\begin{aligned}\lambda_2 &= 0.1565 \times 40\ 093 + 0.8435 \times 41\ 0810 \\ &= 40\ 926.5 \text{ kJ/kmol}\end{aligned}$$

Adjust vapour flow from tray-2 with molar latent heats of vapour from tray-1 and tray-2.

$$\begin{aligned}\bar{V}_2 &= 70.325 \times 41\ 062.3 / 40\ 926.5 \\ &= 70.558 \text{ kmol/h}\end{aligned}$$

Table 8.47 Material Balance of Tray-2 (from Bottom)

Component	\bar{L}_2 kmol/h	\bar{V}_2 mol. fr.	\bar{V}_1		\bar{L}_3	
			kmol/h	kmol/h	kmol/h	mol. fr.
IPA	1.409	0.1565	11.042	1.343	11.108	0.0814
Water	134.851	0.8435	59.516	68.982	125.385	0.9186
Total	136.260	1.0000	70.558	70.325	136.493	1.0000

With $x_1 = 0.0814$ and $x_2 = 0.9186$

$$\gamma_1 = 5.189\ 08 \text{ and } \gamma_2 = 1.027\ 35$$

At $t_3 = 84.75^\circ\text{C}$, $y_1 = 0.4661$ and $y_2 = 0.5339$

$$\lambda_3 = 40622.5 \text{ and } \bar{V}_3 = 71.086 \text{ kmol/h}$$

Table 8.48 Material Balance of Tray-3 (without Fresh Feed)

Component	\bar{L}_3 kmol/h	\bar{V}_3 mol. fr.	\bar{V}_2		\bar{L}_4	
			kmol/h	kmol/h	kmol/h	mol. fr.
IPA	11.108	0.4661	33.133	11.042	33.199	0.2423
Water	125.385	0.5339	37.953	59.516	103.822	0.7577
Total	136.493	1.0000	71.086	70.558	137.021	1.0000

Composition of liquid mixture on tray-4 is 0.2423 mole fraction IPA which is higher than mole fraction 0.2306 of IPA in the fresh feed. Hence, feed is to be introduced on 3rd tray.

Table 8.49 Liquid Composition on Tray-4 after Addition of Fresh Feed on Tray-3

Component	\bar{L}_4	F	L	
	kmol/h	kmol/h	kmol/h	mol. fr.
IPA	33.199	23.06	10.139	0.2739
Water	103.822	76.94	26.882	0.7261
Total	137.021	100.00	37.021	1.0000

Tray-to-tray calculations for the rectification section can be carried out in analogous manner to the stripping section. These are summarised in Table 8.50.

Example 8.17

Refer Table 8.45 in Example 8.16. It can be seen that boiling point of 10 mole % IPA mixture is 83.85°C. This indicates that boiling point variation of only 1.35°C takes place from 0.1 mole fraction to the azeotropic point. Thus if the column is operated to separate up to 0.1 mole fraction, the column operation is nearly isothermal. In the overhead condenser, cooling medium at 72°C (max.) can be used while in the reboiler, heating medium at 95°C should be adequate.

For the above reason it is decided to operate the main column in the range of 0.1 mole fraction to 0.675 mole fraction IPA. It will be provided its own reboiler as shown in Fig. 8.47.

Liquid mixture, containing 0.1 mole fraction, is fed to another column in which open steam will be used for stripping to achieve 0.1 mole % (0.0033 mass %) IPA in the bottoms. Vapour from this column will be fed to the main column to supplement the heat.

For the new arrangement, rework the number of theoretical stages, required for both the columns for $R = 3$.

Solution:

Note: It can be noted that cooling medium at 72°C is required in the overhead condenser while heating medium at 95°C is required in the reboiler. This suggests water as an auxiliary heat transfer medium in the heat pump (refer Sec. 8.8.1(f)). Saturation pressure of water at 72°C is 0.34 bar a and at 95°C is 0.845 bar a. Compression ratio across the turbo-compressor will be just 2.485.

In the second column, since live steam is to be injected, its temperature can be just above 100°C. This permits use of flash steam at low pressure (may be at 105°C or 1.21 bar a) as live steam.

Use of live steam at low pressure and water as the auxiliary heat transfer medium in the heat pump will make distillation quite energy efficient. Thermal energy requirement will be considerably reduced with this arrangement.

From Fig. 8.46 number of theoretical stages required for the separation up to 10 mole %. IPA from 23.06% mole IPA (feed) $N = 8$.

Second column is isothermal steam stripper. In stripping liquid, solution is contacted with vapour or gas in a counter current manner to strip off the most volatile component of liquid solution. Most volatile component of liquid solution is transferred to gas or vapour. Stripping is a reverse of gas absorption. For stripping, low pressure and high temperature are favourable.

Table 8.50 Summary of Tray-to-Tray Calculations

Tray No.	Temperature, °C	Liquid flow rate, kmol/h	Vapour flow rate, kmol/h	Average molar latent heat of vaporization kJ/kmol	Liquid composition		Activity coefficients		Vapour composition	
					$x_1(\text{IPA})$	$x_2(\text{H}_2\text{O})$	$\gamma_1(\text{IPA})$	$\gamma_2(\text{H}_2\text{O})$	$y_1(\text{IPA})$	$y_2(\text{H}_2\text{O})$
Stripping Section:										
1	99.46	65.965	70.325	41 062.3	0.0010	0.9990	9.99657	1.00000	0.0191	0.9809
2	95.54	136.260	70.558	40 926.5	0.0103	0.9897	9.15797	1.00005	0.1565	0.8435
Fresh feed Satd.		100.000	—	—	0.2306	0.7694	—	—	—	—
Rectification Section:										
3	84.75	136.493	71.086	40 622.5	0.0814	0.9186	5.18908	1.02735	0.4661	0.5339
Fresh feed Satd.		100.000	—	—	0.2306	0.7694	—	—	—	—
Rectification Section:										
4	81.62	37.021	71.237	40 536.7	0.2739	0.7261	2.06218	1.23978	0.5503	0.4497
5	80.99	37.172	71.287	40 508.0	0.4360	0.5640	1.40286	1.52530	0.5810	0.4190
6	80.77	37.222	71.317	40 490.9	0.4950	0.5050	1.28304	1.6483	0.5981	0.4019
7	80.67	37.252	71.337	40 479.0	0.5278	0.4722	1.23140	1.72067	0.6093	0.3907
8	80.60	37.272	71.349	40 472.2	0.5493	0.4507	1.20216	1.76960	0.6175	0.3825
9	80.56	37.284	71.359	40 466.6	0.5650	0.4350	1.18282	1.80606	0.6239	0.3761
10	80.53	37.294	71.367	40 462.0	0.5772	0.4228	1.16888	1.83481	0.6291	0.3709
11	80.51	37.302	71.375	40 457.6	0.5872	0.4128	1.15810	1.85864	0.6335	0.3665
12	80.49	37.310	71.385	40 452.1	0.5956	0.4044	1.14948	1.87884	0.6373	0.3627
13	80.48	37.320	71.385	40 451.1	0.6029	0.3971	1.14230	1.89654	0.6407	0.3593
14	80.46	37.320	71.392	40 448.1	0.6094	0.3906	1.13614	1.91240	0.6438	0.3562
15	80.45	37.327	71.402	40 442.3	0.6153	0.3847	1.13072	1.92688	0.6467	0.3533

(Contd.)

Table 8.50 (Contd.)

Tray No.	Temperature, °C	Liquid flow rate, kmol/h	Vapour flow rate, kmol/h	Average molar latent heat of vaporization kJ/kmol	Liquid composition		Activity coefficients		Vapour composition	
					$x_1(\text{IPA})$	$x_2(\text{H}_2\text{O})$	$\gamma_1(\text{IPA})$	$\gamma_2(\text{H}_2\text{O})$	$y_1(\text{IPA})$	$y_2(\text{H}_2\text{O})$
1	80.44	37.337	71.404	40 441.2	0.6209	0.3791	0.12574	1.94070	0.6495	0.3505
16	80.44	37.339	71.808	40 438.4	0.6262	0.3738	1.12116	1.95385	0.6521	0.3479
17	40.44	37.743	71.813	40 435.7	0.6314	0.3686	1.11690	1.96650	0.6547	0.3453
18	80.42	37.748	71.820	40 431.5	0.6364	0.3636	1.11270	1.97934	0.6574	0.3426
19	80.42	37.755	71.824	40 429.4	0.6415	0.3585	1.10865	0.99217	0.6601	0.3399
20	80.42	37.759	71.828	40 427.2	0.6467	0.3533	1.10463	2.00532	0.6629	0.3371
21	80.41	37.763	71.834	40 424.0	0.6520	0.3480	1.10064	2.01878	0.6658	0.3312
22	80.40	37.769	71.840	40 420.6	0.6575	0.3425	1.09663	2.03282	0.6688	0.3312
23	80.40	37.775	71.845	40 417.6	0.6632	0.3368	1.09260	2.04744	0.6720	0.3280
24	80.40	37.780		0.6693	0.3307	1.08841	2.06316	0.6755	0.3245	
25	80.39									

Note: It may be noted that in this method, number of theoretical trays are worked out to be 25 with fresh feed entry on 3rd tray from the bottom. This number is much larger than that obtained with McCabe-Thiele diagram. It is therefore advisable to carry out tray-to-tray calculations for design of an azeotropic column with non-ideal behaviour.

With distillate flow of 65.935 kmol/h, difference in calculated L_{25} and that of reflux (34.065 kmol/h) is about 3 kmol/h. This is due to not carrying out actual material and energy balances on each tray.

This type of repetitive calculation can be conveniently carried out on a spread sheet (such as Excel®) on the computer.

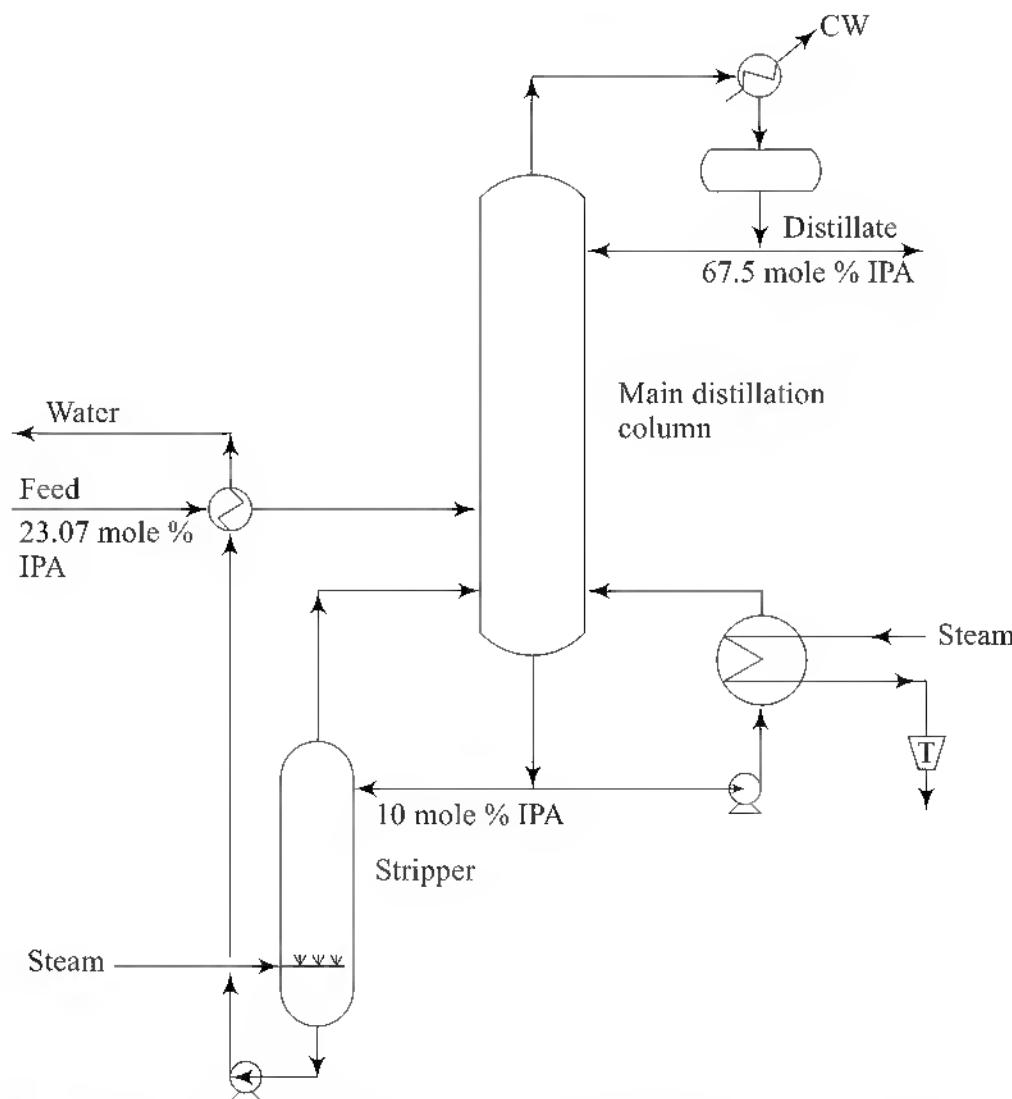


Fig. 8.47 Azeotropic Distillation of Aqueous Isopropyl Alcohol (Conventional System)

Industrial strippers are of two types.

- In first type of stripper, hot gas/steam (can be called stripping agent) is introduced at the bottom of stripper and the same is obtained from external source.
- In second type, hot vapour is generated by partial vaporization of liquid solution in reboiler at bottom. This type of stripper is like a bottom section (stripping section) of distillation column. Feed solution is introduced from top and reboiler is provided at the bottom. Reflux is not provided at the top. If the component to be separated from feed is having very high relative volatility, then this type of stripper can be selected.

In this example first type of stripper is selected. Stripping can be assumed as isothermal at 100°C.

Let, G_s = Molar flow rate of steam, kmol/h

L_s = Molar flow rate of water of feed, kmol/h

$$Y = \frac{y}{1-y} = \frac{\text{moles of IPA in vapour phase}}{\text{moles of steam in vapour phase}}$$

$$X = \frac{x}{1-x} = \frac{\text{moles of IPA in liquid phase}}{\text{moles of water in liquid phase}}$$

Equilibrium data can be approximated from Table 8.45.

x	y	X	Y
0	0	0	0
0.02	0.2495	0.0204	0.3324
0.05	0.3993	0.0526	0.6647
0.075	0.4561	0.081	0.8385
0.1	0.489	0.1111	0.957

Equilibrium curve is plotted in Fig. 8.48.

Minimum amount of steam required for desired separation can be obtained by following equation.

$$G_{sm}(Y_{1m} - Y_2) = L_s(X_1 - X_2) \quad (8.111)$$

where Y_1 and X_1 represent the molar ratios at top of the stripper and Y_2, X_2 represent the molar ratios at the bottom of stripper.

Here equilibrium curve is concave upward, hence Y_{1m} is equilibrium value of Y corresponding to the value of X_F .

$$x_F = 0.1, X_F = \frac{x_F}{1-x_F} = \frac{0.1}{1-0.1} = 0.1111$$

$$Y_{1m} = 0.957 \text{ (From Fig. 8.48)}$$

$$Y_2 = 0, X_1 = X_F = 0.1111, X_2 = \frac{0.001}{1-0.001} \cong 0.001$$

$$G_{sm} = \frac{L_s(X_F - X_2)}{Y_{1m} - Y_2}$$

$$\text{or } \left(\frac{G_s}{L_s} \right)_m = \frac{0.1111 - 0.001}{0.957 - 0} = 0.115$$

Let actual amount of steam, $G_s = 1.5 G_{sm}$

$$\frac{G_s}{L_s} = 1.5 \times 0.115 = 0.1725 = \frac{X_F - X_2}{Y_1 - Y_2}$$

$$0.1725 = \frac{0.1111 - 0.001}{Y_1 - 0}$$

$$Y_1 = 0.6383$$

Draw the operating line starting from (X_2, Y_2) to (X_1, Y_1) . Staircase construction between equilibrium curve and operating line, starting from (X_1, Y_1) to (X_2, Y_2) gives number of theoretical stages required for the desired separation. From Fig. 8.48, no. of theoretical stages required in the steam stripper; $N = 6$.

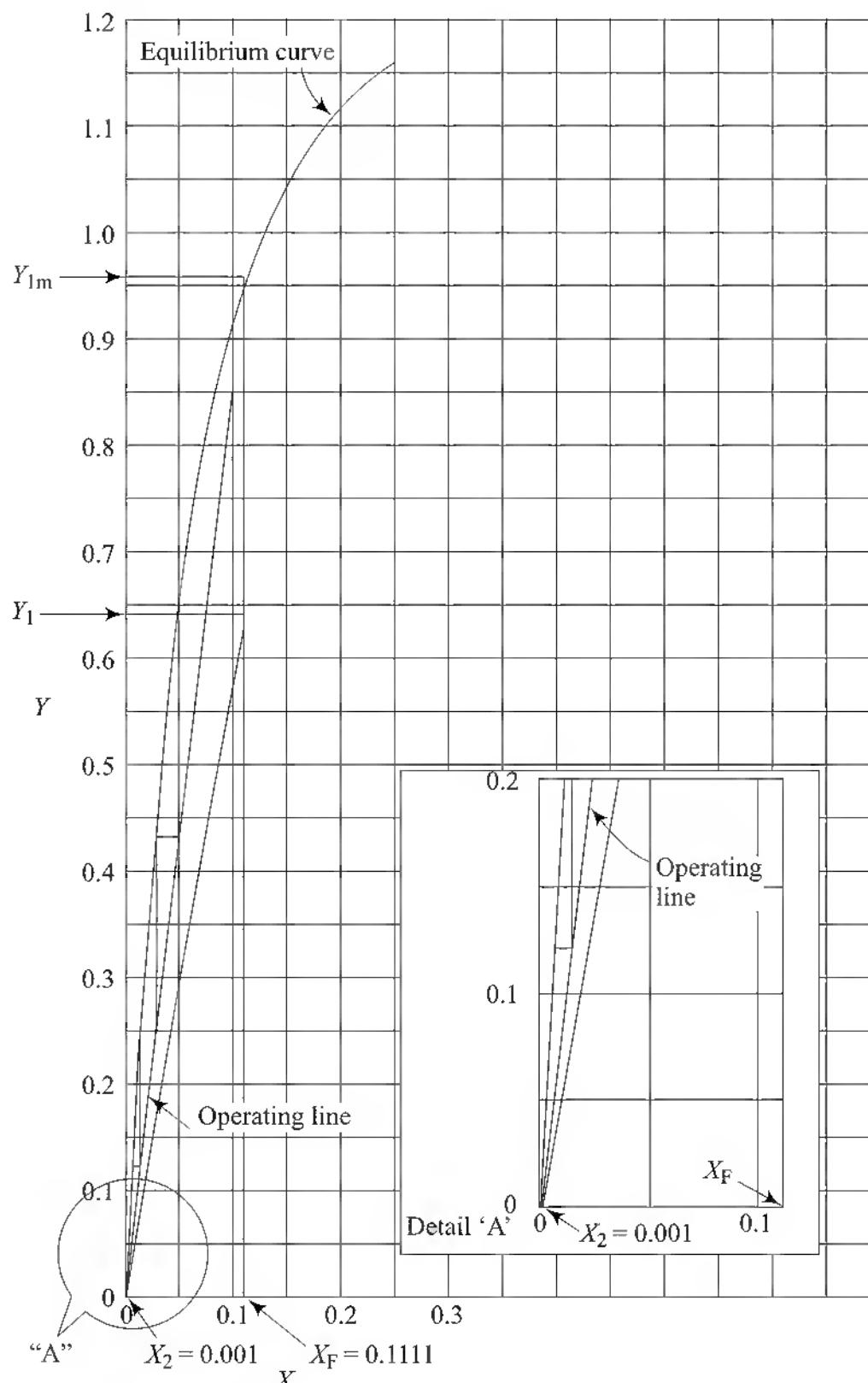


Fig. 8.48 Stripping of Dilute IPA with Live Steam

Example 8.18

Ethanol forms a minimum boiling azeotrope at 78.15°C and 101.325 kPa, with water having composition of 10.57 mole % water. Vapour pressure of ethanol can be calculated using Antoine equation¹³.

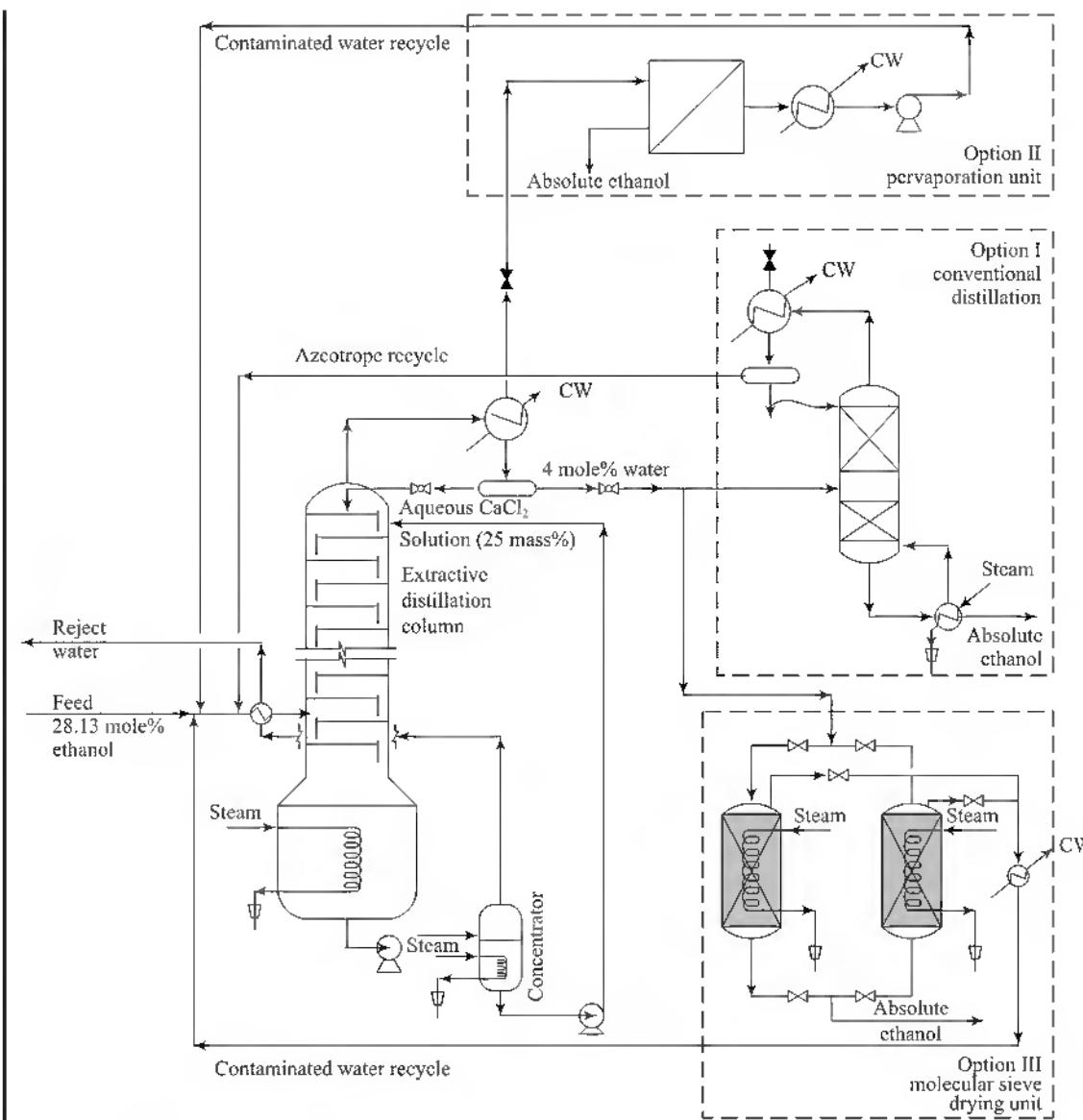


Fig. 8.49 Extractive Distillation of Aqueous Ethanol with Aqueous Calcium Chloride Solution (25 mass %)

$$\log p_v = 7.2371 - \frac{1592.90}{(T - 46.95)}$$

where, p_v = Vapour pressure, kPa
 T = Saturation temperature, K

Extractive distillation technique is known for separation of an azeotrope using a solvent. In a newly developed process, aqueous inorganic salt solution is used as a solvent to break the azeotrope. Lowering of vapour pressure is experienced when an inorganic salt is dissolved in water. Vapour pressures of aqueous calcium chloride solutions are given in Table 8.51.

It is proposed to use aqueous 25% CaCl_2 solution (by mass) as a solvent for dehydration of aqueous ethanol. Proposed flow diagram is shown in Fig. 8.49.

Plot x-y diagrams of ethanol-water system at 101.325 kPa, with and without the solvent. Superimpose both the diagrams and study the effect of addition of aqueous CaCl_2 solution on the separation.

Table 8.51 Vapour Pressure of Water over Aqueous Calcium Chloride Solution³⁹

Temperature, °C	Calculated vapour pressure of water, torr			
	Strength of CaCl_2 Solution, mass %			
	0	20	25	35
40	55.3	47.9	41.5	30.1
70	233.7	197.7	179.6	136.4
90	525.9	448.4	408.7	318.0
100	760.0	660.6	593.9	466.1
110	1074.6	905.9	841.9	629.5
120	1489.2	—	—	886.6

- (a) A feed consisting of 50 mass % ethanol under saturated liquid conditions is to be distilled to give 99.8 mole % (99.92 mass %) ethanol as distillate and 0.1 mole % ethanol in bottoms in the extractive distillation column. Using McCabe-Thiele technique and for $R = 2 R_m$, find the number of theoretical stages required for the separation at 101.325 kPa and locate the feed stage.
- (b) If the distillate quality is restricted to 96.0 mole % (98.4 mass %) ethanol for the case (a), find the number of theoretical stages required for the separation and locate the feed stage.

Solution:

Azeotrope contains 0.1057 mole fraction (0.0442 mass fraction) ethanol. Let suffix 1 represent ethanol and suffix 2 represent water.

At the boiling point of the azeotrope, $t = 78.15^\circ\text{C}$ or $T = 351.3 \text{ K}$,

$$\log p_{v1} = 7.2371 - \frac{1592.90}{(T - 46.95)} = 2.0033$$

$$p_{v1} = 100.763 \text{ kPa} = 755.78 \text{ torr}$$

From steam tables, $p_{v2} = 43.93 \text{ kPa} = 329.50 \text{ torr}$

$$\gamma_1 = 760/755.78 = 1.0056$$

$$\gamma_2 = 760/329.50 = 2.3065$$

van Laar equations:

$$\ln(1.0056) = A \left[\frac{0.1057}{\left(\frac{A}{B} \right) 0.8943 + 0.1057} \right]^2$$

$$\ln(2.3065) = B \left[\frac{0.8943}{\left(\frac{B}{A} \right) 0.1057 + 0.8943} \right]^2$$

Solving by Mathcad,

$$A = 1.95034 \text{ and } B = 0.9329$$

Using values of A and B , t - x - y data can be generated in an analogous manner to the IPA-water system (Example 8.14). When use of aqueous CaCl_2 solution (25 mass %) is considered, p_{v2} will be replaced by p_{v2}' , i.e. vapour pressure of water over the aqueous

solution. For this purpose a graph of p_{v2}' vs temperature can be plotted and p_{v2}' values are read at the required temperature.

Sample calculations for $x_1 = 0.9$ and $x_2 = 0.1$:

$$\gamma_1 = 1.00498 \text{ and } \gamma_2 = 2.31896$$

Assume $t = 78.2^\circ\text{C}$ or $T = 351.35 \text{ K}$.

$$p_{v1} = 757.22 \text{ torr}$$

$$p_{v2}' = 254.6 \text{ torr against } p_{v2} = 330.15 \text{ torr}$$

$$\begin{aligned} p &= \sum x_i \cdot \gamma_i \cdot p_{vi} \\ &= (0.9 \times 757.22 \times 1.00498) + (0.1 \times 254.6 \times 2.31896) \\ &= 684.89 + 59.04 = 743.93 \text{ torr} \neq 760 \text{ torr} \end{aligned}$$

Revise $t = 78.8^\circ\text{C}$ or $T = 351.9 \text{ K}$.

$$p_{v1} = 775.49 \text{ torr}$$

$$p_{v2}' = 260.8 \text{ torr}$$

$$p = 761.9 \text{ torr} \approx 760 \text{ torr}$$

$$y_1 = (0.9 \times 775.49 \times 1.00498)/760 = 0.923$$

Similarly t - x - y data are generated for ethanol-water-solvent system. Both t - x - y data are presented in Table 8.52 and Fig. 8.50(a).

Table 8.52 Vapour-Liquid Equilibrium for Ethanol-Water System at 1 atm

Without solvent, mole fraction ethanol			With solvent, mole fraction ethanol		
In Liquid x	In Vapour y	Boiling point, $^\circ\text{C}$ t	In Liquid x	In Vapour y	Boiling point, $^\circ\text{C}$ t
0	0	100.0	0	0	107.0
0.025	0.2503	92.8	0.025	0.298	97.7
0.05	0.365	88.8	0.05	0.4234	92.95
0.1	0.468	85.0	0.1	0.531	88.35
0.2	0.542	82.3	0.2	0.6026	85.1
0.3	0.578	81.2	0.3	0.638	83.75
0.4	0.6096	80.35	0.4	0.668	82.7
0.5	0.647	79.65	0.5	0.703	81.8
0.6	0.693	79.05	0.6	0.745	80.9
0.7	0.75	78.6	0.7	0.7945	80.05
0.8	0.817	78.25	0.8	0.8513	79.3
0.8943	0.8943	78.15 - azeotrope	—	—	—
0.96	0.953	78.1	0.9	0.921	78.8
1.0	1.0	78.1	1.0	1.0	78.1

Case (a):

Refer Fig. 8.50(b), $x_D = 0.998$, $z_F = 0.2811$ (or 0.5 mass fraction) and $x_W = 0.001$

$$\frac{x_D}{R_m + 1} = 0.2005$$

$$R_m = 3.98$$

$$R = 2 R_m = 2 \times 3.98 = 7.96$$

$$\frac{x_D}{R + 1} = 0.1114$$

Total 29 theoretical stages are required for the separation and the feed will be introduced on 14th stage.

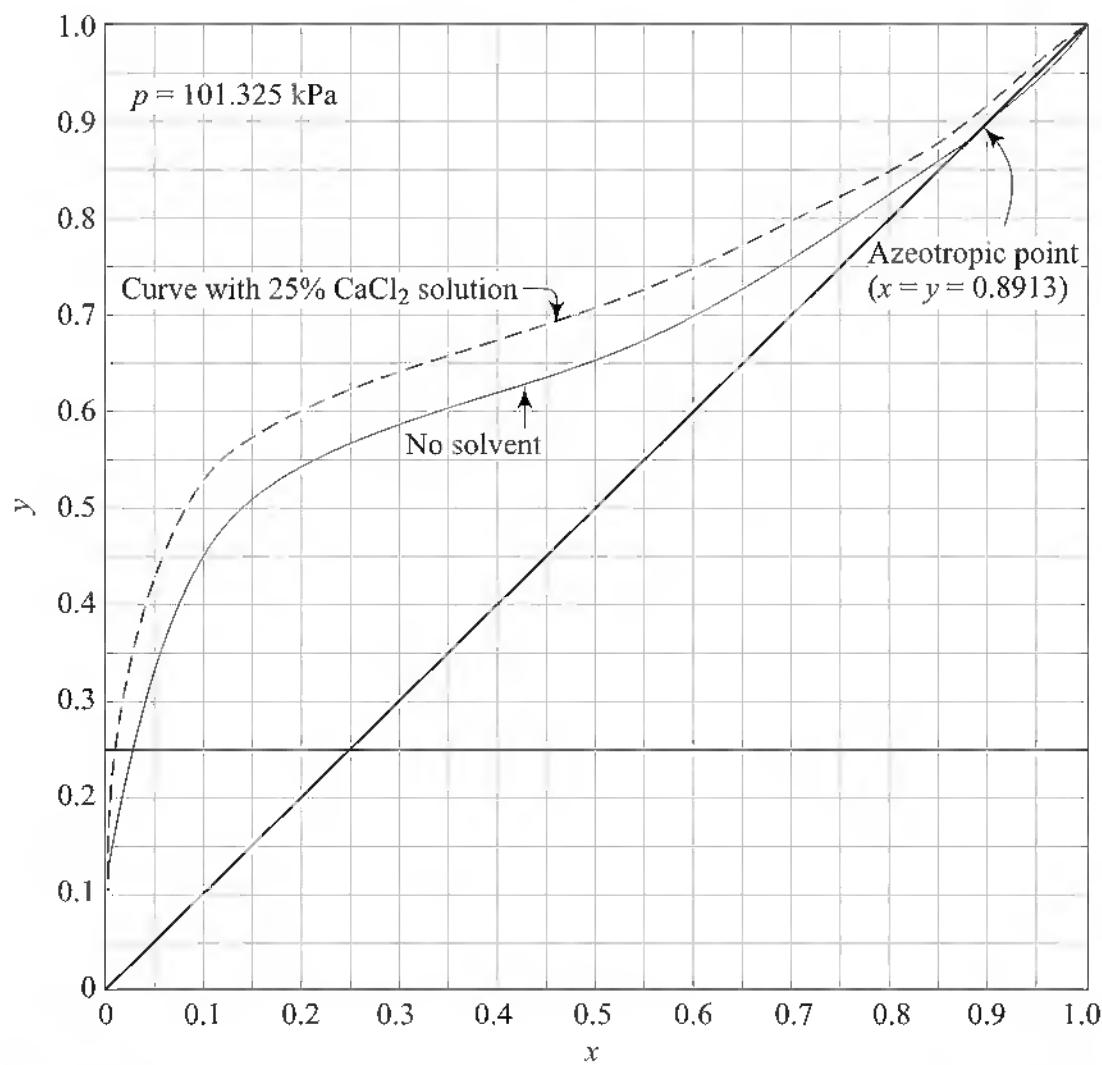


Fig. 8.50(a) Vapour Liquid Equilibrium Curves for Ethanol Water System with and without Aqueous Calcium Chloride Solution (25 mass%)

Case (b):

For $x_D = 0.96$, the graph [similar to Fig. 8.50(c)] was redrawn and following data were collected.

$$\begin{aligned}\frac{x_D}{R_m + 1} &= 0.2701 \\ R_m &= 2.554 \\ R &= 2 \times 2.554 = 5.108 \\ \frac{x_D}{R + 1} &= 0.1572\end{aligned}$$

With these value of R , number of theoretical stages were calculated to be 17 and the feed will have to be introduced on 14th stage.

Required number of theoretical stages for both the cases were found using McCabe-Thiele diagrams [Figs 8.50(b) and (c)]. However, it is recommended to recalculate the requirement by tray-to-tray calculations for actual process design.

Note: It may be noted that single stage extractive distillation (ED) column is adequate to produce ethanol with 99.8 mole % purity while in most ED systems, two columns are required for the same degree of separation. Boiling point elevation at atmospheric

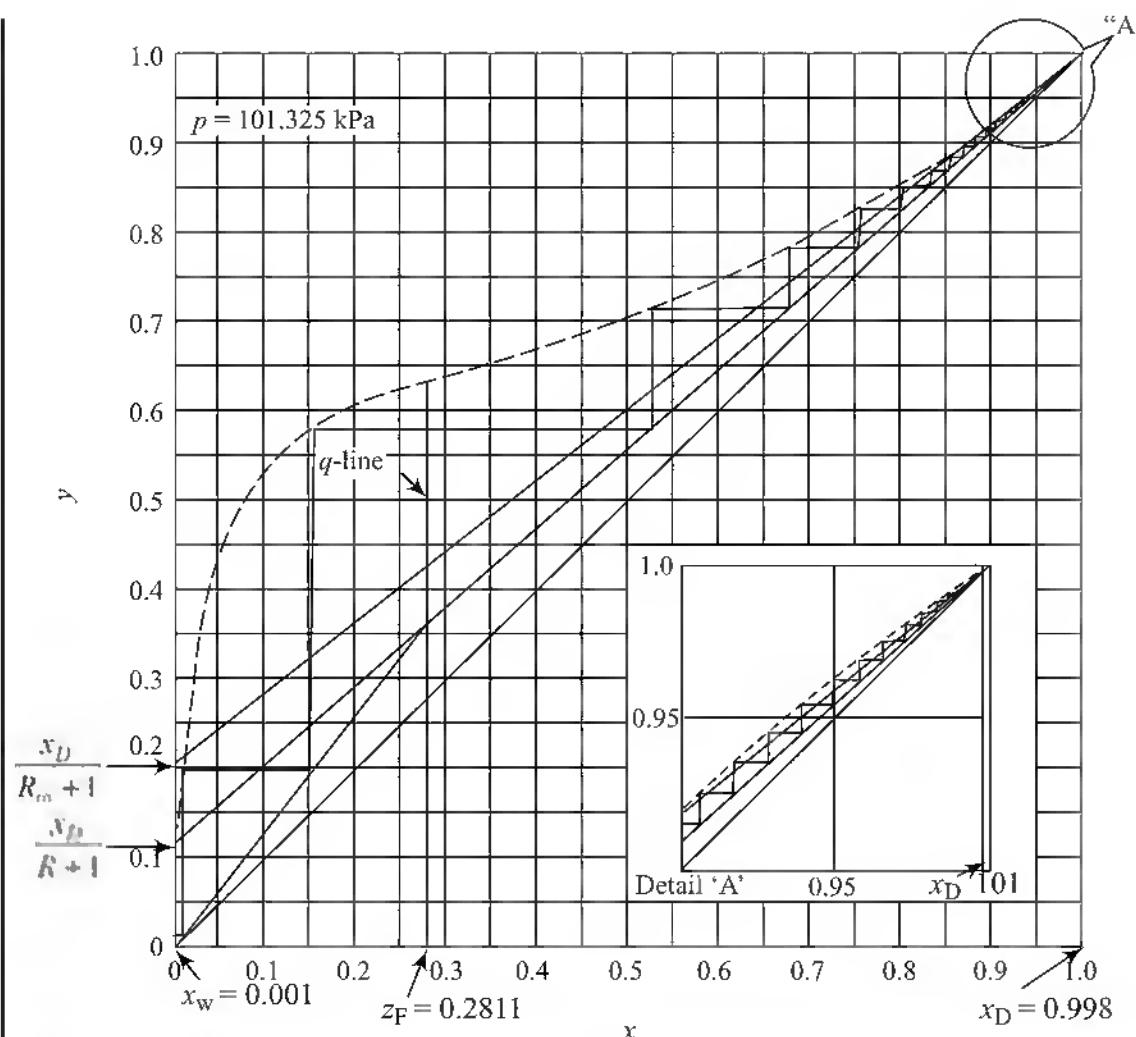


Fig. 8.50(b) Extractive Distillation with Aqueous Calcium Chloride Solution (25 mass %)

pressure (at the bottom of the column) is just 7°C. Also this ED system calls for 50% or less energy than the conventional ED columns.

Presence of high chlorides means corrosive nature of the aqueous system. The column, reboiler and concentrator should be built with material of construction, resistant to chlorides. Special stainless steel, such as Ti stabilized SS 316-L or high nickel alloy, could be used for such corrosive service. Circulation pumps of Alloy-20 could be used. However, cost of such materials of construction is higher than that of normally used materials. Nevertheless, overall operating cost of the ED system with aqueous CaCl_2 solution will be less than the conventional ED system.

Results of Case (a) and Case (b) indicate that the ED column will be more than 70% taller in Case (a). Also reflux ratio of Case (a) is nearly 55 % more than Case (b). Both these factors call for higher investment and higher energy consumption for Case (a).

Distillate of Case (b) contains 4 mole % (1.6 mass % or 16 032 ppm or 12 650 mg/L) water. To dehydrate ethanol further, three options are shown in Fig. 8.49. In a conventional manner (Option I), distillate with 4 mole % water will be distilled in another column to yield the azeotrope at the top (to be recycled) and anhydrous ethanol at the bottom. In Option II, part of the vapour from the ED column will be fed to a pervaporation unit. Here again, contaminated water (with some ethanol), removed in the membrane unit, can be recycled to the feed of the ED column. Drying of the distil-

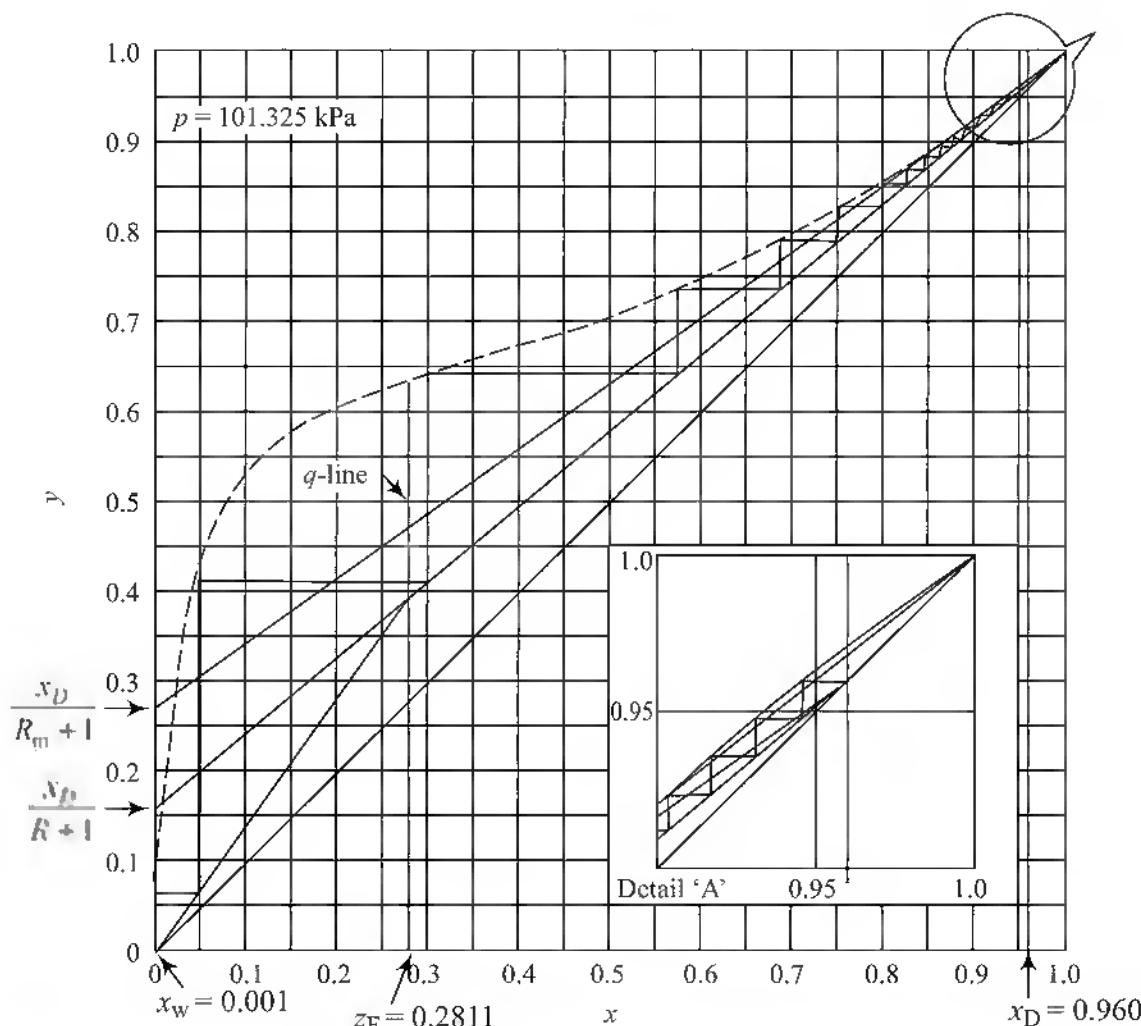


Fig. 8.50(c) Extractive Distillation with Aqueous Calcium Chloride Solution (25 mass %)

late from ED column by molecular sieves is shown as Option III. All three options are well proven in the industry and the selection should be made on the basis of minimum total operating cost. Please refer Sec. 8.8.3 for detailed discussions on various options.

8.9 ENERGY CONSERVATION IN DISTILLATION

It was stated earlier that distillation is the most widely used separation technique in chemical process industries but energy consumption in distillation is relatively high. Hence saving of energy in distillation operation is important for lowering overall cost of process plant. This aspect is also becoming important in biotechnology routes.

For saving the energy in distillation systems there are two options.

- Make the distillation more energy efficient; either by design or by operating the column more efficiently.
- Replace the distillation partially or completely by a new separation technique.

8.9.1 Energy Efficient Distillation by Design

To decrease the energy consumption of distillation various design options can be considered

- (a) Optimum design of the system
- (b) Heat integration
- (c) Use of high efficiency trays or high efficiency packings
- (d) Advanced process control
- (e) Use of thermally coupled distillation columns
- (f) Use of heat pumps (ejector/thermocompressor).

(a) Optimum Design of System

Process engineers have a general tendency to overdesign the system but at the expense of energy. For example, very high purities are specified for the distillate or bottom product which may not be warranted. At times, solvents are recycled in the system and very high purity may not be required. By designing the system for the desired purity only, reflux ratio can be trimmed thereby energy consumption can be reduced. In Sec. 8.7.2, a case of extractive distillation of cyclohexane and benzene mixture was discussed. As cyclohexane is a product, its purity of 99.3% was specified as a distillate from the first column. However, since benzene is a reactant which is to be recycled for further conversion to cyclohexane, its purity of 85% was specified as a distillate from the second column. If benzene purity of over 99% is specified, second column will be much taller with high reflux ratio, resulting in high energy consumption in the reboiler. Take another example of ethanol-water mixture. When ethanol is a desired product for mixing with petrol, very high purity of ethanol will be needed.

For heterogeneous azeotropic distillation, first column is used as a concentrator to produce distillate of near azeotropic composition. Subsequently two more columns are required for separation of the components. Cho and Jeon³² have proved after rigorous simulation by varying design parameters that while separating aqueous IPA solution, the first column (the concentrator) should be designed to produce distillate containing 38.7 mole% IPA (and not the azeotrope) which should be subsequently processed in a two-column system to dehydrate IPA. This design is claimed to consume lowest energy for the entire distillation system.

Knapp and Doharty³⁷ have proposed a two-column system (and not three column system) for concentrating dilute aqueous ethanol solution (containing just 4.2 mole % ethanol) from fermentor by adding approximately 0.043 kmol acetone per kmol feed and applying pressure swing distillation technique. This system is claimed to consume much less energy than conventional system.

Several such distillation case studies can be cited from the literature in which it has been demonstrated that by careful selection of process parameters at the design stage and the right technique, substantial energy savings can be achieved in operation. However, most energy efficient design could be expensive from equipment cost point of view. Nevertheless, energy saving outweighs capital cost in most cases.

(b) Heat Integration

Heat integration is low risk technology and it results in substantial energy saving. Reboiler of distillation column can act as a *cooler* for other unit. For example in

acetaldehyde plant, exothermicity of the reaction is removed by circulating saturated liquid leaving from the bottom of distillation column in tubeside of shell and tube type reactor. Liquid is vaporized and vapour is sent back to the distillation column. Thus reactor act as a reboiler for the distillation column. An additional reboiler is provided for the distillation column for supplying the balance heat (vapour) to the column.

Similarly overhead condenser can act as a *heater* or *reboiler* for other unit. Overhead vapour of distillation column can be partially used as heating medium of reboiler of other distillation column. This can be achieved conveniently with an ejector as discussed in Sec. 8.7.1 (f).

In Sec. 8.7.3, it was seen that pressure swing distillation technique offers an opportunity for thermal integration of condensers and reboilers of two columns as they operate at different temperatures.

(c) Use of High Efficiency Trays or High Efficiency Packings

Number of equilibrium stages of existing distillation column can be increased by replacing sieve trays or bubble cap trays with high efficiency packings or trays. For the fixed extent of separation, increase in number of equilibrium stages reduces reflux ratio and consequently reduces reboiler energy. Other advantage of using high efficiency packings (such as structured packings) or high efficiency trays (modified sieve trays) is to increase in capacity of the distillation column. Disadvantage of this replacement is higher cost of structured packings or of high efficiency trays. Generally savings in energy cost outweigh the cost of high efficiency packings/trays.

Example⁴⁰: Replacement of sieve trays of 400 mm diameter distillation column of styrene plant by a packed column, packed with structured packings, resulted in saving of energy consumption by 17.7%. This column was operating at its capacity of 24 500 kg/h of styrene. Its feed composition was 54% ethyl benzene and 46% styrene (by mole), distillate composition was 96.9% ethyl benzene and residue composition was 99.7% styrene (by mole). After replacement of sieve trays by a packed column with structured packings, reflux ratio was reduced from 7.3 to 5.8 and reboiler energy was reduced from 1605 kJ/kg of styrene to 1321 kJ/kg of styrene. In this modification, feed flow rate and purity of products were kept same.

(d) Advanced Process Control

Plant operators normally operate the distillation columns with a sufficient safety margin by achieving actual compositions of distillation products which are better than specifications. Operating a distillation column in this manner requires extra reflux and energy, but some margin is necessary so that the plant operator can cope with process upsets and other problems. The ‘safety’ margin is smaller in the plants which are properly designed and operated.

Installation of advanced process control systems based on accurate analytical sensors, reliable control loop hardware and instrumentation and good process models increases confidence in operators. In many instances, this added confidence translated to a reduced margin of ‘safety’, reflux ratio and reboiler energy.

Example⁴⁰: A conventional distillation column was used for separating a binary mixture of propane and propylene. Feed composition was 73% propylene and 27% propane (by mole). Required purity of top product was 99.7 mole % propylene for which reflux ratio was 13.5. With a sufficient margin conventional distillation column was operated with excess reflux ratio of 15.5 to get the purity of propylene of 99.9 mole % instead of the required purity of 99.7 mole %. After installation of distributed control system (DCS) on this column more accurate control of the purity of propylene at 99.7 mole % could be achieved which reduced energy consumption up to 13.5%. However, from a practical stand point, operators cannot control product purity precisely at required composition in the conventional system.

(e) Use of Thermally Coupled Distillation⁴¹

In the separation of multicomponent mixture (more than 2), thermally coupled distillation columns require less energy and fixed cost as compared to conventional distillation of multicomponent mixture. To understand this consider the separation of a ternary mixture having components A, B and C by distillation. A is the most volatile component and C is the least volatile component. Component B is having intermediate volatility. Conventional methods for separating the ternary mixture by distillation is either via direct sequence or indirect sequence, as shown in Figs 8.51 (a) & (b).

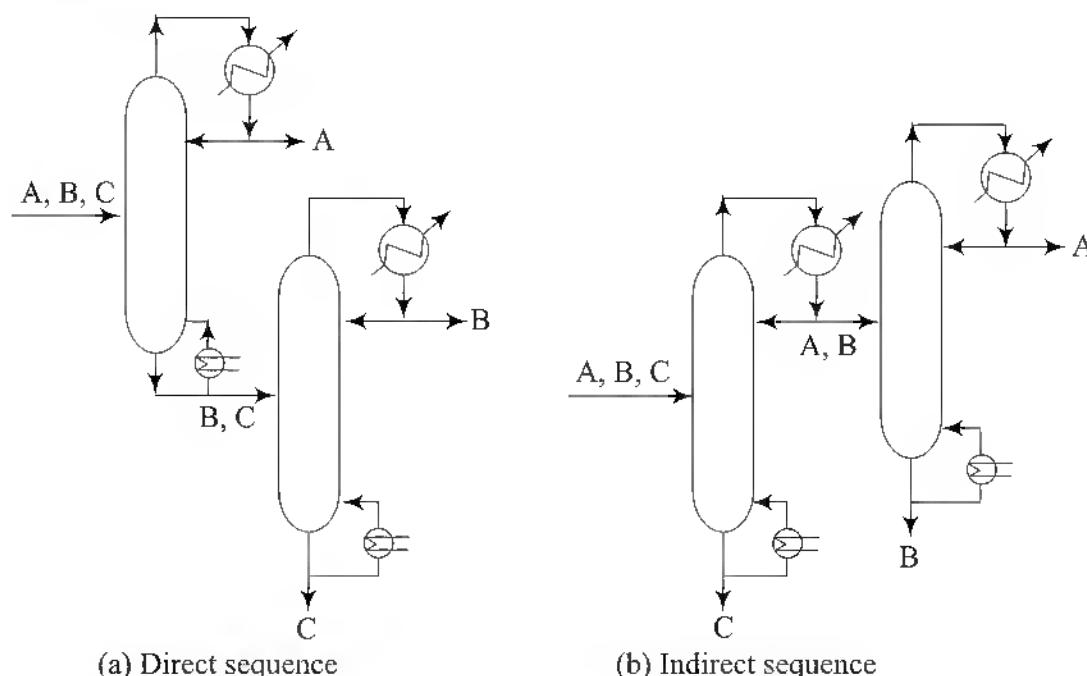


Fig. 8.51 Multicomponent Distillation in Sequence

For the given ternary mixture, selection between these two conventional arrangement of distillation column depends on composition of the ternary mixture, relative volatilities, latent heats of vaporization, boiling points, heat sensitivities, etc. Compared to these conventional arrangements of distillation columns thermally coupled distillation columns require less energy consumption and fixed

capital investment. For the separation of the same ternary mixture of A, B and C by thermally coupled distillation, two types of arrangement are possible, as shown in Figs 8.52 (a) and (b).

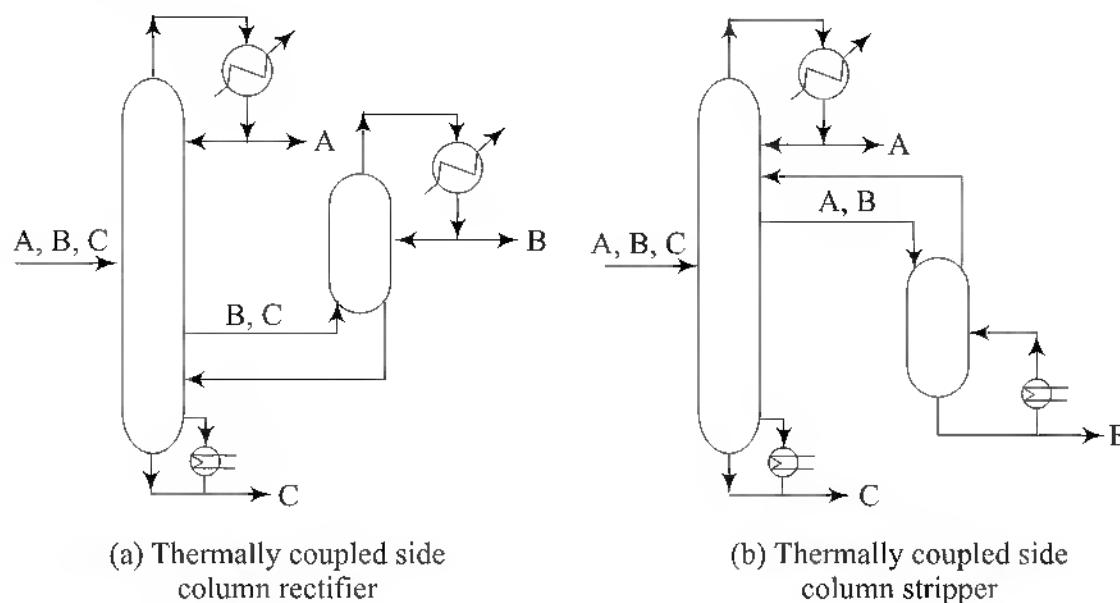


Fig. 8.52 Thermally Coupled Distillation Columns

In thermally coupled side column rectifier, vapour stream is withdrawn from the stripping section of main column and is sent for rectification in side column where an overhead condenser is provided. Liquid stream from the bottom of side column is sent back to the main column.

In thermally coupled side column stripper, liquid stream from the main column is withdrawn and is sent to side column for stripping where reboiler is provided at bottom. Vapour stream from side column is sent back to the main column.

Selection between these two arrangements depend on concentration profile of intermediate component B. If concentration of component B is maximum at any point of stripping section of the main or upstream column, then side column rectifier is selected. By withdrawing a side stream from the point at which the concentration of B is maximized, the distillation of B from either C or A is made much easier.

Side rectifiers and side strippers can be cost-effective on any ternary separation where less than half of the feed leaves in middle product or where relatively low purity of middle product is acceptable. Compared to conventional column arrangements side stripper or side rectifier can reduce the energy consumption up to 50%. Saving in fixed cost is also achieved because thermally coupled distillation columns avoid the use of one condenser or reboiler and require reduced column diameter or fewer stages for at least one column. Concept of thermally coupled distillation is also applicable to any pseudoternary systems—i.e. multicomponent mixtures requiring separation into three products. Hence it is applicable to the distillation of any mixture of more than two components.

Refer Example 8.7 in which separation of fatty acids from a mixture is dealt. Such a system can be considered for thermal coupling. In this case thermally coupled side column is recommended.

In a petroleum refinery, this technique is extensively practiced to produce different cuts (products like kerosene, naphtha, petrol, etc.) from crude by having number of side stream rectifiers and strippers.

As an alternate to the thermally coupled columns, a novel idea in design of tray type distillation column is to have partitioned distillation column (Ref. 42) (also called a divided-wall column or Petlyuk column) for multicomponent system. In such a column, a vertical baffle is placed (Fig. 8.53), which separates the feed location from that at which the intermediate boiling product is withdrawn. On the feed side of the partition, separation is achieved between the light (A) and heavy (C) fractions while the intermediate boiling fraction (B) is allowed to migrate to top and bottom sides of the partition. On the otherside of the partition, the light fraction is washed down, thereby intermediate boiling fraction (B) of desired purity is withdrawn. Such a column is larger in diameter than the otherwise required two columns but overall capital and operating costs are significantly (up to 30%) lower. Process design of such columns is quite complex and is not available in open literature.

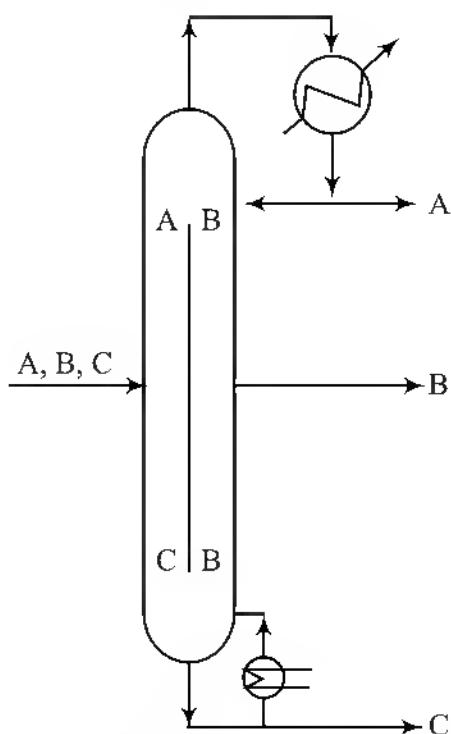


Fig. 8.53 Partitioned Distillation Column

(f) Use of Heat Pumps in Distillation

Steam ejectors have been used in limited distillation applications as inexpensive heat pumps. An ejector uses high pressure fluid to entrain low pressure fluid. Steam ejectors can be operated in different ways.

Simplest way to use an ejector is to operate with live steam. Figure 8.54 shows the proposed arrangement. Part of the top vapours are compressed in an ejector utilizing live (fresh) steam as motive fluid. Two or more ejectors can be used in parallel to improve flexibility. Use of such an ejector system requires that (i) top product is water and (ii) temperature difference between the bottom and top of the column is low. Hydrogen peroxide–water distillation column can be fitted with a live steam ejector.

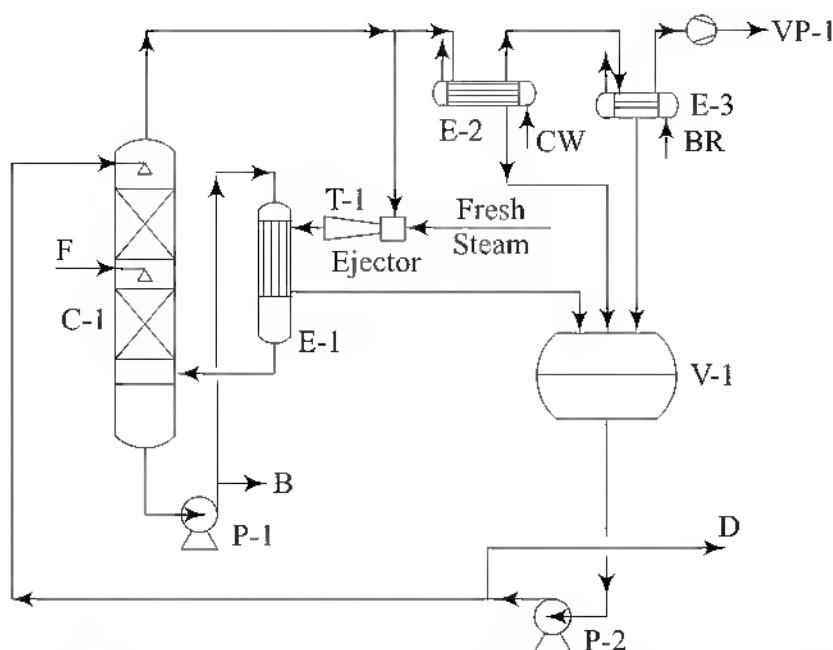


Fig. 8.54 Ejector System with Live Steam as the Driving Medium⁴³
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Figure 8.55 is a capacity ratio curve for the thermocompressor (ejector), operating with saturated steam at 20.7 bar g.

In Fig. 8.56, a system is shown in which top vapour product is used as a motive as well as entrained fluid. In this case, it should be possible to heat the top product to a substantially higher temperature and pressure than the column's operating conditions. Also the temperature difference between top and bottom of the column should be low. Ethyl benzene/styrene splitter can be fitted with such an ejectors system. A 225×10^6 t/a styrene monomer plant when operated with such an ejector system reduced steam and cooling water requirements by 25% and 32%, respectively. Capital investment for the modifications is expected to be repaid in less than 18 months. Acetic acid–water, ethylene glycol–water systems can also be operated in this manner.

In a third alternative, bottom product can be used to generate motive fluid, for the ejector (Fig. 8.57). Part of the top vapours is condensed in a heat exchanger with bottom product which operated under reduced pressure (say sub-atmospheric). High pressure bottom product vapour provides the driving medium for the ejector. For this system to operate satisfactorily, three conditions need be satisfied. (i) It should be possible to heat the bottom product to a substantially higher temperature and pressure than the operating conditions of the column.

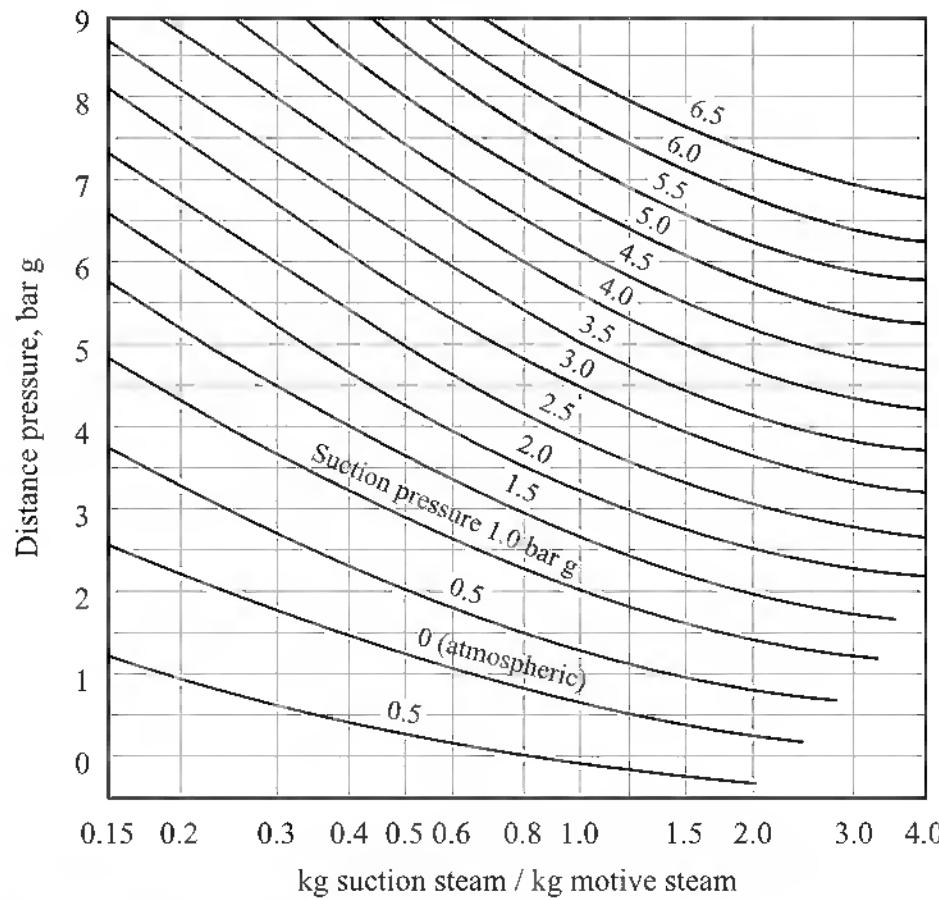


Fig. 8.55 Capacity Ratios of Steam Jet Thermocompressors Operating with Saturated 20.7 bar g Motive Steam
(Courtesy: Mazda-Croll Reynolds, Ahmedabad)

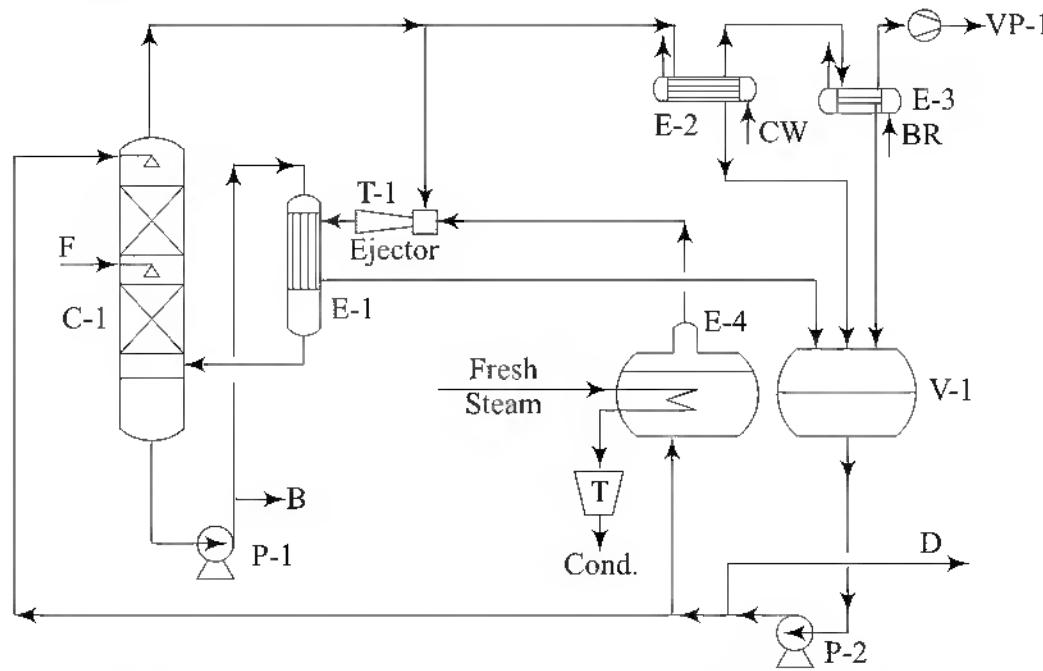


Fig. 8.56 Ejector System with Use Top Product as the Driving Medium⁴³
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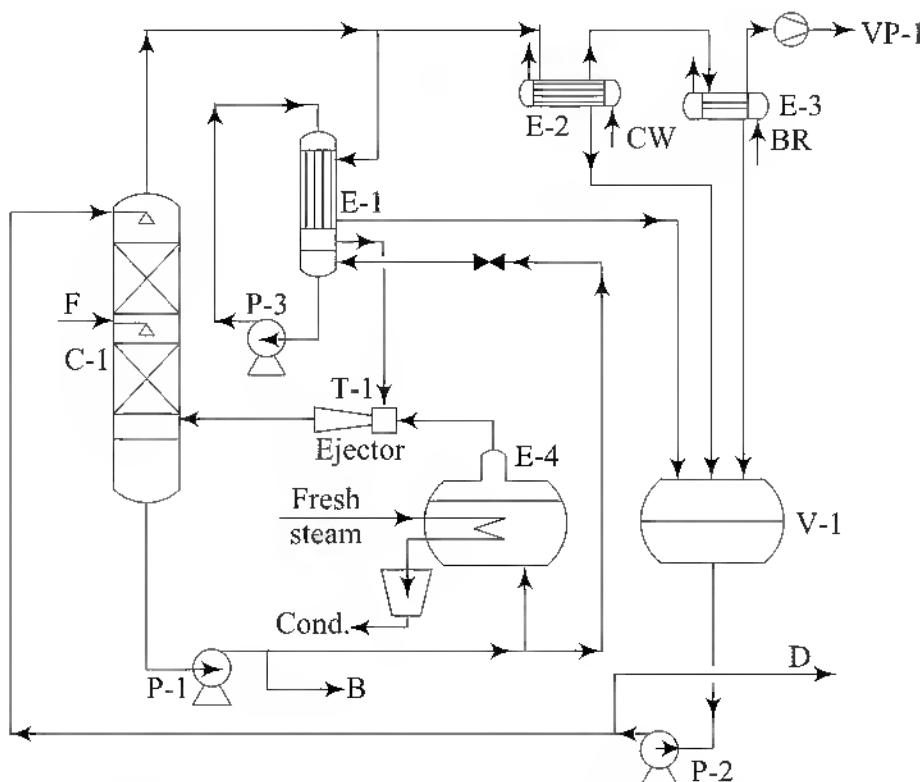


Fig. 8.57 Ejector System with Bottom Product as the Driving Medium⁴³
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(ii) Top product is not suitable for compression with the ejector. (iii) Temperature difference between bottom and top of the column is low. Alcohol (methanol or ethanol)—water distillation column can be fitted with such an ejector system.

Innovative flow sheets were presented as Fig. 8.54, Fig. 8.56 and Fig. 8.57 for use of an ejector for energy conservation. Process engineer should therefore always look for innovative designs which are technically feasible and select the best option based on all aspects, including operational convenience.

It may be noted that in all the applications, it is necessary to have low temperature difference between top and bottom products of the column. Also the ejectors should be large in size so that with low differential pressure between motive and entrained fluids is permitted. In Figs 8.54, Fig. 8.56 and Fig. 8.57, number of peripheral equipments are shown which require substantial capital expenditure. Because of these reasons, ejector installation is of lesser interest for the smaller columns. However, when large distillation column and substantial energy input are required, use of ejector(s) could prove economically viable. (Ref. 43)

In Fig. 8.54, live steam is used for compressing the overhead vapours in an ejector to the reboiler for its use as heat transfer medium. As an alternate to the ejector, a vapour compressor (Ref. 44) can be used to compress the overhead vapours (Fig. 8.58). The compressor could be a radial or an axial-flow turbocompressor or a screw compressor, driven directly or via gear box by an electric motor or a steam turbine. For overhead vapour having low molar mass (such as water), compression is rather restricted up to 10 bar. For high molar mass vapours, multistage compressors can be used to compress them up to 80

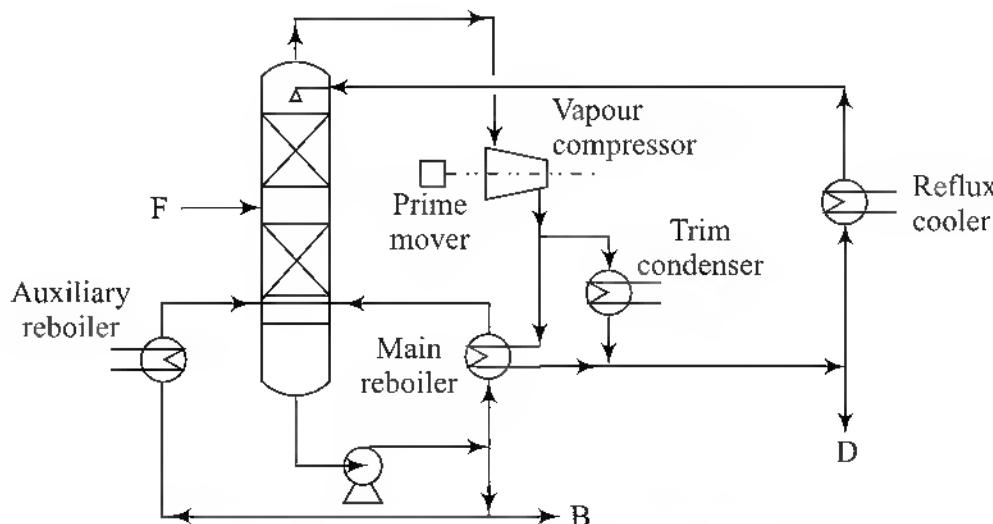


Fig. 8.58 Distillation Columns with Direct Vapour Compressor⁴⁴
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bar. For partial load operation, the compressor should have a variable speed or kick-back arrangement. Performance of Vapour Recompression System (VRC) is measured by calculating the ratio of reboiler duty (kW) to the power consumption (kW) of the compressor. This ratio is called Coefficient of Performance (COP) and typical value ranges from 6 to 15. Although VRC system can be used for any distillation column on technical ground, it is favoured in the following cases due to the economic factors.

- (a) When reboiler duty is high (> 2 MW), VRC system is promising.
- (b) Lower temperature rise across the compressor is preferred.
- (c) When chilled water or brine is used to condense overhead vapours in the condenser, use of VRC system is advantageous.
- (d) Columns that separate products with low separation factors require larger number of stages and high reflux ratio. Such columns are most suitable for adoption of VRC system. Separation of propylene–propane, ethylene–ethane, ethyl benzene–xylene, *iso*–propanol–water (refer Example 8.17), etc. are the systems which can benefit significantly by adoption of the VRC system.

In a typical case, 60% (by mass) *iso*–propanol feed (balance water) is distilled to achieve azeotrope (87.5% *iso*–propanol) with bottom product containing 15% *iso*–propanol. When overhead azeotropic mixture is compressed and used as a heating medium in the reboiler, coefficient of performance in excess of 10 could be achieved.

Where low cost heat or free waste energy is available for use in the reboiler, VRC system is not recommended. Also when the column is operated under high vacuum (> 710 torr), VRC is not economical because compression ratio becomes high.

One more option for reducing energy consumption in distillation is to adopt heat pump with an auxiliary heat transfer medium (Ref. 45). In Fig. 8.59, distillation column with auxiliary heat transfer medium is shown. In the overhead condenser, vapours are condensed by evaporating the auxiliary medium. A compressor compresses auxiliary medium vapours. In the reboiler, auxiliary medium

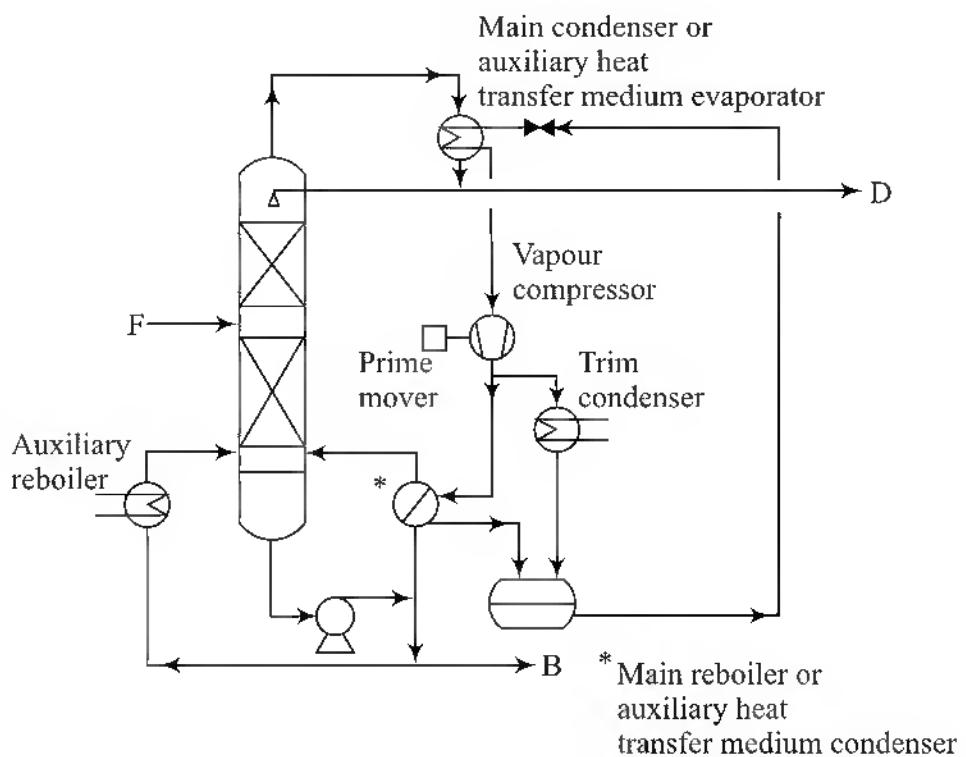


Fig. 8.59 Distillation Column Heat Pump with Auxiliary Heat Transfer Medium⁴⁵
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vapours are condensed and liquefied medium is recycled back to the overhead condenser through an expansion valve, thus completing the cycle.

Typical auxiliary heat transfer media are refrigerants. Water (R-718), ammonia (R-717), R-134a, R-22, R-502, etc. can be used for the purpose. Among these, water (R-718) has many advantages over others. Water is chemically and thermally stable. No negative environmental effect in case of leakage, no special material of construction is required, cheap, high heat transfer co-efficients and high latent heat of vaporization are chief factors that favour its selection. Performance of heat pump with auxiliary medium is measured by Rankin coefficient of performance. Generally, COP of water is higher than that of other refrigerants.

Considering capital investment for the auxiliary heat-transfer medium recycle system and larger condenser and reboiler (as compared to conventional system), heat pump system with auxiliary medium is cost effective when reboiler duty is high (say > 2 MW). In this case also when close boiling components are to be separated, the heat pump is economical. Separation of *p*-xylene and ethyl benzene mixture and styrene and ethyl benzene mixture can be good examples of the system. For a 225 000 t/a styrene monomer plant, COP of heat pump with water as auxiliary medium could be in excess of 5.5.

Example 8.19

A distillation column is to be designed for aromatics separation. Material balance around the column is fixed and compositions of various streams are given below.

Table 8.53 Composition of Distillation Column Streams

Component	Composition, mole %		
	Feed	Distillate	Bottoms
Benzene	2.2	22.8	0.
Toluene	7.4	72.2	0.5
Ethyl benzene	43.4	5.0	47.5
Styrene	47.0	0	52.0

Feed flow rate is 70 kmol/h as saturated liquid. Column must be operated under vacuum to avoid thermal cracking and polymerization.

It is planned to use a heat pump (similar to one shown in Fig. 8.59) for the system. Refrigerant R-134a (1,1,1,2-tetrafluoroethane) is to be used as an auxiliary heat transfer medium. Bubble point of the distillate will be 12°C in the overhead condenser and accordingly operating pressure of the condenser will be fixed.

Determine the required number of theoretical stages for the desired separation. Also find the location of the feed stage. Based on NTS, fix the height of packed bed, packed with fibreglass reinforced plastic (FRP) Pall rings of 25 mm size. Assume pressure drop in the column to be 0.04 atm and calculate the operating pressure of the reboiler. In the reboiler, compressed R-134a vapour will be condensed.

Assume approach of 10°C in the condenser and the reboiler for effective heat transfer. Calculate heat duties of the condenser and reboiler and the circulation rate of the refrigerant. Consider ideal behaviour of all liquid and gaseous mixtures.

Solution:

Bubble point of distillate: 12°C

Considering ideal VLE,

$$\sum x_i p_{vi} = p_t \quad (A)$$

$x_{DB} = 0.228, x_{DT} = 0.722 \text{ and } x_{DEB} = 0.05$

Table 8.54 Antoine Constants¹³

Component	Antoine Constants		
	A	B	C
Benzene	14.1603	2948.78	-44.5633
Toluene	14.2515	3242.38	-47.1806
Ethyl benzene	6.0821	1424.3	-59.95
Styrene	14.3284	3516.43	-56.1529

$$\ln p_v = A - \frac{B}{(T + C)} \text{ for benzene, toluene and styrene} \quad (B)$$

$$\text{and } \log p_v = A - \frac{B}{(T + C)} \text{ for ethyl benzene} \quad (C)$$

where, p_v is in kPa and T is in K.

At $T = 12 + 273 = 285$ K,

$$p_{VB} = 6.659 \text{ kPa}, p_{VT} = 1.8547 \text{ kPa} \text{ and } p_{VEB} = 0.567 \text{ kPa}$$

Operating pressure of the condenser,

$$p_t = 0.228 \times 6.659 + 0.722 \times 1.8547 + 0.05 \times 0.567 = 2.8857 \text{ kPa}$$

Pressure drop in the column = 0.04 atm = 4.053 kPa

Operating pressure at the bottom of the column, $p_t = 2.8857 + 4.053 = 6.9387 \text{ kPa}$

This pressure (p'_t) will fix the bubble point of the liquid in the reboiler.

$$\sum x_{wi} p_{vi} = p'_t \quad (\text{D})$$

By trial and error, bubble point is to be fixed satisfy Eq. (D).

At $t_B = 62.5^\circ\text{C}$, $T_B = 335.5 \text{ K}$

$$p_{VB} = 55.9708 \text{ kPa},$$

$$p_{VT} = 20.2005 \text{ kPa}, p_{VEB} = 8.1878 \text{ kPa} \text{ and } p_{VS} = 5.7 \text{ kPa}$$

$$\sum x_{wi} p_{vi} = 0.005 \times 20.2005 + 0.475 \times 8.1878 + 0.52 \times 5.7$$

$$= 6.954, \text{ close enough to } 6.9387 \text{ kPa.}$$

In this example, toluene is the light key component and ethyl benzene is the heavy key component.

Table 8.55 Relative Volatilities

Component	$\infty_{\text{top}} = \frac{p_i}{p_{vhk}}$	$\infty_{\text{bottom}} = \frac{p_i}{p_{vhk}}$	∞_{av}
Benzene	11.744	6.836	8.96
Toluene (LK)	3.271	2.467	2.8407
Ethyl benzene (HK)	1	1	1
Styrene	0.625	0.6962	0.6596

FUG method:

Minimum number of theoretical stages can be found, using Fenskey's equation.

$$N_m = \frac{\log \left[\left(\frac{x_{LK}}{x_{HK}} \right)_D \left(\frac{x_{HK}}{x_{LK}} \right)_B \right]}{\log \infty_{LK}} \quad (8.23)$$

$$N_m = \frac{\log \left[\left(\frac{0.722}{0.05} \right) \left(\frac{0.475}{0.005} \right) \right]}{\log (2.8407)} = 6.919, \text{ say } 7$$

Underwood method:

$$\sum \frac{\infty_i x_{if}}{\infty_i - \vartheta} = 1 - q \quad (8.15)$$

$$\frac{8.96 \times 0.022}{8.96 - \vartheta} + \frac{2.8407 \times 0.074}{2.8407 - \vartheta} + \frac{1 \times 0.434}{1 - \vartheta} + \frac{0.6596 \times 0.47}{0.6596 - \vartheta} = 0$$

Solving by Mathcad,

$$\vartheta = 2.4052$$

$$\begin{aligned} \text{Also} \quad \vartheta &\in \{1, \dots, 2.8407\} \\ \vartheta &= R_m + 1 \end{aligned}$$

$$\sum \frac{\infty_i x_{iD}}{\infty_i - \vartheta} = R_m + 1 \quad (8.14)$$

$$\frac{8.96 \times 0.228}{8.96 - \vartheta} + \frac{2.8407 \times 0.722}{2.8407 - \vartheta} + \frac{1 \times 0.05}{1 - \vartheta} = R_m + 1$$

For $\vartheta = 2.4052$, $R_m = 3.9856$

Gilliland correlation:

$$f(N) = \frac{N - N_m}{N + 1} = 1 - \exp \left[\left(\frac{1 + 54.4 \psi}{11 + 117.2 \psi} \right) \times \left(\frac{\psi - 1}{\psi^{0.5}} \right) \right] \quad (8.25)$$

where, $\psi = \frac{R - R_m}{R + 1}$ (8.26)

Table 8.56 Evaluation of Parameters

R	ψ	f(N)	N
4.5	0.093 53	0.5603	17
6	0.2878	0.39	12
7	0.3768	0.3288	10.76
8	0.446	0.2819	10
9	0.50144	0.2483	9.53

Referring Table 8.56, $R = 8$ is considered optimum and required number of theoretical stages are 10.

Feed tray location can be determined by Kirkbride equation,

$$\log \left(\frac{N_r}{N_s} \right) = 0.206 \log \left[\frac{W}{D} \left(\frac{x_{HK,F}}{x_{LK,F}} \right) \left(\frac{x_{LK,B}}{x_{HK,D}} \right)^2 \right] \quad (8.27)$$

For determining W and D , material balance calculations are to be carried out.

Overall material balance:

$$\begin{aligned} F &= D + W \\ 70 &= D + W \end{aligned} \quad (\text{E})$$

Benzene balance:

$$\begin{aligned} 0.022 \times F &= 0.228 \times D + 0 \times W \\ 0.022 F &= 0.228 D \end{aligned} \quad (\text{F})$$

Solving Eq. (E) and Eq. (F),

$$D = 6.7544 \text{ kmol/h and } W = 63.2456 \text{ kmol/h}$$

$$\log \left(\frac{N_r}{N_s} \right) = 0.206 \log \left[\frac{63.2456}{6.7544} \times \frac{0.434}{0.074} \times \left(\frac{0.005}{0.05} \right)^2 \right]$$

$$\frac{N_r}{N_s} = 0.8839$$

$$N_r + N_s = 10$$

$$N_s = 5.308. \text{ Let } N_s = 6$$

Therefore, sixth stage from bottom will be the feed stage. Pressure drop per stage is calculated to be 0.405 kPa which is reasonable.

Heat duty of condenser, ϕ_c

$$\phi_c = (R + 1) D \lambda_{av}$$

Using Watson equation, latent heats of vaporization for the components of distillate are calculated as under at 12°C.

$$\lambda_B = 34\ 252.6 \text{ kJ/kmol},$$

$$\lambda_T = 38\ 452.8 \text{ kJ/kmol and}$$

$$\lambda_{EB} = 42\ 497.0 \text{ kJ/kmol}$$

$$\begin{aligned}\lambda_{av} &= \sum \lambda_i \cdot y_i = 0.228 \times 34\ 252.6 + 0.722 \times 38\ 452.8 + 0.05 \times 42\ 497.0 \\ &= 37\ 697.3 \text{ kJ/kmol distillate}\end{aligned}$$

$$\phi_c = (8 + 1) \times 6.7544 \times 37\ 697.3 = 2291\ 603.8 \text{ kJ/h} \equiv 636.56 \text{ kW}$$

Let $t = 12^\circ\text{C}$ be the reference temperature

$$H_D = 0, H_D \cdot D = 0$$

Refer Table 6.29 of Chapter 6.

At $T = 335.5 \text{ K}$

$$H_w = -17.712 (335.5 - 285) + 1054.193 \times 10^{-3} \left(\frac{335.5^2 - 285^2}{2} \right)$$

$$- 1838.075 \times 10^{-6} \left(\frac{335.5^3 - 285^3}{3} \right)$$

$$+ 1694.332 \times 10^{-9} \left(\frac{335.5^4 - 285^4}{4} \right)$$

$$= 9240 \text{ kJ/kmol mixture}$$

$$H_w \cdot W = 9240 \times 63.2456 = 584\ 389.3 \text{ kJ/h} \equiv 162.33 \text{ kW}$$

Feed is saturated and hence enthalpy of feed at its bubble point is to be calculated.

At bubble point,

$$\sum \frac{x_i p_{vi}}{p_t''} = \sum y_i = 1$$

Since feed is nearly in the middle of the column, assume average pressure at the feed point

$$p_t'' = \frac{2.8857 + 6.9387}{2} = 4.9122 \text{ kPa}$$

$$0.022 p_{VB} + 0.074 p_{VT} + 0.434 p_{VEB} + 0.47 p_{VS} = 4.9122$$

This equation can be solved by trial and error calculations. At $= 49^\circ\text{C}$ (322 K),

$$p_{VB} = 34.18, p_{VT} = 11.6264, p_{VEB} = 4.435, p_{VS} = 3, \text{ all in kPa}$$

$$\sum x_i p_{vi} = 4.947, \text{ close enough to } 4.9122 \text{ kPa}$$

Refer Table 6.30 of Chapter 6.

$$\begin{aligned}
 H_F &= -16.023 (322 - 285) + 1030.422 \times 10^{-2} \left(\frac{322^2 - 285^2}{2} \right) \\
 &\quad - 1809.476 \times 10^{-6} \left(\frac{322^3 - 285^3}{3} \right) \\
 &\quad + 1693.124 \times 10^{-6} \left(\frac{322^4 - 285^4}{4} \right) \\
 &= 6561.5 \text{ kJ/kmol feed}
 \end{aligned}$$

$$H_F \cdot F = 6561.5 \times 70 = 459\ 305 \text{ kJ/h} \equiv 127.6 \text{ kW}$$

Assume 5% heat loss to surrounding and no heat of mixing.

$$\begin{aligned}
 \phi_B &= (\phi_C + H_D \cdot D + H_W \cdot W - H_F \cdot F) 1.05 \\
 &= (636.56 + 0 + 162.33 - 127.6) 1.05 = 704.85 \text{ kW} \equiv 2537\ 460 \text{ kJ/h}
 \end{aligned}$$

Approach for heat transfer in the overhead condenser and reboiler is 10°C. Since distillate bubble point is 12°C, auxiliary heat transfer medium (R-134a) should evaporate at 2°C. Similarly in reboiler, boiling takes place at 62.5°C and hence R-134a vapours are to be condensed at 72.5°C.

Table 8.57 Properties of R-134a²³

Temperature °C	Pressure, bar a	Enthalpy, kJ/kg			Vapour, C_p/C_v
		Liquid	Latent	Vapour	
2.0	3.150 (satd.)	202.68	197.07	399.75	1.182
72.5	22.385 (satd.)	308.57	120.42	428.99	1.653
130.0	23.0	—	—	500.00	—

(Reference state: Enthalpy of saturated R-134a at 0°C = 200 kJ/kg)

In the condenser, R-134a liquid from the reboiler (at 72.5°C) will be flashed to 2°C and vapours will be produced.

Heat, taken up in condenser,

$$H_1 = 399.75 - 308.57 = 91 \text{ kJ/kg}$$

$$\text{Evaporation rate of R-134a, } \dot{m}_R = \frac{\phi_c}{H_1}$$

$$\dot{m}_R = 2291\ 603.8/91 = 25\ 182.5 \text{ kg/h}$$

These vapours will be superheated by 10°C before taking to the compressor to avoid any liquid carryover. This can be done by exchanging heat with liquid from reboiler. However, for the present, this difference is neglected. Vapour at 3.15 bar a will be compressed to 23 bar a.

Overall compression ratio = 23/3.15 = 7.302

Since this is high, it will be compressed in a 2-stage compressor.

Compression ratio in each stage = $\sqrt{7.302} = 2.702$

Vapours will be compressed from 3.15 bar a to 8.511 bar a in the first stage, cooled suitably and compressed to 23.0 bar a in the second stage. Its temperature is expected to be 130°C.

$$\begin{aligned}
 \text{Heat, given up in the reboiler, } H_2 &= 500 - 308.57 \\
 &= 191.43 \text{ kJ/kg}
 \end{aligned}$$

Heat available, $\phi'_B = 25 \times 182.5 \times 191.43 = 4820\,686 \text{ kJ/h} \equiv 1339.1 \text{ kW}$

Since ϕ'_B is much higher (by 90%) than ϕ_B , a trim condenser will be needed to take care of the excess heat load. This heat (low level) can be utilised to heat the feed or some other stream in the plant. In addition, during start-up, overhead condenser will not have any heat-load but reboiler will have to be commissioned. Therefore, an additional auxiliary reboiler will be needed with some other heat source, say with low pressure steam for the start-up. It can be seen that adoption of heat pump system will thus call for a number of additional equipments.

Notes:

- (i) Since required number of equipments are many more than for the conventional distillation system, initial investment will be substantially high.
- (ii) Operating pressure of the reboiler shell will be 23 bar a. This will call for thick shell, calling for higher investment.
- (iii) Adequate instrumentation and controls (I&C) will be needed for the refrigerant circulation system in addition to the I&C for distillation system, calling for additional investment.
- (iv) Cost of Refrigerant R-134a is much higher than conventional utilities. Enough care is required to prevent its leakage from the circulation system.

Fixed, and variable cost for driving the compressor and extra cost of the heat pump system should be carefully weighed against savings in normal utilities, used in the conventional distillation system. Also skilled personnel will be required for operating the integrated system.

Heat pumps save energy in distillation but at a definite cost. Careful economic evaluation is therefore necessary before its implementation.

8.9.2 Efficient Operation of Distillation Columns

Existing distillation columns can be operated in such a manner that substantial energy can be saved.

- (a) Many distillation columns are operated to excessively purify the products which might call for high reflux ratio and hence high reboiler duty. This should be avoided by having adequate I&C.
- (b) Damaged or worn column internals can be costly. Over a period of operation, the column internals may get damaged. For example, in bubble cap columns, top nut may loosen and cap may open up, in valve trays, valves may flyoff, in packed columns, ceramic packings may crumble, scaling on trays may take place over a period of operation, etc. Due to similar reasons, the existing columns should be opened for inspection periodically and preventive maintenance should be carried out. Such preventive maintenance practice will pay rich dividends in terms of energy saving in operation.

8.9.3 Replace the Distillation Partially or Completely by New Separation Technique

Replacing of distillation by supercritical extraction or by reactive distillation can result in significant energy savings. The same is discussed in Sec. 7.6 and Sec 8.6 of this text. Similarly energy savings can be achieved by replacing distillation

partially or completely by new separation techniques like membrane separation techniques (reverse osmosis, pervaporation, membrane distillation, membrane reactor, etc.), molecular sieve adsorption, crystallization, etc.

Example⁴⁰: Conventionally absolute alcohol (ethanol) is separated from azeotropic mixture of ethanol-water (95.6% ethanol, 4.4% water by mass) by azeotropic distillation using benzene or cyclohexane as an entrainer. This conventional method uses three distillation columns and requires very high energy consumption. One of the new methods for separating the same mixture is by Pressure Swing Adsorption (PSA) which utilize 3 Å molecular sieves. Molecular sieve adsorption consumes less energy and eliminates the use of the entrainer. Benzene is a proven carcinogen and therefore an air pollutant. For the production of 3400 kg/h of fuel-grade ethanol, installation of molecular sieve—pressure swing adsorption system instead of azeotropic distillation unit allows a reduction in energy consumption of boiler from 2257 to 147 kW. But this new technique requires additional electrical energy in the amount of about 100 kW to drive the vacuum pumps and other equipments for the PSA unit. The use of benzene or cyclohexane as entrainer is eliminated.

For the separation of same azeotropic mixture, pervaporation can also be used. For the production of fuel grade ethanol with the same rate; 3400 kg/h, installation of pervaporation allows a reduction in energy consumption of boiler from 2257 to 264 kW. But fresh electrical energy, equivalent to 150 kW is required to drive the vacuum pumps and other equipments for the pervaporation unit.

Iso-propyl-alcohol (IPA) is yet another application for which pervaporation can be used. IPA forms an azeotrope with water having composition of 87.9% (by mass) IPA. Dehydration of IPA azeotrope with zeolite membrane is commercially carried out.

Table 8.58 Comparison of Pervaporation and Adsorption with Distillation

Parameter	Distillation	Pervaporation	Adsorption
Capacity, m ³ /d	3 to 30	0.5 to 30	10 to 30
Feed water content, %	5–95%	0.5 to 30	0.05 to 5
Product water content, mg/L	< 2000	< 1000	< 100
Operating cost	High	Low	Medium
Capital cost	Low	Medium	Medium
Floor space requirement	Large	Small	Large
Safety	Medium	High	Medium
Environment friendliness	Low	Excellent	Medium
Ease of installation	Medium	Easy	Medium
Operation	Skilled	Easy	Skilled
Turndown capability	Medium	Excellent	Poor
Start-up and shut-down time	Long	Short	Long
Maintenanace requirements	High	Low	High
Availability/Uptime	Medium	High	Medium
Flexibility	Limited	Excellent	Medium

Freeze crystallization is yet another technique which can be used for recovery of organic species, such as acetic acid, acrylic acid, etc. from very dilute solutions with 75 to 90% energy reduction as compared to conventional distillation. Heist⁴⁶ has given a process flow sheet for 95% recovery of acetic acid from 1% aqueous solution (by mass) by freeze crystallization. Normally 1% solution could be considered as effluent with high non-biodegradable COD and its disposal will be a serious threat to the industry. Recovery will turn effluent disposal problem into an economical proposal by adoption of freeze crystallization.

EXERCISES

8.1 Fill the gaps in the following crossword puzzle.

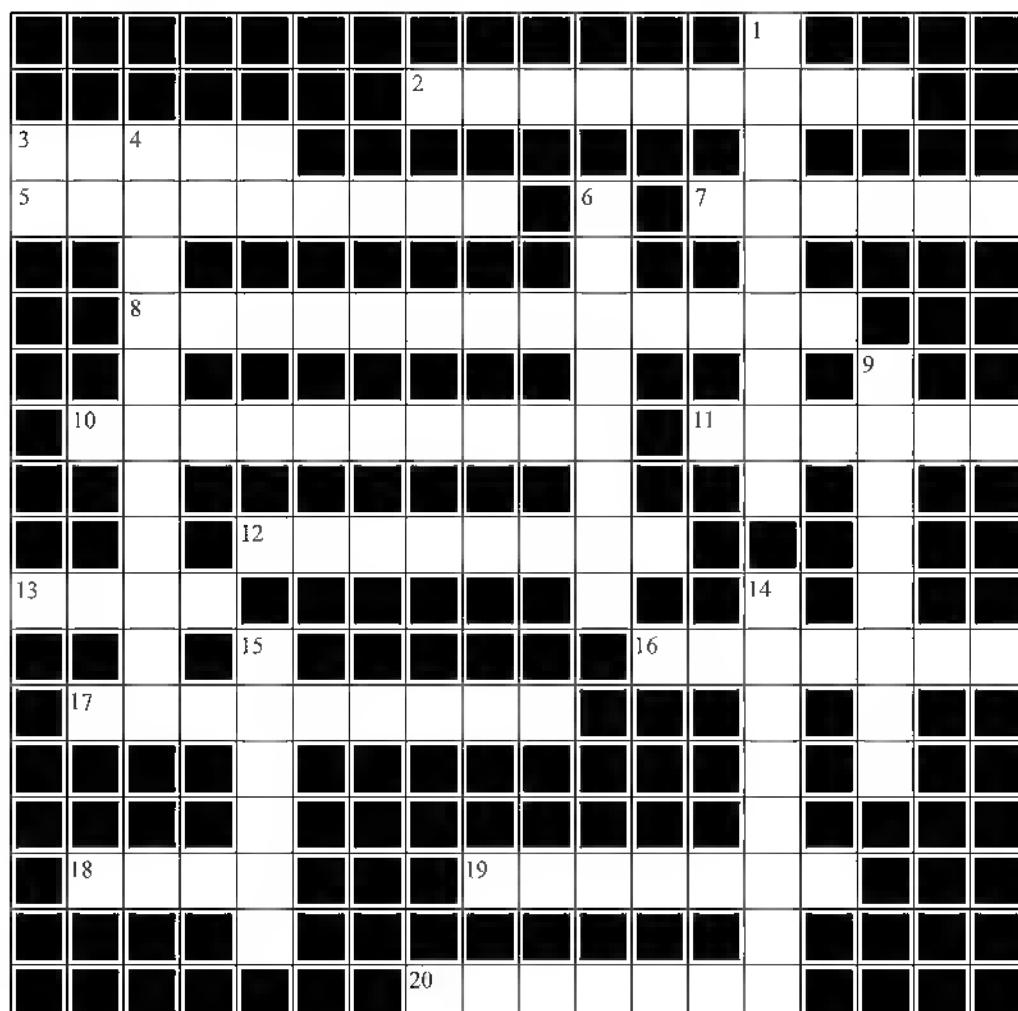


Fig. 8.60 Crossword Puzzle⁴⁷

(Reproduced with the Permission of Dr. Ming Tham of School of Chemical Engineering and Advanced Materials, University of Newcastle upon Tyne, UK)

KEYS:

Down:

1. A conduit that directs liquid from one tray to another.
4. Liquid carried by vapour up to the tray above.
6. This is determined by the amount of material going through the column.

9. Brought about by excessive vapour flow.
14. An alternative to the use of trays or plates.
15. Made-up of the two components.

Across:

2. A liquid mixture which when vaporized produces the same composition as the liquid.
3. A type of tray used in distillation/absorption.
5. An equipment used to cool vapours, coming off the top of the column.
7. Distillation operations require lots of this.
8. Operation performed by the top section of the column.
10. Product stream taken off the top of the column.
11. Liquid that is fed back to the top of the column.
12. An equipment used to raise vapour.
13. A mixture that is to be separated.
16. Expansion of liquid due to vapour or gas.
17. Operation performed by the bottom section of the column.
18. Ensures that there is sufficient liquid on a distillation tray.
19. Caused by low vapour flow.
20. Product stream taken off bottom of the column.

8.2 Distillation column is used to separate aqueous solution of dimethyl formamide (DMF). Determine the following based on given data.

- (a) Minimum reflux ratio.
- (b) Number of theoretical stages required for desired separation for $R = 2 R_m$.
- (c) Overhead condenser duty.
- (d) Reboiler duty.

Data:

- (i) Feed composition: 20% by mass DMF, Molar mass of DMF = 73.1 kg/kmol
Feed is saturated liquid. Feed flow rate = 100 kmol/h
- (ii) Distillate composition: 99% water (by mass).
- (iii) Residue composition: 99% DMF (by mass).
- (iv) Operating pressure at top: 650 torr vacuum or 110 torr a.
- (v) For vapour pressure data refer Table 8.59.
- (vi) Properties of DMF

Latent heat of vaporization $\lambda = 576.85 \text{ kJ/kg}$ at the top most temperature of distillation column

Specific heat $C_L = 2 \text{ kJ/(kg} \cdot ^\circ\text{C)}$

8.3 NRTL parameters for dimethyl formamide (1) and water (2) are as follows:

$$\begin{aligned}\tau_{12} &= b_{12}/RT, \quad \tau_{21} = b_{21}/RT, \quad R = 8.314 \text{ kJ/(kmol} \cdot \text{K)} \\ b_{12} &= -267.667, \quad b_{21} = 470.31\end{aligned}$$

$$\ln \gamma_1 = x_2^2 \left[\tau_{21} \left(\frac{G_{21}}{x_1 + x_2 G_{21}} \right)^2 + \frac{G_{12} \tau_{12}}{(x_2 + x_1 G_{12})^2} \right] \quad (8.112)$$

$$\ln \gamma_2 = x_1^2 \left[\tau_{12} \left(\frac{G_{12}}{x_2 + x_1 G_{12}} \right)^2 + \frac{G_{21} \tau_{21}}{(x_1 + x_2 G_{21})^2} \right] \quad (8.113)$$

where, $G_{12} = \exp(-\infty \tau_{12})$, $G_{21} = \exp(-\infty \tau_{21})$
 $\infty = 0.277$

Operating pressure in distillation column = 650 torr vacuum

Table 8.59 Vapour Pressure Data of DMF and Water

Temperature °C	p_v of DMF torr	p_v of water torr
53.7	18	110
60	26	149.38
70	42	233.7
80	65	355.1
90	100	525.76
92	110	567
100	165	760

Solve Exercise 8.2 considering nonideality of DMF-water system. To find the VLE data following equation can be used.

$$y = \frac{\gamma p_v}{p_t} x$$

y, x = Mole fraction of more volatile component (water) in vapour and liquid phase, respectively.

γ = Activity coefficient of water in solution.

p_t = Operating pressure = 110 torr a

- 8.4 In the production of chloromethanes, a saturated liquid mixture consisting of 60% methyl chloride, 28% methylene dichloride, 9% chloroform and 3% carbon tetrachloride (by mass), is sent do distillation section for the separation of pure products. In first distillation column 99.9% (by mass) pure methyl chloride is separated as top product. Determine the followings for this distillation column.

(a) Operating pressure of distillation column.

(b) Minimum reflux ratio by Underwood's method.

(c) For $R = 3 R_m$ calculate the numer of theoretical stages required for desired separation.

(d) Determine the tower diameter of distillation column, selecting sieve tray tower for the same.

Feed is a saturated vapour mixture and its flow rate is 5000 kg/h.

- 8.5 Determine the minimum reflux ratio and number of theoretical stages required for reflux ratio, $R = 2 R_m$, for the following given separation by.

(i) Ponchon – Saverit method based on given H - x - y data and equilibrium x vs y data, given in Table 8.60.

(ii) McCabe – Thiele method using phase equilibrium data x vs y , given in Table 8.60.

(iii) McCabe – Thiele method by using vapour–liquid equilibrium data x vs. y , which can be derived by using following equation.

$$y_i p_t = \gamma_i p_{vi} x_i$$

where, γ_i = Activity coefficient can be determined by van Laar equations. van Laar constants A = 1.978, B = 1.401.

In Table 8.60, "1" stands for ethanol and "2" for benzene.

Data:

- (i) Feed: 100 kmol/h contains 10% ethanol and 90% benzene (by mole)
Feed is saturated liquid
- (ii) Distillate: It contains 44% ethanol (by mole)
- (iii) Residue: 99.9% benzene (by mole)
- (iv) VLE and enthalpy data at 760 torr:

Table 8.60 VLE Data and Enthalpy Data for Ethanol-Benzene System

Mole Fraction of ethanol x_1	y_1^*	Temperature $t, ^\circ\text{C}$	Enthalpy of mixture, kJ/kmol	
			\bar{H}_L	\bar{H}_G
0	0	79.7	-15 870	19 090
0.01	0.075	78.0	-15 910	18 380
0.023	0.150	76.0	-16 120	17 580
0.045	0.217	74.0	-16 410	17 170
0.095	0.305	71.0	-17 170	16 540
0.200	0.395	69.0	-18 630	15 910
0.300	0.425	68.4	-20 050	15 740
0.447	0.447	68.2	-22 150	15 570

- 8.6 1000 kg of feed containing 30% by mass ethylene glycol and 70% by mass water, is to be separated in batch distillation with rectification column. Bottom product must contain 98.8% by mass of ethylene glycol. Composition of distillate should remain constant throughout the batch distillation. Desired composition of distillate is 98% by mass water.
- (a) Design the rectification column.
 - (b) Determine the operating (actual) reflux ratio vs time data.
 - (c) Compare the resulting data of this example (batch distillation with rectification with the resulting data of Example 8.12 (simple batch distillation)).
- Operating pressure in rectification column is 30.4 kPa a (maximum). Vapour liquid equilibrium data for ethylene glycol–water system at 30.4 kPa a are given in Example 8.12.
- 8.7 Chloroform and methanol form a minimum boiling azeotrope at 101.325 kPa and 53.5°C containing 65.17 mole % chloroform. Using van Laar equations, generate t - x - y relationship and plot y vs x diagram.

Table 8.61 Antoine Equation Constants¹³

Component	Antoine Constants			Normal Boiling Point at 101.325 kPa, $^\circ\text{C}$
	A	B	C	
Chloroform	14.5014	2938.55	-36.9972	61.15
Methanol	16.4948	3593.39	-35.2249	64.65

$$\text{Antoine equation: } \ln p_v = A - \frac{B}{(T + C)}$$

where, p_v = Vapour pressure, kPa

T = Saturation temperature, K

- 8.8 Formic acid and water form a maximum boiling azeotrope at 101.325 kPa and 107.1°C, containing 43.3 mole % water. Using van Laar equations generate t - x - y relationship and plot y vs x diagram.

Table 8.62 Antoine Equation Constants¹³

Component	Antoine Constants			Normal Boiling Point at 101.325 kPa °C
	A	B	C	
Formic acid	6.7067	1699.20	-12.45	100.8
Water	7.0436	1636.90	-48.25	100.0

$$\text{Antoine equation: } \log p_v = A - \frac{B}{(T + C)}$$

where, p_v = Vapour pressure, kPa

T = Saturation temperature, K

- 8.9 In Example 8.19, refrigerant R-134a was selected. Consider use of refrigerant R-22 (chlorodifluoromethane) or refrigerant R-717 (ammonia) as alternate auxiliary heat transfer mediums. Properties of R-22 and R-717 are given below.

Table 8.63 Properties of R-22²³

Temperature °C	Pressure bar a	Enthalpy, kJ/kg			Vapour Cp/Cv
		Liquid	Latent	Vapour	
2.0	5.315 (satd.)	202.36	202.65	405.01	1.296
72.5	31.51 (satd.)	296.44	118.15	414.59	2.147
130.0	32.0 —	—	—	478.7	—

(Reference state: Enthalpy of saturated liquid at 0°C = 200 kJ/kg.)

Assume 10°C approach in the overhead condenser and reboiler for R-22.

Table 8.64 Properties of R-717²³

Temperature °C	Pressure bar a	Enthalpy, kJ/kg			Vapour Cp/Cv
		Liquid	Latent	Vapour	
7.0	5.54 (satd.)	232.55	1236.31	1468.86	1.415
72.5	35.06 (satd.)	558.85	923.10	1481.95	1.834
130.0	35.50 —	—	—	1692.34	—

(Reference state: Enthalpy of saturated liquid at 0°C = 200 kJ/kg.)

Ammonia evaporator is normally a flooded one. Therefore, assume approach of 5°C in the overhead condenser and approach of 10°C in the reboiler.

Calculate required circulation rates of R-22 and R-717 for the same heat duties. Also review the performances of all three auxiliary heat transfer mediums and critically evaluate system requirements, including environment friendliness.

- 8.10 Extractive distillation of 50 mass % aqueous isopropyl alcohol (IPA) is to be carried out with the help of aqueous 35 mass % CaCl₂ solution. Vapour pressure data of aqueous calcium chloride solution are given in Table 8.51. Calculate *t-x-y* data for the system and plot the equilibrium (*x*, *y*) curve.
- 8.11 In a refinery, a gas stream, having composition 6.84% ethane, 40.81% propane, 9.70% *i*-butane and 42.65% *n*-butane (by mole), is to be fractionated at a rate of 300 kmol/h in a column to separate LPG fraction⁴⁸. Feed gas is compressed, cooled

and flashed in the fractionating column at 9.7 bar a and 45°C. Overhead vapours from the column are taken to partial condenser from which C₂ and C₃-fractions are separated in gaseous form. Composition of the gaseous stream from the condenser is 28.25% ethane, 68.55% propane, 1.6% *i*-butane and 1.6% *n*-butane (by mole). Flow rate of overhead vapour is 68.52 kmol/h. It leaves the condenser at 9.4 bar a and 20°C. Saturated liquid from the condenser at 20°C is recycled as reflux to the column at the rate of 1.2 kmol/kmol vapours separated. Pressure drop in the overhead condenser may be considered negligible (< 10 kPa) for calculation purpose. Bottom residue (LPG fraction) is removed from the reboiler at 10 bar a. Use Fig. 6.20 for dew point and bubble point calculations.

- Make complete material balance calculations of the column.
- Determine minimum reflux ratio.
- Determine number of theoretical stages for the reflux ratio 1.2 kmol/kmol and the stage at which the feed should be introduced by FUG method.
- In Tables 8.65 and 8.66, enthalpies of liquid and gaseous hydrocarbons are given. Using these values, calculate the heat duties of the overhead condenser and the reboiler.
- Considering low temperature of the vapours (20°C), leaving the overhead condenser, use of ammonia (R-717) is proposed as an auxiliary heat transfer medium in the overhead condenser and the reboiler. Evaporation of ammonia at 15°C in the overhead condenser and condensation of ammonia at 65°C in the reboiler are fixed as design parameters. Consider conditions of ammonia at the compressor discharge as 30 bar a and 125°C. Properties of ammonia are given in Table 8.67. Calculate ammonia circulation rates, required in the overhead condenser and the reboiler. Comment on the results.

Table 8.65 Enthalpy of Liquid Hydrocarbons at Saturated Conditions⁴⁹ (in kJ/kmol)

Temperature, K	Ethane	Propane	<i>i</i> -Butane	<i>n</i> -Butane
290	304	1918	2521	2525
300	1840	3112	3939	3941
305.34 ^c	4154			
310	—	4353	5397	5391
320	—	5650	6896	6878
330	—	7015	8442	8404

^cCritical temperature of ethane

Reference state: Enthalpy of saturated liquid hydrocarbon at 0°C = 0 kJ/kmol

Table 8.66 Enthalpy of Gaseous Hydrocarbons⁴⁹ (in kJ/kmol)

Hydrocarbon	Temperature, K	Pressure, bar		
Ethane		1	2	3
	280	11 700	11 630	11 560
	300	12 730	12 680	12 620
	320	13 820	13 770	13 720
	330	14 380	14 330	14 280

(Contd.)

Table 8.66 (Contd.)

Hydrocarbon	Temperature K	Pressure, bar		
		5	6	8
Propane				
	290	17 785	17 615	—
	300	18 585	18 425	18 105
	320	20 205	20 085	19 825
	340	21 885	21 785	21 565
<i>i</i> -Butane		0.1	0.5	1.0
	280	21 935	21 835	21 695
	300	23 825	23 745	23 635
	320	25 825	25 755	25 665
	340	27 935	27 875	27 805
<i>n</i> -Butane		0.1	0.5	1.0
	280	23 545	23 425	22 845
	300	25 475	25 385	25 255
	305	—	—	—
	320	27 515	27 435	27 325
	340	29 655	29 575	29 485
				2.0
				3.0
				—
				—
				25 245
				26 875
				29 105

(Reference state: Enthalpy of saturated liquid hydrocarbon at 0°C = 0 kJ/kmol)

Table 8.67 Properties of Ammonia (R-717)²³

Temperature, °C	Pressure bar a	Condition	Enthalpy, kJ/kg		C_p/C_v
			Liquid	Vapour	
15	7.286	Saturated	270.2	1475.9	1.438
65	29.476	Saturated	518.6	1487.1	1.748
125	30.0	Superheated	—	1695.0	—

(Reference state: Enthalpy of saturated liquid ammonia at 0°C = 200 kJ/kg)

- 8.12 Tetrahydrofuran (also known as 1, 4-epoxy butane; formula: C_4H_8O) is widely used as a solvent and also as a reaction medium in chemical industry. Typical THF recovery method involves adsorption on activated carbon, followed by steam stripping of the bed. Resulting THF-water mixture is a dilute aqueous stream of THF which is required to be separated in pure components for reuse of THF.

THF forms a minimum boiling azeotrope at 63.5°C, containing 5.3% by mass of water at atmospheric pressure. For the separation of THF from the mixture, pressure swing distillation process⁵⁰ is used. Basic flow sheet of the process is shown in Fig. 8.61.

In the first column, THF-water mixture, containing 0.06 mole fraction THF is fed. Distillate D_1 from the top has near azeotropic composition (0.805 mole fraction THF). Reflux ratio of 0.26 kmol/kmol distillate is maintained in this column. Bottom product of the first column is pure water (in which THF content is 1×10^{-6} mole fraction).

Distillate D_1 is fed to the second column, operating at 7.908 bar a. At this pressure, THF-water mixture forms a minimum boiling azeotrop at 136°C, containing

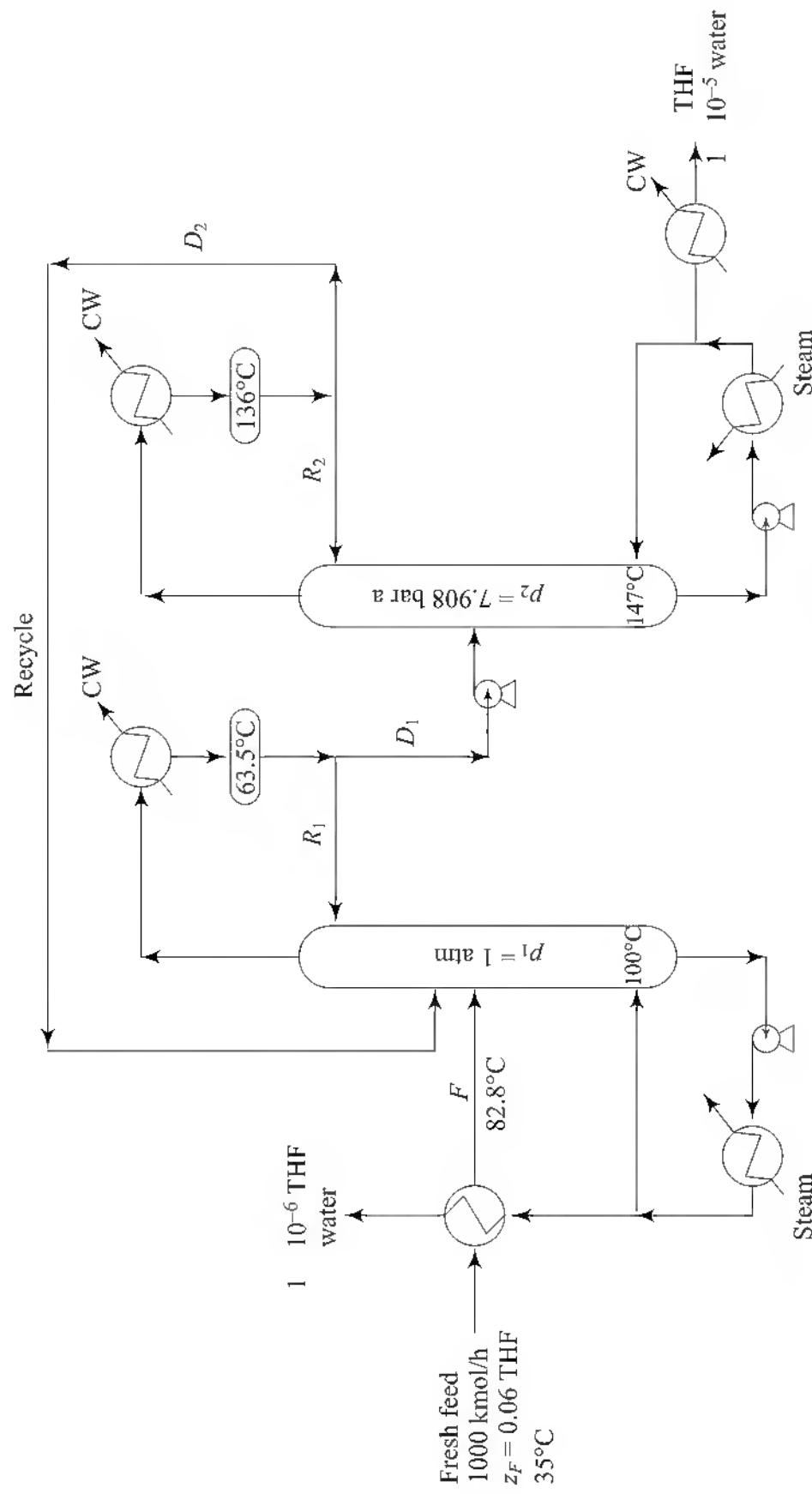


Fig. 8.61 Pressure Swing Distillation of THF - Water

Note: All the concentrations are expressed as mole fractions.

12% by mass water. Distillate D_2 from the top has near azeotropic composition (0.65 mole fraction THF) and is recycled to the first column. Bottom product from the second column is pure THF (in which water content is 1×10^{-5} mole fraction). Reflux ratio in the second column is maintained at 0.65 kmol/kmol distillate.

Assume that van Laar correlations represent non-ideality of THF-water mixtures in both the columns. Carryout tray-to-tray calculations for both the columns and fix feed locations of F and D_1 for fresh feed rate of 1000 kmol/h.

Table 8.68 Properties of THF and Water

	THF			Water		
Normal boiling point, °C	66			100		
Freezing point, °C	−108.5			0		
Density, kg/L	0.889			1.0		
Antoine constants	Temperature range, °C					
	60–150	60–70	70–80	80–90	90–100	100–150
A	6.995 15	7.1365	7.1095	7.0883	7.0733	7.0436
B	1202.29	1695.2	1678.9	1665.9	1656.4	1636.9
C	226.25	−42.75	−44.15	−45.35	−46.25	−48.25
Average latent heat of vaporization, λ , kJ/kmol	27 901			41 453		

$$\text{Antoine equations: } \log p_v = A - \frac{B}{(t + C)} \text{ for THF}$$

$$\log p'_v = A - \frac{B}{(T + C)} \text{ for water}$$

where, p_v = Vapour pressure of THF, torr

p'_v = Vapour pressure water, kPa

t = Saturation temperature, °C

T = Saturation temperature, K

- 8.13 A plant employs a batch reactor with refluxing arrangement (with cooling water) to manufacture amino resins. One of the products is melamine-formaldehyde (MF) resin. Melamine, aqueous formaldehyde (37% by mass) and *n*-butanol are the chief reactants. Phosphoric acid (98% pure, 0.5% by mass) is used as a catalyst. In a typical batch of 4 kL, average consumptions for a specific grade MF resin are reported to be 0.58 t *n*-butanol, 0.215 t melamine and 0.24 t formaldehyde (100%) per t MF product. Etherification and esterification reaction take place in the reactor at atmospheric pressure and in the temperature range of 90 to 105°C. Product water and water of aqueous formaldehyde solution are removed from the reactor in the form of an azeotrope with *n*-butanol. Condensed azeotrope separates in two layers; compositions of both the layers depend on the temperature. Butanol rich layer is recycled and aqueous layer at 40°C is collected in a separate tank for recovery of *n*-butanol by distillation. In the batch, 100% excess *n*-butanol is used for completion of reaction. At the end of reaction excess *n*-butanol is recovered by distillation. Batch is normally completed in 8 to 10 h.

Product contains up to 1.25% formaldehyde and 2 to 2.5% *n*-butanol (by mass). Since the reaction takes longer time, substantial crosslinking takes place which is more than desired.

It is decided to adopt a continuous system for production of MF resin as the product demand has shot up. For this purpose, process research was carried out in the laboratory. Use of strong cation exchange resin (up to 4% by mass), suitable for continuous operation at 140°C, was made as a catalyst instead of phosphoric acid. Results of the 20-litre flask showed that while consumptions of the reactants were nearly same as that of batch process (for the same grade), better quality product was produced in 3 h. MF resin so produced had less crosslinking and low formaldehyde (<0.5%) content. Further experiments with a glass column, packed with glass Raschig rings, confirmed that 3 theoretical stages for stripping are required for achieving bottom product (MF resin) with less than 1% *n*-butanol (by mass). Data on *n*-butanol-water azeotrope are covered in Sec. 8.7. Consider: (i) Excess *n*-butanol will be just sufficient to remove water in the form of the azeotrope and (ii) Residence time of reaction mass in the distillation column in contact with the resin will be 3 h.

Based on above information, develop a process flow diagram for the reactive distillation system for continuous production of MF resin along with desired instrument and control system with technical reasons. Consider whether pilot plant studies are required to implement the project commercially. Also study the advantages and disadvantages/limitations of proposed reactive distillation system over the batch system.

REFERENCES

1. Treybal, R. E., *Mass Transfer Operations*, 3rd Ed., McGraw-Hill, 1980.
2. Ludwig, E. E., *Applied Process Design for Chemical and Petrochemical Plants*, Vol. 2, 3rd Ed., Gulf Publishing Co., USA, 2002.
3. Smith, B. D., *Design of Equilibrium Stage Processes*, McGraw-Hill, USA, 1963.
4. Gmehling and Onken, *DECHEMA Chemistry Data Series*, DECHEMA, Germany Vol.1, 1997.
5. Chu, J. C. Wang, S. L., Levy, S. L. and Pual, R., *Vapour—Liquid Equilibrium Data*, Edwards Inc., USA., 1956.
6. Hala, E., Wichterle, I. and Lineks, J., *Vapour—Liquid Equilibrium Data Bibliography*, Elsevier, The Netherlands, 1976.
7. Hala, E., Wichterle, I., Polak, J. and Boublík, T., *Vapour-Liquid Equilibrium Data at Normal Pressure*, Pergamon, UK. 1968.
8. Gmehling, J., *Azeotropic Data*, 3 Vol., Wiley-VCH, Germany, 2004.
9. Sinnott, R. K. *Coulson and Richardson's Chemical Engineering*, Vol. 6, Revised 2nd Ed., Asian Books Pvt. Ltd. 1988.
10. McCade, W. L., Smith, J. C., Harriott, P., *Unit Operation of Chemical Engineering*, 6th Ed., McGraw-Hill, USA, 2001.
11. King, C. J., *Separation Processes*, 2nd Ed., McGraw-Hill, USA, 1980.
12. Deshpande, P. B., *Distillation Dynamics and Control*, Arnold, USA, 1985.
13. Bhatt, B. I. and S. M. Vora, *Stoichiometry*, 4th Ed., Tata McGraw-Hill Publishing Co. Ltd., New Delhi, 2004.
14. Perry, R. H. and Green D., *Perry's Chemical Engineer's Handbook*, 6th Ed., McGraw-Hill, USA 1984.

15. Kroschwitz, J. I. (Ed.) *Kirk and Othmer's Encyclopedia of Chemical Technology*, 4th Ed., Vol. 5, John Wiley, USA, 1993. p. 175.
16. Kister, H. Z., *Distillation Design*, McGraw-Hill, USA, 1992.
17. Fair J. R., *Chem. Engg.*, **70** (14), July 8, 1963, p. 119.
18. Van Winkle, M., *Distillation*, McGraw-Hill, 1967.
19. Billet, R., *Distillation Engineering*, Chemical Publishing Co., USA, 1979.
20. Mukherjee, S., *Chem. Engg.*, **112** (9), 2005, p. 52.
21. Fischer, W., Saminar on "Short Path Distillation", Mumbai, 1996.
22. Kukla, N., *Chemical Plant Processing*, 1996, p. 74.
23. Bhatt, B. I., *Design Databook: Properties of Steam, Selected Refrigerants' n-Hexane and Brines*, CBS Publishers and Distributors, New Delhi, 2007.
24. Degarmo J. L., Parwleker V. N., Pinjala V, *Chem. Engg. Progr.*, **86** (3), 1992, p. 45.
25. Mahajani S. M., Kolah A. K., *Ind. Engg. Chem. Res.*, **35**, 1996, p. 4587.
26. Grogins, *Unit Process in Organic Synthesis*, McGraw-Hill, USA.
27. Jacobs R., Krishna R., *Ind. Engg. Chem. Res.*, **32**, 1993, p. 1706.
28. Snessby M. G., Tade M. J., Datta R., Smith T. N., *Ind. Engg. Chem. Res.*, **36**, 1997 p. 1855.
29. Bhanvase, B. A., Y. R. Soman and R. V. Naik, *Chem. Engg. World*, **41** (2), p. 43, 2006.
30. Coates, J., *Chem. Engg.*, **67** (10), May 16, 1960, p. 121.
31. Norman, W. S., *Absorption, Distillation and Cooling Towers*, Longmans, USA, 1961.
32. Cho, J. and Jong-Ki Jeon, *Korean J. Chem. Engg.*, **23** (1), 2006, p. 1.
33. Mcketta, J. J. and W.A. Cunningham, *Encyclopedia of Chemical Processing and Design*, Vol. 5, Marcel Dekker, Inc., USA, 1977, p. 257.
34. Private Communication with Dr. Jen-Uwe Repke, TU Berlin, Germany.
35. Repke, Jen-Uwe and A. Klein, *Proceedings of the 15th European Symposium on Computer Aided Process Engineering*, (Ed. L. Puigjaner and A. Espuna), Elsevier Science B.V., 2005.
36. Repke, Jen-Uwe, F. Forner and A. Klein, *Chem. Engg. & Tech.*, **28** (10), 2005, p. 1151.
37. Knapp, P. J. and M. F. Doherty, *Ind. Engg. Chem. Res.*, **31**, 1992, p. 346.
38. Levy, S. G., D. B. Van Dongen and M. F. Doherty, *Ind. Engg. Chem. Fundam.*, **24**, 1985, p. 463.
39. *International Critical Tables*, 1st Ed., Vol. III, McGraw-Hill, USA, 1928, p. 292.
40. Humprey, J. L. and Seibert, A. F., *Chem. Engg. Progr.*, **88** (3), March, 1992, p. 32.
41. Finn, A. J., *Chem. Engg. Progr.*, **89** (10), October, 1993, p. 41.
42. H. Becker, S. Godorr, H. Kreis and J. Vaughan, *Chem. Engg.*, **108** (1), January 2001, p. 68.
43. Meszaros, I. and A. Meili, *Hydrocarbon Processing*, **81** (3), March 2002, p. 51,
44. Meili, A. and A. Stuecheli, *Chem. Engg.*, **94** (2), February 16, 1987, p. 133.
45. Meili, A. and A. Stuecheli, *Chem. Engg. Progr.*, **89** (5), 1993, p. 49.
46. Heist, J. A., *Chem. Engng.*, **86** (10), May 7, 1979, p. 72.
47. Private Communication with Dr. Ming Tham, University of Newcastle Upon Tyne, UK.
48. Kaiser V. and J. P. Gourlia, *Chem. Engg.*, **92** (17), August 19, 1985, p. 45.
49. Younglove, B. A. and J. K. Ely, *J. Phy. Chem. Ref. Data*, **16** (4), 1987, p. 577.
50. Samir, I., Abu-Elshah and W. L. Luyber, *Ind. Engg. Chem., Process Des. Dev.*, **24** (1), 1985, p. 132.



Process Design of Absorbers

9.1 INTRODUCTION

Absorption is an important unit operation of chemical process industries. In absorption a gas mixture (or a gas–vapour mixture) is contacted with a suitable liquid called solvent to separate one or more components of the gas mixture by dissolving them in the liquid. Hence, after the absorption, components of gas mixture are transferred to the solution. Dissolved component in the solution can be separated by distillation or stripping (reverse of absorption). For example, in the manufacturing of synthesis gas for ammonia from the gas mixture containing nitrogen, hydrogen, carbon dioxide and traces of other components, carbon dioxide is removed or separated by absorption in ethanol amine solution. Then carbon dioxide from the resulting solution is separated by stripping. “Absorber” and “scrubber” are sometimes used as synonyms. Truly speaking, if a component of the gas mixture, transferred to liquid, is a gas at operating conditions of mass transfer, then it is called absorption. If a component transferred is vapour at operating conditions then it is called scrubbing. Below critical temperature, a gas is known as vapour (which could be saturated or superheated). For example, separation of acetone vapour from air. Acetone vapour mixture in contact with water as solvent is called scrubbing. Operating conditions of this scrubber is atmospheric pressure and ambient temperature. Critical temperature of acetone ($T_c = 508.1\text{ K}$) is well above the ambient temperature. Absorption or scrubbing is chiefly carried out in a packed tower, although plate (tray) columns are not uncommon. Various types of equipment used as absorbers or scrubbers are:

- (i) Packed tower
- (ii) Tray tower
- (iii) Falling film absorber (Shell and tube heat exchanger)
- (iv) Spray chamber
- (v) Venturi scrubber
- (vi) Stirred tank

9.2 CRITERIA OF SELECTION

9.2.1.1 In majority of applications, packed tower is selected because of the following reasons.

- (i) Tower diameter required for the majority of the applications is less than 0.75 m. Packed tower is generally cheaper than tray tower when tower diameter is less than 0.75 m.
- (ii) Many absorption or scrubbing systems are corrosive systems. For corrosive systems plastic packed towers (for example FRP packed towers) with plastic packing are cheaper than metallic tray towers.
- (iii) In majority of absorption with chemical reaction systems, overall rate of gas – liquid reaction is totally controlled by rate of mass transfer (i.e., rate of chemical reaction $>>$ rate of mass transfer). Packed tower provides higher rate of mass transfer due to higher interfacial area of contact compared to other equipments.
- (iv) For the liquids having high foaming tendency and also for a heat sensitive liquid, packed tower is a better choice than tray tower.
- (v) For removing obnoxious gases from the vent gases or from the flue gases by absorption, available pressure of feed gas, fed to absorber or scrubber, is usually very low; close to atmospheric pressure. Hence, for such a case, pressure drop in absorber is the main criteria for selecting the equipment. Packed tower provides less pressure drop compared to a tray tower. Hence for such applications, packed towers are preferred.

9.2.1.2 For removing sulphur dioxide (SO_2) from boiler flue gases in large scale plants, available pressure of flue gases is very low; close to the atmospheric pressure. For this case, even packed tower is not suitable because pressure drop offered by packed tower is normally greater than 100 mm WC. Normally for such an application spray tower or spray chamber is used. Alternatively, a venturi scrubber can be used which induces vacuum in the suction.

9.2.1.3 Tray towers are selected as absorber in the following cases.

- (i) In case of exothermic absorption, it can be improved by providing external cooling during absorption. Cooling coils can be more readily built into tray towers and liquid can readily be removed from the trays to be passed through coolers and returned, than from packed towers. Take for example absorption of acetaldehyde from acetaldehyde vapour–gas mixture by using ethanol–water solution as solvent. This absorption is exothermic and hence tray tower with intermediate coolers is used as absorber. Similarly, absorption of formaldehyde in water is exothermic which can be conveniently carried out in a plate column. Refer Example 9.5 and Figs 4.2 and 4.7. Absorption of carbon dioxide in ammonia plant is preferably carried out in a tray column which is exothermic in nature, although packed columns are also in use.

- (ii) For the absorption systems involving low liquid flow rate, tray towers are preferred to get the better gas–liquid contacting efficiency. Low liquid rate may lead to incomplete wetting of column packing in case of packed tower and can give lower contacting efficiency.
- (iii) If solids are present in liquid or gas then tray towers are preferred because they permit easier cleaning.

9.2.1.4 For highly exothermic absorption, e.g. absorption of hydrogen chloride gas in water and absorption of ammonia gas in water, more efficient heat removal system is required to get the higher efficiency and higher concentration of solution. For such applications, falling film absorbers are used. These are shell and tube heat exchangers, in which absorption is carried out inside the tubes while cooling medium is circulated on shell side. Design of a falling film absorber is dealt later in this chapter (Example 9.4).

9.2.1.5 Venturi scrubbers are normally preferred for removing particulate matter from a gas stream as opposed to absorbing soluble vapours from vapour–gas mixture. Venturi scrubber provides higher gas–liquid contact or higher absorption efficiency but at the expense of relatively large gas side and liquid side pressure drops and consequently it requires higher power consumption.

9.2.1.6 Stirred tanks and sparged towers are preferred for slow gas–liquid reactions. Normally, in cases of absorption with chemical reaction, solvents or liquid reactants are selected in such a way that they offer fast or very fast chemical reaction with the solute gas. Hence, these equipments are normally not used as absorbers.

9.3 DESIGN OF PACKED TOWER TYPE ABSORBER

Process design of packed tower type absorbers involve two major steps; (i) determination of tower diameter and (ii) determination of height of packing. Method for determining tower diameter is same for all types of packed tower absorbers. But methods for finding the height of packing are different for the following cases.

- (a) Physical absorption from dilute gas mixture in isothermal conditions
- (b) Physical absorption from concentrated gas mixture in isothermal conditions
- (c) Physical absorption in non-isothermal condition. Example: absorption of HCl or NH₃ in water
- (d) Absorption with chemical reaction. Example: absorption of chlorine in sodium hydroxide solution

9.3.1 Packed Tower Type Absorber for Physical Absorption

Example of an absorber for physical absorption from dilute gas mixture in isothermal condition is the absorption of acetone vapour in water from air–acetone

vapour mixture containing less acetone (say 6%). Step wise design for this case is as follows.

(i) From the given gas mixture data, find G_1 , G_s , y_1 and Y_1 .

where, G_1 = Molar flow rate of gas mixture, kmol/h

G_s = Molar flow rate of non solute gas mixture, kmol/h

y_1 = Mole fraction of solute in feed gas

$$G_s = G_1 (1 - y_1) \quad (9.1)$$

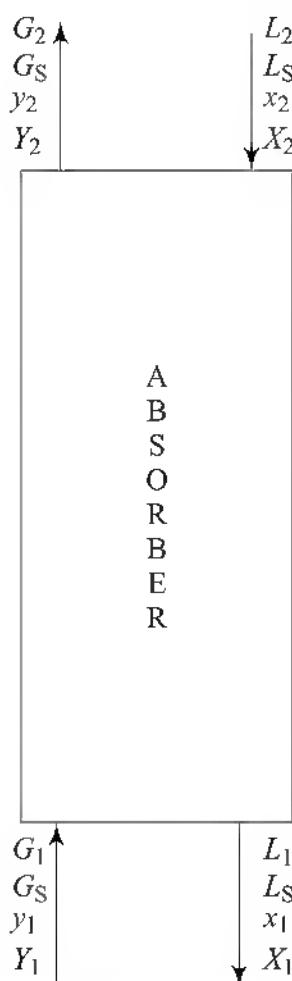


Fig. 9.1 Variables Used for Material Balance and Design

$$Y_1 = \frac{y_1}{1 - y_1}$$

Y_1 is the ratio of the moles of solute gas to the same of non-solute gas for incoming gas,

$$G_1 = G_s(1 + Y_1) \quad (9.2)$$

Decide the degree of absorption to be carried out in absorber, if it is not specified. Find y_2 , Y_2 and G_2 .

where, y_2 = Mole fraction of solute in outgoing gas mixture

$Y_2 = \frac{y_2}{1 - y_2}$, is the ratio of the moles of solute gas to non-solute gas for outgoing gas mixture

$$G_2 = G_s (1 + Y_2) \quad (9.3)$$

where, G_2 = Molar flow rate of outgoing gas mixture, kmol/h

If absorber is a pollution control tower then value of y_2 or Y_2 is decided by pollution control norms.

(ii) Select a suitable solvent. In the selection of solvent, the important properties to be considered are solubility of solute gas in the solvent, volatility, cost, corrosiveness, etc¹. Solvent which reacts with the solute by chemical reaction provides high solubility. If recovery of solvent or solute gas is essential then chemical reaction should be reversible. For example, for the absorption of CO₂ gas, caustic soda solution provides higher solubility or absorptivity than ethanol amine solution. But in actual application ethanolamine solution is selected as solvent rather than caustic soda solution because CO₂ gas can be easily separated from ethanolamine solution at higher temperature and lower pressure in a stripping column while caustic soda solution does not easily release CO₂ gas in stripping operation.

(iii) Find the gas–liquid equilibrium data. Draw the equilibrium curve in terms of Y vs X .

(iv) Find the minimum amount solvent required for the desired separation. Minimum amount of solvent is the maximum amount of solvent for which infinite number of equilibrium stages are required for the desired separation. In other words, it is the maximum amount of solvent for which desired degree of absorption is not possible. Minimum amount of solvent, in physical absorption is determined by graphical method.

(a) In this method first draw the equilibrium curve Y vs X .

(b) Mark (X_2, Y_2) point, where, X_2 = ratio of the moles of solute to moles of solvent for incoming solvent, Y_2 = ratio of the moles of solute to nonsolute gas for outgoing gas mixture.

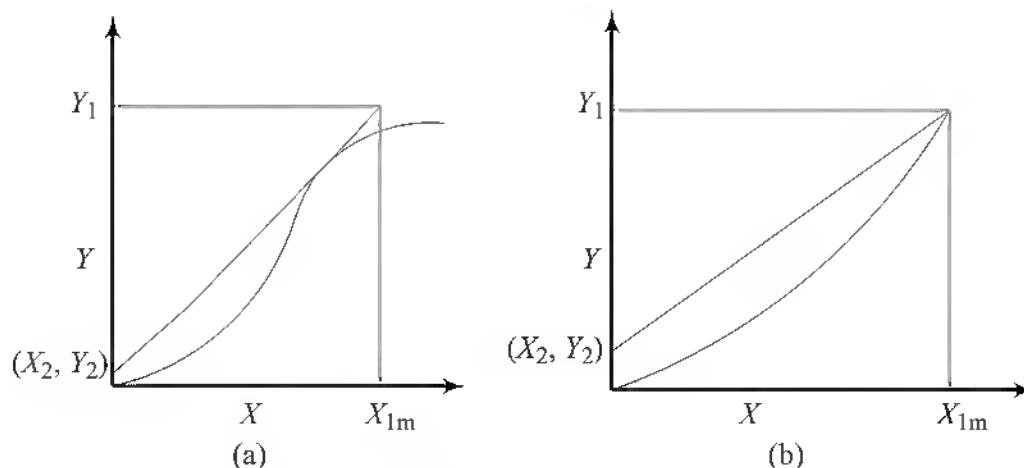
(c) Draw the tangent starting from point (X_2, Y_2) and allow it to intersect with horizontal line from Y_1 , if the equilibrium curve is concave upward [Fig. 9.2 (a)]. But if the curve is concave downward as shown in Fig. 9.2 (b), draw the line from (X_2, Y_2) and join it with the point of intersection of equilibrium curve and horizontal line from Y_1 .

In any case intersection point is represented as (X_{1m}, Y_1) . Where X_{1m} is the value of X_1 corresponding to minimum amount of solvent and X_1 is the ratio of the moles of solute to solvent in outgoing solution.

(d) Write the overall solute balance equation

$$L_{sm}(X_{1m} - X_2) = G_s (Y_1 - Y_2) \quad (9.4)$$

substitute the value of X_{1m} obtained from the graph and find the value of L_{sm} from the above equation.

**Fig. 9.2 Minimum Amount of Solvent**

(e) Decide the actual molar flow rate of solvent; L_s . Ideally it should be optimum. Optimum amount of solvent is the amount of solvent which gives minimum total cost of absorption. Total cost includes fixed cost, operating cost, interest on fixed capital, etc. At design stage, if it is not possible to find the optimum solvent flow rate, then actual flow rate L_s should be the greatest of the following values.

$$L_s = 1.5 L_{sm} \quad (9.5a)$$

$$L_s = \frac{1 \times \rho}{M_w} \quad (9.5b)$$

where, M_w = Molar mass of solvent

Certain minimum flow rate is required to facilitate the use of a centrifugal pump.

*Minimum wetting rate (MWR) is the liquid rate, required to form a liquid film over all the packing. No hundred per cent reliable method is available to find MWR. Some designers take minimum liquid rate for any tower packing as $7340 \text{ kg}/(\text{h} \cdot \text{m}^2 \text{ of tower cross section})^2$. Packing wetting rates are related to packing material surface

Table 9.1 Minimum Wetting Rate for Different Packings³

Surface	Minimum wetting rate (MWR), $\text{m}^3/(\text{m}^2 \cdot \text{h})$
Unglazed ceramic	0.5
Glazed ceramic	2.0
Glass	2.5
Stainless steel	3.0
Carbon steel	0.7
Polypropylene	4.0
Polyvinyl chloride	3.5
Polytetrafluoro ethylene (PTFE)	4.0

Morris and Jackson equation gives minimum solvent rate required in $\text{m}^3/(\text{h} \cdot \text{m}^2 \text{ of cross section of tower})$ by following equation^{2, 3}

$$L_s = \text{MWR. } a_t \quad (9.5c)$$

where, a_t = Packing surface area per unit volume, m^2/m^3 .

Table 9.2 Characteristics of Dumped (Randomly Packed) Tower Packings³

Packing type	Nominal size, mm	Wall thickness, mm	Outside diameter & length, mm	Approximate No. of elements per m ³	Approximate mass per m ³ , kg	Approximate surface area, a, m ² /m ³	Percent void space	Packing factor F _p , m ⁻¹
							ε	
Berl saddles, ceramic	6	—	—	3.78 × 10 ⁶	900	900	60	2950
	13	—	—	590 000	865	465	62	790
	25	—	—	77 000	720	250	68	360
	38	—	—	22 800	640	150	71	215
	50	—	—	8 800	625	105	72	150
Intalox saddles, ceramic	6	—	—	4.15 × 10 ⁶	865	984	75	2380
	13	—	—	730 000	720	625	78	660
	25	—	—	84 000	705	255	77	320
	38	—	—	25 000	670	195	80	170
	50	—	—	9 400	760	118	79	130
	75	—	—	1 870	590	92	80	70
Intalox saddles, metal	(No. 25)	—	—	168 400	—	—	97	135
	(No. 40)	—	—	50 100	—	—	97	82
	(No. 50)	—	—	14 700	—	—	98	52
	(No. 70)	—	—	4 630	—	—	98	43
Intalox saddles, plastic (polypropylene)	25	—	—	55 800	76	206	91	105
	50	—	—	7 760	64	108	93	69
	75	—	—	1 520	60	88	94	50
Pall rings,	16	26 gauge	16	—	—	—	92	230
	25	24	25	49 600	480	205	94	157
	38	22	38	13 000	415	130	95	92
	50	20	50	6 040	385	115	96	66
	90	90	90	1170	270	92	97	53

(Contd.)

Table 9.2 (Contd.)

Packing type	Nominal size, mm	Wall thickness, mm	Outside diameter & length, mm	Approximate No. of elements per m ³	Approximate mass per m ³ , kg	Approximate surface area, $a, \text{m}^2/\text{m}^3$	Percent void space ϵ	Packing factor F_p, m^{-1}
Pall rings, plastic (polypropylene)	16	—	16	214 000	116	340	87	310
	25	—	25	50 100	88	205	90	170
	38	—	38	13 600	76	130	91	105
	50	—	50	6 360	72	100	92	82
Raschig rings, ceramic	90	—	90	1 170	68	85	92	52
	6	1.6	6	3.02×10^6	960	710	62	5250
	13	2.4	13	378 000	880	370	64	2000
	19	2.4	19	109 000	800	240	72	840
Raschig rings, steel	25	3.2	25	47 700	670	190	74	510
	38	6.4	38	13 500	740	120	68	310
	50	6.4	50	5 800	660	92	74	215
	75	9.5	75	1 700	590	62	75	120
Hy-Pac, steel (No.1)	100	9.5	100	700	580	46	80	730
	19	1.6	19	111 000	1500	245	80	450
	25	1.6	25	46 300	1140	185	86	270
	38	1.6	38	14 100	785	130	90	187
Leva packing (No.2)	50	1.6	50	5 900	590	95	92	105
	75	1.6	75	1 800	400	66	95	141
	(No.1)	30	30	30 000	300	177	96	59
	(No.2)	60	3 780	225	95	97		
Low-density polyethylene Tellerettes	25			34 000	270	164		
				10 500	210	118		
				39 700	160	250	83	

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Values of MWR in m^2/h for rings less than 75 mm size is 0.08 and for packing more than 75 mm size it is 0.12.

Kister has suggested values of MWR in $\text{m}^3/(\text{m}^3 \cdot \text{h})$, 1.26 to 5.4 for random packings and 0.252 to 0.504 for structured packing⁴.

(f) Find the value of X_2 based on overall solute balance.

$$L_s(X_1 - X_2) = G_s(Y_1 - Y_2) \quad (9.6)$$

(g) Find the N_{OG} or N_{OL} .

where, N_{OG} = Number of overall gas phase transfer units

N_{OL} = Number of overall liquid phase transfer units

Height of packing required for the given absorption duty is determined by using one of the following equations.

$$Z = H_{OG} \cdot N_{OG} \quad (9.7)$$

or

$$Z = H_{OL} \cdot N_{OL} \quad (9.8)$$

If overall resistance to mass transfer is controlled by the gas film, then Eq. (9.7) is used to find the packing height Z for the desired absorption duty. If the gas is highly soluble in liquid (solvent), then major resistance to mass transfer is provided by the gas film. For example HCl (hydrogen chloride gas) is highly soluble in water. Hence, in this absorption, over all resistance to mass transfer is governed by hydrogen chloride gas film resistance. Another example of gas-film controlled absorption is absorption of acetone vapour in water from air-acetone vapour mixture. In the same case, if the concentration of solute gas in gas mixture is less than 10% (by mole) then molar flow rate of gas can be assumed as constant and height of packing (Z), is given by following equation.

$$Z = H_{OG} \cdot N_{OG} = \frac{G_m}{K_G a p} \int_{y_2}^{y_1} \frac{dy}{y - y_e} \quad (9.9)$$

where,

$$H_{OG} = \frac{G_m}{K_G a p} \quad (9.10)$$

and

$$N_{OG} = \int_{y_2}^{y_1} \frac{dy}{y - y_e} \quad (9.11)$$

where, G_m = Molar flow rate of gas per unit cross sectional area, $\text{kmol}/(\text{m}^2 \cdot \text{s})$

K_G = Over all gas phase mass transfer coefficient, $\text{kmol}/(\text{atm} \cdot \text{m}^2 \cdot \text{s})$

a = Interfacial surface area per unit volume, m^2/m^3 (Ref. Table 9.2)

p = Operating pressure, atm

H_{OG} = Height of an over all gas phase transfer units, m

N_{OG} = Number of an overall gas phase transfer units

N_{OG} can be determined by Eq. (9.11), for which area under the curve of $1/(y - y_e)$ vs y within limits from y_2 to y_1 is determined. In the special case, if equilibrium curve and operating line, both can be assumed as straight lines, N_{OG} can be calculated by equation⁴

$$N_{OG} = \frac{1}{1 - \left(\frac{mG_m}{L_m} \right)} \ln \left[\left(1 - \frac{mG_m}{L_m} \right) \frac{y_1}{y_2} + m \frac{G_m}{L_m} \right] \quad (9.12)$$

where m is the slope of the equilibrium line and G_m/L_m is the slope of the operating line.

If the overall resistance to mass transfer is controlled by liquid film then Eq. (9.8) is used to find the packing height. If the solubility of gas in liquid phase is very low then overall resistance to mass transfer is governed by liquid film resistance. For example; (i) absorption of carbon dioxide gas (CO_2) in water from CO_2 rich gas mixture and (ii) absorption of dimethyl ether in water. In such cases normally a chemical reagent is added into liquid phase which reacts with solute gas and thereby increases the rate of mass transfer dramatically. For example, for the absorption of CO_2 , potassium carbonate (K_2CO_3) or ethanol amine is added into water to improve the rate of the absorption of carbon dioxide (CO_2). In the same case, if the concentration of solute gas in gas mixture fed to absorber is less than 10% (by mole) or for the dilute system, molar flow rate of liquid can be assumed as constant and height of packing required can be calculated by the following equations.

$$Z = H_{OL} \cdot N_{OL} \quad (9.8)$$

where,

$$H_{OL} = \frac{L_m}{K_L a C_t} \quad (9.13)$$

and

$$N_{OL} = \int_{x_2}^{x_1} \frac{dx}{x_e - x} \quad (9.14)$$

where, L_m = Molar liquid flow rate per unit cross sectional area, $\text{kmol}/(\text{m}^2 \cdot \text{s})$

N_{OL} = Number of overall liquid phase transfer units

H_{OL} = Height of an overall liquid phase transfer unit, m

K_L = Overall liquid – phase mass transfer coefficient, m/s

a = Interfacial surface area per unit volume m^2/m^3

C_t = Total molar concentration, kmol/m^3

x_1 = Mole fraction of solute in the liquid phase at the outlet from absorber

x_2 = Mole fraction of solute in the liquid phase at the inlet to the absorber

x_e = Equilibrium mole fraction of solute in liquid phase

N_{OL} can be determined by Eq. (9.14), for which area under the curve of $\frac{1}{x_e - x}$

vs x within limits x_1 to x_2 should be determined.

(h) Determination of Tower Diameter:

For packed tower type absorber, tower diameter is determined based on flooding velocity. Actual velocity of gas through packed tower is kept about 60 to 70% of flooding velocity. Recommended range of pressure drop for packed tower type absorber is 15 to 50 mm water column per m of packing height depending on the application. At the time of flooding, one of two conditions may occur; (i) Liquid phase may occupy the entire cross section of tower. Continuous phase of liquid body rises in the column. The change in pressure drop is very high with only a slight change in gas rate. (ii) Phase inversion occurs and gas bubbles through the liquid. Pressure drop rises rapidly as phase inversion occurs. To find the tower diameter, at first the following factor is determined.

$$F_{LG} = \frac{L_w}{G_w} \sqrt{\frac{\rho_G}{\rho_L}} \quad (9.15)$$

where, L_w = Mass velocity of liquid, $\text{kg}/(\text{m}^2 \cdot \text{s})$
 G_w = Mass velocity of gas, $\text{kg}/(\text{m}^2 \cdot \text{s})$
 ρ_G = Density of gas, kg/m^3
 ρ_L = Density of liquid, kg/m^3

Using Fig. 9.3, corresponding value of K at flooding (K_F) is determined. Actual value of K is determined as follows.

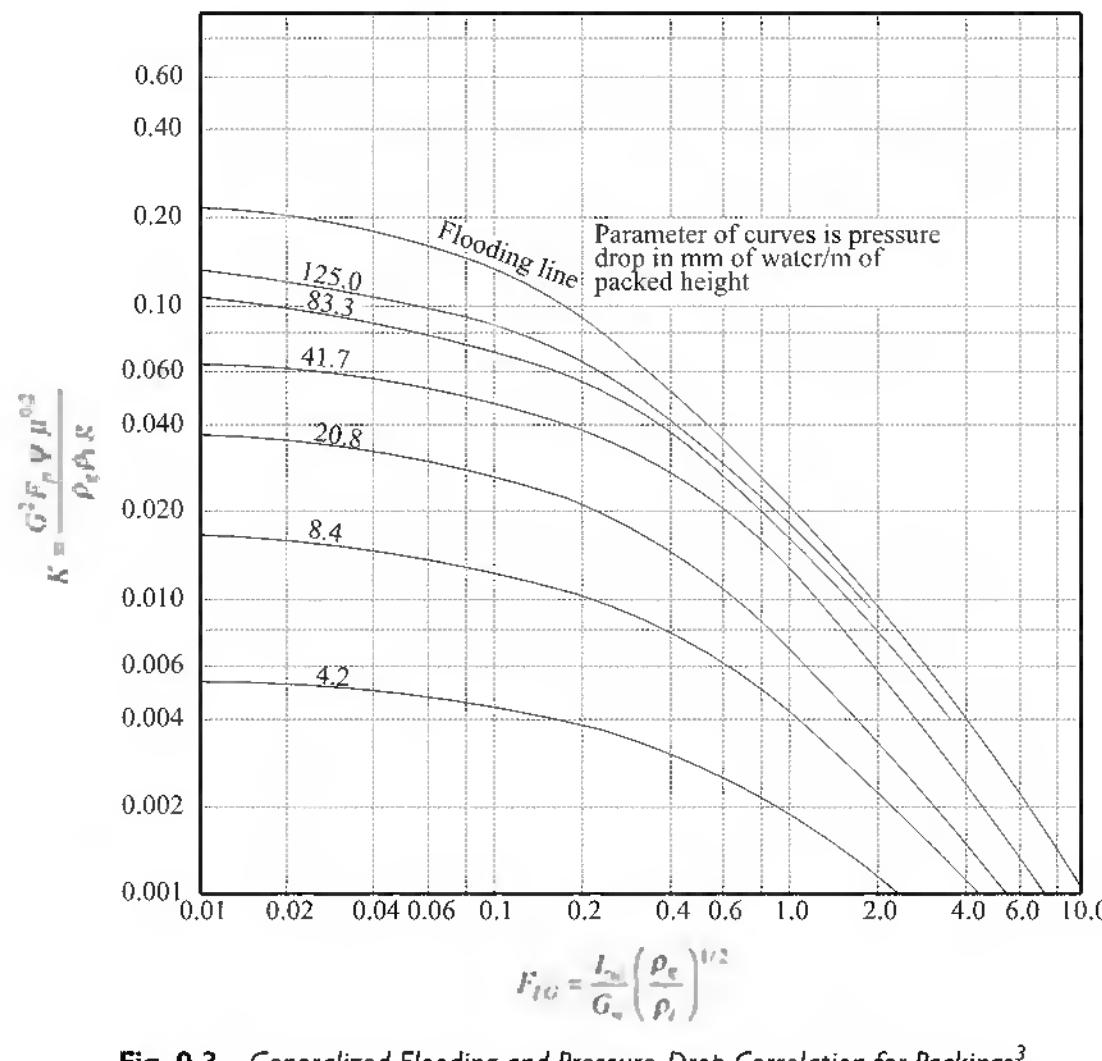


Fig. 9.3 Generalized Flooding and Pressure-Drop Correlation for Packings³
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Let the actual velocity of gas through tower be 66% of flooding velocity.

$$K = (0.66)^2 K_F \quad (9.16)$$

Corresponding value of pressure drop per m of packed height can be determined by using Fig. 9.3.

K is a function of G

$$K = \frac{G_w^2 F_p \psi \mu_L^{0.2}}{\rho_G \rho_L g} \quad (9.17)$$

where, G_w = Mass velocity of gas, $\text{kg}/(\text{m}^2 \cdot \text{s})$

K = Function of velocity of gas through tower

$\rho_G \rho_L$ = Density of gas and liquid, respectively, kg/m^3

$$G_w = \left(\frac{K \cdot \rho_G \rho_L g}{F_p \psi \mu_L^{0.2}} \right)^{1/2} \quad (9.18)$$

$g = 9.81 \text{ m/s}^2$

F_p = Packing factor, m^{-1} (Ref: Table 9.2)

ψ = Ratio of density of water to density of liquid

μ_L = Viscosity of liquid, $\text{m Pa} \cdot \text{s}$ (= cP)

(i) Determination of H_{OG} or H_{OL} :

Many different correlations are available to find out the height of transfer unit or to find out the mass transfer coefficients. But, these correlations are not as reliable as the same for finding the heat transfer coefficients. Hence, it is suggested to find out the height of transfer unit of mass transfer coefficient by using the correlation which is derived for the same or a similar system. But, if the correlation for the same or a similar system is not available, then, use more than one generalized correlations for finding the height of transfer unit (H_{OG} or H_{OL}) and the maximum value obtained from the different correleations is considered for finding the height of packing. Two generalized correlations are given as follows:

(a) Onda Takeuchi and Okumoto Correlation:³

Height of overall gas phase transfer unit H_{OG} and height of overall liquid phase transfer unit H_{OL} are related with H_G and H_L by following equation.

$$H_{OG} = H_G + m \frac{G_m}{L_m} H_L \quad (9.19)$$

$$H_{OL} = H_G + \frac{L_m}{m G_m} H_G \quad (9.20)$$

where, m is the slope of equilibrium line, G_m/L_m is the slope of operating line and G_m and L_m are the molar flow rates of gas and liquid, respectively per unit cross-sectional area of tower in $\text{kmol}/(\text{m}^2 \cdot \text{s})$. Height of gas phase transfer unit (H_G) is determined by following equation.

$$H_G = \frac{G_w}{K_G a_e p M_g} \quad (9.21)$$

and height of liquid phase transfer unit H_L is determined by following equation

$$H_L = \frac{L_w}{K_L a_e \rho_L} \quad (9.22)$$

where, G_w = Superficial mass velocity of gas, $\text{kg}/(\text{s} \cdot \text{m}^2)$

L_w = Superficial mass velocity of liquid, $\text{kg}/(\text{s} \cdot \text{m}^2)$

p = Operating pressure, Pa

M_g = Molar mass of gas

ρ_L = Liquid density, kg/m³

a_e = Effective area for mass transfer, m²/m³

K_G = Gas phase mass transfer coefficient, kmol/(m² · s · Pa)

K_L = Liquid phase mass transfer coefficient, m/s

Effective area for mass transfer are is obtained by following equation

$$\frac{a_e}{a_t} = 0.31 \frac{\sigma_L^{0.5}}{Z^{0.4}} (C_{aL} \cdot Re_G)^{0.392} \quad (9.23)$$

where, a_e = Effective area for mass transfer, m²/m³

a_t = Total area of packing, m²/m³ (Ref. Table 9.2)

σ_L = Liquid surface tension, m N/m (= dyn/cm)

Z = Packed height, m

$$C_{aL} = \text{Liquid capillary number} = \mu_L L_w / (\rho_L \sigma_L) \quad (\text{dimensionless group}) \quad (9.24)$$

ρ_L = Density of liquid, kg/m³

μ_L = Viscosity of liquid, mPa · s (= cP)

$$Re_G = \text{Gas Reynolds number} = \frac{6G_w}{a_t \mu_G} \quad (9.25)$$

G_w = Mass velocity of gas, kg/(m² · s)

μ_G = Gas viscosity Pa · s

Gas phase mass transfer coefficient is determined by the following equation:

$$\frac{K_G RT}{a_t D_G} = C_1 \left(\frac{G_w}{a_t \mu_G} \right)^{0.7} \left(\frac{\mu_G}{\rho_G D_G} \right)^{1/3} (a_t d_p)^{-2} \quad (9.26)$$

where, K_G = Gas phase mass transfer coefficient, kmol/(m² · s · Pa)

R = 8314 J/(kmol · K)

D_G = Gas phase diffusion coefficient, m²/s

T = Operating temperature, K

ρ_G = Density of gas, kg/m³, d_p = Packing size, m

C_1 = 2 for random packings of size less than 12 mm

= 5.23 for random packings of size more than 12 mm

Liquid phase mass transfer coefficient is determined by following equation.

$$K_L \left(\frac{\rho_L}{\mu_L g} \right)^{1/3} = 0.0051 \left(\frac{L_w}{a_t \mu_L} \right)^{2/3} \left(\frac{\mu_L}{\rho_L D_L} \right)^{-0.5} (a_t d_p)^{0.4} \quad (9.27)$$

μ_L = Liquid viscosity, kg/(m · s)

g = 9.81 m/s²

L_w = Mass velocity of liquid, kg/(m² · s)

D_L = Liquid phase diffusion coefficient, m²/s

d_p = Equivalent diameter of packing, m

a_w = Wetted surface of packing, m^2/m^3
 a_w is determined by following equation.

$$\frac{a_w}{a_t} = 1 - \exp \left[-1.45 \left(\frac{\sigma_C}{\sigma_L} \right)^{0.75} Re_L^{0.1} Fr_L^{-0.05} We_L^{0.2} \right] \quad (9.28)$$

where, σ_C = Critical surface tension of the packing material, m N/m
(Ref. Table 9.3)

σ_L = Surface tension of liquid, m N/m

$$Re_L = \text{Liquid Reynolds number} = \frac{L_w}{a_t \mu_L} \quad (\text{dimensionless})$$

$$Fr_L = \text{Froude number for liquid} = L_w^2 a_t / (\rho_L^2 g) \quad (\text{dimensionless})$$

$$We_L = \text{Weber number for liquid} = L_w^2 / (\rho_L \sigma_L a_t) \quad (\text{dimensionless})$$

Table 9.3 Critical Surface Tension of Packing Materials³

Material of Packing	σ_c , m N/m or dyn/cm
Carbon	56
Ceramic	61
Glass	73
Polyethylene	33
Polypropylene	33
Polyvinyl chloride	40
Steel	75

(b) Cornell's method

In this method height of overall gas phase transfer unit H_{OG} or height of overall liquid phase transfer unit H_{OL} is calculated by the same equations Eq. (9.19) and (9.20) that are used for Onda's method but H_G and H_L are determined by the following equations.

Height of gas phase transfer unit H_G is calculated by following equations.

For Raschig rings and Pall rings,

$$H_G = \frac{0.017 \psi D^{1.24} Z^{0.33} Sc_G^{0.5}}{(L_w f_1 f_2 f_3)^{0.6}} \quad (9.29)$$

For saddle type packings,

$$H_G = \frac{0.029 \psi D^{1.11} Z^{0.33} Sc_G^{0.5}}{(L_w f_1 f_2 f_3)^{0.5}} \quad (9.30)$$

where H_G = Height of gas phase transfer unit, m

$$Sc_G = \text{Gas phase Schmidt number} = \frac{\mu_G}{\rho_G D_G} \quad (\text{dimensionless})$$

D_G = Gas phase diffusion coefficient, m^2/s

ρ_G = Density of gas, kg/m^3

μ_G = Viscosity of gas, $\text{kg}/(\text{m} \cdot \text{s})$ or $\text{Pa} \cdot \text{s}$

D = Column diameter, m

Z = Packed height, m

L_w = Mass velocity of liquid, $\text{kg}/(\text{m}^2 \cdot \text{s})$

$$f_1 = \left(\frac{\mu_l}{\mu_w} \right)^{0.16} \quad \text{with } \mu_w = 1.0 \text{ mPa} \cdot \text{s}$$

$$f_2 = \left(\frac{\rho_w}{\rho_L} \right)^{1.25} \quad \text{with } \rho_w = 1000 \text{ kg}/\text{m}^3$$

$$f_3 = \left(\frac{\sigma_w}{\sigma_L} \right)^{0.8} \quad \text{with } \sigma_w = 72.8 \text{ m N/m}$$

ψ = Correlation parameter can be obtained from Fig. 9.4 for various types of random packings.

Height of liquid phase transfer unit is calculated by following equation.

$$H_L = \frac{\phi C}{3.28} \left(\frac{\mu_L}{\rho_L D_L} \right)^{0.5} \left(\frac{Z}{3.05} \right)^{0.15} \quad (9.31)$$

where, H_L = Height of liquid phase transfer unit, m

ϕ = Correlation parameter for a given packing, m (ϕ can be obtained from Fig. 9.5)

C = Correlation factor for high gas rates which can be obtained from Fig. 9.6

μ_L = Liquid viscosity Pa.s

ρ_L = Liquid density kg/m^3

D_L = Liquid diffusion coefficient, m^2/s

Z = Height of packing, m

In this method, if the column diameter (D) is greater than 0.6 m then take $D = 0.6$ m for the calculation of H_G .

(j) Design or selection of internals of packed tower:

Internals of packed tower are packings, packing support, liquid distributor, liquid redistributor, mist eliminator, hold-down plate, etc.

(I) *Suitable packing must be selected before the determination of tower diameter.* Packings can be divided in two broad classes.

(i) Regular packings: Grids, structured packings, stacked rings, etc. are of these type. Grids are used for high gas rates as they offer very low pressure drop. They are used in cooling towers. Structured packings are widely used in chemical process industries. They are made from corrugated sheets with some perforations or wire mesh. They provide high surface area with high void fraction. HETP (Height Equivalent to Theoretical Plate) of structured packing is generally less than 0.5 m.

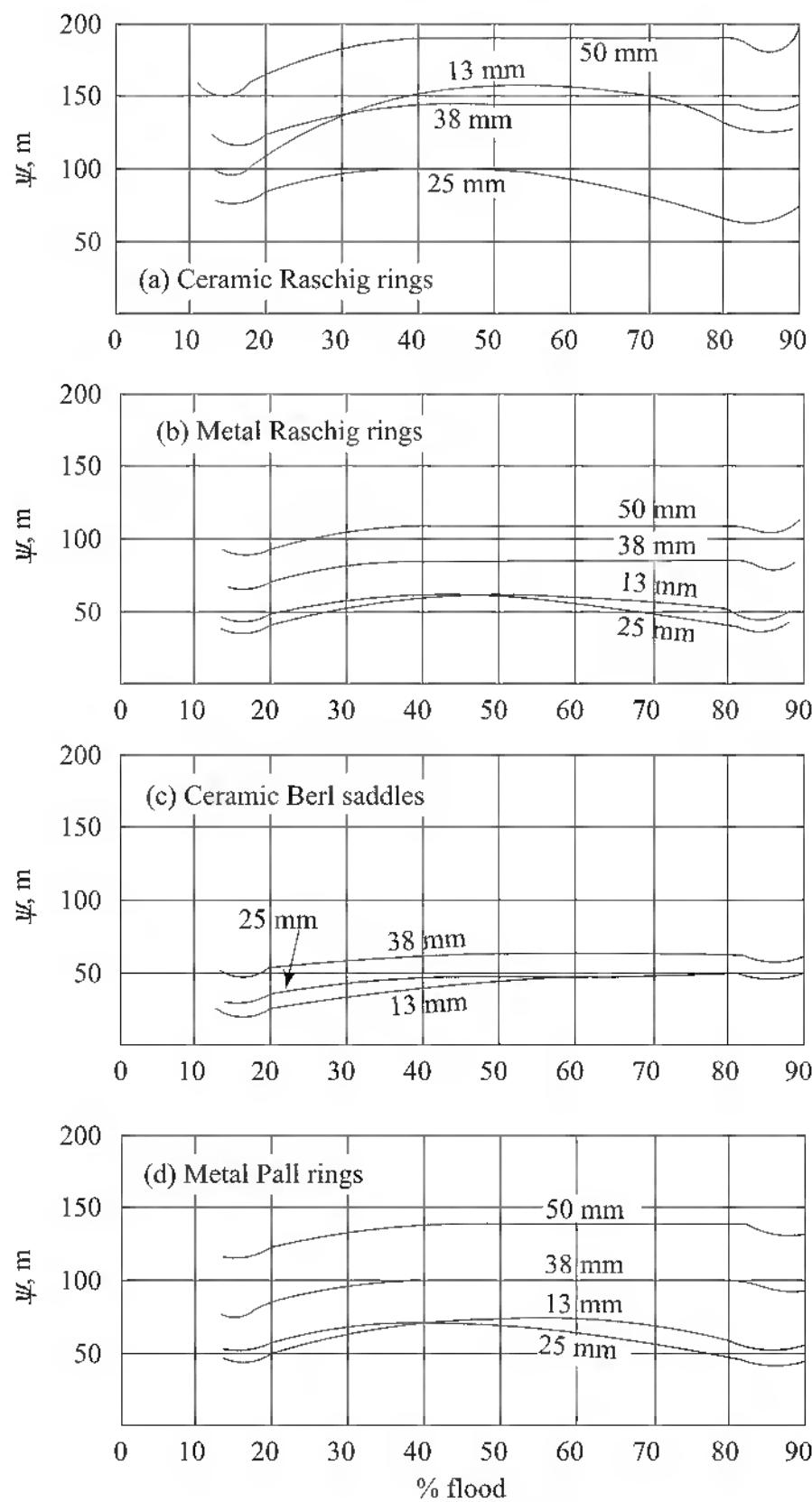
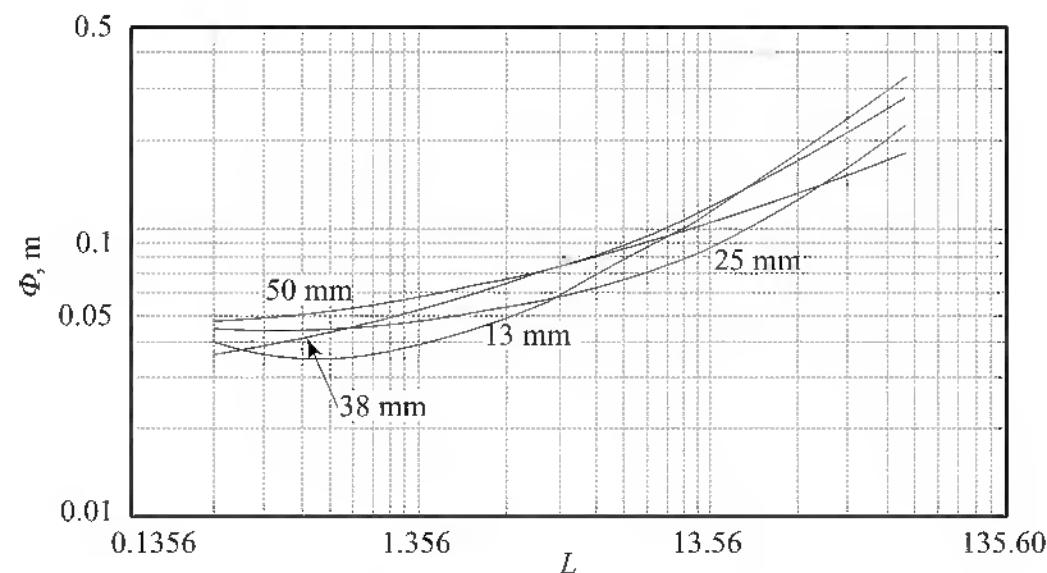
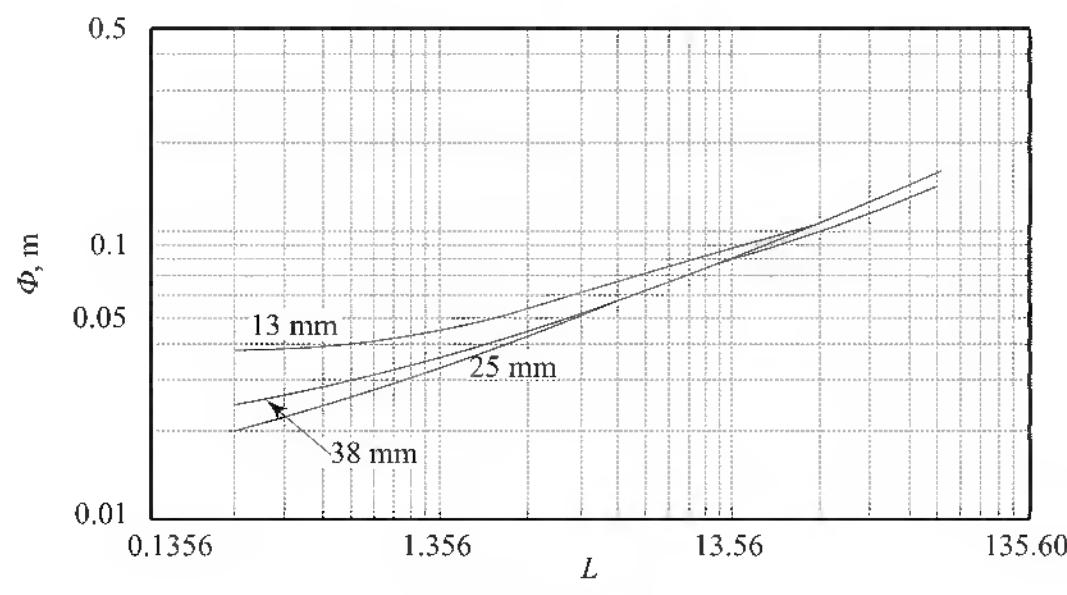


Fig. 9.4 Correlation Parameter ψ for Cornell's Method³
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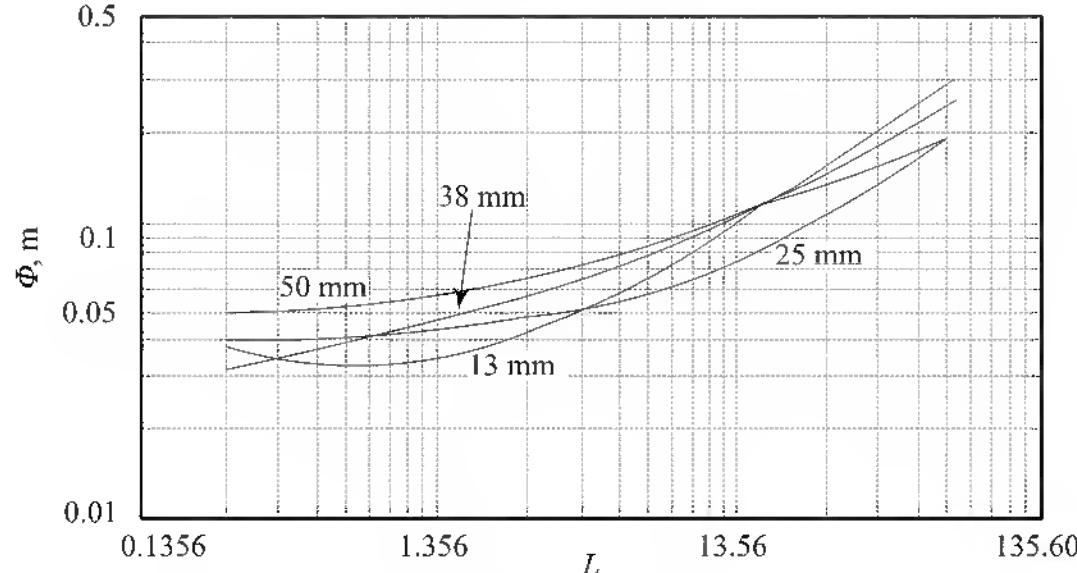
They are produced by many manufacturers. They are preferred for difficult distillation operations, for high vacuum distillation, for high efficiency absorption with low allowable pressure drop and also for increasing the capacity of existing



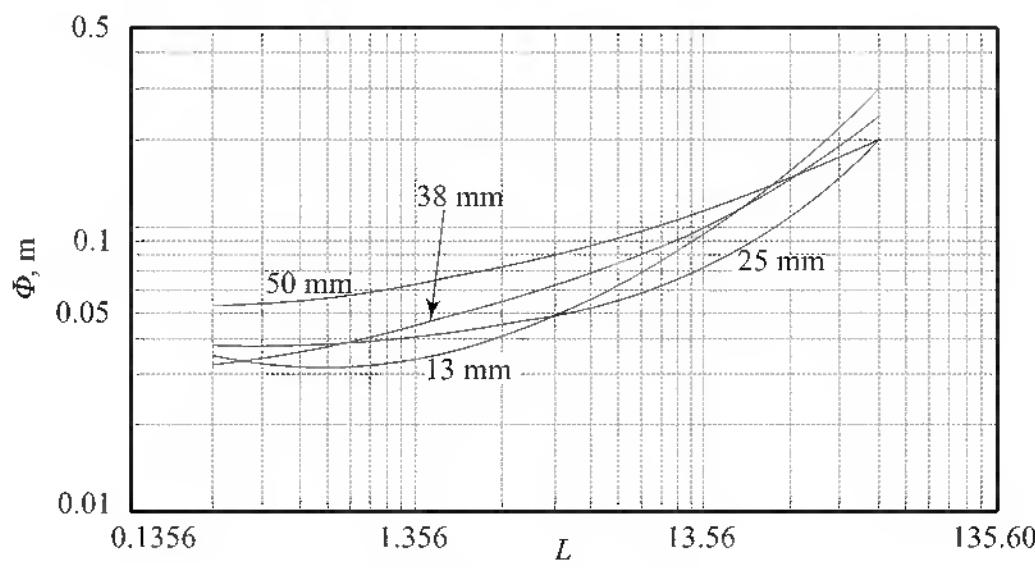
(a) Ceramic Raschig Rings



(b) Ceramic Berl Saddles



(c) Metallic Raschig Rings



(d) Metal Pall Rings

 $L = \text{Liquid Rate, kg/(m} \cdot \text{s}^2\text{)}$

Fig. 9.5 Correlation Parameter for Various Packings³
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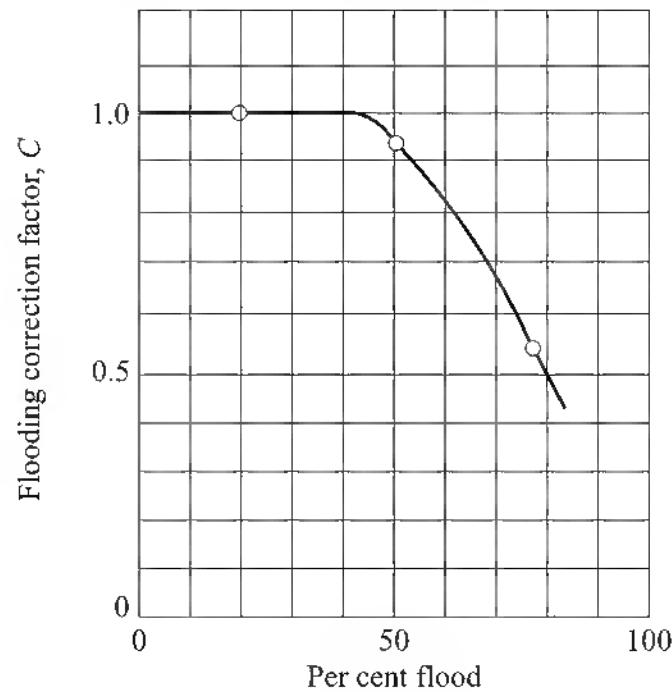
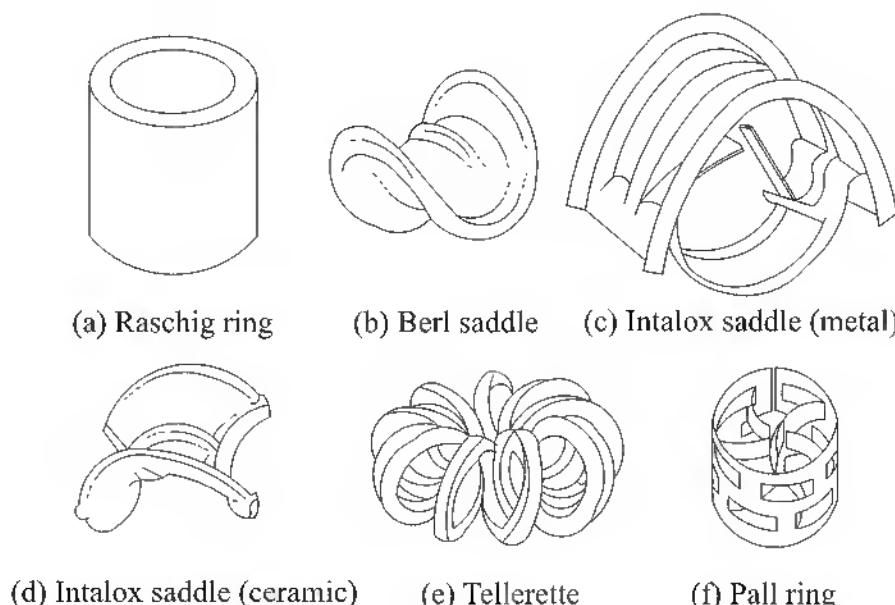


Fig. 9.6 Flooding Correction Factor, C^3
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distillation column. Necessary data, correlations or graphs for calculating tower diameter can be obtained from the manufacturer.

(ii) Random Packings:

Various types of random packings are used with packed towers. Most commonly used are Pall rings, Berl saddles, Intalox saddles, Raschig rings, Tellerettes, Hy-Pac, etc. Design data of these packings are given Table 9.2.

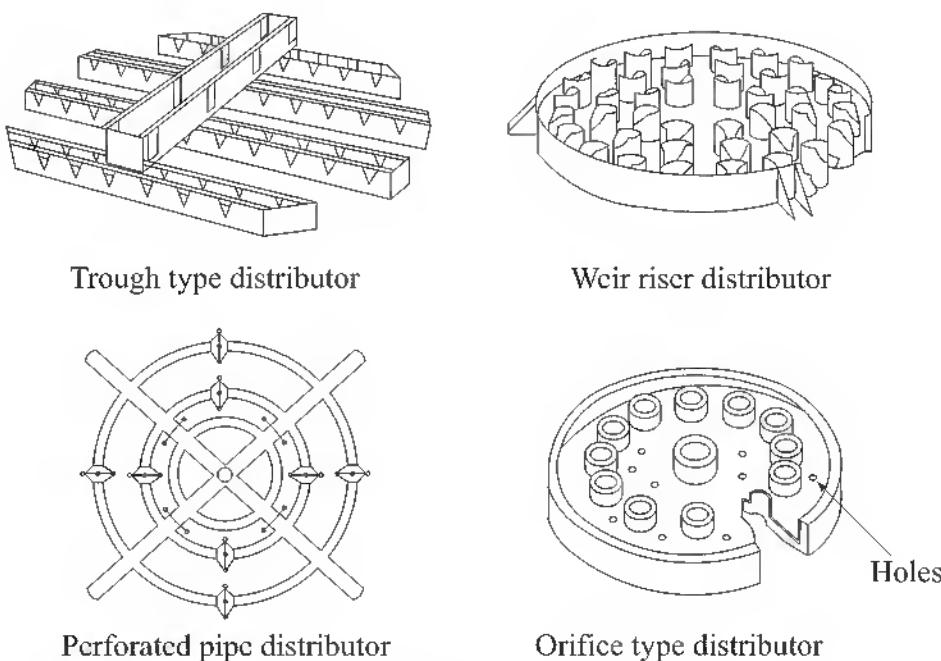
**Fig. 9.7 Random Packings**

Pall rings are the preferred and commonly used random packings but their cost per unit volume is high. Pall rings require minimum diameter and minimum height for the given absorption or distillation duty as compared to other types of random packings. Pall rings are available in metals and plastics. For high temperature services polyethylene or polypropylene Pall rings cannot be used. Plastic packings are also attacked by some organic solvents. For corrosive services ceramic Berl saddles are cheaper than metallic Pall rings. For example in distillation of the mixture of phosphorous trichloride and phosphorous oxychloride, packed tower with ceramic packing is used. Ceramics are brittle at high temperature and have poor strength. For very high temperature and corrosive services glass packed columns with glass Raschig rings are preferred against packed column of special alloy. For example, in vacuum distillation (for extractive distillation) of concentrated nitric acid using concentrated sulfuric acid as solvent, it is carried out in glass packed tower with glass Raschig rings. Raschig rings are the cheapest packing material but are less efficient. Raschig rings require the maximum diameter and height for the given absorption or distillation duty compared to other types of random packings. Hence, total fixed cost of packed column is higher. Diameter of tower is recommended to be more than 10 times the packing size.

(II) Liquid distributors:

Various types of liquid distributors, used with packed tower, are shown in Fig. 9.8. Uniform initial distribution of liquid at the top of packed bed is essential for the efficient mass transfer operation.

For small diameter packed columns (having diameter less than 0.3 m) single point distributor like one spray nozzle is adequate. For large diameter packed columns, multi point distributors like perforated pipe distributor, trough type distributor, orifice distributor, etc. are used. As per the Perry's Handbook³, for each 194 cm^2 cross sectional area one distribution point is required. As per the Eckert's

**Fig. 9.8** Different Types of Liquid Distributors

recommendations, number of streams or number of distribution points required in packed tower are given as follows:

Table 9.4 Number of Distribution Points in Packed Tower³

Column diameter, m	No. of points/m ²
0.4	340
0.75	170
≥ 1.2	40

Perforated pipe distributors are preferred for column diameter ranging from 0.3 m to 1 m. It is used with clean liquids and offers minimum restriction to gas flow. They are preferred with reasonably constant flow rate.

Trough type distributors are preferred for columns having diameter greater than or equal to 1.2 m. It can be used with liquids having solid in suspension. Trough type distributors are fabricated from metal sheets, plastics or ceramics. This distributor consists of a series of troughs containing side notches. It provides good liquid distribution with a large free area for gas flow.

Orifice type liquid distributor is also preferred for large diameter packed column. This type of distributor consists of flat perforated tray equipped with a number of risers or short stand pipes. Ideally gas rises upward through risers or short stand pipes, while liquid maintains the certain level over the perforated plate and flows down through perforations. The risers should be sized to give sufficient area for gas flow without creating a significant pressure drop. The holes (perforations) should be small enough to ensure that there is a level of liquid on the plate at the lowest liquid rate but large enough to prevent the distributor overflowing at the highest rate.

In **weir type distributor** gas and liquid (both) are flowing through the same stand pipes. Notched weirs are provided in upper portion of stand pipes. Certain level of liquid is maintained over the tray and liquid flows over notched weirs and falling down through the same pipes from which gas rises upward. Weir type distributor is preferred with fluctuating liquid flow rates.

(III) Liquid redistributor:

After travelling a certain distance in a packed tower, considerable fraction of liquid is migrated to the column wall and flows down in the vicinity of column wall while the gas rises upwards through the central portion. This phenomena is called channelling. Liquid redistributor collects the liquid that has migrated to the column wall and redistributes it evenly over the next bed of the packing.

For small diameter coloumn ($D \leq 0.6$ m) wall wiper type liquid redistributor is preferred. It collects liquid from the column wall and redistributes it into the central portion. Sometimes packing support plate itself acts as liquid redistributor. Different types of liquid redistributors are shown in Fig. 9.9.

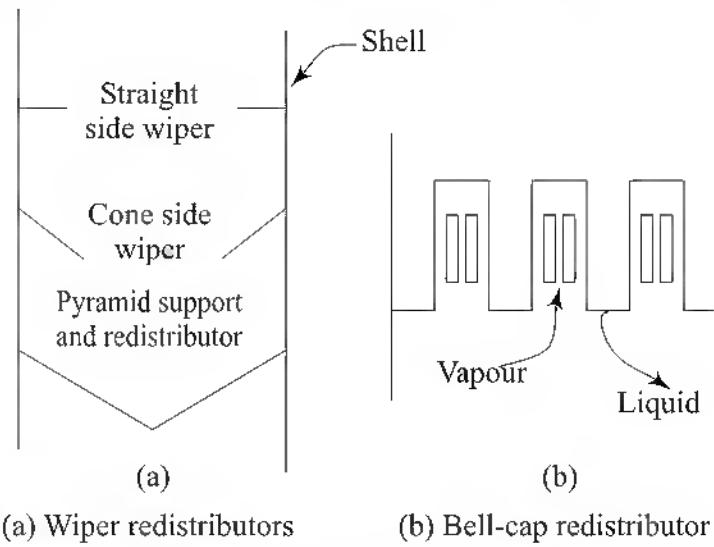


Fig. 9.9 Different Types of Liquid Redistributors

(IV) Packing support:

The function of packing support is to carry the load of wet packing bed without providing excessive restriction to gas and liquid flows. It also acts as distributor for both streams. Poor design of packing support provides higher pressure drop and can cause premature local flooding. Two types of packing supports are used. (Fig. 9.10)

(i) Countercurrent type: Example; Mesh type packing support

(ii) Separate flow or gas injection type: Example; Cap type packing support, beam type gas injection support, etc.

In counter current type packing support liquid and gas both are flowing through the same openings in counter current direction. Major open area of counter current type packing support is occluded by packing pieces resting on it. Hence, this type of packing support provides the low value of net free area (50% or less). Column diameter is decided based on the characteristics of packing material. Berl saddles, Raschig rings, etc. provide the lesser free area for flow of gas,

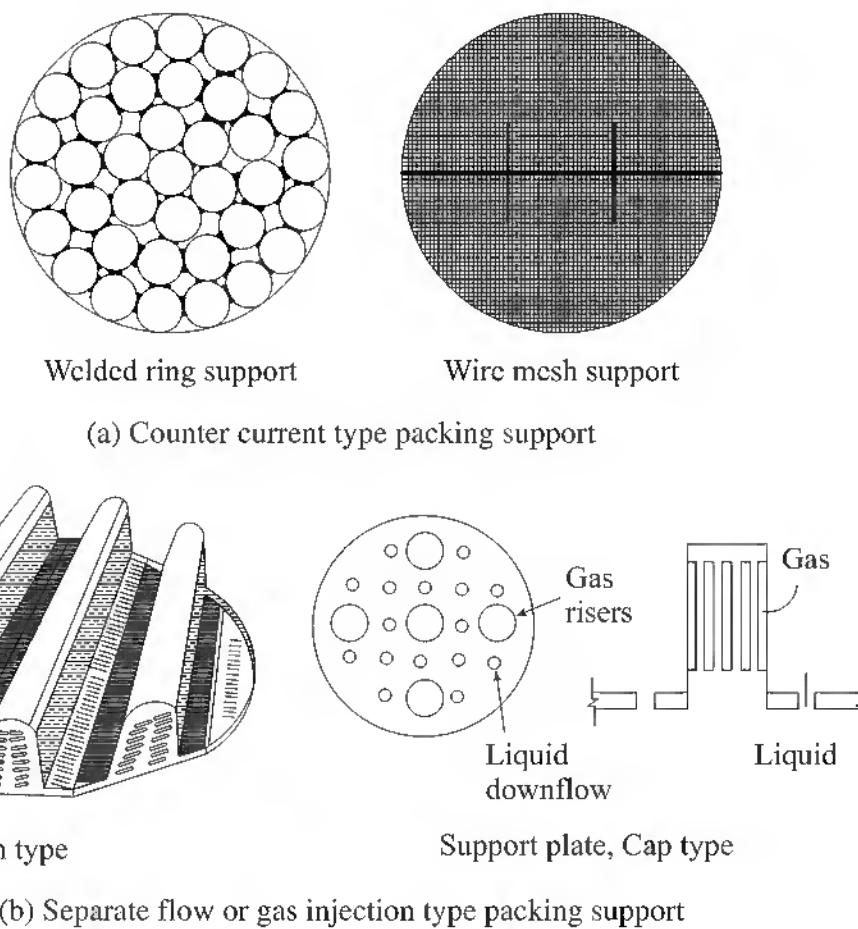


Fig. 9.10

hence with such packings, counter current type packing support can be used. But other packings like Pall rings, structured packings, etc. provide higher % free area for the flow of gas (85% or more). Hence, with these type of packings, counter flow type packing support cannot be used. Gas injection type packing support can be designed for free area up to 90% and because of their geometry there will have very little occlusion by the packing. In this type of packing support separate flow passages or openings are provided for gas and liquid streams. Gas inlets are provided above the liquid level. With packing materials like Pall rings, structured packings, etc. gas injection type packing support is recommended.

(V) Hold-down Plate:

Hold-down plate is required to prevent damage to the packing which can result due to a breakdown in normal operating conditions. At high gas or vapour flow rates, packing at the top can be fluidized. This may result in breaking or deshaping of packing. Ceramic packings can be easily broken which may settle in voids of the packed bed and plug the flow channels. In case of metal packings, deshaping may take place and the deformed pieces can plug the flow channels. In case of plastic packings, they may fly away with gas or vapour and seat at various locations in the column. Also plastic packing may break and seat randomly in the tower.

Hold down plate, generally in the grid form or the screen form, can be used to prevent such fluidization. While heavier hold down plates are used for ceramic

and metal packings, lighter hold down plates of similar construction are used for packed towers with plastic packing. Hold down grids/screens have generally poor open area (< 50% of internal cross-section of the tower). However, size of opening in the grid/plate are fixed in such a manner that fly off of the packing material can be prevented.

Example 9.1

Design a scrubber for absorbing acetone vapour from air-acetone vapour mixture by using pure water as solvent. The temperature in the scrubber is 25°C and scrubbing is isothermal. Operating pressure of scrubber is near atmospheric. A mixture of air with acetone vapour containing 6% by volume of acetone is passed through the scrubber. This mixture contains 1400 m³/h of air. The scrubber is required to absorb 98% of the acetone.

Solution:

Refer Fig. 9.1.

Let G_s = Molar flow rate of air, kmol/h.

G_1 = Molar flow rate of incoming air-vapour mixture, kmol/h

y_1 = Mole fraction of solute (vapour) in incoming air-vapour mixture

$$Y_1 = \frac{y_1}{1 - y_1} \frac{\text{kmol of vapour}}{\text{kmol of air}}$$

G_2 = Molar flow rate of outgoing air-vapour mixture, kmol/h

y = Mole fraction of vapour in outgoing air-vapour mixture

$$Y_2 = \frac{y_2}{1 - y_2} \frac{\text{kmol of acetone}}{\text{kmol of air}}$$

x_2 = Mole fraction of solute in incoming solvent = 0

$$X_2 = \frac{x_2}{1 - x_2} = 0 \frac{\text{kmol of acetone}}{\text{kmol of water}}$$

$L_2 = L_s$ = Molar flow rate of solvent at top, entering the scrubber, kmol/h

L_1 = Molar flow rate of solution leaving the scrubber, kmol/h

x_1 = Mole fraction of acetone in liquid solution leaving the absorber

$$X_1 = \frac{x_1}{1 - x_1} \frac{\text{kmol of acetone}}{\text{kmol of water}}$$

Flow rate of air G_s = 1400 m³/h at 1 atm and 25°C

$$\text{Density of air } \rho_A = \frac{pM}{RT} = \frac{1 \times 29}{0.08206 \times (273 + 25)}$$

$$\rho_A = 1.1859 \text{ kg/m}^3$$

$$G_s = \frac{1400 \times 1.1859}{29} = 57.25 \text{ kmol/h}$$

$$y_1 = 0.06 \text{ (mole \% = volume \%)}$$

$$Y_1 = \frac{y_1}{1 - y_1} = 0.063\ 83 \frac{\text{kmol of acetone}}{\text{kmol of air}}$$

Scrubber is required to absorb 98% of acetone.

$$y_2 = 0.02 \times y_1 = 0.0012, \quad Y_2 = \frac{y_2}{1 - y_2} = 0.001\ 201\ 44 \frac{\text{kmol of acetone}}{\text{kmol of air}}$$

$$X_2 = 0, \quad L_s = ?$$

L_s : To find the value of L_s , first the minimum amount of solvent required L_{sm} for the desired separation is to be determined.

Phase equilibrium equation

$$y = mx = \frac{p_v}{p_t} x$$

At 25°C, vapour pressure of acetone⁴

$$\ln p_v = 16.6513 - \frac{2940.46}{T - 34.93} \quad (\text{Antoine equation})$$

where, p_v = Vapour pressure of acetone at 25°C, torr
 T = Temperature, K

$$\ln p_v = 16.6513 - \frac{2940.46}{(273 + 25) - 35.93}$$

$$p_v = 228.416 \text{ torr}$$

$$m = \frac{228.416}{760} = 0.3$$

$$y = 0.3 x \quad \text{and} \quad \frac{Y}{1+Y} = 0.3 \frac{X}{1+X}$$

Table 9.5 Equilibrium Data Calculations

y	$Y = \frac{y}{1-y}$	$x = \frac{y}{m}$	$X = \frac{x}{1-x}$
0	0	0	0
0.0012	0.001 2014	0.004	0.004 016
0.01	0.0101	0.033	0.034 12
0.02	0.0204	0.067	0.0718
0.03	0.0309	0.1	0.111
0.04	0.041 67	0.133	0.1534
0.05	0.052 63	0.167	0.2
0.06	0.063 83	0.2	0.25

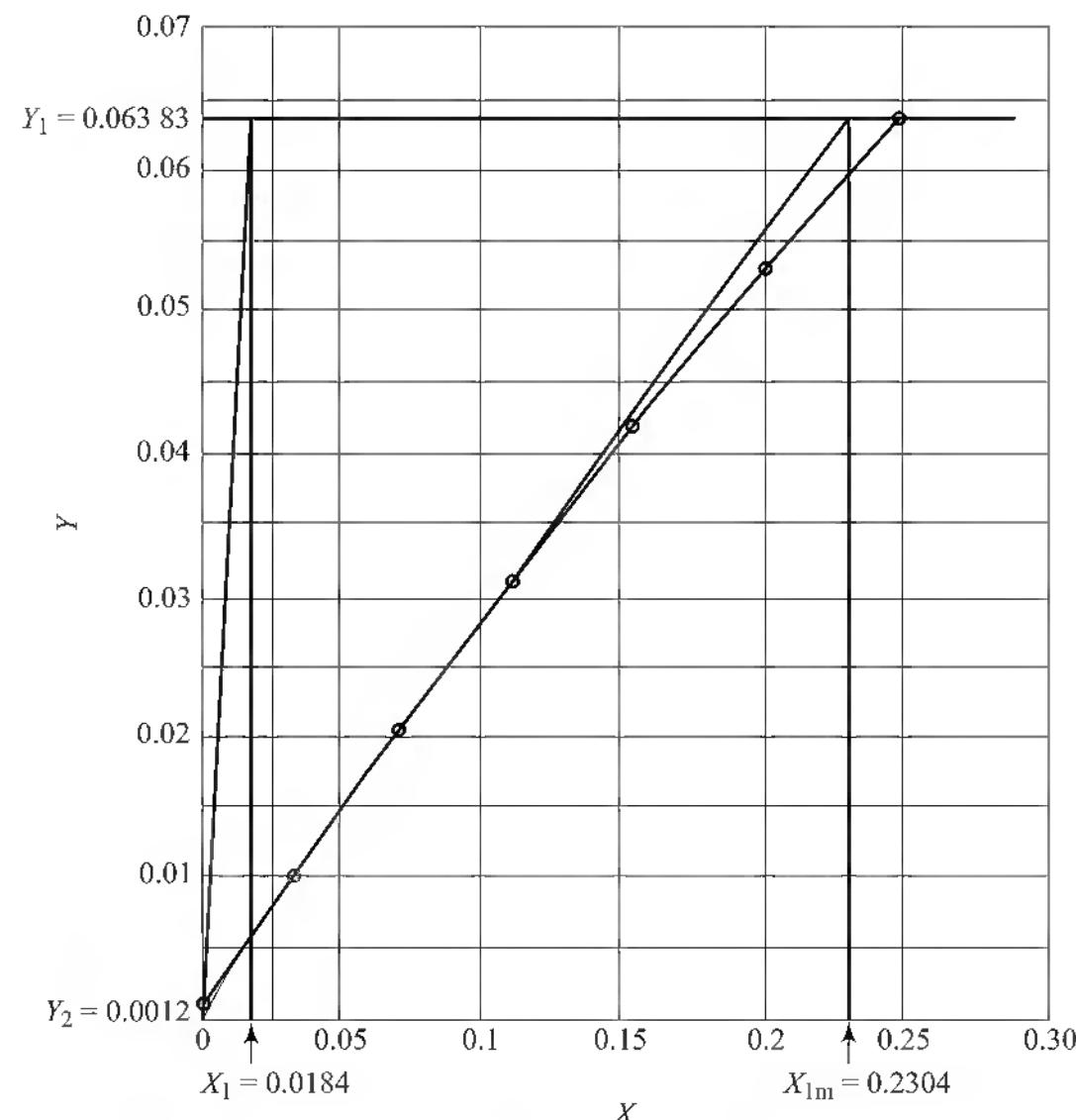


Fig. 9.11 Determination of Minimum Amount of Solvent

Solute balance at minimum solvent rate

$$G_s(Y_1 - Y_2) = L_{sm}(X_{1m} - X_2)$$

$$57.22(0.06383 - 0.00120144) = L_{sm}(X_{1m} - 0)$$

where L_{sm} = Minimum amount of solvent required, kmol/h

X_{1m} = Value of X_1 corresponding to the minimum amount of solvent

Value of X_{1m} can be obtained from the graph. Here, the curve of Y vs X (equilibrium curve; Fig. 9.11) is concave downward, hence the tangent is drawn from (X_2, Y_2) point and is allowed to intersect the horizontal line at Y_1 .

Intersection point is (X_{1m}, Y_1)

$$X_{1m} = 0.2304 \text{ (from Fig. 9.11)}$$

$$57.22(0.06383 - 0.00120144) = L_{sm}(0.2304 - 0)$$

$$L_{sm} = 15.55 \text{ kmol/h}$$

Actual amount of solvent should be the greatest of the following values.

$$(i) L_s = 1.5 L_{sm} = 23.33 \text{ kmol/h } L_2 \equiv 23.33 \times 18 \equiv 419.94 \text{ kg/h}$$

- (ii) $L_s = \frac{1 \times 1000}{18} = 55.55 \text{ kmol/h}$, i.e. about $1 \text{ m}^3/\text{h}$ (minimum flow rate required to facilitate the use of a centrifugal pump)
- (iii) L_s = Minimum wetting rate required to form a liquid film over all the packing. But minimum wetting rate required can be calculated after finding the tower diameter. Tower diameter can be determined after deciding the actual amount of solvent. Hence, it requires trial and error calculations.

For the first trial let $L_s = 55.55 \text{ kmol/h} = 1000 \text{ kg/h}$

Tower diameter required at top

$$F_{LG} = \frac{L_w}{G_w} \sqrt{\frac{\rho_G}{\rho_L}} \quad (9.15)$$

$$L_w = \text{Mass velocity of liquid } \text{kg}/(\text{m}^2 \cdot \text{s}) = \frac{1000/3600}{(\pi/4)D^2}$$

$$L_w = 0.2778 / \left(\frac{\pi}{4} D^2 \right)$$

$$G_M = \text{Mass velocity of gas, } \text{kg}/(\text{m}^2 \cdot \text{s})$$

Molar flow rate for gas – vapour mixture at the top

$$G_2 = G_s (1 + Y_2) = 57.25 (1 + 0.00120144) = 57.3189 \text{ kmol/h}$$

Average molar mass of gas – vapour mixture at the top

$$M_{av} = \sum (M_i y_i)$$

$$M_{av} = 0.012 \times M \text{ of acetone} + 0.9988 \times M \text{ of air}$$

$$M_{av} = 0.0012 \times 58 + 0.9988 \times 29$$

$$M_{av} = 29.0348 \text{ kg/kmol}$$

$$G_w = \frac{(57.3189 \times 29.0348)/3600}{(\pi/4)D^2} = \frac{0.462}{(\pi/4)D^2} \text{ kg}/(\text{m}^2 \cdot \text{s})$$

Density of vapour gas mixture at top

$$\rho_G = \frac{p M_{av}}{RT} = \frac{1 \times 29.0348}{0.082057 \times (273 + 25)} = 1.187 \text{ kg/m}^3$$

$$F_{LG} = \frac{L_w}{G_w} \sqrt{\frac{\rho_G}{\rho_L}} = \frac{0.2778}{0.462} \times \sqrt{\frac{1.187}{1000}} = 0.02072$$

From Fig. 9.3, $K_F = 0.2$

Let actual velocity of gas = 66% of folding velocity

$$\sqrt{\frac{K}{K_F}} \times 100 = 66\%$$

$$K = (0.66)^2 \times 0.2 = 0.08712$$

For $K = 0.08712$ and $F_{LG} = 0.02072$

Pressure drop/m of packing height = 75 mm H₂O/m of packing (From Fig. 9.3)

Mass velocity of gas through tower

$$G_w = \left(\frac{K \rho_G \rho_L g}{F_p \psi \mu_L^{0.2}} \right)^{1/2} \quad (9.18)$$

$$\psi = \frac{\text{Density of liquid}}{\text{Density of water}} = 1, \rho_L = 1000 \text{ kg/m}^3$$

$$g = 9.81 \text{ m/s}^2$$

Viscosity of water at 20°C, $\mu_L = 1 \text{ cP} = 10^{-3} \text{ kg/(m} \cdot \text{s)}$

Select 25 mm polypropylene Pall rings as packing,

Packing factor $F_P = 170 \text{ m}^{-1}$

(Ref. Table 9.2)

$$G_w = \left(\frac{0.08712 \times 1.187 \times 1000 \times 9.81}{170 \times 1 \times 1^{0.2}} \right)^{1/2}$$

$$= 2.4428 \text{ kg/(m}^2 \cdot \text{s})$$

$$\text{Tower area required at top} = \frac{\text{Mass flow rate of gas - vapour mixture}}{G_w}$$

$$= \frac{(57.3189 \times 29.0348)/3600}{2.4428} = 0.1892 \text{ m}^2$$

Tower diameter required at top = 0.49 m ≈ 0.5 m

$$\frac{\text{Tower diameter}}{\text{Packing size}} = \frac{350}{25} = 14 > 10 \quad (\text{satisfactory})$$

$$\text{Liquid rate} = \frac{1}{\frac{\pi}{4}(0.50)^2} = 5.09 \text{ m}^3/(\text{m}^2 \cdot \text{h})$$

Hence, it is greater than $4 \text{ m}^3/(\text{m}^2 \cdot \text{h})$ (minimum wetting rate required for polypropylene surface)

$$\text{Liquid rate} = \frac{1000}{\frac{\pi}{4}(0.50)^2} = 5093 \text{ kg/(h} \cdot \text{m}^2)$$

It is less than $7340 \text{ kg/(h} \cdot \text{m}^2)$, minimum wetting rate specified in literature (Ref. 2).

Based on Morris and Jackson's equation, minimum solvent required

$$L_{\min} = \text{MWR} \cdot a_t \quad (9.5c)$$

For 25 mm polypropylene poll rings $a_t = 205 \text{ m}^2/\text{m}^3$ (From Table 9.2)

For 25 mm polypropylene Pall rings MWR = 0.08

$$L_{sm} = 0.08 \times 205 = 16.4 \text{ m}^3/(\text{m}^2 \cdot \text{h})$$

Considering the last method for predicting minimum wetting rate, let $L_s = 3500 \text{ kg/h} = 194.4 \text{ kmol/h}$

$$\text{At top } F_{LG} = \frac{3500/3600}{0.462} \times \sqrt{\frac{1.187}{1000}} = 0.0725$$

From Fig. 9.3, $K_F = 0.17$

Let actual velocity = 66% of flooding velocity

$$K = (0.66)^2 \times 0.17 = 0.074$$

Pressure drop = 66 mm H₂O/m of packing

Mass velocity of gas through tower

$$G_w = \left(\frac{K \rho_G \rho_L g}{F_p \psi \mu_L^{0.2}} \right)^{1/2} = \left(\frac{0.074 \times 1.187 \times 1000 \times 9.81}{170 \times 1 \times 1^{0.2}} \right)^{1/2} \\ = 2.2514 \text{ kg/(m}^2 \cdot \text{s)}$$

Tower area required at top, $A_1 = \frac{0.462}{2.2514}$

$$A_1 = 0.2052 \text{ m}^2 = \left(\frac{\pi}{4} \right) D^2$$

$$D = 0.511 \text{ m} \approx 0.52 \text{ m}$$

$$\text{Liquid rate} = \frac{3500}{\frac{\pi}{4}(0.52)^2} = 16480.54 \text{ kg/(m}^2 \cdot \text{h}) \equiv 16.48 \text{ m}^3/(\text{m}^2 \cdot \text{h})$$

Hence it satisfies the conditions.

Tower diameter required at the bottom:

Average molar mass of gas-vapour mixture

$$M_{av} = \sum M_i y_i = y_1 \times 58 + (1 - y_1) \times 29 = 0.06 \times 58 + (1 - 0.06) \times 29$$

$$M_{av} = 30.74 \text{ kg/mol}$$

Density of gas-vapour mixture at bottom

$$\rho_{G_1} = \frac{PM_{av}}{RT} = \frac{1 \times 30.74}{0.082057 \times (273 + 25)} = 1.257 \text{ kg/m}^3$$

For $L_s = 3500 \text{ kg/h} = 194.44 \text{ kmol/h}$

Acetone balance

$$G_s(Y_1 - Y_2) = L_s(X_1 - X_2)$$

$$57.25(0.06383 - 0.00120144) = 194.44(X_1 - 0)$$

$$X_1 = 0.01844$$

$$x_1 = \frac{X_1}{1 + X_1} = 0.0181$$

Density of water = 1000 kg/m^3

Density of acetone = 790 kg/m^3

Average density of liquid solution leaving the scrubber

$$\rho_{L_1} = \frac{1}{\sum(W_i/\rho_i)}$$

$$W_1 = x_1 \times 58 / [x_1 \times 58 + (1 - x_1) \times 18]$$

$$= \frac{0.0181 \times 58}{0.0181 \times 58 + 0.9819 \times 18} = 0.056$$

$$\rho_{L_1} = \frac{1}{\frac{0.056}{790} + \frac{(1 - 0.056)}{1000}} = 985.33 \text{ kg/m}^3$$

$$L_1 = L_s(1 + X_1) = 194.44(1 + 0.01844) = 198.025 \text{ kmol/h}$$

Average molar mass of liquid solution

$$M_{av} = 0.0181 \times 58 + 0.9819 \times 18 = 18.724$$

$$L_{1_w} = \frac{(198.025 \times 18.724)/3600}{(\pi/4)D^2} = \frac{1.03}{(\pi/4)D^2} \text{ kg/(m}^2 \cdot \text{s})$$

$$G_1 = G_s(1 + Y_1) = 57.25(1 + 0.06383) = 60.904 \text{ kmol/h}$$

$$G_{1_w} = \frac{(60.904 \times 30.74)/3600}{(\pi/4)D^2} = \frac{0.52}{(\pi/4)D^2} \text{ kg/(m}^2 \cdot \text{s})$$

$$\text{At bottom, } F_{LG} = \frac{L_{1_w}}{G_{1_w}} \sqrt{\frac{\rho_{G_1}}{\rho_{L_1}}} = \frac{1.03}{0.52} \times \sqrt{\frac{1.257}{985.33}} \quad (9.15)$$

$$F_{LG} = 0.0707$$

$$K_F = 0.175 \quad (\text{From Fig. 9.3})$$

Let actual velocity of gas = 66% of flooding velocity

$$K = (0.66)^2 \times 0.175 = 0.07623$$

Δp of packing \equiv 66 mm of H₂O/m of packing

mass velocity of gas through tower

$$G_{1_w} = \left(\frac{K \rho_{G_1} \rho_{L_1} g}{F_p \psi \mu_L^{0.2}} \right)^{1/2} = \left(\frac{0.07623 \times 1.257 \times 985.33 \times 9.81}{170 \times \left(\frac{1000}{985.33} \right) \times 1^{0.2}} \right)^{1/2} \\ = 2.316 \text{ kg/(m}^2 \cdot \text{s})$$

$$\text{Tower area required at bottom } A = \frac{0.52}{2.316} = 0.2245 \text{ m}^2$$

$$A = \frac{\pi}{4} D^2 = 0.2245 \text{ m}^2$$

$$D = 0.5346 \text{ m}$$

Thus the diameter of the entire tower, $D = 0.54 \text{ m}$

N_{OG} : (Number of overall gas phase transfer units)

To calculate the N_{OG} , draw the operating line on the graph which starts from (X_2, Y_2) and terminates at (X_1, Y_1) , i.e. starts from $(0, 0.00120144)$ and terminates at $(0.01843, 0.06383)$. (Ref. Fig. 9.11)

Given system is a dilute system ($x_1 < 0.1$). Here the portion of equilibrium curve under the operating line can be approximated as straight line. Hence, in the given case N_{OG} can be calculated by the following equation.

$$N_{OG} = \frac{1}{1 - \left(\frac{m G_m}{L_m} \right)} \ln \left[\left(1 - \frac{m G_m}{L_m} \right) \frac{y_1}{y_2} + \frac{m G_m}{L_m} \right] \quad (9.12)$$

$$\frac{m G_m}{L_m} = 0.3 \times \frac{\left(\frac{G_1 + G_2}{2}\right) / \frac{\pi}{4} D^2}{\left(\frac{L_1 + L_2}{2}\right) / \frac{\pi}{4} D^2} = 0.3 \times \frac{\left(\frac{60.904 + 57.3189}{2}\right)}{\left(\frac{198.025 + 194.45}{2}\right)}$$

$$= 0.09038$$

$$N_{OG} = \frac{1}{1 - 0.09038} \ln \left[(1 - 0.09038) \times \frac{0.06}{0.0012} + 0.09038 \right]$$

$$= 4.1988$$

Here, acetone is highly soluble in water. Hence liquid film resistance is negligible as compared to the gas film resistance. Hence, height of packing in the given case should be determined by equation;

$$Z = N_{OG} \cdot H_{OG} \quad (9.7)$$

H_{oG}: Height of Overall Gas Phase Transfer Unit

$$H_{OG} = H_G + m \frac{G_m}{L_m} H_L \quad (9.19)$$

Based on Onda, Takeuchi and Okumoto's method,
Height of gas phase transfer unit

$$H_G = \frac{G}{K_G a_e p M_g} \quad (9.21)$$

G = Superficial mass velocity of gas, kg/(m² · s)

$$= \frac{(0.462 + 0.52)/2}{\frac{\pi}{4}(0.540)^2} = 2.1439 \text{ kg}/(\text{m}^2 \cdot \text{s})$$

Effective area of mass transfer a_e is determined by following equation.

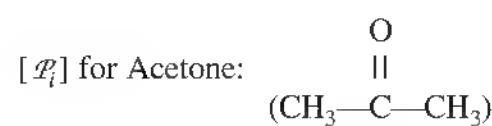
$$\frac{a_e}{a_t} = 0.31 \frac{\sigma_L^{0.5}}{Z^{0.4}} (C_{a_L} \cdot R_{eG})^{0.392} \quad (9.23)$$

$$a_t = 205 \text{ m}^2/\text{m}^3$$

Surface tension data for acetone at the scrubbing temperature can be calculated by a group-contribution method, given in Ref. 3.

$$\sigma_L = \text{Liquid surface tension in } \frac{\text{m N}}{\text{m}} \text{ or } \frac{\text{dyn}}{\text{cm}}$$

$$x_i = x_{av} = \frac{x_1 + x_2}{2} = \frac{0.0181 + 0}{2} = 9.05 \times 10^{-3} = 0.0095$$



$$[\mathcal{P}_i] = 55.5 \times 2 + (9 + 22.3) = 141.7$$

(From Table 3.343 of Ref. 3)

Surface tension of Acetone

$$\sigma^{1/4} = [\mathcal{P}_i](\rho_L - \rho_G) \text{ (Ref. Eq. 3.151 of Ref. 3)}$$

where, ρ_L, ρ_G are in mol/cm³

$$\sigma^{1/4} = 141.7 \left[\frac{(790 - 2.372) \times 10^{-3}}{58} \right]$$

$$\text{Density of pure acetone vapour, } \rho_G = \frac{1 \times 58}{0.082057 \times (273 + 25)} = 2.372 \text{ kg/m}^3$$

$$\sigma^{1/4} = 1.9242$$

$$\sigma = 13.71 \text{ dyn/cm}$$

Actual surface tension data are reported by Timmermans (Ref. 5)

$$\sigma = 24.5 \text{ dyn/cm at } 25^\circ\text{C}$$

Thus it can be seen that group-contribution method may give erroneous results. It is advised to use the actual value as far as possible.

For pure water, $\sigma = 70 \text{ dyn/cm}$

$$\sigma_L = \sum (\sigma_i x_i) = 24.5 \times 0.00905 + (1 - 0.00905)70$$

$$\sigma_L = 69.58 \text{ dyn/cm or m N/m}$$

For dilute aqueous solution, surface tension of water may be taken for calculations.

$$C_{aL} = \text{Liquid capillary number} = \frac{\mu_L L_W}{\rho_L \sigma_L} \quad (9.24)$$

$$x_{av} = 0.00905$$

$$W_{av} = (0.00905 \times 58) / (0.00905 \times 58 + (1 - 0.00905) \times 18)$$

$$W_{av} = 0.02858 \text{ (average mass fraction)}$$

$$\rho_L = \frac{1}{\sum (w_i / \rho_i)} = \frac{1}{\frac{0.02858}{790} + \frac{(1 - 0.02858)}{1000}} = 992.46 \text{ kg/m}^3$$

$$\sigma_L = 69.58 \times 10^{-3} \text{ N/m}$$

$$L_W = \frac{(3500/3600 + 1.03)/2}{(\pi/4)(0.54)^4} = 4.3712 \text{ kg/(m}^2 \cdot \text{s)}$$

μ_L = Viscosity of liquid mixture in kg/(m · s)

$$\ln \mu_{mix} = \sum (w_i \ln \mu_i) \quad (\text{Eq. 3.107 of Ref. 3})$$

Viscosity of acetone at 25°C = 0.33×10^{-3} kg/(m · s)

Viscosity of water $\mu_w = 0.95 \text{ cP} = 0.95 \times 10^{-3} \text{ kg/(m} \cdot \text{s)}$

$$\ln \mu_L = 0.02858 \ln (0.33 \times 10^{-3}) + (1 - 0.02858) \ln (0.95 \times 10^{-3})$$

$$\mu_L = 9.217 \times 10^{-4} \text{ kg/(m} \cdot \text{s)}$$

$$C_{aL} = \frac{9.217 \times 10^{-4} \times 4.3712}{992.46 \times 69.58 \times 10^{-3}}$$

$$= 5.834 \times 10^{-5}$$

$$\text{Reynolds number, } Re_G = \frac{6 G_W}{a_t \mu_G}$$

$$G_W = 2.1439 \text{ kg/(m}^2 \cdot \text{s}), a_t = 205 \text{ m}^2/\text{m}^3$$

Viscosity of gas mixture

$$\mu_G = \sum_{i=1}^n \frac{(y_i \mu_i)}{(y_j \phi_{ij})} \quad (9.32)$$

$$y_{av} = \frac{y_1 + y_2}{2} = \frac{0.06 + 0.0012}{2} = 0.0306$$

$$\phi_{ij} = \frac{\left[1 + (\mu_i / \mu_j)^{1/2} (M_j / M_i)^{1/4}\right]^2}{\left[8(1 + M_i / M_j)\right]^{1/2}} \quad (9.33)$$

$$\text{and } \phi_{ji} = \left(\frac{\mu_j}{\mu_i}\right) \left(\frac{M_i}{M_j}\right) \phi_{ij}$$

Note that here ϕ is only a parameter (and does not represent heat load)

Viscosity of acetone, $\mu_{ac} = 750 \times 10^{-7} \text{ P} = 750 \times 10^{-5} \text{ cP}$

Viscosity of $\mu_{air} = 1850 \times 10^{-7} \text{ P} = 1850 \times 10^{-5} \text{ cP}$

$$\phi_{ij} = \frac{\left[1 + \left(\frac{750}{1850}\right)^{1/2} \left(\frac{29}{58}\right)^{1/4}\right]^2}{\left[8(1 + 58/29)\right]^{1/2}} = 0.4812$$

$$= \left(\frac{1850}{750}\right) \left(\frac{58}{29}\right) \times 0.4812 = 2.374 \text{ (Here } i = \text{acetone, } j = \text{air)}$$

$$\mu_G = \frac{0.0306 \times 750 \times 10^{-5} + (1 - 0.0306) \times 1850 \times 10^{-5}}{(1 - 0.0306) \times 0.4812 + 0.0306 \times 2.374}$$

$$= 0.03369 \text{ cP} \equiv 0.03369 \times 10^{-3} \text{ kg/(m} \cdot \text{s)}$$

$$Re_G = \frac{6 G}{a_t \mu_G} = \frac{6 \times 2.1439}{205 \times 0.03369 \times 10^{-3}} = 1862$$

$$Z = N_{OG} \cdot H_{OG} = 4.1988 H_{OG}$$

$$\frac{a_e}{a_t} = 0.31 \frac{\sigma_L^{0.5}}{Z^{0.4}} (C_{aL} Re_G)^{0.392} \quad (9.23)$$

$$\frac{a_e}{205} = 0.31 \frac{69.58^{0.5}}{Z^{0.4}} (5.834 \times 10^{-5} \times 1862)^{0.392}$$

$$= \frac{1.0832}{Z^{0.4}}$$

For the first trial calculations, let $H_{OG} = 0.6 \text{ m}$

Approximate values of H_{OG} for random packings of size 25 mm is 0.3 to 0.6 m, for 38 mm size it is 0.5 to 0.75 m and for 50 mm size it is 0.6 to 1.0 m (Ref. 2).

$$Z = 4.1988 \times 0.6 = 2.52 \text{ m}$$

$$\frac{a_e}{205} = \frac{1.0832}{2.52^{0.4}} = 0.74842$$

$$a_e = 153.43 \text{ m}^2/\text{m}^3 \text{ of packing}$$

Gas phase transfer coefficient K_G is determined by following equation.

$$\frac{K_G RT}{a_t D_G} = C_1 \left(\frac{G_w}{a_t \mu_G} \right)^{0.7} \left(\frac{\mu_G}{\rho_G D_G} \right)^{1/3} (a_t d_p)^{-2} \quad (9.26)$$

where, $R = 8314 \text{ J/(kmol} \cdot \text{K)}$

$T = \text{operating temperature} = 273 + 25 = 298 \text{ K}$

$$\rho_G = \frac{p M_{av}}{RT}, M_{av} = \sum y_i M_i = 0.0306 \times 58 + (1 - 0.0306) \times 29 = 29.8874$$

$$= \frac{1 \times 29.8874}{0.082057 \times 298} = 1.2222 \text{ kg/m}^3$$

D_G = Gas phase diffusion coefficient, m^2/s

From Table 3.318, of Ref. 3,

At 25°C diffusivity of acetone in air

$$D_G = 0.109 \text{ cm}^2/\text{s} = 0.109 \times 10^{-4} \text{ m}^2/\text{s}$$

$$D_G \propto T^{1.75}$$

$$D_G = 0.109 \times 10^{-4} \times \left(\frac{298}{273} \right)^{1.75} = 1.2706 \times 10^{-5} \text{ m}^2/\text{s}$$

$C_1 = 5.23$ for $d_p > 12 \text{ mm}$

$$K_G \times \frac{8314 \times 298}{205 \times 1.2706 \times 10^{-5}} = 5.23 \left(\frac{2.1439}{205 \times 0.03369 \times 10^{-3}} \right)^{0.7} \times \left(\frac{0.03369 \times 10^{-3}}{1.2222 \times 1.2706 \times 10^{-5}} \right)^{1/3} \times (205 \times 0.025)^{-2}$$

$$K_G \times 9.51182 \times 10^8 = 5.23 \times 55.509 \times 1.2945 \times 0.03807.$$

$$K_G = 1.504 \times 10^{-8} \text{ kmol}/(\text{m}^2 \cdot \text{s} \cdot \text{Pa})$$

Height of gas phase transfer unit

$$H_G = \frac{G_w}{K_G a_e p M_g} \quad (9.21)$$

G_w = Average superficial mass velocity of gas, $\text{kg}/(\text{m}^2 \cdot \text{s})$

$$G_w = 2.1435 \text{ kg}/(\text{m}^2 \cdot \text{s})$$

$$K_G = 1.504 \times 10^{-8} \text{ kmol}/(\text{m}^2 \cdot \text{s} \cdot \text{Pa})$$

$$a_e = 153.43 \text{ m}^2/\text{m}^3$$

p = operating pressure = 101.325×10^3 Pa
 M_g = average molar mass of gas = 29.8874 kg/mol

$$H_G = \frac{2.1439}{1.504 \times 10^{-8} \times 153.43 \times 101.325 \times 10^3 \times 29.8874}$$

$$= 0.3068 \text{ m}$$

Height of liquid phase transfer unit

$$H_L = \frac{L_w}{K_L a_e \rho_L} \quad (9.22)$$

L_w = Average mass velocity of liquid through tower

$$= \frac{[(3500/3600) + 1.03]/2}{\pi/4(0.54)^2} = \frac{8.7425}{2} = 4.3712 \text{ kg/(m}^2 \cdot \text{s)}$$

$$a_e = 153.41 \text{ m}^2/\text{m}^3, \quad \rho_L = 992.46 \text{ kg/m}^3$$

$$H_L = \frac{4.3712}{K_L \times 153.41 \times 992.46} = \frac{2.871 \times 10^{-5}}{K_L}$$

Liquid phase mass transfer coefficient K_L can be determined by the following equation

$$K_L \times \left(\frac{\rho_L}{\mu_L g} \right)^{1/3} = 0.0051 \left(\frac{L_w}{a_w \mu_L} \right)^{2/3} \left(\frac{\mu_L}{\rho_L D_L} \right)^{-0.5} (a_t d_p)^{0.4} \quad (9.27)$$

$$\rho_L = 992.46 \text{ kg/m}^3, \quad \mu_L = 9.217 \times 10^{-4} \text{ kg/(m} \cdot \text{s)}$$

$$L_w = 4.3712 \text{ kg/(m}^2 \cdot \text{s}), \quad g = 9.81 \text{ m/s}^2, \quad a_t = 205 \text{ m}^2/\text{m}^3$$

$$d_p = 0.025 \text{ m}$$

$$a_w = \text{wetted surface of packing, m}^2/\text{m}^3$$

$$\frac{a_w}{a_t} = 1 - \exp \left(-1.45 \left(\frac{\sigma_c}{\sigma_L} \right)^{0.75} Re_L^{0.1} Fr_L^{-0.05} We_L^{0.2} \right) \quad (9.28)$$

For polypropylene packing, $\sigma_c = 33 \times 10^{-3}$ N/m (Table 9.3)

$$\sigma_L = 69.58 \times 10^{-3} \text{ N/m}$$

$$\text{Liquid Reynolds number, } Re_L = \frac{L_w}{a_t \mu_L} = \frac{4.3712}{205 \times 9.217 \times 10^{-4}}$$

$$= 23.134$$

Froude number of liquid, $Fr_L = L_w^2 a_t / (\rho_L g)$

$$= \frac{4.3712^2 \times 205}{992.46^2 \times 9.81} = 4.0538 \times 10^{-4}$$

Weber number for liquid,

$$We_L = L_w^2 / (\rho_L \sigma_L a_t) = \frac{4.3712^2}{992.46 \times 69.58 \times 10^{-3} \times 205} = 1.35 \times 10^{-3}$$

$$\frac{a_w}{a_t} = 1 - \exp \left[-1.45 \left(\frac{33}{69.58} \right)^{0.75} \times 23.134^{0.1} \times (4.0538 \times 10^{-4})^{-0.05} \times (1.35 \times 10^{-3})^{0.2} \right]$$

$$\frac{a_w}{a_t} = 1 - \exp (-0.4472)$$

$$\frac{a_w}{a_t} = 0.3606$$

$$a_w = 0.3606 \times 205 = 73.923 \text{ m}^2/\text{m}^3$$

D_L = Diffusivity of acetone in water, m^2/s

D_L can be determined by Wilke and Chang equation

$$D_L = \frac{(117.3 \times 10^{-18})(\phi M_s)^{0.5} T}{\mu V_A^{0.6}}$$

(Equation 2.44, of Ref. 1)

where, V_A = Molar volume of solute at normal boiling point, m^3/kmol

ϕ = Association factor for solvent = 2.26 for water (a parameter)

M_s = Molar mass of solvent = 18 kg/kmol

μ = Solution viscosity, $\text{kg}/(\text{m} \cdot \text{s}) = 9.217 \times 10^{-4} \text{ kg}/(\text{m} \cdot \text{s})$

T = Temperature, $\text{K} = 273 + 25 = 298 \text{ K}$

For calculating V_A , a group contribution method, given in Treybal is recommended.
From Table 2.3 of Ref. 1

Table 9.6 Group Contributions for Acetone¹

Atom	Volume, m^3/kmol	Total No. of atoms
C	0.0148	3
H	0.0037	6
O	0.0074	1

$$V_A = 0.0148 \times 3 + 0.0037 \times 6 + 0.0074 \times 1 = 0.074 \text{ m}^3/\text{kmol}$$

$$D_L = \frac{(117.3 \times 10^{-18}) \times (2.26 \times 18)^{0.5} \times 298}{9.217 \times 10^{-4} \times (0.074)^{0.6}} = 1.154 \times 10^{-9} \text{ m}^2/\text{s}$$

$$K_L \left(\frac{992.46}{9.217 \times 10^{-4} \times 9.81} \right)^{1/3} = 0.0051 \left(\frac{4.3712}{73.923 \times 9.217 \times 10^{-4}} \right)^{2/3}$$

$$\times \left(\frac{9.217 \times 10^{-4}}{992.46 \times 1.154 \times 10^{-9}} \right)^{-0.5} \times (205 \times 0.025)^{0.4}$$

$$K_L \times 47.88 = 0.0051 \times 16.02 \times 0.03525 \times 1.9225 \\ = 1.1564 \times 10^{-4} \text{ kmol/(m}^2 \cdot \text{s} \cdot \text{Pa})$$

$$H_L = \frac{2.871 \times 10^{-5}}{1.1564 \times 10^{-4}}$$

$$H_L = 0.248 \text{ m}$$

$$H_{OG} = H_G + m \frac{G_m}{L_m} H_L \quad (9.19)$$

$$H_{OG} = 0.3068 + 0.09038 \times 0.248 = 0.329 \text{ m}$$

$$Z = N_{OG} \cdot H_{OG} = 4.1988 \times 0.329 = 1.38 \text{ m}$$

For second trial calculations, let $Z = 1.4 \text{ m}$

$$a_e = \frac{1.0832 \times 205}{1.4^{0.4}} = 194 \text{ m}^2/\text{m}^3$$

$$H_G = 0.3068 \times \frac{153.43}{194} = 0.2426 \text{ m}$$

$$\text{and } H_L = 0.248 \times \frac{153.43}{194} = 0.196 \text{ m}$$

$$H_{OG} = 0.2426 + 0.09038 \times 0.196 = 0.2603 \text{ m}$$

$$Z = 4.1988 \times 0.2603 = 1.093 \text{ m} < 1.382 \text{ m}$$

H_{OG} for 25 mm size random packing ranges from 0.3 to 0.6 m.

Let $H_{OG} = 0.3 \text{ m}$

$$Z = 0.3 \times 4.1986 = 1.26 \text{ m} \approx 1.3 \text{ m}$$

This is the value of packed bed height based on Onda, Takeuchi and Okumoto's method.

Cornell's method:

For Pall rings, height of gas phase transfer unit

$$H_G = \frac{0.017 \psi D^{1.24} Z^{0.33} Sc_G^{0.5}}{(L f_1 f_2 f_3)^{0.6}} \quad (9.29)$$

ψ = Correlation parameter = 60 m for metal Pall rings (Fig. 9.4)

For polypropylene Pall rings, data for ψ are not available. Hence, assume that the value of ψ for polypropylene Pall rings and ψ for metal Pall rings is same.

For PP Pall rings $\psi = 60 \text{ m}$

$D = 0.54 \text{ m}, z = 1.3 \text{ m}$ (Assume for the 1st trial calculations)

$$Sc_G = \frac{\mu_G}{\rho_G D_G} = \frac{0.03369 \times 10^{-3}}{1.2222 \times 1.2706 \times 10^{-5}} \\ = 2.1695$$

$$f_1 = \left(\frac{\mu_L}{\mu_w} \right)^{0.16} \text{ with } \mu_w = 10^{-3} \text{ Pa} \cdot \text{s}$$

$$= \left(\frac{9.217 \times 10^{-4}}{10^{-3}} \right)^{0.16} = 0.987$$

$$f_2 = \left(\frac{\rho_w}{\rho_L} \right)^{1.25} \quad \text{with } \rho_w = 1000 \text{ kg/m}^3$$

$$= \left(\frac{1000}{992.46} \right)^{1.25} = 1.01$$

$$f_3 = \left(\frac{\sigma_w}{\sigma_L} \right)^{0.8} \quad \text{where } \sigma_w = 72.8 \text{ mN/m}$$

$$= \left(\frac{72.8}{69.49} \right)^{0.8} = 1.038$$

L = Liquid phase mass velocity = $4.3712 \text{ kg}/(\text{m}^2 \cdot \text{s})$

$$H_G = \frac{0.017 \times 60 \times 0.54^{1.24} \times 1.3^{0.33} \times 2.1695^{0.5}}{(4.3712 \times 0.987 \times 1.01 \times 1.038)^{0.6}} = 0.3085 \text{ m}$$

Height of liquid phase transfer unit,

$$H_L = \frac{\phi C}{3.28} \left(\frac{\mu_L}{\rho_L D_L} \right)^{0.5} \left(\frac{Z}{3.05} \right)^{0.15}$$

From Fig. 9.5, for $L = 4.3712 \text{ kg}/(\text{m}^2 \cdot \text{s})$,

Correlation parameter ϕ for metal Pall rings = 0.05 m

Assume that ϕ for PP Pall rings is ϕ for SS Pall ring

$$\phi = 0.05 \text{ m}$$

$C = 0.75$ for 66% Flooding (From Fig. 9.6)

$$H_L = \frac{0.05 \times 0.75}{3.28} \left(\frac{9.217 \times 10^{-4}}{992.46 \times 1.154 \times 10^{-9}} \right)^{0.5} \times \left(\frac{1.3}{3.05} \right)^{0.15}$$

$$= 0.2854 \text{ m}$$

$$H_{OG} = H_G + m \frac{G_m}{L_m} H_L = 0.3085 + 0.09038 \times 0.2854$$

$$= 0.334 \text{ m}$$

Height of packed bed, $Z = N_{OG} \cdot H_{OG}$

$$Z = 4.1988 \times 0.334 \text{ m} = 1.4 \text{ m}$$

For second trial calculations, let $Z = 1.4 \text{ m}$

$$H_G = 0.3058 \times \left(\frac{1.4}{1.3} \right)^{0.33} = 0.3134 \text{ m}$$

$$H_L = 0.2854 \times \left(\frac{1.4}{1.3} \right)^{0.15} = 0.2856 \text{ m}$$

$$H_{OG} = 0.3134 + 0.09038 \times 0.2856 = 0.3392 \text{ m}$$

$$Z = 4.1988 \times 0.3392 = 1.424 \text{ m}$$

Let height of packed bed $Z = 1.5$ m (Higher than the values of Z obtained based on Cornell's method as well as based on Onda, Takeuchi and Okumoto's method.)

No. of packing sections required = one

Liquid distributor: For clean liquid solvent and for small diameter tower perforated pipe type distributor is satisfactory. It is recommended that one liquid stream must be provided for each 194 cm^2 area. (Ref. 3)

No. of holes (distribution points) required in pipe type distributor,

$$n_h = \frac{\text{Cross sectional area of tower, cm}^2}{194}$$

$$n_h = \frac{(\pi/4)(0.54)^2 \times 10^4}{194} = 11.8 \approx 12$$

Let the velocity of water through the pipe = 2 m/s

Flow rate of water (solvent) = 3500 kg/h

$$\text{Volumetric flow rate of solvent} = \frac{3500}{1000} \times \frac{1}{3600} = 9.7222 \times 10^{-4} \text{ m}^3/\text{s}$$

$$d = \sqrt{\frac{(9.722 \times 10^{-4}) \times 4}{2 \times \pi}} = 0.025 \text{ m}$$

$$d = 25 \text{ mm}$$

Let the velocity of solvent through holes = 3 m/s

Let d_h = diameter of hole, m

$$12 \times \frac{\pi}{4} d_h^2 = \frac{9.7222 \times 10^{-4}}{3}$$

$$d_h = 0.006 \text{ m} \approx 6 \text{ mm}$$

Resulting pipe type liquid distributor is shown in Fig. 9.12.

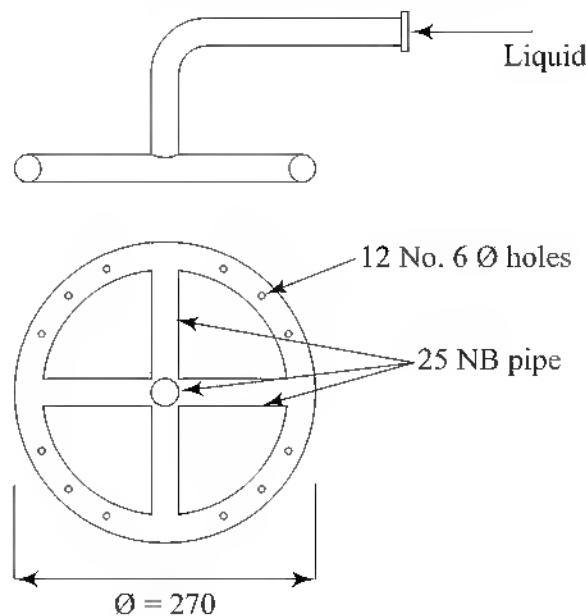


Fig. 9.12 Pipe Type Liquid Distributor

Packing support:

Packing support should be selected such that flow area provided by packing support for the flow of gas should be greater than flow area (i.e. void) provided by packing material.

% Void space for 25 mm size polypropylene Pall rings = 90% (Ref. Table 9.2)

With Pall rings any gas-injection type packing support can be used.

Let type of packing support: Cap type packing support

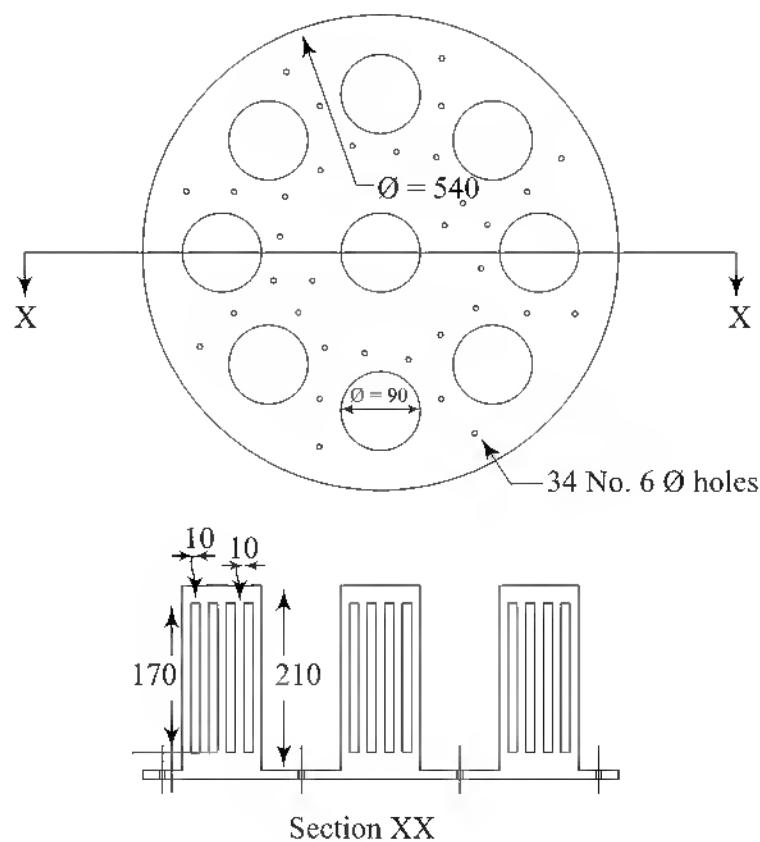


Fig. 9.13 Cap Type Packing Support

Actual outer diameter of packing support is greater than 540 mm as some portion of packing support is sandwiched between two flanges.

Let h = height of slot in riser

x = width of slot in riser

d = diameter of riser

n = number of risers.

D_C = Inside diameter of column = 540 mm

If $d = D_C/6$, then nine number of risers can be provided on packing support.

$$= \frac{540}{6} = 90 \text{ mm}$$

Let, n_s = number of slots per riser

Total area of slots of risers = $n_s \cdot n h x$

Let $x = 10 \text{ mm}$

$$n_s = \frac{\pi d}{2x} = \frac{\pi \times 90}{2 \times 10} \cong 14$$

(If the distance between two successive slots is $x \text{ mm}$)

$$\text{Total area of slots of risers} = 14 \times 9 \times h \times 10 \times \geq 0.9 \times \frac{\pi}{4} (540)^2$$

$$h \geq 163.58 \text{ mm}$$

$$\text{Take, } h = 170 \text{ mm}$$

Let diameter of weep hole = 6 mm, velocity of liquid through weep hole = 1 m/s

$$\text{No. of weeping holes required} = \frac{(9.722 \times 10^{-4})/1}{\frac{\pi}{4}(0.006)^2} = 34$$

9.4 PROCESS DESIGN OF SPRAY CHAMBER OR SPRAY TOWER TYPE ABSORBER^{3, 6}

Spray absorbers are used for removing SO₂ (sulphur dioxide gas) from boiler flue gases that is exhausted from a boiler. When very low pressure drop is essential and when incoming gas stream is contaminated by particulate matter, spray absorbers are preferred over the packed tower type absorber. In the large scale plant, boiler flue gases entering the absorber for the removal of SO₂ are available at very low pressure (100 to 200 mm WC) and with very high volumetric flow rate. These gases are also contaminated by particulate matters.

Spray chambers (absorbers) are having either circular or rectangular cross section. Normally there is no packing in the spray absorber.

They are of two types.

- (a) Vertical spray tower
- (b) Horizontal spray chamber

Liquid phase residence time in spray absorbers is in the range of 1 to 10 s which is very low. To compensate this, liquid phase resistance must be negligible. Hence, a chemical reagent is added in the liquid phase to get the higher rate of absorption.

9.4.1 Process Design of Vertical Spray Tower³

In vertical spray tower gas stream is flowing vertically upward and liquid is sprayed downward in various sections within the tower as well as from the sides by spray nozzles.

Process design steps for vertical spray tower are as follows:

(i) Determination of tower diameter: Maximum permissible gas velocity through vertical spray tower is 2.3 m/s. Actual velocity of gas through tower should be less than 2.3 m/s to avoid the excessive entrainment of liquid droplets in the exit gas. On fixing the actual velocity, tower diameter can be determined by following equation.

$$D = \sqrt{\frac{q_v \times 4}{u_g \times \pi}} \quad (9.34)$$

where, D = Tower diameter, m

q_v = Volumetric flow rate of gas, m³/s

u_g = Velocity of gas through tower, m/s < 2.3 m/s

- (ii) Determine the number of gas phase transfer units required for the desired separation by following equation

$$N_G = \frac{y_1 - y_2}{y_2} \quad (9.35)$$

where, N_G = Number of gas phase transfer units

y_1 = Gas phase solute mole fraction at inlet (at bottom)

y_2 = Gas phase solute mole fraction at outlet (at top)

- (iii) Calculate the total volume of spray section by following equation.

$$V = \frac{N_G \times G_m}{K_G a} \quad (9.36)$$

where, V = Volume of spray section, m^3

N_G = Number of gas-phase mass transfer units

$K_G a$ = Overall volumetric gas phase mass transfer coefficient $\text{kmol}/[(\text{s} \cdot \text{m}^3) (\text{mole fraction of solute in gas})]$

G_m = Flow rate of gas phase, kmol/s

The best method for finding the $K_G a$ is from the similar type of commercial gas absorption unit noting that same value of $K_G a$ can be used only with same value of liquid flow rate to gas flow rate ratio.

9.4.2 Process Design of Horizontal Cross Flow Spray Chamber⁶

For the given absorption duty total volume required by horizontal cross flow chamber is considerably less than that of a vertical spray tower. Horizontal chamber is having either rectangular or circular cross section. Gas passes horizontally through it and liquid is sprayed vertically downward perpendicular to the direction of gas flow. Process design steps for horizontal cross flow chamber are as follows.

(i) Determination of duct area or diameter:

Maximum permissible gas velocity through horizontal spray chamber is 7 m/s. On fixing the actual gas velocity through horizontal chamber, duct area or diameter can be determined.

- (ii) Determine the number of gas phase transfer units, required for the desired separation by following equation.

$$N_G = \ln\left(\frac{y_1}{y_2}\right) \quad (9.37)$$

where N_G = number of gas phase transfer unit

y_1 = Gas phase solute mole fraction at inlet

y_2 = Gas phase solute mole fraction at outlet

- (iii) Total volume of spray section is determined by the same equation that is used for vertical spray tower. (Eq. 9.36)

Example 9.2

Design the spray chamber for the following duty.

Volumetric flow rate of boiler flue gas = 24 000 Sm³/h

Pressure of gas = 150 mm WC (gauge)

Temperature = 80 to 90 °C

SO₂ concentration in boiler flue gas = 4000 ppm = 4000×10^{-6} kmol/kmol gas

Desired concentration of SO₂ in the outgoing gas = 200 ppm = 200×10^{-6} kmol/kmol gas

Solvent = 1% lime solution

Solution:

Type of scrubber: Spray chamber

Position: Horizontal or vertical

(a) Tower diameter

(i) For vertical spray chamber:

Volumetric flow rate of gas = 24 000 Sm³/h

Volumetric flow rate of gas at actual operating condition

$$q_v = \frac{24\ 000 \times 10\ 333}{273} \times \frac{(273 + 90)}{(10\ 333 + 150)} = 31\ 455.5 \text{ m}^3/\text{h} \equiv 8.7376 \text{ m}^3/\text{s}$$

Maximum permissible gas velocity through vertical spray chamber = 2.3 m/s (to avoid the excessive entrainment)

$$u_{g \max} = 2.3 \text{ m/s}$$

Let actual velocity of gas through spray tower.

$$u_g = 66\% \text{ of } u_{g \max}$$

$$u_g = 0.66 \times 2.3 = 1.518 \text{ m/s}$$

$$\frac{\pi}{4} D_i^2 = \frac{q_v}{u_g}$$

where D_i = Inside diameter of spray tower

$$D_i = \sqrt{\frac{4 \times q_v}{\pi \times u_g}} = \sqrt{\frac{4 \times 8.7376}{\pi \times 1.518}} = 2.707 \text{ m}$$

Let $D_i = 2.71 \text{ m} \equiv 2710 \text{ mm}$

(ii) Horizontal cross flow spray chamber:

Maximum permissible gas velocity through horizontal spray chamber = 7 m/s

$$u_{g \ max} = 7 \text{ m/s}$$

Let actual velocity of gas through horizontal spray chamber, $u_g = 66\% \text{ of } u_{g \ max}$

Let cross sectional area of horizontal spray chamber, $A = W^2$ for square duct

where, W = Width of chamber = Height of chamber

$$W^2 = \frac{q_v}{u_g} = \frac{8.7376}{4.62} = 1.8913$$

$$W = \sqrt{\frac{q_v}{u_g}}$$

$$W = 1.375 \text{ m}$$

(b) Number of gas phase transfer units, N_G

(i) Number of gas phase transfer units required for desired separation, for vertical spray tower

$$\begin{aligned} N_G &= \frac{y_1 - y_2}{y_2} \\ &= \frac{4000 \times 10^{-6} - 200 \times 10^{-6}}{200 \times 10^{-6}} = 19 \end{aligned} \quad (9.35)$$

(ii) For horizontal spray chamber

$$N_G = \ln\left(\frac{y_1}{y_2}\right) = \ln\left(\frac{4000 \times 10^{-6}}{200 \times 10^{-6}}\right) = 3 \quad (9.37)$$

(c) Circulation rate of 1% lime solution

(i) For vertical spray chamber:

To decide the circulation rate and height of gas phase transfer unit duplication of similar/same type of commercial gas absorption unit is the best method.

From the existing vertical spray tower of one of the large scale industry, following data are available.

Flow rate of boiler flue gas = 12 000 Sm³/h

Temperature of incoming gas = 80 to 90°C

SO₂ concentration in inlet stream = 2500 to 4000 ppm

SO₂ concentration in outlet stream = Less than 400 ppm

Circulation rate of 1% lime solution = 30 m³/h

Tower diameter = 1.7 m

Height of spray zone = 3.5 m

Pressure drop = 100 mm WC (maximum)

Based on these data, circulation rate required for new tower

$$\text{circulation rate} = 30 \times \frac{24\,000}{12\,000} = 60 \text{ m}^3/\text{h}$$

(ii) For Horizontal spray chamber

For the trial run, let the circulation rate of 1% lime solution for horizontal spray chamber = 60 m³/h

(d) Height of tower:

(i) Vertical spray tower:

$$V = \frac{N_G \times G_m}{K_G a} \quad (9.36)$$

For existing tower

$$N_G = \frac{4000 \times 10^{-6} - 400 \times 10^{-6}}{400 \times 10^{-6}} = 9$$

$$V = \frac{\pi}{4} (1.7)^2 \times 3.5 = 7.944 \text{ m}^3$$

$$G_m = \frac{12\,000}{22.414} = 535.38 \text{ kmol/h} \equiv 0.1487 \text{ kmol/s}$$

For existing tower, $K_G a = \frac{9 \times 0.1487}{7.944} = 0.1685 \text{ kmol/(m}^3 \cdot \text{s)}$

Volume of spray section required for new tower

$$V = \frac{N_G G_m}{K_G a} = \left(19 \times \frac{24000}{22.414} \times \frac{1}{3600} \right) / 0.1685 \\ = 33.54 \text{ m}^3 = (\pi/4) D_i^2 H$$

where, H = Height of spray section required

$$33.54 \text{ m}^3 = (\pi/4) \times (2.7)^2 \times H$$

$$H = 5.86 \text{ m}$$

Let $H = 6 \text{ m}$

(ii) For horizontal spray chamber:

Mass transfer data are available for vertical spray tower only. Assume the overall volumetric mass transfer coefficient $K_G a$ for cross flow arrangement in horizontal spray chamber to be $0.1 \text{ kmol/(m}^3 \cdot \text{s)}$.

(less than the same for vertical position).

$$V = \frac{N_G \times G_m}{K_G a} = \frac{3 \times 0.2974}{0.1} = 8.922$$

$$V = 8.922 \text{ m}^3 = W^2 L = (1.375)^2 L$$

Length of spray section required with horizontal position, $L = 4.72 \text{ m}$

Let $L = 5 \text{ m}$

(c) Approximate pressure drop calculations:

(i) For vertical spray chamber

Pressure drop $\Delta p \propto G^{1.84} d^{-4.84} L$ (Ref. Eq. 5.5)

Pressure drop in actual vertical spray tower is 100 mm WC (maximum).

Pressure drop in proposed new vertical spray tower,

$$\Delta p = 100 \times \left(\frac{24}{12} \right)^{1.84} \times \left(\frac{2.71}{1.7} \right)^{-4.84} \times \left(\frac{6}{3.5} \right)$$

$$\Delta p = 64.24 \text{ mm WC (within permissible limit)}$$

(ii) For horizontal spray chamber

$$\Delta p = 100 \times \left(\frac{24}{12} \right)^{1.84} \times \left(\frac{1.375}{1.7} \right)^{-4.84} \times \left(\frac{5}{3.5} \right)$$

$$\Delta p = 1428 \text{ mm WC (very high)}$$

To decrease the pressure drop, duct area must be increased. Let $W = 2 \text{ m}$ and $L = 2.5 \text{ m}$

$$\Delta p = 100 \times \left(\frac{24}{12} \right)^{1.84} \times \left(\frac{2}{1.7} \right)^{-4.84} \times \left(\frac{2.5}{3.5} \right)$$

$$= 116.45 \text{ mm WC (within permissible limit)}$$

(Note: For this approximate calculation width or height of chamber W is considered as diameter.)

Actually duct area (W^2) is higher than circular cross sectional area $\left(\frac{\pi}{4}W^2\right)$ and hence

it will offer less pressure drop than the circular cross section.

Volume of spray section, $V = (2)^2 \times 2.5 = 10 \text{ m}^3$ (revised)

(f) Make-up of lime solution:

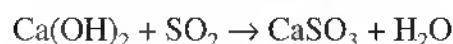
Lime required = Lime consumed in reaction + Drift loss.

Liquid particles entrained in the out going gas. Let drift loss = 0.2% of circulation rate (based on experience in cooling tower).

$$\text{Drift loss} = \frac{0.2}{100} \times 60 = 0.12 \text{ m}^3/\text{h} \approx 120 \text{ kg/h}$$

Loss of lime as drift = 1.2 kg

Lime is consumed in reaction.



$$\text{SO}_2 \text{ consumed in reaction} = \frac{24\,000}{22.414} \times (4000 - 200) \times 10^{-6} = 4.07 \text{ kmol/h}$$

$$\text{Ca(OH)}_2 \text{ consumed in reaction} = 4.07 \text{ kmol/h} = 4.07 \times 74 = 301.18 \text{ kg/h}$$

$$\text{Lime required as CaCO}_3 = (301.18 + 1.2) \times \frac{100}{74} + 1.2 = 408.62 \text{ kg/h}$$

Loss of water = Drift loss + Evaporation rate + Blow down $\approx 1\%$ of circulation rate

CaCO₃ addition required = 408.62 kg/h

Water addition required = 6 m³/h = 6000 kg/h

Summary of results:

<i>Vertical spray tower</i>	<i>Horizontal spray chamber</i>
1. Tower diameter = 2.71 m	1. Duct width and height = 2 m
2. Height of spray section = 6 m	2. Length of spray section = 2.5 m
3. Volume of spray section = 33.54 m ³	3. Volume of spray section = 10 m ³
4. Pressure drop = 64 mm WC	4. Pressure drop = 116.45 mm WC
5. Circulation rate = 60 m ³ /h	5. Circulation rate = 60 m ³ /h
6. Lime as CaCO ₃ required = 408.62 kg/h	6. Lime as CaCO ₃ required = 408 kg/h
7. Makeup water required = 6 m ³ /h	7. Makeup water required = 6 m ³ /h

Comments: From the resulting data, it is clear that horizontal spray chamber is a better option. But it is less common in operation as compared to vertical spray tower. Here mass transfer data used for calculation are available for vertical spray tower. Hence, design of the vertical spray tower is more reliable as compared to the horizontal spray chamber.

9.5 VENTURI SCRUBBER^{3, 7}

Venturi scrubber is normally preferred for removing particulate matter from gas stream as opposed to absorbing soluble vapour. Efficient contact between liquid and gas is obtained in venturi scrubber. Gas is drawn into the throat of venturi by a stream of absorbing liquid solvent sprayed into the convergent duct section. Compared to spray absorber more efficient contact between gas and liquid is

provided by the venturi scrubber but at the expense of relatively large gas-side pressure drop and a consequent higher power consumption. Spray absorber cannot remove very fine particles (below 10 μm size) while the venturi scrubber can remove the fine particles up to the size 2 μm effectively.

Venturi scrubber provides cocurrent contact (over all) between liquid and gas. The fractional solute removal for a dilute system in venturi scrubber is given by following equation.

$$\text{Fractional solute removal} = \frac{y_1 - y_2}{y_1} = \frac{\eta(1 - mx_2/y_2)}{\left(1 + \frac{mG_M}{L_M}\right)}$$

where, y_2 = Mole fraction of solute in gas phase at outlet

y_1 = Mole fraction of solute in gas phase at inlet

m = Slope of equilibrium curve

x_2 = Mole fraction of solute in incoming solvent

G_M = Molar gas phase velocity, kmol / ($\text{m}^2 \cdot \text{s}$)

L_M = Molar liquid phase velocity, kmol / ($\text{m}^2 \cdot \text{s}$)

η = Mass transfer efficiency, calculated by following equation.

$$\eta = 1 - e^{-N_G} \quad (9.38)$$

where, N_G = Number of overall gas-phase mass transfer units can be calculated by following equation.

$$N_G = (K_G a RT / p_t) \theta_c \quad (9.39)$$

where, $K_G a$ = Overall volumetric gas-phase mass-transfer coefficient for dilute systems, $\text{kmol}/(\text{s} \cdot \text{m}^3)$

R = Ideal gas constant = 8314 J/(kmol · K)

T = Operating temperature of gas · K

θ_c = Effective gas-liquid contact time, s

p_t = Operating pressure, Pa

The number of transfer units, N_G that can be achieved in venturi scrubber is in the range of 1 to 2.

The liquid to gas ratio for venturi scrubber is in between 0.7 to 2.7 L/m³. Throat velocity is kept in between 60 to 150 m/s. The estimated pressure drop across the venturi scrubber is given by following equation (Hesketh equation⁷).

$$\Delta P = 2.584 \times 10^{-3} v_G^2 \rho_G A_{th}^{0.133} (L'/G')^{0.78} \quad (9.40)$$

where, ΔP = Venturi pressure drop from inlet duct to the outlet duct, cm WC

v_G = Gas velocity in the throat, m/s

ρ_G = Gas density, kg/m³

A_{th} = Venturi throat area, cm²

L'/G' = Liquid to gas ratio, L/m³

Example 9.3

Predict the fractional solute removal and pressure drop in a venturi scrubber based on the following data.

Volumetric flow rate of boiler flue gas = 24 000 Nm³/h

Discharge pressure of gas from venturi = Atmospheric

Temperature of gas = 80 to 90°C

SO₂ concentration in boiler flue gas = 4000 ppm (or mg/kg)

Solvent = 1 % lime solution

Solvent to gas ratio = 1.4 L/m³

Throat velocity of gas phase = 100 m/s

Average molar mass of flue gas = 29.48 kg/kmol

Solution:

Volumetric flow rate of gas at actual operating condition.

$$q_v = \frac{24\ 000}{273} \times (273 + 90) = 31\ 912.1 \text{ m}^3/\text{h}$$

$$\equiv 8.8645 \text{ m}^3/\text{s}$$

Gas velocity in throat = 100 m/s

Inside diameter of throat,

$$d_i = \sqrt{\frac{4 \times 8.8645}{\pi \times 100}} = 0.336 \text{ m}$$

Flow rate of solvent (1% lime solution) = 1.4 × 8.8645 = 12.41 L/s = 44.676 m³/h

Density of 1% lime solution = 1012.5 kg/m³

Mass flow rate of solution = 45 234.5 kg/h

$$\dot{m}_L = 12.565 \text{ kg/s}$$

$$\text{Molar flow rate of Liquid, } L_M = \frac{12.565}{\left(\frac{1}{(0.99/18) + (0.01/74)} \right)} = 0.693 \text{ kmol/s}$$

$$\text{Molar flow rate of Gas, } G_M = \frac{p q_v}{RT} = \frac{1 \times 8.8645}{0.082 \times (273 + 90)}$$

$$G_M = 0.2978 \text{ kmol/s}$$

Number of transfer units, N_G that can be achieved in venturi scrubber is in the range of 1 to 2.

Assuming $N_G = 1$ (safe value)

Mass transfer efficiency,

$$\eta = 1 - e^{-N_G}$$

$$\eta = 1 - e^{-N_E} = 1 - e^{-1} = 0.6321 \quad (9.38)$$

Fractional solute removal,

$$1 - \frac{y_2}{y_1} = E \frac{(1 - mx_2/y_2)}{\left(1 + m \frac{G'_M}{L'_M} \right)}$$

$$\frac{G'_M}{L'_M} = \frac{G_M / \text{Flow area}}{L_M / \text{Flow area}} = \frac{G_M}{L_M} = \frac{0.2978}{0.693} = 0.43$$

$$x_2 = 0, \quad y_2 = ?$$

$$1 - \frac{y_2}{y_1} = \frac{E}{1 + m \frac{G'_M}{L'_M}} = \frac{0.6321}{1 + 0.43m}$$

$$y_1 = 4000 \times 10^{-6} = 0.004$$

Partial pressure of SO_2 in gas phase $\cong 0.004 \times 760 = 3.04$ torr

Assume the average temperature of scrubber = 50°C

Equilibrium mass of SO_2 per 100 mass of $\text{H}_2\text{O} = 0.035$

(Table 3–144 of Ref. 3)

$$\text{Equilibrium mass fraction} = \frac{0.035}{100.035} \cong 3.5 \times 10^{-4}$$

$$\text{Equilibrium mole fraction} = \frac{3.5 \times 10^{-4} / 64}{(3.5 \times 10^{-4} / 64) + (1 - 3.5 \times 10^{-4}) / 18}$$

$$x_e = 9.846 \times 10^{-5}$$

$$y_e = 0.004$$

$$m = \frac{0.004}{9.846 \times 10^{-5}} = 40.63$$

Equilibrium constant for physical absorption at 50°C and 1 atm pressure, $m = 40.63$

For low concentration range m can be assumed constant.

$$1 - \frac{y_2}{y_1} = \frac{0.6321}{1 + 0.43 \times 40.63} = 0.03422$$

$$\frac{y_2}{y_1} = 0.9658$$

$$y_2 = 0.00386$$

Here, presence of 1% lime in solution or chemical reaction between lime and SO_2 is not considered. Chemical reaction enhances the rate and extent of absorption considerably. To find this, lab-scale or pilot plant scale data are required on the same system and also on the same type of absorber. Reliable theoretical equations are not available for the same.

Pressure drop

$$\Delta P = 2.584 \times 10^{-3} v_G^2 \rho_G A_{\text{th}}^{0.133} \left(\frac{L'}{G'} \right)^{0.78} \quad (9.40)$$

$$v_G = 100 \text{ m/s}$$

$$A_{\text{th}} = \text{Throat area in cm}^2 = \frac{\pi}{4} (33.6)^2 = 886.7 \text{ cm}^2$$

Density of gas, ρ_G kg/m³

$$\rho_G = \frac{PM}{RT} = \frac{1 \times 29.48}{0.082 \times (273 + 90)} = 0.99 \text{ kg/m}^3$$

$$\frac{L'}{G'} = 1.4 \frac{L}{m^3}$$

$$\Delta P = 82 \text{ cm WC} \equiv 820 \text{ mm WC}$$

Comparison with spray absorber:

Pressure drop provided by venturi scrubber (820 mm WC) is very much greater than the same provided by vertical spray absorber (64 mm WC).

9.6 PROCESS DESIGN OF FALLING FILM ABSORBER^{8, 9}

Falling film absorber is a shell and tube heat exchanger in which absorption of gas in liquid is carried out inside the tubes while cooling medium like cooling water is circulated on the shell side. It is used for highly exothermic absorption like absorption of hydrogen chloride gas in water, absorption of ammonia gas in water, absorption of sulphur trioxide in alpha olefins, absorption of sulphur tri-oxide in methyl esters of saturated fatty acids, etc.

9.6.1 Advantages of Falling Film Absorber Over Packed Tower Type Adiabatic Absorber

- (i) Falling film absorber gives better quality of product solution. Concentration of HCl in product solution from falling film absorber is 35% to 37% (by mass), while the same from adiabatic packed tower ranges from 25% to 30%.
- (ii) Absorption is more efficient in falling film absorber for the given absorption duty. Falling film absorber requires less space compared to adiabatic packed tower type absorber.
- (iii) Other advantages of falling film absorber are lower temperatures, lower pressure drops and higher turn down.

9.6.2 Disadvantages of Falling Film Absorber

- (i) More difficult to control and is sensitive to liquid and gas distribution problems.
- (ii) It has greater tendency to absorb undesirable water soluble low boiling impurities.

9.6.3 Exothermic Absorption of Gases Like HCl and NH₃

In case of exothermic absorption of gas like HCl and NH₃ in water, there are two requirements

- (i) Large liquid surface area to get the higher rate of mass transfer
- (ii) Rapid removal of the heat of absorption

Both can be done most effectively by causing the liquid to flow down as a film inside of a vertical tube which is cooled on the outside.

Falling film absorber with counter current flow of the gas and liquid suffer from the limitation that flooding occurs when the gas velocity exceeds a limiting value of the order of **4.5 m/s** for 25 mm OD tube. A sample calculation shows that at the usual ratio of liquid and gas flow rates, the liquid rate is barely sufficient to maintain a liquid film on the tube wall. Hence counter current contact is not preferred. Cocurrent contact of gas and liquid allows the gas velocity up to **18 m/s**.

For the absorption of hydrogen chloride gas falling film absorber (with cocurrent contact of gas and liquid) followed by tail gas scrubber is preferred if the concentration of hydrogen chloride gas in feed gas mixture is more than 40% (by mole). Nearly 66 to 85% of absorption is carried out in falling film absorber while the balance is carried out in the tail gas scrubber. If the concentration of hydrogen chloride in feed gas mixture is less than 40% (by mole), then it is difficult to produce the product solution of commercial strength. With cocurrent contact, partial pressure of hydrogen chloride in exist gas is greater than or equal to exit gas equilibrium partial pressure of hydrogen chloride in product solution.

While most HCl gas will be scrubbed in these two scrubbers, local environmental standards could mandate an additional vent scrubber in which final wash of the outgoing gas mixture is carried out by spraying small quantity of water. However, the bottom solution from the vent scrubber will contain some HCl and its disposal may require neutralization with alkali.

Low concentration of HCl in exist gas gives low concentration of HCl in product solution. The arrangement of falling film absorber with tail gas scrubber is shown in Fig. 9.14.

Feed gas and weak acid from the tail gas scrubber (5 to 10% HCl solution) is supplied at the top head of falling film absorber. Tubes are extended above the top tube sheet to facilitate the uniform distribution of liquid in each tube. Notches may be cut in the top ends of the tubes. Tubes are made from graphite, glass or tantalum. As per the recommendations tubes must be extended at least by 125 mm above the top tube sheet to minimize the effect of hydraulic gradient in the liquid. With cocurrent contact in upper portion of tubes, rate of absorption is very high. It may lead to the overheating of tubes in upper portion. Overheating breaks the glass tubes. To avoid this, inverted U in the discharge line of water is essential. Inside the tubes there is a two phase flow of liquid and gas in which liquid forms the film over the inside periphery of tubes while gas flows in the central portion. Absorption of gas in liquid film generates the heat. Temperature of liquid film is controlled by cooling water circulated on shell side. In the bottom head, gas and product solution are separated. Exist gas is in the equilibrium of product solution at outlet condition. Hence, complete absorption of HCl with this arrangement is impossible. Remaining HCl is absorbed by contact with pure water in tail gas scrubber which is commonly used and is normally a packed tower type absorber.

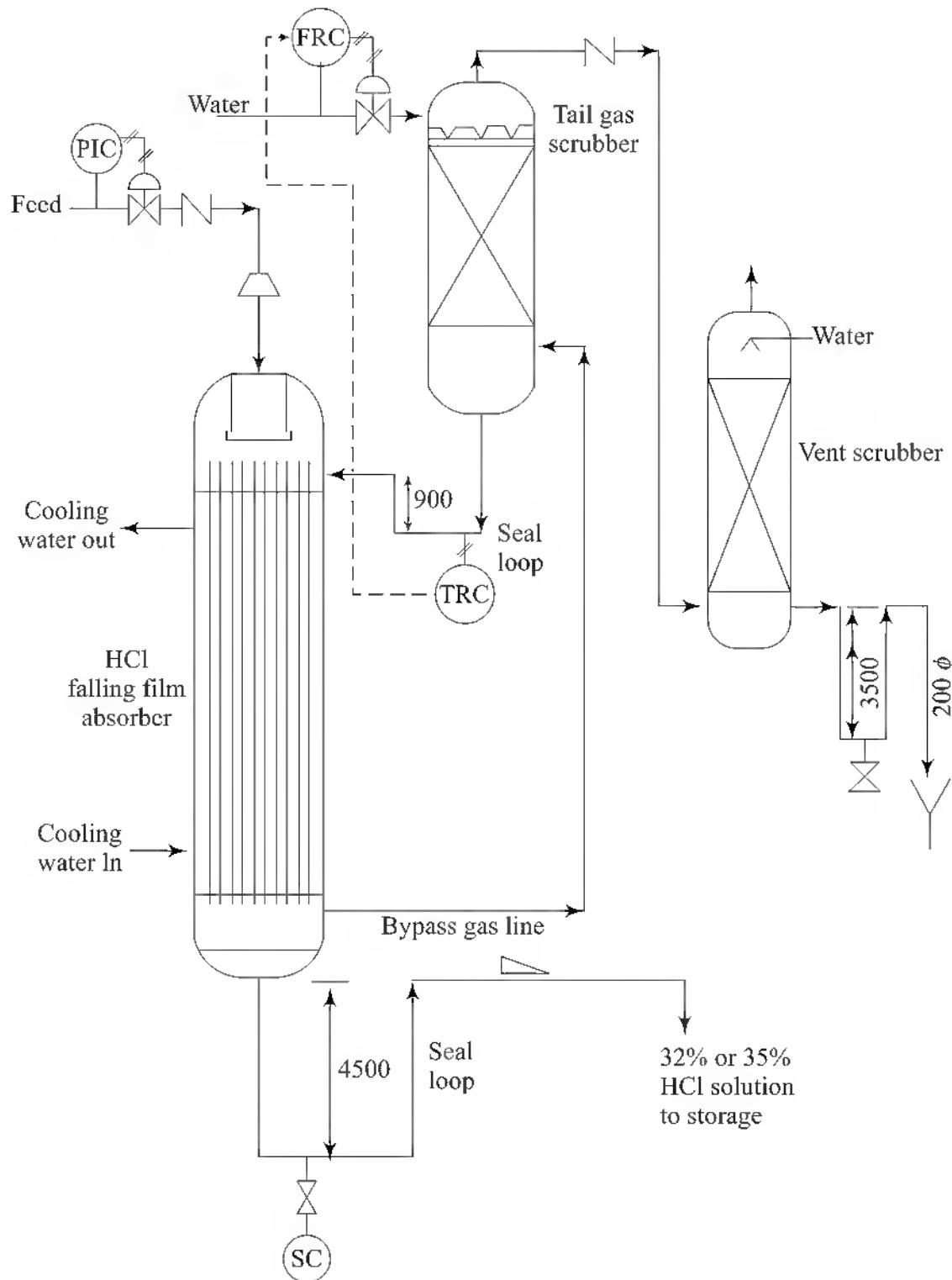


Fig. 9.14 Typical Falling Film Absorption System

9.6.4 Process Design of Falling Film Absorber

Consider absorption of HCl in water.

- Fix the inlet and outlet concentrations of hydrochloric acid. Also, fix the concentration of hydrogen chloride in the exist gas stream. Partial pressure of hydrogen chloride in the exist gas stream at bottom should be greater than vapour pressure of hydrogen chloride in product solution at outlet

temperature. Based on this fact, outlet temperature and concentrations of liquid and gas streams can be fixed.

- (ii) Determine the overall rate of absorption and based on the same, determine the total heat duty. Actually rate of absorption and hence rate of heat transfer required are changing from point to point of the heat exchanger (i.e. falling film absorber)
- (iii) Based on the heat balance, find the flow rate of cooling medium.
- (iv) In the end, heat transfer area of falling film absorber must be determined

$$A = N_t \pi d_o L = \frac{\phi}{U \Delta T_m} \quad (9.41)$$

To facilitate two phase flow, d_o (tube outside diameter) can be selected as 25.4 mm. Number of tubes must be decided based on following criteria

- (a) For cocurrent contact velocity of feed gas mixture at inlet should be less than or equal to 18.3 m/s. To get the higher rate of mass transfer gas velocity at tube, inlet should be around 15 m/s as per the recommendations. (p. 409 of Ref. 8)
- (b) For efficient operation of falling film absorber certain minimum liquid flow rate is required to maintain a continuous film over the tube wall. Minimum required flow rate of liquid is 150 kg/(h · m), i.e. m of inside periphery of tubes.

Actually once velocity of gas is fixed, flow rate of liquid per unit periphery of tube is fixed by material balance and reverse is also true.

Tube length can be easily determined based on average value of overall heat transfer coefficient U and based on the overall values of heat duty (ϕ) and overall mean temperature difference (ΔT_m) (difference between temperature of liquid film and temperature of water). Actually rate of absorption, rate of heat transfer, overall heat transfer coefficient and temperature difference across the heat transfer wall are changing from point to point. Hence, for the precise calculations, heat exchanger can be divided in different zones and for each zone, tube length is determined. For each zone, rate of absorption, heat duty and mean temperature difference are calculated. Over all coefficient U is assumed as constant. In trial and error calculations, film temperature is assumed. Based on that rate of absorption per unit area, heat duty per unit area and ΔT_m are calculated. Check $\phi/A = U\Delta T_m$. Rate of absorption of gas is given by equation.

$$\text{Rate of absorption, } N_A = K_G M (\overline{p_{AG}} - \overline{p_{AL}^*}) \text{ kg}/(\text{m}^2 \cdot \text{s}) \quad (9.42)$$

where, K_G = Overall mass transfer coefficient based on the arithmetic mean partial pressure difference in $\text{kmol}/(\text{m}^2 \cdot \text{atm} \cdot \text{s})$

M = Molar mass of gas absorbed kg/kmol = 36.5 for HCl or 17 for NH_3

$\overline{p_{AG}}$ = Partial pressure of solute gas in gas mixture, atm

$\overline{p_{AL}}$ = Equilibrium partial pressure of solute A at gas liquid interface, atm

Mass transfer coefficient for hydrogen chloride absorption in the tubular absorber is calculated by following equation⁸

$$K_G = \frac{1.654 \times 10^{-5}}{M_{av}^{1.75}} \left(\frac{d_i G_t}{\mu} \right) \text{ kmol/(m}^2 \cdot \text{atm} \cdot \text{s}) \quad (9.43)$$

where, d_i = Inside diameter of tube, m

G_t = Mass velocity of gas mixture at inlet, kg/(m² · s)

μ = Viscosity of gas mixture kg/(m · s)

M_{av} = Average molar mass of gas mixture at inlet, kg/kmol

Heat transfer coefficient of liquid film can be calculated by McAdams, Drew and Bay's equation (Ref. 8).

$$h = 9136 \times \tau^{1/3} \quad (9.44)$$

where, h = Tube side heat transfer coefficient, W/(m² · °C)

τ = Liquid flow rate per unit periphery, kg/(m · s)

This correlation is valid for values of τ , ranging from 0.25 to 6.2 kg/(m · s)

Example 9.4

Gas mixture, obtained after the chlorination reaction and separation of organic vapours, contains 20% Cl₂ and 80% HCl by mole. From the mixture, hydrogen chloride gas is to be separated by absorption in water. For the absorption of HCl, falling film absorber, followed by tail gas scrubber are used. Design the falling film absorber.

Flow rate of gas mixture = 2500 kg/h

Temperature of gas mixture = 10°C

% of HCl in the liquid stream leaving the tail gas scrubber and entering at the top of falling film absorber = 5% (by mass)

Desired concentration of HCl in product stream = 32% (by mass)

Operating pressure = 30 kPa g (at inlet)

Solution:

Amount of product solution = q_m kg/h

HCl balance

$$2500 \times \left(\frac{0.8 \times 36.5}{0.8 \times 36.5 + 0.2 \times 71} \right) = q_m \times 0.32$$

$$2500 \times 0.6728 = q_m \times 0.32$$

$$q_m = 5256.25 \text{ kg/h}$$

Heat of formation of aqueous-hydrochloric Acid (Table 5.53 of Ref. 10)

Heat of solution = (ΔH_f°) of 32% solution – (ΔH_f°) of HCl gas

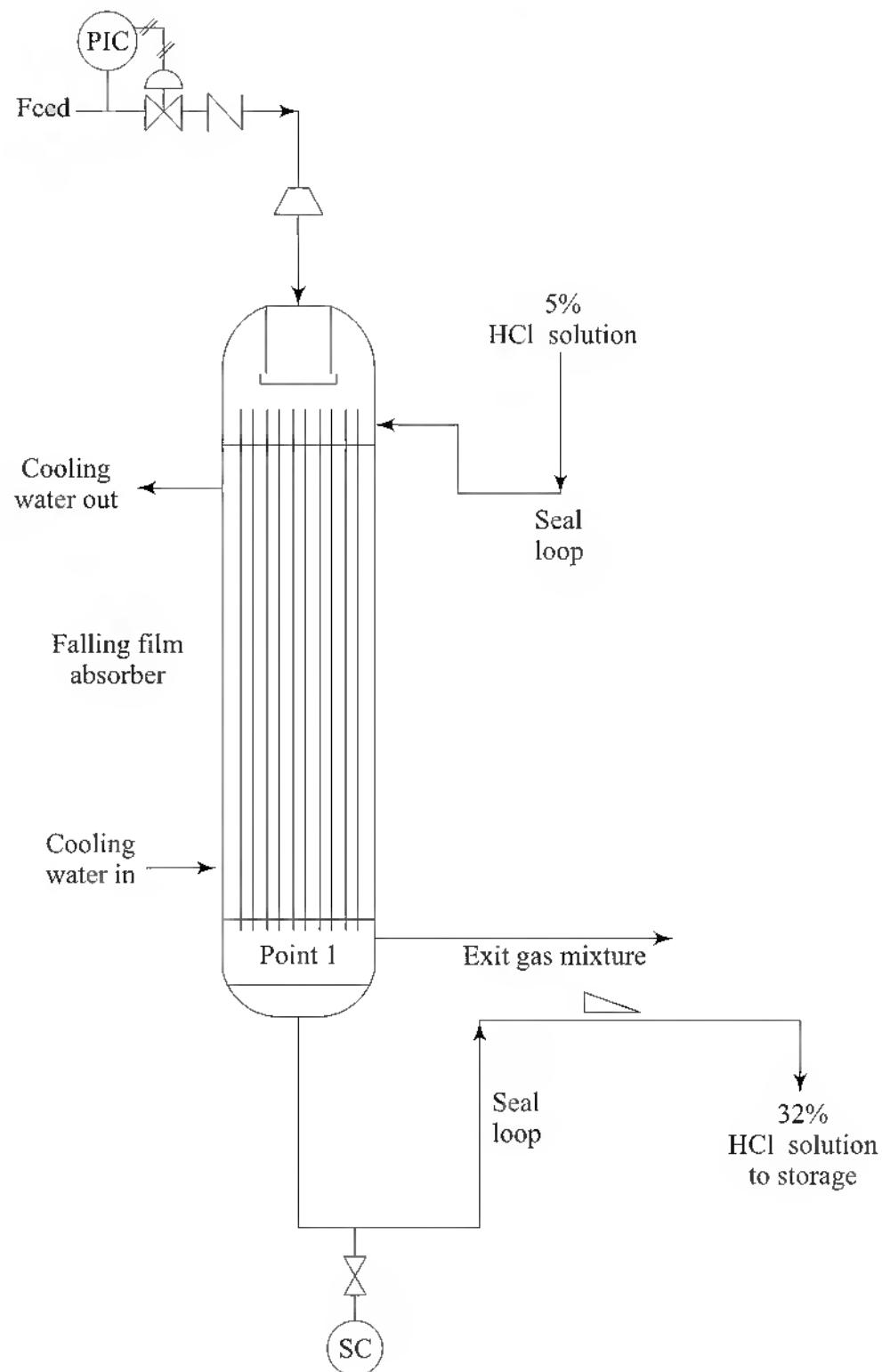


Fig. 9.15 Falling Film Absorption System

Heat of formation of 32% HCl solution = $-153.9 \text{ kJ/mol HCl}$
 Heat of solution = $(-153.89) - (-92.31) = -61.58 \text{ kJ/mol HCl}$

$$\text{Moles of HCl absorbed} = \frac{2500 \times 0.6728}{36.5} = 46.082 \text{ kmol/h}$$

Table 9.7 Heat of Formation of Aqueous Hydrochloric Acid Solution¹⁰

Formula	State	Heat of formation (ΔH_f°) at 298 K, kJ/mol HCl	Mass % HCl in aqueous solution
HCl. 75 H ₂ O	aq	-165.72	2. 63
HCl. 50 H ₂ O	aq	-165.36	3. 90
HCl. 40 H ₂ O	aq	-165.10	4. 82
HCl. 30 H ₂ O	aq	-164.67	6. 33
HCl. 25 H ₂ O	aq	-164.34	7. 50
HCl. 20 H ₂ O	aq	-163.85	9. 21
HCl. 15 H ₂ O	aq	-163.03	11. 9
HCl. 10 H ₂ O	aq	-161.32	16. 9
HCl. 8 H ₂ O	aq	-160.00	20. 2
HCl. 6 H ₂ O	aq	-157.68	25. 3
HCl. 5 H ₂ O	aq	-155.77	28. 9
HCl. 4 H ₂ O	aq	-152.92	33. 6
HCl. 2 H ₂ O	aq	-140.96	50. 3
HCl. H ₂ O	aq	-121.55	67. 0
HCl	gas	-92.31	100.0

Note: The number in the first column indicates the number of moles of water mixed with one mole of hydrogen chloride.

Heat duty of falling film absorber (including that of tail gas scrubber),

$$\phi_f = 61.58 \times 1000 \times 46.082 = 2837\ 729.6 \text{ kJ/h} \equiv 788.26 \text{ KW}$$

(Actually in falling film absorber, concentration of HCl in aqueous solution is increased from 5% to 32% but tail gas scrubber is operated in adiabatic manner hence heat of solution generated on increasing the concentration from 0 to 5% of HCl is to be added in heat duty of falling film absorber)

Temperature of 5% weak acid:

Heat of formation for 5% HCl solution = -165.05 kJ/mol

Heat of solution = (-165.05) - (-92.31) = -72.74 kJ/mol

Moles of HCl absorbed in adiabatic absorber

$$\begin{aligned} &= \left(\frac{46.082 \times 36.5}{0.32} - 46.082 \times 36.5 \right) \times \frac{0.05}{0.95} \times \frac{1}{36.5} \\ &= 5.154 \text{ kmol/h} \\ &\equiv 188.121 \text{ kg/h} \end{aligned}$$

Heat generated in adiabatic absorber

$$\phi_1 = 72.74 \times 1000 \times 5.154 = 374\ 902 \text{ kJ/h} \equiv 104.14 \text{ kW}$$

Mass flow rate of pure water entering to adiabatic absorber = Mass flow rate of 32% solution $\times (1 - 0.32)$

$$q_w = 5256.35 \times (1 - 0.32) = 3574.25 \text{ kg/h}$$

Mass flow rate of 5% HCl solution

$$q_{w1} = \frac{3574.25}{(1 - 0.05)} = 3762.37 \text{ kg/h}$$

Enthalpy of 5% solution

$$= \text{Enthalpy of pure water} + \text{Enthalpy of incoming gas mixture} - \text{Enthalpy of exist gas from adiabatic absorber} + \text{Heat of solution}$$

Let reference temperature = 30°C

Here pure water enters at room temperature or at a temperature close to 30°C. Temperature of incoming and exist gas mixture are also close to 30°C. Heat of solution must be determined at average temperature. Neglect the change in the value of heat of solution with temperature.

Heat capacity of 5% solution = 4.19 kJ/(kg · °C)

Enthalpy of 5% solution = $q_w \cdot C_L (t - 30)$

$$3762.37 \times 4.19(t - 30) = 374\,902 \text{ kJ/h}$$

$$t = 53.78^\circ\text{C}$$

Mass flow rate of cooling water:

Let inlet temperature of cooling water to absorber = 32°C and outlet temperature of cooling water from absorber = 38°C

Mass flow rate of cooling water,

$$\begin{aligned} q_w &= \frac{\phi_t}{C_L \Delta t} = \frac{788.26}{4.1868 \times (38 - 32)} = 31.379 \text{ kg/s} \\ &= 112\,964.4 \text{ kg/h} \end{aligned}$$

Material of tube = Graphite

Outside diameter of tube $d_o = 25 \text{ mm}$

Inside diameter of tube $d_i = 20 \text{ mm}$

It is reported in literature that if the velocity of gas through the tubes is kept more than 15.24 m/s, then higher tube side or film side heat transfer coefficient is obtained. There is a sharp increase in the tube side heat transfer coefficient when the velocity of gas at the tube inlet exceeds 15.24 m/s.

Let the velocity of gas at tube inlet = 16 m/s

Average molar mass of feed gas mixture at inlet

$$M_{av} = \sum(M_i \cdot y_i) = 0.2 \times 71 + 0.8 \times 36.5 = 43.4 \text{ kg/kmol}$$

Density of feed gas at inlet

$$\begin{aligned} \rho &= \frac{p M}{R T} = \frac{(30 + 101.325) \times 43.4}{(273 + 10)} \times \frac{273}{101.325 \times 22.414} \\ &= 2.42 \text{ kg/m}^3 \end{aligned}$$

Volumetric flow rate of gas mixture at tube inlet, $q_v = \frac{2500}{2.42}$

$$q_v = 1033 \text{ m}^3/\text{h}$$

$$\text{Velocity of gas at tube inlet, } v = \frac{q_v}{N_t \times \frac{\pi}{4} d_i^2}$$

$$N_t = \frac{1033/3600}{16 \times \frac{\pi}{4} (0.02)^2} = 57$$

Tube side mass velocity, $G_t = u_t \rho = 16 \times 2.42 = 38.72 \text{ kg/(m}^2 \cdot \text{s)}$

Tube side heat transfer coefficient:

Tube side or falling film side heat transfer coefficient

$$h_i = 9136 \times \tau^{1/3} \quad (9.44)$$

where, h_i is in $\text{W}/(\text{m}^2 \cdot ^\circ\text{C})$

τ = Liquid flow rate per unit periphery, $\text{kg}/(\text{m} \cdot \text{s})$

Liquid flow rate at inlet = $3762.37 \text{ kg/s} = q_{w_1}$

$$\tau = \frac{q_{w_1}}{N_t \pi d_i} = \frac{3762.37 / 3600}{57 \times \pi \times 0.02}$$

$$\tau = 0.2918 \text{ kg}/(\text{m} \cdot \text{s}) (= 1050.5 \text{ kg}/(\text{m} \cdot \text{h}) > 150 \text{ kg}/(\text{m} \cdot \text{h}))$$

$$h_i = 9136 \times (0.2918)^{1/3} = 6059.7 \text{ W}/(\text{m}^2 \cdot ^\circ\text{C})$$

Shell side heat transfer coefficient

$$\text{Shell side flow area, } A_S = \frac{(P_t - d_o) B_s D_s}{P_t} \quad (6.29)$$

$$P_t = \text{Tube pitch} = 1.25 \text{ } d_O = 1.25 \times 25 = 31.25 \text{ mm}, \quad d_O = 25 \text{ mm}$$

Tube bundle diameter

$$D_b = d_o \left(\frac{N_t}{K_1} \right)^{1/n_1} \quad (6.1)$$

For $P_t/d_O = 1.25$, triangular pitch, 1-1 shell and tube fixed tube sheet heat exchanger, $k_1 = 0.319$, $n_1 = 2.142$ (From Table 6.2)

$$D_b = 25 \left(\frac{57}{0.319} \right)^{\frac{1}{2.142}} = 281.4 \text{ mm}$$

$$D_S = D_b + 18.6 \text{ mm} = 300 \text{ mm}, \quad \text{Let } B_S = 100 \text{ mm}$$

$$A_S = \frac{(31.25 - 25)}{31.25} \times 0.1 \times 0.3 = 0.006 \text{ m}^2$$

Shell side equivalent diameter

$$d_e = \frac{1.1}{d_o} (P_t^2 - 0.907 d_o^2) \quad (6.32)$$

$$= \frac{1.1}{25} (31.25^2 - 0.907 \times 25^2) = 18.026 \text{ mm}$$

$$\text{Shell side mass velocity, } G_S = \frac{q_w}{A_s} = \frac{31.379}{0.006} = 5229.8 \text{ kg}/(\text{m}^2 \cdot \text{s})$$

$$\text{Shell side velocity, } u_S = \frac{G_s}{\rho} = \frac{5229.8}{994.032} = 5.26 \text{ m/s} > 3 \text{ m/s (too high)}$$

For the liquid flow on shell side, it is better to restrict the shell side velocity below 3 m/s to avoid the liquid induced vibrations.

Change the baffle spacing from 100 to 300 mm.

$$B_S = 300 \text{ mm}$$

$$A_S = \frac{31.25 - 25}{31.25} \times 0.3 \times 0.3 = 0.018 \text{ m}^2$$

Shell side mass velocity, $G_s = \frac{31.379}{0.018} = 1743.3 \text{ kg}/(\text{m}^2 \cdot \text{s})$

$$u_s = \frac{G_s}{\rho} = \frac{1743.3}{994.032} = 1.7538 \text{ m/s}$$

$$\text{Shell side Reynolds number, } Re = \frac{d_e G_s}{\mu} = \frac{0.018026 \times 1743.3}{(0.8 \times 10^{-3})} = 39280.9$$

(Viscosity of water at 35°C, $\mu = 0.8 \text{ cP}$)

Prandtl number for water at 35°C

$$Pr = \frac{C_p \mu}{k} = \frac{4.1868 \times (0.8 \times 10^{-3}) \times 10^3}{0.628} = 5.333$$

Evaluation of h_o (shell side heat transfer coefficient):

$$\frac{h_o d_e}{k} = 0.36 Re^{0.55} Pr^{0.33} \left(\frac{\mu}{\mu_w} \right)^{0.14} \quad (6.35)$$

$$h_o = 0.36 \times \frac{0.628}{0.018026} \times (39280.9)^{0.55} \times (5.333)^{0.33} \times \left(\frac{\mu}{\mu_w} \right)^{0.14}$$

where $\frac{\mu}{\mu_w} \approx 1$

$$h_o = 7329.3 \text{ W}/(\text{m}^2 \cdot ^\circ\text{C})$$

Overall heat transfer coefficient

$$\frac{1}{U_o} = \frac{1}{h_o} + \frac{1}{h_{od}} + \frac{d_o \ln(d_o/d_i)}{2 k_w} + \frac{d_o}{d_i} \times \frac{1}{h_{id}} + \frac{d_o}{d_i} \times \frac{1}{h_i} \quad (6.42)$$

Fouling coefficients, $h_{od} = h_{id} = 5000 \text{ W}/(\text{m}^2 \cdot ^\circ\text{C})$

Thermal conductivity of graphite = 150 W/(m · k) from Appendix of Ref. 11.

$$\frac{1}{U_o} = \frac{1}{7329.3} + \frac{1}{5000} + \frac{0.025 \ln\left(\frac{25}{20}\right)}{2 \times 150} + \frac{25}{20} \times \frac{1}{5000} + \frac{25}{20} \times \frac{1}{6059.7}$$

$$U_o = 1232.6 \text{ W}/(\text{m}^2 \cdot ^\circ\text{C})$$

The temperature of the acid film on inside surface of tubes attains an equilibrium value at which the rate of heat transfer to the cooling water balances the rate of heat release due to absorption. This temperature is calculated by trial-and-error method for a series of points in the absorber.

Point 1: Bottom most point of tubes: At this point aqueous acid film concentration of liquid film is 32% (by mass). Liquid composition at point 1.

Table 9.8 Composition of Bottom Solution (Point 1)

Component	kg/h	mass %	kmol/h
HCl	1682.00	32	46.082
H ₂ O	3574.25	68	198.570
Total	5256.25	100	244.650

HCl amount in 5% acid stream = 188.12 kg/h

HCl balance around falling film absorber:

$$\text{HCl in exist gas stream} = \text{HCl in inlet gas stream} + \text{HCl in 5\% acid stream} \\ - \text{HCl in product stream}$$

$$\text{HCl in exist gas stream} = 2500 \times 0.6728 + 188.12 - 1682 = 188.12 \text{ kg/h}$$

$$\text{Cl}_2 \text{ in exist gas stream} = 2500 (1 - 0.6728) = 818 \text{ kg/h}$$

Table 9.9 Gas Composition at Point I

Component	kg/h	mass %	kmol/h	mole %
HCl	188.12	18.7	5.154	30.91
Cl ₂	818.00	81.3	11.520	69.09
Total	1006.12	100.0	16.674	100.00

Partial Pressure of HCl gas in gas mixture at point 1

$$\bar{p}_{AG} = p_t \times y_{\text{HCl}} = 131.325 \times 0.30966 = 40.666 \text{ kPa} = 0.4013 \text{ atm}$$

Equilibrium Partial Pressure of HCl (\bar{p}_{AL}^*):

Equilibrium Partial Pressure of HCl gas over aqueous solution of Hydrochloric acid can be calculated by following equation

$$\log \bar{p}_{AL}^* = A - \frac{B}{T}$$

where \bar{p}_{AL}^* is in torr and T in K (Table 3.11 of Ref. 3)

Table 9.10 Antoine Equation Constants³

% HCl by mass	A	B
2	11.8037	4736
4	11.6400	4471
6	11.2144	4202
8	11.0406	4042
10	10.9311	3908
12	10.79	3765
14	10.6954	3636
16	10.6261	3516
18	10.4957	3376
20	10.3833	3245
22	10.3172	3125
24	10.2185	2995
26	10.1303	2870
28	10.0115	2732
30	9.8763	2593
32	9.7523	2457
34	9.6061	2316
36	9.5262	2279

Rate of heat transfer at point 1 between acid film and cooling water

$$\frac{\phi}{A} = U\Delta t = 1232.6 \times (35.5 - 32) = 4314.1 \text{ W/m}^2 \equiv 4.314 \text{ kW/m}^2$$

Heat release due to absorption $\neq U\Delta t$

2nd trial:

Let acid film temperature at point 1 = 36.5°C

Equilibrium partial pressure of HCl for 32% solution at 36.5°C:

$$\bar{p}_{AL}^* = 65.116 \text{ torr} = 0.0857 \text{ atm}$$

$$\begin{aligned} \text{Heat release due to absorption} &= 2.8233 \times 10^{-4} \times 36.5 (0.4013 - 0.0857) \\ &= 3.252 \times 10^{-3} \text{ kg/(m}^2 \cdot \text{s}) \end{aligned}$$

$$\text{Heat release due absorption} = 3.254 \times 10^{-3} \times 1687.12 = 5.49 \text{ kW/m}^2$$

$$\frac{q}{A} = U\Delta t = 1232.6 \times (36.5 - 32) = 5546.7 \text{ W/m}^2 = 5.546 \text{ kW/m}^2$$

Heat release due to absorption $\cong U\Delta t$

Temperature of acid film at point 1 = 36.5°C

Point 2: At this point concentration of Acid film is 26% (by mass).

$$\text{Mass flow rate of 26% HCl solution} = \frac{5256.25 \times 0.68}{(1 - 0.26)} = 4830.07 \text{ kg/h}$$

Amount of HCl absorbed in between point 1 and 2 = 5256.25 - 4830.07 = 426.18 kg/h

Composition of liquid mixture at point 2:

Table 9.11 Liquid Composition at Point 2

Component	kg/h	mass %
HCl	1255.818	26
Water	3574.25	74
Total	4830.07	100

Table 9.12 Composition of Gas Mixture at Point 2

Component	kg/h	kmol/h	mole %
HCl	614.3	16.83	59.365
Cl ₂	818.0	11.52	40.635
Total	1432.3	28.35	100

For the first trial calculations let the temperature of 26% acid film at point 2 = 40°C

$$\log \bar{p}_{AL}^* = 10.1303 - \frac{2870}{(40 + 273)}$$

$$\bar{p}_{AL}^* = 9.14 \text{ torr} = 0.012 \text{ atm}$$

$$\bar{p}_{AG} = y_{HCl} \times p_t = 0.59365 \times 131.325 = 77.96 \text{ kPa} = 0.7694 \text{ atm}$$

Average molar mass of gas mixture

$$M_{av} = \Sigma(y_i M_i) = 0.59365 \times 36.5 + 0.40335 \times 71 = 50.306 \text{ kg/kmol}$$

Mass velocity of gas mixture

$$G_t = \frac{q_m}{a_t} = \frac{1432.3/3600}{57 \times \frac{\pi}{4}(0.02)^2} = 22.218 \text{ kg/(m}^2 \cdot \text{s)}$$

At point 2

$$K_G = \frac{1.654 \times 10^{-5}}{M_{\text{av}}^{1.75}} \left(\frac{d_i G_t}{\mu} \right) = \frac{1.654 \times 10^{-5}}{50.306^{1.75}} \times \left(\frac{0.02 \times 22.218}{0.0145 \times 10^{-3}} \right)$$

(At 40°C, $\mu_{\text{Cl}_2} = 0.0145 \text{ cP}$, $\mu_{\text{HCl}} = 0.0145 \text{ cP}$)

$$K_G = 5.334 \times 10^{-4} \text{ kmol/(atm} \cdot \text{m}^2 \cdot \text{s})$$

Rate of absorption, $N_A = K_G M (\bar{p}_{AG} - \bar{p}_{AL}^*)$

$$N_A = 5.334 \times 10^{-4} \times 36.5 \times (0.7694 - 0.012) = 0.014746 \text{ kg/(m}^2 \cdot \text{s})$$

Heat of solution for 26% HCl solution

$$\begin{aligned} \Delta H_S &= (\Delta H_f^\circ)_{26\% \text{ HCl}} - (\Delta H_f^\circ)_{\text{HCl gas}} \\ &= (-157.3) - (92.31) = -65 \text{ kJ/mol} \\ &= -65000 \text{ kJ/kmol} \equiv -1780.82 \text{ kJ/kg} \end{aligned}$$

Heat release due to absorption (per unit area)

$$\phi = N_A \times (-\Delta H_S) = 0.014746 \times 1780.82 = 26.26 \text{ kW/m}^2$$

Heat duty required in between 1 and 2

ϕ_{12} = Total heat duty of falling film absorber – Heat duty required to form 26% HCl solution

ϕ_{12} = 788.26 kW – $(-\Delta H_S) \times \text{kmol/h}$ of HCl absorbed to form 26% solution

$$\begin{aligned} \phi_{12} &= 788.26 - 65000 \times \left(46.082 - \frac{426.18}{36.5} \right) \times \frac{1}{3600} \\ &= 167.0435 \text{ kW} = q_{cw} C_L(t - 32) \\ 167.0435 &= 31.379 \times 4.1868 \times (t - 32) \end{aligned}$$

Temperature of cooling water at point 2, $t = 33.27^\circ\text{C}$

$$\begin{aligned} \frac{\phi}{A} &= U \Delta t = 1232.6 \times (40 - 33.27) = 8295.4 \text{ W/m}^2 \equiv 8.295 \text{ kW/m}^2 \\ &= 8.295 \text{ kW/m}^2 \neq \text{Heat release due to absorption.} \end{aligned}$$

2nd trial

Let temperature of 26% acid film at point 2 = 54°C

Equilibrium partial pressure of HCl over solution

$$\log \bar{p}_{AL}^* = 10.1303 - \frac{2870}{(54 + 273)}$$

$$\bar{p}_{AL}^* = 22.57 \text{ torr} \equiv 0.0297 \text{ atm}$$

At 54°C, $\mu_{\text{HCl}} = \mu_{\text{Cl}_2} = 0.015 \text{ cP}$ (Fig. 3.14 of Ref. 3)

$$K_G = 5.334 \times 10^{-4} \times \frac{0.0145}{0.0155} = 4.9899 \times 10^{-4} \text{ kmol/(m}^2 \cdot \text{atm} \cdot \text{s})$$

$$N_A = 4.9899 \times 10^{-4} \times 36.5 \times (0.7694 - 0.0297) = 0.01347 \text{ kg/(m}^2 \cdot \text{s})$$

Heat release due to absorption = $0.01347 \times 1780.82 = 23.9876 \text{ kW/m}^2$

$$\frac{\phi}{A} = U \Delta t = 1232.6 \times (54 - 33.27) = 25551.8 \text{ W/m}^2 = 25.55 \text{ kW/m}^2$$

3rd trial

Let temperature of 26% Acid film = 52.5°C

$$\bar{p}_{AL}^* = 20.563 \text{ torr} = 0.027 \text{ atm}$$

$$N_A = 4.9899 \times 10^{-4} \times 36.5 (0.7694 - 0.027) = 0.01352 \text{ kg/(m}^2 \cdot \text{s)}$$

Heat release due to absorption = $0.01352 \times 1780.82 = 24.076 \text{ kW/m}^2$

$$\frac{\phi}{A} = U \Delta t = 1232.6 \times (52.5 - 33.27) = 23703 \text{ W/m}^2 \equiv 23.7 \text{ kW/m}^2$$

$$\frac{\phi}{A} = U \Delta t \equiv \text{Heat release due to absorption}$$

Temperature of acid film = 52.5°C

Heat transfer between point 1 and 2:

$$\phi_{12} = 167.0435 \text{ kW}, \quad U_O = 1232.6 \text{ W/(m}^2 \cdot {}^\circ\text{C)}$$

$$\Delta T_{ln} = \frac{(52.5 - 33.27) - (36.5 - 32)}{\ln\left(\frac{52.5 - 33.27}{36.5 - 32}\right)} = 10.142^\circ\text{C}$$

$$\text{Area required } A_{12} = \frac{\phi_{12}}{U_O \Delta T_{ln}} = \frac{167.0434 \times 10^3}{1232.6 \times 10.142} = 13.362 \text{ m}^2$$

$$N_t \pi d_O L_{12} = A_{12} = 13.362 \text{ m}^2 \text{ or } L_{12} = \frac{13.362}{57 \times \pi \times 0.025} = 2.984 \text{ m}$$

Point 3: At this point acid film contains 20% by mass HCl

Let liquid film temperature at point 3 = 68°C

Table 9.13 Liquid Composition at Point 3

	kg/h	mass %	kmol/h
HCl	893.56	20%	24.48
H ₂ O	3574.25	80%	198.57
	4467.81	100%	223.05

Table 9.14 Gas Composition at Point 3

	kg/h	kmol/h	mole %
HCl	976.56	26.755	69.9
Cl ₂	818.00	11.52	30.1
	1794.56	38.275	100%

Equilibrium partial pressure of HCl over 20% HCl solution

$$\log \bar{p}_{AL}^* = 10.3833 - \frac{3245}{T} = 10.3833 - \frac{3245}{(273 + 68)}$$

$$\bar{p}_{AL}^* = 7.365 \text{ torr} \equiv 0.0097 \text{ atm}$$

$$\bar{p}_{AL} = p_t \times y_{\text{HCl}} = 131.325 \times 0.699 = 91.8 \text{ kPa} \equiv 0.906 \text{ atm}$$

Average molar mass of gas mixture,

$$M_{\text{av}} = \sum(M_i y_i) = 0.699 \times 36.5 + 0.301 \times 71 = 46.885$$

$$\text{Mass velocity, } G_t = \frac{1794.56/3600}{57 \times (\pi/4)(0.02)^2} = 23.8375 \text{ kg/(m}^2 \cdot \text{s})$$

$$\text{At } 68^\circ\text{C, } \mu = \mu_{\text{HCl}} \equiv \mu_{\text{Cl}_2} = 0.0162 \text{ cP}$$

$$K_G = \frac{1.654 \times 10^{-5}}{M_{\text{av}}^{1.75}} \left(\frac{d_t G_t}{\mu} \right) = \frac{1.654 \times 10^{-5}}{46.885^{1.75}} \times \left(\frac{0.02 \times 23.8375}{0.0162 \times 10^{-3}} \right)$$

$$K_G = 5.7943 \times 10^{-4} \text{ kmol/(atm} \cdot \text{m}^2 \cdot \text{s})$$

Rate of absorption,

$$N_A = K_G M (\bar{p}_{AG} - \bar{p}_{AL}^*) = 5.7943 \times 10^{-4} \times 36.5 \times (0.906 - 0.0097) \\ = 0.018956 \text{ kg/(m}^2 \cdot \text{s})$$

Heat of solution of 20% HCl solution $\Delta H_s = (-160.08) - (-92.31)$

$$\Delta H_s = -67.77 \text{ kJ/mol} \equiv -67770 \text{ J/mol} \equiv -1856.7 \text{ kJ/kg}$$

$$\text{Rate of heat generated due to absorption} = N_A \times (-\Delta H_s) \\ = 0.018956 \times 1856.7 = 35.1956 \text{ kW/m}^2$$

Amount of HCl absorbed in between point 3 and 2 = 362.26 kg/h

Heat duty required in between point 2 and 3

$$\phi_{23} = \phi_t - \phi_{12} - \text{Heat required to form 20% HCl solution}$$

$$\text{Heat required to form 20% HCl solution} = -\Delta H_s \times 893.56 \text{ kg/h} \\ = 1856.7 \times 893.56 = 1659072.9 \text{ kJ/h} \equiv 460.85 \text{ kW}$$

$$\phi_{23} = 788.26 - 167.044 - 460.85 = 160.366 \text{ kW}$$

$$= 160.366 \text{ kW} = q_{cw} \cdot C_L (t - 33.27)$$

$$160.366 = 31.379 \times 4.1868 \times (t - 33.27)$$

$$t = 34.49^\circ\text{C} = \text{Temperature of cooling water at point 3}$$

$$\frac{\phi}{A} = U \Delta t = 1232.6 \times (68 - 34.49) = 41304 \text{ W/m}^2 \equiv 41.304 \text{ W/m}^2$$

\neq Rate of heat generated due to absorption

Ind trial: Let the temperature of 20% Acid film at point 3 = 63°C

$$\log \bar{p}_{AL}^* = 10.3833 - \frac{3245}{(63 + 273)}$$

$$\bar{p}_{AL}^* = 5.3157 \text{ torr} = 0.007 \text{ atm}$$

$$\text{At } 63^\circ\text{C, } \mu = \mu_{\text{HCl}} = \mu_{\text{Cl}_2} = 0.0161 \text{ cP}$$

$$K_G = 5.7943 \times 10^{-4} \times \frac{0.0162}{0.0161} = 5.8303 \times 10^{-4} \text{ kmol}/(\text{atm} \cdot \text{m}^2 \cdot \text{s})$$

Temperature of acid film = 52.5°C

$$N_A = 5.8303 \times 10^{-4} \times 36.5 \times (0.906 - 0.007) = 0.01913 \text{ kg}/(\text{m}^2 \cdot \text{s})$$

$$\begin{aligned} \text{Rate of heat generated due to absorption} &= N_A \times (-\Delta H_s) \\ &= 0.01913 \times 1856.7 = 35.52 \text{ kW/m}^2 \end{aligned}$$

$$\frac{\phi}{A} = U \Delta t = 1232.6 \times (63 - 34.49) = 35141 \text{ W/m}^2 \equiv 35.141 \text{ kW/m}^2$$

$$\frac{\phi}{A} = U \Delta t \equiv \text{Rate of heat generated due to absorption}$$

Temperature of acid film at point 3 = 63°C

$$\phi_{23} = 160.336 \text{ kW}, \quad U = 1232.6 \text{ W}/(\text{m}^2 \cdot ^\circ\text{C})$$

$$\Delta T_{ln} = \frac{(63 - 34.49) - (52.5 - 33.27)}{\ln\left(\frac{63 - 34.49}{52.5 - 33.27}\right)} = 23.566^\circ\text{C}$$

$$A_{23} = \frac{\phi_{23}}{U \Delta T_{ln}} = \frac{160.336 \times 10^3}{1232.6 \times 23.566} = 5.5198 \text{ m}^2 = N_t \pi d_o L_{23}$$

$$57 \times \pi \times 0.025 \times L_{23} = 5.5198 \text{ m}^2$$

$$L_{23} = 1.233 \text{ m}$$

Point 4: Assume that at this point acid contains 12% HCl.

Table 9.15 Liquid Composition at Point 4

	kg/h	mass %
HCl	487.4	12
H ₂ O	3574.25	88
	4061.65	100

Table 9.16 Gas Mixture Composition at Point 4

	kg/h	mass %	kmol/h	mole %
Cl ₂	818	37.17	11.52	23.32
HCl	1382.72	62.83	37.883	76.68
	2200.72	100	49.403	100

Let temperature of acid film at point 4 = 72°C

Equilibrium partial pressure of HCl over 12% HCl solution

$$\begin{aligned} \log \bar{p}_{AL}^* &= 10.79 - \frac{3765}{T} = 10.79 - \frac{3765}{(273 + 72)} \\ &= 0.753 \text{ torr} \equiv 9.9 \times 10^{-4} \text{ atm} \end{aligned}$$

$$\bar{P}_{AG} = y_{HCl} \times P_t = 0.7668 \times 131.325 = 100.7 \text{ kPa} = 0.9938 \text{ atm}$$

$$M_{av} = \sum(y_i M_i) = 0.7668 \times 36.5 + 0.2332 \times 71 = 44.55 \text{ kg/mol}$$

$$G_t = \left(\frac{2200.72 / 3600}{57 \times (\pi/4)(0.02)^2} \right) = 34.138 \text{ kg/(m}^2 \cdot \text{s})$$

Mass transfer coefficient

$$K_G = \frac{1.654 \times 10^{-5}}{M_{av}^{1.75}} \left(\frac{d_i G_t}{\mu} \right)$$

At 72°C,

$$\mu = \mu_{HCl} = \mu_{Cl_2} = 0.0163 \text{ cP}$$

$$K_G = \frac{1.654 \times 10^{-5}}{44.55^{1.75}} \times \left(\frac{0.02 \times 34.138}{0.0163 \times 10^{-3}} \right) = 9.0185 \times 10^{-4} \text{ kmol/(m}^2 \cdot \text{atm} \cdot \text{s})$$

Rate of absorption

$$N_A = K_G M (\bar{P}_{AG} - \bar{P}_{AL}^*) = 9.0185 \times 10^{-4} \times 36.5 (0.9938 - 9.9 \times 10^{-4}) \\ = 0.03268 \text{ kg/(m}^2 \cdot \text{s})$$

Heat of formation of 12% HCl solution $\Delta H_f^\circ = -163 \text{ kJ/mol}$

Heat of solution of 12% HCl solution

$$\Delta H_S = (\Delta H_f^\circ)_{12\% \text{ HCl}} - (\Delta H_f^\circ)_{HCl \text{ gas}} \\ = -163 - (-92.31) = -70.69 \text{ kJ/mol} \equiv -70690 \text{ kJ/mol} \equiv -1936.7 \text{ kJ/kg}$$

Heat release due to absorption

$$= 0.03268 \times 1936.7 = 63.29 \text{ kW/m}^2$$

Heat duty required in between points 3 and 4

$$\phi_{34} = \phi_i - \phi_{12} - \phi_{23} - \text{Heat duty required to form 12% HCl solution}$$

$$\phi_{34} = 788.26 - 167.044 - 160.366 - (1936.7 \times 487.4) \times \frac{1}{3600} \\ = 198.64 \text{ kW} = q_{cw} C_l \Delta t_{34} = 31.379 \times 4.1868 \times (t - 34.49)$$

$t = 36^\circ\text{C}$ = Temperature of cooling water at point 4

$$\frac{\phi}{A} = U \Delta t = 1232.6 \times (72 - 36) = 44374 \text{ W/m}^2 \equiv 44.374 \text{ kW/m}^2$$

Hence $U \Delta t \neq$ heat release due to absorption

2nd trial

Let film temperature of 12% HCl solution = 85°C

$$\log \bar{P}_{AL}^* = 10.79 - \frac{3765}{(85 + 273)}$$

$$\bar{P}_{AL}^* = 1.876 \text{ torr} \equiv 0.0025 \text{ atm}$$

Viscosity of gas mixture $\mu = 0.017 \text{ cP}$

$$K_G = 9.0185 \times 10^{-4} \times \frac{0.0163}{0.017} = 8.647 \times 10^{-4} \text{ kmol/(m}^2 \cdot \text{atm} \cdot \text{s})$$

$$N_A = K_G M (\bar{P}_{AG} - \bar{P}_{AL}^*) = 8.647 \times 10^{-4} \times 36.5 \times (0.9938 - 0.0025) \\ = 0.03129 \text{ kg/(m}^2 \cdot \text{s})$$

Heat release due to absorption = $N_A \times (-\Delta H_S) = 0.03129 \times 1936.7 = 60.6 \text{ kW/m}^2$

$$\frac{\phi}{A} = U\Delta t = 1232.6 \times (85 - 36) = 60397 \text{ W/m}^2 = 60.397 \text{ kW/m}^2$$

\neq Heat release due to absorption

Let film temperature of 12% HCl solution = 85.1°C

$$\bar{p}_{AL}^* = 1.889 \text{ torr} = 0.0025 \text{ atm}$$

$$N_A = 8.647 \times 10^{-4} \times 36.5 \times (0.9938 - 0.0025) = 0.031287 \text{ kg/(m}^2 \cdot \text{s})$$

Heat release due to absorption = $0.031287 \times 1936.7 = 60.59 \text{ kW/m}^2$

$$\frac{\phi}{A} = U\Delta t = 1232.6 \times (85.1 - 36) = 60521 \text{ W/m}^2 \equiv 60.52 \text{ kW/m}^2$$

$$\frac{\phi}{A} = U\Delta t = \text{Heat release due to absorption}$$

$$\phi_{34} = 198.64 \text{ kW}, U_O = 1232.6 \text{ W/m}^2$$

$$\Delta T_{ln} = \frac{(85.1 - 36) - (63 - 34.49)}{\ln\left(\frac{85.1 - 36}{63 - 34.49}\right)} = 37.877^\circ\text{C}$$

$$A_{34} = \frac{\phi_{34}}{U_O \Delta T_{ln}} = \frac{198.64 \times 10^3}{1232.6 \times 37.877} = 4.2547 \text{ m}^2 = N_t \pi d_O L_{34}$$

$$L_{34} = \frac{4.2547}{57 \times \pi \times 0.025} = 0.95 \text{ m}$$

Point 5: At this point concentration of Acid film is 5% HCl (by mass).

Table 9.17 Liquid Composition at Point 5

	kg/h	mass %
HCl	188.12	5
H ₂ O	3574.25	95
	3762.37	100

Let film temperature of 5% HCl solution = 97.5°C

For 5% HCl solution

$$\log \bar{p}_{AL}^* = 11.4272 - \frac{4336.5}{T}$$

$$\bar{p}_{AL}^* = 0.5281 \text{ torr} = 6.95 \times 10^{-4} \text{ atm}$$

$$\bar{p}_{AG} = 0.8 \times 131.325 = 105.06 \text{ kPa} = 1.0369 \text{ atm}$$

$$M_{av} = 0.8 \times 36.5 + 0.2 \times 71 = 43.4 \text{ kg/kmol}$$

$$G_t = \frac{\left(\frac{2500}{3600}\right)}{57 \times \frac{\pi}{4} (0.02)^2} = 38.78 \text{ kg/(m}^2 \cdot \text{s)}$$

Viscosity of gas mixture at 97.5°C, $\mu = 0.018 \text{ cP}$

$$K_G = \frac{1.654 \times 10^{-5}}{43.4^{1.75}} \left(\frac{0.02 \times 38.78}{0.018 \times 10^{-3}} \right) = 9.7116 \times 10^{-4} \text{ kmol/(atm} \cdot \text{m}^2 \cdot \text{s})$$

$$N_A = 9.7116 \times 10^{-4} \times 36.5 \times (1.0369 - 6.95 \times 10^{-4})$$

Rate of heat generated due to absorption = $N_A \times (-\Delta H_s)$

$$= 0.03673 \times \left(72.74 \times 1000 \times \frac{1}{36.5} \right) \\ = 73.2 \text{ kW/m}^2$$

At point 5,

$$\frac{\phi}{A} = U \Delta t = 1232.6 (97.5 - 38) = 73340 \text{ W/m}^2 \equiv 73.34 \text{ kW/m}^2$$

\cong Heat release due to absorption

Table 9.18 Gas Composition at Point 5

	kg/h	kmol/h	mole %
HCl	1682	46.082	80
Cl ₂	818	11.521	20
	2500	57.603	100

Equilibrium acid film temperature at point 5 = 97.5°C

$$\phi_{45} = 788.26 - 167.044 - 160.366 - 198.64$$

$$\phi_{45} = 262.2 \text{ kW}, \quad U = 1232.6 \text{ W/(m}^2 \cdot ^\circ\text{C})$$

$$\Delta T_{LM} = \frac{(97.5 - 38) - (85.1 - 36)}{\ln \left(\frac{97.5 - 38}{85.1 - 36} \right)} = 54.134^\circ\text{C}$$

$$A_{45} = \frac{\phi_{45}}{U_o \Delta T_{ln}} = \frac{262.2 \times 10^3}{1232.6 \times 54.134} = 3.9295 \text{ m}^2$$

$$L_{45} = \frac{A_{45}}{N_t \pi d_o} = \frac{3.9295}{57 \times \pi \times 0.025} = 0.878 \text{ m}$$

5 % HCl solution enters the falling film absorber at 53.78°C. Hence, it will absorb the heat to reach equilibrium temperature (97.5°C). Actually this direct heating of liquid film will reduce some heat duty of heat exchanger. So, further addition in heat transfer area is not required.

Table 9.19 Summary of Zonewise Calculations

Point	% HCl	Water, kg/s	Temperature of acid film	Temperature of water °C	Area, m ²	Length from, m bottom
1	32	31.379	36.5°C	32		
2	26	31.379	52.5°C	33.27	13.362	2.984
3	20	31.379	63°C	34.49	5.5198	1.233
4	12	31.379	85.1°C	36	4.2547	0.95
5	5	31.379	97.5°C	38	3.9295	0.878
Total					27.066	6.045

Normally 10% excess area is provided.

$$\text{Desired HTA} = 27.066 \times 1.1 = 29.773 \text{ m}^2$$

Since tube length of 6.65 m in carbate heat exchanger is not desirable, select two heat exchangers with equal HTA in series.

$$\text{HTA per heat exchanger} = 29.773/2 = 14.9 \text{ m}^2$$

Let tube length be L .

$$57 \times L \times \pi \times 0.025 = 14.9$$

$$L = 3.328 \text{ m}$$

Carbate tubes of length 3.328 m (min.) will have to be provided.

Comments:

For the given case, falling film absorber with glass tubes can be considered as another option. But glass falling film absorber with once through operation for the given case cannot be used. Conductive coefficient, offered by glass tubes, is very low hence it will control the value of overall coefficient. For absorption of HCl in water with once through operation in glass tube heat exchanger, temperature of acid film in upper portion of absorber reaches boiling temperature. However, glass falling film absorber can be used for the given case with only recirculation of hydrochloric acid solution. To reduce the acid film temperature, gas feed can be diluted by air. Recirculation of hydrochloric acid solution or addition of air in gas feed decreases acid film temperatures but increases heat transfer area required for the falling film absorber.

Example 9.5

Design a sieve plate type absorber for the absorption of formaldehyde from the Reactor Exit Gas Mixture by water to make 37% (by mass) of formaldehyde solution in water. Material balance data around absorber of formaldehyde plant (Via Formox process) are given as follows.

Reactor exit gas is at 110°C (from recuperative heat exchanger) and at 135 kPa a pressure.

Enthalpies of reactor exit gas mixture, absorber outlet gas mixture, bottom product solution and fresh water with reference to 298.15 K (25°C) are 1141.77 kW, 295.38 kW, 216.35 and 32.17 kW respectively. Exothermic heat of solution of formaldehyde gas in water is substantially independent of concentration and has a value of 62.75 kJ/mol HCHO at 298.15 K up to a concentration of about 40% (by mass) of HCHO. Fresh water enters at 30°C and product solution leaves the absorber at 50°C. Temperature of exit gas from absorber is 50°C (Ref: 10)

Table 9.20 Stream Compositions Across Absorber¹⁰

Component	Reactor Exit Gas		Absorber Outlet Gas		Bottom Product	
	kg/h	mass %	kg/h	mass %	kg/h	mass %
CH ₃ OH	40	0.09	nil	—	40.0	0.44
O ₂	6806.8	15.81	6806.8	17.21		
N ₂	29 626.8	68.81	29 626.8	74.9		
H ₂ O	2766.0	6.42	2649.0	6.7	5649.1	62.56
HCHO	3341.3	7.76	Nil	—	3341.3	37.00
CO ₂	386.6	0.9	386.6	0.98		
CO	27.7	0.07	27.7	0.07		
H ₂	4.0	0.01	4.0	0.01		
CH ₄	9.9	0.02	9.9	0.02		
(CH ₃) ₂ O	45.5	0.11	45.5	0.11		
Total	43 054.6	100.00	39 556.3	100.00	9030.4	100.00

Solution:

$$\text{Total moles of formaldehyde absorbed} = \frac{3341.3}{30} = 111.376 \text{ kmol/h}$$

Heat of solution = 62.75 kJ / mol

Total heat evolved by absorption

$$= 111.376 \times 62.75 \times 1000 = 6988 844 \text{ kJ/h} \equiv 1941.35 \text{ kW}$$

This exothermic physical absorption is carried out either in tray tower or packed tower in actual practice. In packed tower or tray tower, recirculation streams are provided and these recirculation streams are passing through, external coolers. Recirculation streams are provided to control the temperature of absorption. To achieve the complete absorption, final contact of exit gas is with fresh water, which is fed from the top.

In tray tower, intercoolers are provided. These intercoolers may be shell and tube type or plate type or spiral heat exchangers. Here tray tower with sieve trays is designed.

Total heat to be removed by coolers

$$\begin{aligned} &= \text{Total enthalpy in} + \text{Heat generated due to absorption} - \text{Total enthalpy out} \\ &= (1141.77 + 32.17) + 1941.35 - (295.38 + 216.35) = 2603.56 \text{ kW} \end{aligned}$$

Flow rate of fresh water fed at the top of tower = 5649.1 + 2649 - 2766 = 5532.1 kg/h

Absorption of formaldehyde in water is highly exothermic but it can be carried out approximately in isothermal condition by using intercoolers and providing recirculation streams.

Let the average temperature of absorption in the entire tower is 45°C.

Consider the top most section of tower [Refer Figures (4.2) and (4.7)]. To achieve the complete absorption of formaldehyde, gas mixture must be finally contacted with pure water. Recirculation cannot be provided in the top most section. If dilute solution of formaldehyde is fed from the top with fresh water, then it limits the concentration of formaldehyde in the exist gas.

Assuming that the heat of solution is utilized only in increasing the temperature of formaldehyde solution. Fix the temperature of formaldehyde solutions leaving the top-most section is 60°C.

Enthalpy of solution leaving the top most (first) section – Enthalpy of fresh water

$$= \text{Heat of solution} \times \text{Formaldehyde absorbed in top section}$$

$$\begin{aligned} & \left(\frac{5532.1}{3600} \right) C_L (60 - 25) - \frac{(5532.1 \times 4.1868 (30 - 25))}{3600} \\ & = \frac{62.75 \times 1000}{30 \times 3600} \left(\frac{5532.1}{1 - \frac{w_1}{100}} - 5532.1 \right) \quad (a) \end{aligned}$$

where, w_1 = Mass % of formaldehyde in solution leaving the 1st section
Specific heat of formaldehyde solution is given by following equation¹².

$$C_L = 4.1868 [1 - 0.00534 w + (0.004 + 0.00001 w) t] \quad (b)$$

where, C_L is in $\text{kJ}/(\text{kg} \cdot ^\circ\text{C})$, w is mass%, t is temperature in $^\circ\text{C}$.

Here, $t = \frac{60 + 25}{2} = 42.5^\circ\text{C}$ and $w = w_1$

$$C_L = 4.9 - 0.0206 w_1 \quad (c)$$

From (a) and (c)

$$\begin{aligned} \frac{1.5367}{(1 - 0.01 w_1)} (4.9 - 0.0206 w_1) (60 - 25) - 32.17 &= 0.581 \times 5532.1 \left(\frac{1}{1 - 0.01 w_1} - 1 \right) \\ \frac{53.7845}{1 - 0.01 w_1} (4.9 - 0.0206 w_1) - 32.17 &= 3214.15 \left(\frac{1}{1 - 0.01 w_1} - 1 \right) \end{aligned}$$

By trial and error calculations

For $w_1 = 7.0$, LHS \equiv RHS = 242 kW

Hence mass % of formaldehyde in solution leaving the topmost (1st) section = 7.0 %
This solution is leaving at 60°C .

Provide the first intercooler to cool this solution from 60°C to 40°C by cooling water.

Heat duty of first intercooler.

$$\phi_I = \dot{m} C_L (60 - 40)$$

$$\dot{m} = \frac{5532.1}{1 - 0.01 \times 7.0} = 5948.5 \text{ kg/h} \equiv 1.6524 \text{ kg/s}$$

$$C_L = 4.1868 \left[(1 - 0.00534 \times 7.0) + (0.004 + 0.00001 \times 7) \left(\frac{40 + 60}{2} \right) \right]$$

$$C_L = 4.88 \text{ kJ}/(\text{kg} \cdot ^\circ\text{C})$$

$$\phi_I = 1.6524 \times 4.88 (60 - 40) = 161.27 \text{ kW}$$

Stream leaves the first intercooler and enters at the top of second section which is 7% (by mass) formaldehyde solution at 40°C .

This stream is mixed with recirculated stream as shown in Figs (4.2) and (4.7).

Let recirculation ratio = R

Mass flow rate of recirculated stream = $R \times 1.6524$

Total mass flow rate of formaldehyde solution feed to the top of the second section = $(R + 1) \times 1.6524$.

% by mass of formaldehyde in liquid solution leaving the second section = 37%

Mass flow rate of formaldehyde solution leaving the second section

$$= R \times 1.6524 + \frac{\frac{5532.1}{3600}}{1 - \left(\frac{37}{100} \right)} = (R \times 1.6524 + 2.4392) \text{ kg/s}$$

This stream is leaving at 50°C.

Enthalpy balance around second section:

Enthalpy of solution leaving the second section – Enthalpy of recirculated stream
– Enthalpy of solution coming from 1st section. = Heat of solution × Formaldehyde absorbed in 2nd section.

$$(1.6524 \times R + 2.4392) C_L (50 - 25) - 1.6524 R C'_L \times (40 - 25) - 1.6524 C''_L \times (40 - 25)$$

$$= \frac{62.75 \times 1000}{30} (2.4392 - 1.6524) \quad (\text{I})$$

$$\text{where, } C_L = 4.1868 \left[1 - 0.00534 \times 37 + (0.004 + 0.00001 \times 37) \left(\frac{50+25}{2} \right) \right]$$

$$C_L = 4.0457 \text{ kJ/(kg} \cdot ^\circ\text{C}) \quad (\text{II})$$

$$C'_L = 4.1868 \left[1 - 0.00534 \times 37 + (0.004 + 0.00001 \times 37) \left(\frac{40+25}{2} \right) \right]$$

$$C'_L = 3.9542 \text{ kJ/(kg} \cdot ^\circ\text{C}) \quad (\text{III})$$

$$C''_L = 4.1868 \left[1 - 0.00534 \times 7 + (0.004 + 0.00001 \times 7) \left(\frac{40+25}{2} \right) \right]$$

$$C''_L = 4.5841 \text{ kJ/(kg} \cdot ^\circ\text{C}) \quad (\text{IV})$$

From (I), (II), (III) and (IV)

$$(1.6524 \times R + 2.4392) \times 4.0457 (50 - 25) - 1.6524 R \times 3.9542 (40 - 25) - 1.6524 \\ \times 4.5841 (40 - 25)$$

$$= \frac{62.75 \times 1000}{30} (2.4392 - 1.6524)$$

$$69.119 R - 133.085 = 1645.723$$

$$R = 25.7354$$

$$\begin{aligned} \text{Amount of recirculated stream back to the top of second section} &= 25.7354 \times 1.6524 \\ &= 42.525 \text{ kg/s} \end{aligned}$$

Second intercooler will cool this stream from 50°C to 40°C.

Heat duty of second intercooler,

$$\phi_2 = \dot{m} C_L (50 - 40) = 42.525 \times C_L (50 - 40)$$

$$C_L = 4.1868 \left[1 - 0.00534 \times 37 + (0.004 + 0.00001 \times 37) \left(\frac{50+40}{2} \right) \right]$$

$$C_L = 4.1829 \text{ kJ/(kg} \cdot ^\circ\text{C)}$$

$$\phi_2 = 1779 \text{ kW}$$

Let cooling water enters to this cooler at 32°C and leave at 37°C . Mass flow rate of cooling water required.

$$\dot{m}_w = \frac{1779}{4.1868 \times (37 - 32)} = 84.98 \text{ kg/s}$$

Flow rate of cooling water required $= 84.98 \times 3600 = 305\,928 \text{ kg/h} \equiv 306 \text{ m}^3/\text{h}$.

It is planned to use the same stream of cooling water at 37°C as a cooling medium for both, top section inter cooler and inlet gas cooler on parallel basis. Let the outlet temperature of cooling water from top section intercooler is equal to 39°C .

$$\phi_1 = 161.27 \text{ kW} = \dot{m}_1 \times 4.1868 \times (39 - 37)$$

$$\dot{m}_1 = 19.26 \text{ kg/s}$$

Remaining, $\dot{m}_2 = 84.98 - 19.26 = 65.72 \text{ kg/s}$ will be used for the gas cooler.

Calculations for heat duty of gas cooler:

Reactor exist gas is at 110°C and at 135 kPa pressure. This gas mixture is cooled from 110°C to 55°C in cooler.

(a) Dew point temperature calculation:

Mass % of water in Reactor exist gas = 6.42 %

Molar flow rate of reactor exit gas

$$\begin{aligned} &= \frac{40}{32} + \frac{6806.8}{32} + \frac{29626.8}{28} + \frac{2766}{18} + \frac{3341.3}{30} + \frac{386.6}{44} + \frac{27.7}{28} + \frac{4}{2} + \frac{9.9}{16} + \frac{45.5}{46} \\ &= 1550.49 \text{ kmol/h} \end{aligned}$$

Moisture (H_2O) content of gas mixture

$$= \frac{2766/18}{1550.49 - \left(\frac{2766}{18}\right)} = 0.11001 \frac{\text{kmol H}_2\text{O}}{\text{kmol of dry gas}}$$

At dew point

$$\frac{p_v}{p_t - p_v} = 0.11001$$

$$\frac{p_v}{135 - p_v} = 0.11001$$

$$\begin{aligned} 1.11001 p_v &= 135 \times 0.11001 = 14.85135 \\ p_v &= 13.379 \text{ kPa} \end{aligned}$$

At 51.6°C temperature vapour pressure of water is 13.379 kPa. Hence, dew point temperature of gas mixture is 51.6°C . Therefore, on cooling the gas mixture from 110°C to 55°C , no condensation is expected.

Heat duty of cooler,

$$\phi_3 = \int_{(273.15+55)}^{(273.15+110)} C_{mp,i}^0 dT$$

$$\phi_3 = \Sigma a_i (383.15 - 328.15) + \frac{\Sigma b_i (383.15^2 - 328.15^2)}{2} + \frac{\Sigma c_i (383.15^3 - 328.15^3)}{3} + \frac{\Sigma d_i (383.15^4 - 328.15^4)}{4}$$

Σa_i , Σb_i , Σc_i and Σd_i are taken from Ref. 10.

$$\phi_3 = 47\ 588.5 (383.15 - 328.15) - \frac{2267.1 \times 10^{-3}}{2} (383.15^2 - 328.15^2) + \frac{15\ 211.7 \times 10^{-6}}{3} (383.15^3 - 328.15^3) - \frac{5816.3 \times 10^{-9}}{4} (383.15^4 - 328.15^4)$$

$$\phi_3 = 2617\ 367.5 - 44\ 346.20 + 106\ 035.4 - 14\ 476.6$$

$$\phi_3 = 2664\ 580.1 \text{ kJ/h} \equiv 740.16 \text{ kW}$$

Total heat removed by two intercoolers and one gas cooler

$$= \phi_1 + \phi_2 + \phi_3 \\ = 161.27 + 1779 + 740.16 = 2680.4 \text{ kW}$$

$$\phi_3 = \dot{m} \times 4.1868 (t_o - 37) = 740.16$$

$$65.72 \times 4.1868 (t_o - 37) = 740.16$$

$$t_o = 39.69^\circ\text{C}$$

Outlet temperature of cooling water from the gas cooler,

$$t_o = 39.69^\circ\text{C}$$

Calculations for number of trays required

(a) Top section:

Flow rate of fresh water at top, $L_{2w} = 5532.1 \text{ kg/h}$

$$L_2 = L_s = \frac{5532.1}{18} = 307.34 \text{ kmol/h}$$

$$x_2 = 0, X_2 = \frac{x_2}{1-x_2} = 0$$

Mass flow rate of exist gas at top = 39 556.3 kg/h

$$G_{2w} = 39\ 556.3 \text{ kg/h}$$

Molar flow rate of gas mixture at the outlet of absorber,

$$G_2 = \frac{6806.8}{32} + \frac{29\ 626.8}{28} + \frac{2649}{18} + \frac{386.6}{44} + \frac{27.7}{28} + \frac{4}{2} + \frac{9.9}{16} + \frac{45.5}{46} \\ = 1431.36 \text{ kmol/h}$$

Let concentration of formaldehyde in outgoing gas mixture = 120 ppm

$$y_2 = 120 \times 10^{-6} = 0.000\ 12$$

$$Y_2 = \frac{y_2}{1-y_2} = 0.000\ 12$$

Let x'_2 = Mole fraction of formaldehyde in solution entering to the second section.

y'_2 = Mole fraction of formaldehyde in gas mixture leaving the second section.

n_F = Moles of formaldehyde in liquid solution entering to second section

$$n_F = \frac{42.525 \times 0.37 + 1.6524 \times 0.07}{30}$$

$$n_F = 0.52833 \text{ kmol/s}$$

Moles of water in the same solution

$$n_w = \frac{42.525(1 - 0.37) + 1.6524(1 - 0.07)}{18}$$

$$n_w = 1.5737 \text{ kmol/s}$$

$$x'_2 = \frac{0.52833}{0.52833 + 1.5737} = 0.25134$$

$$X'_2 = \frac{x'_2}{1 - x'_2} = 0.33572$$

Formaldehyde absorbed in top section

$$G_S(Y'_2 - Y_2) = \frac{\left(\frac{5532.1}{1 - 0.07} - 5532.1\right)}{30}$$

$$G_S(Y'_2 - Y_2) = 13.88 \text{ kmol/h}$$

$$G_S = G_2(1 - y_2) = 1431.19 \text{ kmol/h}$$

$$1431.19(Y'_2 - 0.00012) = 13.88$$

$$Y'_2 = 0.009818$$

$$y'_2 = \frac{Y'_2}{1 + Y'_2} = 0.00972$$

At the base of tower

$$G_1 = 1550.49 \text{ kmol/h}$$

Mole fraction of formaldehyde in gas mixture

$$y_1 = \frac{\frac{3341.3}{30}}{1550.49} = 0.07183$$

$$Y_1 = \frac{y_1}{1 - y_1} = 0.0774$$

Flow rate of liquid solution leaving the tower

$$L_{1w} = R \times 1.6524 \times 3600 + 9030.4 \quad (R = 25.7354)$$

$$L_{1w} = 162121 \text{ kg/h}$$

(Note: From this stream 9030.4 kg/h is separated as the product stream and the remaining is recirculated back)

Average molar mass of product solution

$$M_{av} = \frac{100}{\frac{37}{30} + \frac{62.56}{18} + \frac{0.44}{32}} = 21.175 \text{ kg/mol}$$

$$L_1 = \frac{162121}{21.175} = 7656.25 \text{ kmol/h}$$

Mole fraction of formaldehyde in this solution.

$$x_1 = \frac{\left(\frac{162121 \times 0.37}{30} \right)}{7656.25} = 0.26116$$

$$X_1 = \frac{x_1}{1-x_1} = 0.35347$$

$$\begin{aligned} \text{Mass \% of formaldehyde at the top of second section} &= \frac{x'_2 \times 30}{x'_2 \times 30 + (1 - x'_2) \times 18} \times 100 \\ &= \frac{0.25134 \times 30 \times 100}{0.25134 \times 30 + (1 - 0.25134) \times 18} \\ w_2 &= 35.878\% \end{aligned}$$

$$\text{Average mass \% of formaldehyde in second section} = \frac{35.878 + 37}{2} = 36.44\%$$

Average temperature of absorption = 45°C

Partial pressure of formaldehyde in atmospheric air over formaldehyde solution is given by Lacy's empirical equation. (Ref: 12).

$$\text{Log } \bar{p}_v = 9.942 - 0.953 (0.488)^{w/10} - 2905/T$$

At $T = 45 + 273 = 318 \text{ K}$ and $w = 36.44\%$

$$\bar{p}_v = 5.46 \text{ torr}$$

At atmospheric pressure,

$$\text{equilibrium constant, } m = \frac{5.46/760}{(36.44/30)/(36.44/30 + (100 - 36.44)/18)}$$

$$m = 0.028 \text{ at atmospheric pressure}$$

Operating pressure in the given absorber is closed to atmospheric, hence the average value of m for second section is 0.028.

$$\frac{Y}{1+Y} = 0.028 \frac{X}{1+X}$$

$$\begin{array}{cccccccc} Y & 0 & 0.005 & 0.01 & 0.015 & 0.02 & 0.025 & 0.028 \\ X & 0 & 0.216 & 0.547 & 1.1177 & 2.336 & 6.76 & \infty \end{array}$$

$$\text{Average mass \% of formaldehyde for top section, } w = \frac{7}{2} = 3.5\%$$

$$\text{Average temperature of absorption} = \frac{30 + 60}{2} = 45^\circ\text{C}$$

$$\log \bar{p}_v = 9.942 - 0.953 (0.488)^{\frac{3.5}{10}} - 2905/T$$

$$\bar{p}_v = 1.1625 \text{ torr} \quad [\text{for } T = (273 + 45) \text{ K}]$$

$$m = \frac{(1.1625/760)}{(3.5/30)/[(3.5/30) + (96.5/18)]} = 0.07182$$

$$\frac{Y}{1+Y} = 0.07182 \frac{X}{1+X}$$

Equilibrium data 1st section

Y	0	0.001	0.002	0.003	0.004	0.006	0.008	0.01
X	0	0.014	0.0286	0.0434	0.0587	0.09	0.1242	0.16

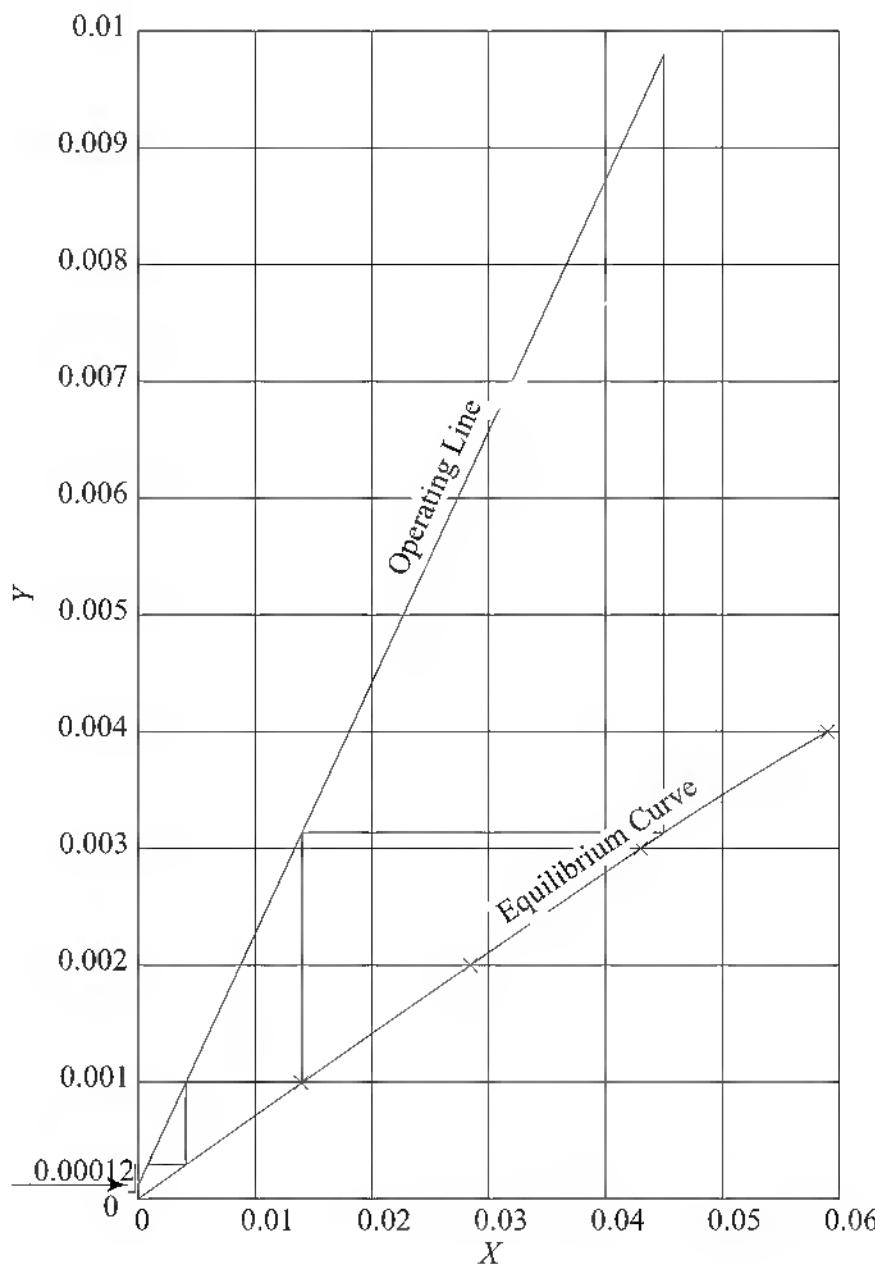


Fig. 9.16 Theoretical Stage Calculations for Top Section of Formaldehyde Absorber

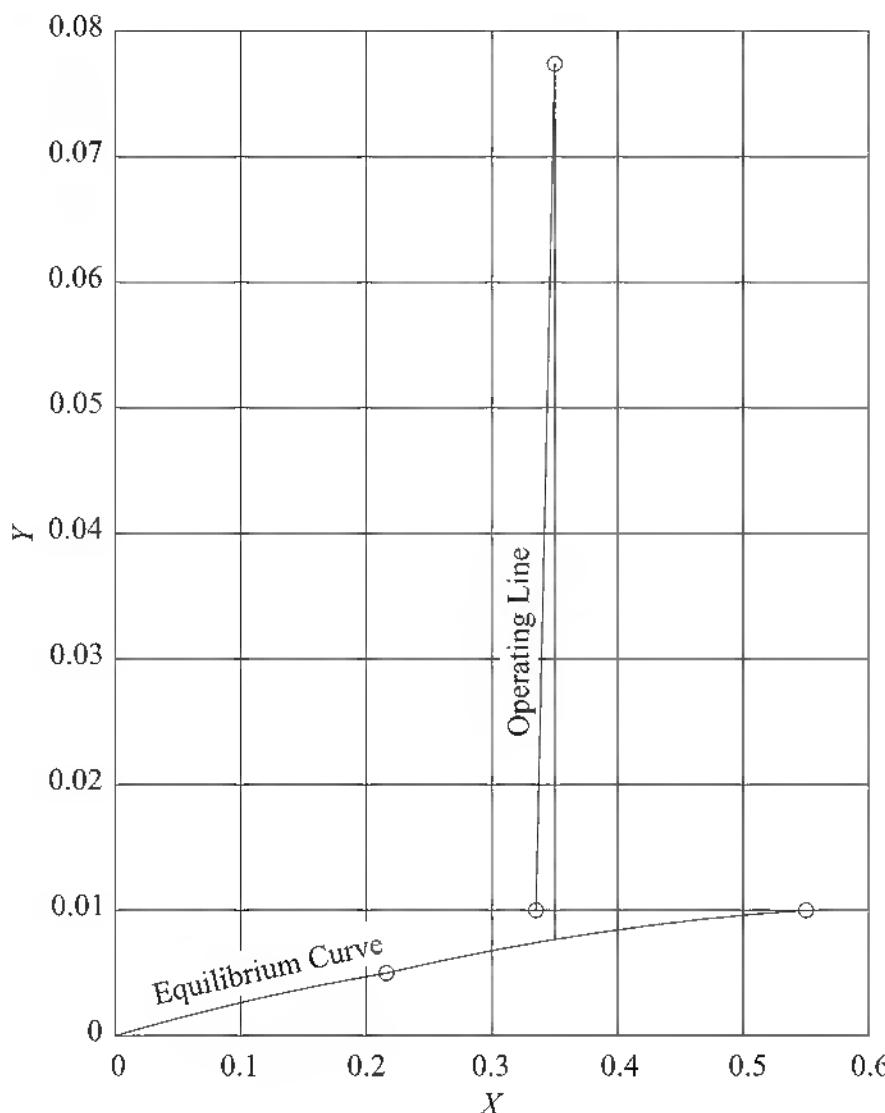


Fig. 9.17 Theoretical Stage Calculations for Bottom Section of Formaldehyde Absorber

For 1st section operating line starts from (0, 0.000 12) to (X, 0.009 817).

$$\text{where } X = \frac{x}{1-x} \text{ and } x = \frac{\frac{7}{30}}{\frac{7}{30} + \frac{93}{18}} = 0.0432$$

$$X = 0.045\ 16$$

Number of theoretical stages required in top section is $n_1 = 3.73$

For 2nd section operating line starts from (0.335 72, 0.009 818) and terminates at (0.353 47, 0.0774).

Number of theoretical stages required in bottom section is $n_2 < 1$ but assumed as 1.

Total no. of theoretical stages required for the desired separation, $n_T = 4.7304$

Tower diameter calculations

At the base of second section

$$G_{1w} = 43\ 054.6 \text{ kg/h}, \quad L_{1w} = 162\ 121 \text{ kg/h}$$

Average molar mass of gas mixture that enters the tower,

$$M_{av} = \frac{43\ 054.6 \text{ kg/h}}{1550.49 \text{ kmol/h}} = 27.768 \text{ kg/mol}$$

Liquid vapour flow factor F_{LV} for sieve tray tower

$$F_{LV} = \frac{L_w}{G_w} \sqrt{\frac{\rho_v}{\rho_L}} \quad (8.66)$$

$$L_w = L_{1w} = 162.121 \text{ kg/h}$$

$$G_w = G_{1w} = 43.054.6 \text{ kg/h}$$

$$\rho_v = \frac{p M_{av}}{RT} = \frac{135 \times 27.768}{8.3145 \times (273 + 55)} = 1.375 \text{ kg/m}^3$$

$$\rho_L = \frac{1}{\sum \frac{w_i}{\rho_{Li}}} = \frac{1}{\frac{0.37}{815} + \frac{0.6256}{988} + \frac{0.0004}{780}}$$

(Density of water at 50°C $\rho_w = 988 \text{ kg/m}^3$, $\rho_{Fo} = 815 \text{ kg/m}^3$, $\rho_{me} = 780 \text{ kg/m}^3$)

$$\rho_L = 919.37 \text{ kg/m}^3$$

$$F_{LV} = \frac{162.121}{43.054.6} \sqrt{\frac{1.375}{919.37}} = 0.1456$$

For tray spacing, $S = 0.45 \text{ m}$

From Fig. 8.16 $C_f = 0.069$

Flooding velocity

$$v_F = C_f \left(\frac{\sigma_L}{0.02} \right)^{0.2} \left(\frac{\rho_L - \rho_v}{\rho_v} \right)^{0.5} \quad (8.65)$$

σ_L = Surface tension of liquid, N/m

$$\sigma_L = \sum \sigma_i x_i = 0.26 \sigma_{Fo} + 0.74 \sigma_{wat}$$

$$\sigma_{Fo}^{1/4} = [\varphi_i] (\sigma'_L - \sigma'_G)$$

$$[\varphi_i] \text{ for HCHO} = 15.5 + 66 = 81.5$$

(Table 3-343 of Ref. 3)

$$\rho'_G = \frac{pM}{RT} = \frac{135 \times 30}{8.3145 \times (273 + 50)} = 1.5 \text{ kg/m}^3$$

$$\sigma_{Fo}^{1/4} = 81.5 \left(\frac{(815 - 1.5) \times 10^{-3}}{30} \right) = 2.21$$

$$\sigma_{Fo} = 23.85 \frac{\text{dyn}}{\text{cm}} = 23.85 \times 10^{-3} \text{ N/m}$$

$$\sigma_L = 0.26 \times 23.85 \times 10^{-3} + 0.74 \times 70 \times 10^{-3} = 58 \times 10^{-3} \text{ N/m}$$

$$v_F = 0.069 \left(\frac{0.058}{0.02} \right)^{0.2} \left(\frac{919.37 - 1.375}{1.375} \right)^{0.5}$$

$$= 2.206 \text{ m/s}$$

Let actual velocity of gas – vapour mixture through tower

$$v_1 = 0.66 v_F = 1.456 \text{ m/s}$$

Volumetric flow rate at bottom (of gas mixture)

$$q_{V1} = \frac{G_{1w}}{\rho_V} = \frac{43054.6/3600}{1.375} = 8.7 \text{ m}^3/\text{s}$$

Net area of tray required at bottom

$$A_n = \frac{q_{V1}}{v} = \frac{8.7}{1.456} = 5.975 \text{ m}^2$$

Let downcomer area, $A_d = 0.12 A_c$

where

A_c = Inside cross sectional area of tower

$$A_n = A_c - A_d = A_c - 0.12 A_c = 0.88 A_c$$

$$0.88 A_c = 5.975 \text{ m}^2$$

$$A_c = 6.79 \text{ m}^2 = \left(\frac{\pi}{4}\right) D_i^2$$

$$D_i = 2.94 \text{ m}$$

Tower diameter required at the base of top section:

At the base of top section,

$$L_w = 1.6524 \text{ kg/s}$$

Gas flow rate at the base of 1st section

G_w = Gas flow rate at top of 1st section + L_w – Solvent flow rate at top

$$G_w = 39556.3 + 1.6524 \times 3600 - 5532.1$$

$$G_w = 39972.84 \text{ kg/h} \equiv 11.1036 \text{ kg/s}$$

Molar flow rate of gas mixture at the entrance of 1st section,

$$G = G_s (1 + Y'_2) = 1431.36 (1 + 0.009818)$$

$$G = 1445.41 \text{ kmol/h}$$

$$M_{av} = \frac{39972.84}{1445.41} = 27.655 \text{ kg/kmol}$$

$$\rho_V = \frac{p M_{av}}{RT} \equiv \frac{135 \times 27.655}{8.3145 \times (60 + 273)} = 1.35 \text{ kg/m}^3$$

$$\rho_L = \frac{1}{\sum \frac{w_i}{\rho_{Li}}} = \frac{1}{\frac{0.07}{815} + \frac{0.93}{983.2}} = 969.2 \text{ kg/m}^3$$

(Density of water at 60°C = 983.2 kg/m³)

$$F_{LV} = \frac{L_w}{G_w} \sqrt{\frac{\rho_v}{\rho_L}} = \frac{1.6524}{11.1036} \sqrt{\frac{1.35}{969.2}} = 5.554 \times 10^{-3}$$

for tray spacing $S = 0.45 \text{ m}$

from Fig. 8.16 $C_f = 0.08$

Surface tension of 7 % formaldehyde solution.

$$\sigma_L = \sum \sigma_i x_i = \sigma_{\text{For}} x + (1 - x) \sigma_{\text{wat}}$$

$$x = \frac{7/30}{7/30 + 93/18} = 0.0432$$

$$\sigma_L = 0.0432 \times 23.843 \times 10^{-3} + (1 - 0.0432) \times 70 \times 10^{-3}$$

$$\sigma_L = 0.068 \text{ N/m}$$

Flooding Velocity

$$v_F = C_F \left(\frac{\sigma_L}{0.02} \right)^{0.2} \left(\frac{\rho_L - \rho_V}{\rho_V} \right)^{0.5}$$

$$= 0.08 \left(\frac{0.068}{0.02} \right)^{0.2} \left(\frac{969.2 - 1.35}{1.35} \right)^{0.5} = 2.736 \text{ m/s}$$

For 66% flooding,

$$v = 0.66 \times 2.736 = 1.8 \text{ m/s}$$

Volumetric flow rate of gas mixture at the base of 1st section.

$$q_V = \frac{11.1036}{1.35} = 8.225 \text{ m}^3/\text{s}$$

Net area of tray required,

$$A_n = \frac{q_v}{v} = \frac{8.225}{1.8} = 4.57 \text{ m}^2$$

Let downcomer area = 12 % of column area (A_c)

$$A_c = \frac{4.57}{0.88} = 5.19 \text{ m}^2 = \frac{\pi}{4} D_i^2$$

$$D_i = \sqrt{\frac{4 \times 5.19}{\pi}} = 2.57 \text{ m}$$

Checking of weeping for bottom section:

The minimum design vapour velocity through holes to avoid weeping is given by following equation.

$$u_h = \frac{K - 0.9(25.4 - d_h)}{\sqrt{\rho_G}} \quad (8.70)$$

u_h = Minimum gas velocity through holes, m/s

K = Constant can be obtained from Fig. 8.19

d_h = hole diameter, mm

Let $d_h = 5 \text{ mm}$, $\rho_G = 1.375 \text{ kg/m}^3$

Height of weir, $h_w = 50 \text{ mm}$

Height of liquid crest over the weir

$$h_{ow} = 750 \left[\frac{L_w}{\rho_L l_w} \right]^{\frac{2}{3}} \quad (8.69)$$

L_w = Liquid flow rate, kg/s

$$L_w = 162.121 \text{ kg/h} = 45.03 \text{ kg/s}$$

ρ_L = Density of liquid = 919.37 kg/m³

l_w = Length of weir, m

$$\text{For } \frac{A_d}{A_c} = 0.12 \Rightarrow \frac{l_w}{D_i} = 0.76$$

$$l_w = 0.76 \times 2.94 \text{ m} = 2.2344 \text{ m}$$

$$h_{ow} = 750 \left[\frac{45.03}{919.37 \times 2.2344} \right]^{2/3} = 58.7 \text{ mm}$$

$$h_w + h_{ow} = 108.7 \text{ mm}, \quad K = 31.2$$

$$u_h = \frac{31.2 - 0.9(25.4 - 5)}{\sqrt{1.375}}$$

$$u_h = 10.95 \text{ m/s},$$

Maximum hole area required

$$A_{hm} = \frac{8.7}{10.95} = 0.7945 \text{ m}^2$$

$$\frac{A_{hm}}{A_c} = \frac{0.7945}{6.79} = 0.117$$

$$\text{Let } A_h = 0.08 A_c = 0.5432 \text{ m}^2$$

Actual velocity, of gas through holes, $V_w = 16 \text{ m/s} > 10.95 \text{ m/s}$

Hence, weeping will not take place.

Checking of weeping for top section:

For the trays of top section, let hole diameter, $d_h = 5 \text{ mm}$

Height of weir, $h_w = 50 \text{ mm}$

Height of liquid crest over the weir

$$h_{ow} = 750 \left[\frac{L_w}{\rho_L l_w} \right]^{2/3} \quad (8.69)$$

L_w = Liquid flow rate = 1.6524 kg/s

ρ_L = 969.2 kg/m³

$$\text{For } \frac{A_d}{A_c} = 0.12 \Rightarrow \frac{l_w}{D_i} = 0.76$$

Length of weir,

$$l_w = 0.76 \times 2.57 = 1.9532 \text{ m}$$

$$h_{ow} = 750 \left[\frac{1.6524}{969.2 \times 1.9532} \right]^{2/3}$$

$$h_{ow} = 6.85 \text{ mm}$$

$$\text{for } h_w + h_{ow} = 56.85 \text{ mm}, \quad K = 30.2$$

Minimum vapour velocity required through holes to avoid weeping

$$u_h = \frac{K - 0.9(25.4 - d_h)}{\sqrt{\rho_G}} \quad (8.70)$$

$$u_h = \frac{30.2 - 0.9(25.4 - 5)}{\sqrt{1.35}}$$

$u_h = 10.19 \text{ m/s}$ under weeping conditions

Maximum hole area which can be provided,

$$A_{hm} = \frac{8.225}{10.19} = 0.8072 \text{ m}^2$$

$$\frac{A_{hm}}{A_c} = \frac{0.8072 \text{ m}^2}{\frac{\pi}{4}(2.57)^2} = 0.1556$$

Let $\frac{A_h}{A_c} = 0.1$

$$A_h = 0.1A_c = 0.51875 \text{ m}^2$$

Actual velocity of gas through holes,

$$v_h = \frac{8.225}{0.51875} = 15.855 \text{ m/s} > 10.10 \text{ m/s}$$

Hence weeping will not take place.

Checking of downcomer flooding for the bottom section:

Clear liquid back-up in downcomer,

$$h_b = h_w + h_{ow} + h_t + h_{dc} \quad (8.81)$$

where, h_t = Total tray pressure drop, mm LC

h_{tC} = head loss in downcomer, mm LC

$$h_t = h_d + (h_w + h_{ow}) + h_r \quad (8.73)$$

where, h_d = Dry tray pressure drop, mm LC

h_r = Residual pressure drop, mm LC

$$h_d = 51 \left(\frac{v_h}{C_o} \right)^2 \frac{\rho_G}{\rho_L} \quad (8.74)$$

where, C_o = Orifice coefficient can be determined by using Fig. 8.20

Let plate thickness = 5 mm,

$$\frac{\text{Plate thickness}}{\text{hole diameter}} = \frac{5}{5} = 1$$

$$\frac{A_h}{A_p} \cong \frac{A_h}{A_a} = \frac{A_h}{A_c - 2A_d} = \frac{A_h}{A_c - 2 \times 0.12 A_c}$$

$$\frac{A_h}{A_p} = \frac{A_h}{0.76 A_c} = \frac{0.08}{0.76} = 0.1053$$

$C_o = 0.845$ (From Fig. (8.20))

$$h_d = 51 \times \left(\frac{15.67}{0.845} \right)^2 \times \frac{1.375}{919.37} = 26.23 \text{ mm LC}$$

Residual pressure drop,

$$h_r = \frac{12.5 \times 10^3}{\rho_L} = \frac{12.5 \times 10^3}{919.37} = 13.6 \text{ mm LC}$$

$$h_t = 26.23 + (50 + 58.9) + 13.6 = 148.73 \text{ mm}$$

$$h_b = 50 + 58.9 + 148.73 + h_{dc}$$

head loss in downcomer,

$$h_{dc} = 166 \left[\frac{L_{wd}}{\rho_L A_m} \right]^2 \quad (8.78)$$

L_{wd} = Liquid flow rate in downcomer, kg/s

$A_m = A_d$ or A_{ap} whichever is smaller

$A_{ap} = h_{ap} l_w$

h_{ap} = Clearance between bottom edge of downcomer apron and tray

$h_{ap} = h_w - (5 \text{ to } 10 \text{ mm})$

$$h_{dc} = 166 \left(\frac{45.03}{919.37 \times A_m} \right)^2$$

$$A_m = A_d (0.47 \text{ m}^2) \text{ or } A_{ap} (0.04 \times 2.2344 = 0.08938 \text{ m}^2)$$

$$A_m = 0.08938 \text{ m}^2$$

$$h_{dc} = 166 \left(\frac{45.03}{919.37 \times 0.08938} \right)^2$$

$$h_{dc} = 49.85 \text{ mm}$$

$$h_b = 50 + 58.9 + 148.73 + 49.85$$

$$h_b = 307.48 \text{ mm}$$

$$S + h_w = 450 \text{ mm} + 50 \text{ mm} = 500 \text{ mm}$$

$$h_b > 0.5 (S + h_w)$$

Hence, downcomer flooding will take place. To avoid flooding, let tray spacing in bottom section, $S = 600 \text{ mm}$

For $F_{LV} = 0.1456$

$C_F = 0.09$

(From Fig. (8.16))

$$\text{Flooding velocity, } v_F = \frac{0.09}{0.069} \times 2.206$$

$$v_F = 2.877 \text{ m/s}$$

$$v_1 = 0.66 v_F = 1.9 \text{ m/s}$$

$$A_n = \frac{8.7}{1.9} = 4.579 \text{ m}^2$$

$$A_n = \frac{4.579}{0.88} = 5.2034 \text{ m}^2$$

$D_i = 2.574 \text{ m}$ (Nearly same as top section)

Length of weir, $l_w = 0.76 \times 2.574 = 1.956 \text{ m}$

$$h_{ow} = 750 \left(\frac{45.03}{919.37 \times 1.956} \right)^{2/3}$$

$$h_{ow} = 64.2 \text{ mm}$$

$$h_w + h_{ow} = 50 + 64.2 = 114.2 \text{ mm}$$

$$K = 31.2 \text{ (from Fig. 8.19)}$$

Minimum vapour velocity required through holes,

$$u_h = \frac{31.2 - 0.9(25.4 - 5)}{\sqrt{1.375}}$$

$$u_h = 10.95 \text{ m/s}$$

Let $\frac{A_h}{A_c} = 0.1$

$$A_h = 0.1 \times \frac{\pi}{4} (2.574)^2 = 0.52 \text{ m}^2$$

Actual gas velocity through holes

$$V_h = \frac{8.7}{0.52} = 16.73 \text{ m/s} > u_h = 10.95 \text{ m/s}$$

Hence, weeping will not take place.

Checking of downcomer flooding:

Dry plate pressure drop,

$$h_d = 51 \left(\frac{v_h}{C_o} \right)^2 \frac{\rho_G}{\rho_L} \quad (8.74)$$

$$\frac{\text{Plate thickness}}{\text{hole diameter}} = \frac{5}{5} = 1$$

$$\frac{A_h}{A_p} = \frac{A_h}{0.76 A_C} = \frac{0.1}{0.76} = 0.1315$$

$$C_o = 0.87 \quad (\text{From Fig. (8.20)})$$

$$h_d = 51 \times \left(\frac{16.73}{0.87} \right)^2 \times \frac{1.375}{919.37} = 28.2 \text{ mm LC}$$

$$h_t = 28.2 + 50 + 64.2 + 13.6 = 156 \text{ mm}$$

$$h_b = 50 + 64.2 + 156 + h_{dc}$$

$$A_m = A_{ap} = 0.04 \times 1.956 = 0.07824 \text{ m}^2$$

$$h_{dc} = 166 \left(\frac{45.03}{919.37 \times 0.07824} \right)^2$$

$$h_{dc} = 65 \text{ mm LC}$$

$$h_b = 50 + 64.2 + 156 + 65 = 335.2 \text{ mm LC}$$

$$S + h_w = 600 + 50 = 650 \text{ mm}$$

$$h_b < 0.5(S + h_w)$$

Let $h_{ap} = h_{ap} - 5 \text{ mm} = 45 \text{ mm}$

$$A_m = A_{ap} = 0.045 \times 1.956 = 0.088 \text{ m}^2$$

$$h_{dc} = 51.4 \text{ mm}$$

$$h_b = 321.6 \text{ mm} < 0.5(600 + 50)$$

Downcomer flooding will not take place.

Downcomer residence time,

$$\theta_r = \frac{A_d h_b \rho_L}{L_{wd}}$$

$$\theta_r = \frac{(0.12 \times 5.2034) \times 0.3216 \times 919.37}{45.03}$$

$$\theta_r = 4.1 \text{ sec} > 3 \text{ s (satisfactory)}$$

Checking of downcomer flooding in top section

Dry plate pressure drop,

$$h_d = 51 \left(\frac{v_h}{C_o} \right)^2 \frac{\rho_G}{\rho_L} \quad (8.74)$$

$$\frac{\text{Plate thickness}}{\text{Hole diameter}} = \frac{5}{5} = 1$$

$$\frac{A_h}{A_p} = \frac{A_h}{0.76 A_c} = \frac{0.1}{0.76} = 0.1315$$

$$C_o = 0.87 \text{ (From Fig. 8.20)}$$

$$h_d = 51 \left[\frac{15.855}{0.87} \right]^2 \frac{1.35}{969.2} = 23.6 \text{ mm}$$

Residual pressure drop,

$$h_r = \frac{12.5 \times 10^3}{\rho_L} = \frac{12.5 \times 10^3}{969.2} = 12.9 \text{ mm}$$

$$h_t = 23.6 + 50 + 6.85 + 12.9 = 93.35 \text{ mm}$$

$$l_w = 1.9532 \text{ m}, A_{ap} = 0.045 \times 1.9532 = 0.088 \text{ m}^2$$

Head loss in down comer,

$$h_{dc} = 166 \left[\frac{L_{wd}}{\rho_L A_m} \right]^2$$

$$= 166 \left[\frac{1.6524}{969.2 \times 0.088} \right]^2$$

$$h_{dc} = 0.0623 \text{ mm}$$

$$h_b = 50 + 6.85 + 93.35 + 0.06 \text{ mm} = 150.26 \text{ mm}$$

$$S + h_w = 450 + 50 = 500 \text{ mm}$$

$$h_b < 0.5(S + h_w)$$

Downcomer flooding will not take place.

Downcomer residence time

$$\theta_r = \frac{A_d h_b \rho_L}{L_{wd}} = \frac{0.12 \left(\frac{\pi}{4} \right) 2.57^2 \times 0.15 \times 969.2}{1.6524}$$

$$\theta_r = 54.77 \text{ s (satisfactory)}$$

Tray efficiency:

Overall tray efficiency based on O'Connell correlation for the absorption in tray tower is given in Fig. 14.7, *Perry's Chemical Engineers' Handbook*, 7th Ed., 1998.

The O'Connell parameter for top section

$$\frac{\rho_L}{K M \mu_L} = \frac{969.2}{K M \mu_L}$$

where, K = Equilibrium constant

M = Average molar mass of liquid solution

μ_L = Viscosity of liquid solution

For top section:

$$K = 0.07182$$

$$M = \frac{0.0432}{2} \times 30 + \left(1 - \frac{0.0432}{2} \right) \times 18 = 18.26$$

$$\mu = \text{Viscosity of liquid solution} = 10^{-3} \text{ Pa} \cdot \text{s}$$

$$\frac{\rho_L}{K M \mu_L} = \frac{969.2}{0.07182 \times 18.26 \times 10^{-3}} = 739\,038.8 \text{ kmol/(m}^3 \cdot \text{Pa} \cdot \text{s})$$

$$= \frac{739\,038.8}{1.6 \times 10^4} = 46.2 \text{ lb mole / (ft}^3 \cdot \text{cP)}$$

Plate efficiency, η is more than 60% (from Fig. 14.7, *Perry's Chemical Engineers Handbook*, 7th Ed.)

For bottom section:

$$\frac{\rho_L}{K M \mu_L} = \frac{919.37}{0.028 \times 21.0756 \times 1.2 \times 10^{-3}} = 1298\,288.2 \text{ kmol/(m}^3 \cdot \text{Pa} \cdot \text{s})$$

$$(M = 0.2563 \times 30 + (1 - 0.2563) \times 18 = 21.07563)$$

$$= 81.143 \text{ (lb mole/(ft}^3 \cdot \text{cP})$$

Plate efficiency, η is more than 60%

(From Fig. 14.7, *Perry's Chemical Engineers Handbook*, 7th Ed.)

However, plate efficiencies of commercial hydrocarbon absorbers are ranging from 15 to 55%.

Let tray efficiencies be 0.34 for top section and 0.33 for bottom section

Hence, actual number of sieve trays in top section will be 11 and in bottom section will be 3. Total numbers of sieve trays required is 14.

Resulting data:

Inside diameter of tower = 2574 mm

No. of trays in top section = 11

No. of trays in bottom section = 3

Flow rate of liquid (37% formaldehyde) solution recirculated and fed to the top of bottom section = 153 239 kg/h

Figure 4.7 is the P&I diagram of the entire formaldehyde manufacturing. It may be noted that entire cooling water is first supplied to HE-3 which has highest heat duty (1779 kW). Cooling water, leaving HE-3, is split into three streams. One stream is sent to HE-4. (746.32 kW), another to HE-2 (161.27 kW) and a small stream to sample cooler SC-1. This will mean a cooling range of for the cooling water flow rate of 306.0 m³/h. Thus cooling water enters at 32°C and leaves the plant at 39.55°C. In an alternate design, fresh cooling water could be supplied at 32°C to all the coolers separately and each cooler could be designed for a temperature rise to 37°C. This will mean a cooling range of 5°C for cooling water flow of 462.0 m³/h for the design of cooling tower.

In cooling tower design wet bulb temperature is an important consideration. Cold water temperature is limited by approach to the wet bulb temperature. Cooling tower with a range of 7.55°C and flow rate 306 m³/h will require lesser floor space than the cooling tower with a range of 5°C and flow rate of 462.0 m³/h. Hence, cost of cooling tower with a cooling range of 7.55°C will be lower. Also, cost of pumping cooling water will be lower.

Above discussion should not mislead to assume that coolers should always be designed for series cooling as heat transfer area requirement of the coolers also will have to be checked vis-à-vis cooling tower design. In general, a cooling range of 10 to 12°C and approach of 5°C to wet bulb temperature of air can be considered optimal for the design of cooling water system.

EXERCISES

- 9.1 Design a scrubber for absorbing dimethyl ether (DME) vapour from nitrogen-dimethyl ether vapour mixture. The temperature in the scrubber is 20°C and scrubbing is approximated as isothermal. Operating pressure of scrubber is atmospheric. A mixture of nitrogen - DME vapour contains 10% DME (by volume) and its flow rate is 60 kmol/h. The scrubber is required to absorb 98% of the DME. Molar mass of DME is 46 kg/kmol. Cold water is used as solvent.
- 9.2 Gas mixture obtained from chlorination process contains 20% hydrogen chloride gas and 80% chlorine (by mole). From the mixture, hydrogen chloride gas is to be separated by absorption in water. For the absorption of HCl, FRP (fiber glass reinforced plastic) packed tower is used. Product hydrochloric acid solution, leaving the packed tower, must contain 30% HCl (by mass). Design the FRP packed tower.
- 9.3 Falling film absorber is used in ammonia-water absorption refrigeration plant¹⁰. Refrigeration duty of this plant is 200 kW (56.87 TR). Flow rate of ammonia vapour, entering to falling film absorber is 0.1935 kg/s. It is absorbed by weak aqueous solution of ammonia having 30% strength (by mass). Product strong solution of ammonia leaving from the bottom of absorber is having 36.5% strength.

Average value of heat of absorption of ammonia for the required concentration change is 2046.6 kJ/kg. Design the falling film absorber. Temperature of ammonia gas at inlet to absorber is -10°C .

Operating pressure = 280 kPa a

Assume that Eq. (9.43) and Eq. (9.44) are also valid for the absorption of ammonia in water (in falling film absorption) to calculate mass transfer coefficient and heat transfer coefficient, respectively.

- 9.4 Venturi scrubber is planned for absorbing NO_x gases from the exist gas stream of nitric acid plant by using 10% NaOH solution (by mass). Exist gas mixture, leaving the absorber of nitric acid plant, is having the following composition (Ref. : Table 2.7 of Chapter 2).

Component	kmol/h
N_2	1275.097
O_2	61.590
Ar	16.330
NO	2.790
N_2O	2.190
H_2O	173.805
Total	1531.802

Discharge pressure of gas from venturi = atmospheric, solvent to gas ratio = 1.5 L/ m^3 .

Temperature of gas mixture, entering the venturi scrubber = 50°C .

Determine: (a) throat diameter of venturi scrubber,

(b) % removal of NO_x gases,

(c) consumption of NaOH and

(d) pressure drop in venturi scrubber.

- 9.5 Design a sour-water stripper based on following information.¹³

Feed liquid mixture flow rate = 50 000 kg/h

% of H_2S in feed = 0.73% (by mass)

% of NH_3 in feed = 0.556% (by mass)

% of H_2O in feed = 98.714% (by mass)

Feed condition = Saturated liquid at operating condition

Stripping steam rate = 5100 kg/h

Operating pressure at bottom = 2.4 atm a

Concentration of H_2S and NH_3 in bottom stream = 5 ppm and 20 ppm, respectively

Operating temperature at bottom = 126°C

Steam is generated by using reboiler at bottom. Stripped overheads are to be condensed in overhead condenser. Condensate and tail gas from this condenser are separated in reflux drum. Liquid phase from drum is recycled back to top tray. Tail gas will be sent to sulphur recovery plant.

- 9.6 From a 10 mole% NH_3 in NH_3 -air mixture, 95% of the ammonia is to be removed by countercurrent scrubbing at 1 atm and 20°C . Entering gas rate is $3600 \text{ kg}/(\text{h} \cdot \text{m}^2)$. Calculate the followings for both solvents, water and 0.1 N H_2SO_4 solution.

For $\text{NH}_3\text{-H}_2\text{O}$ system Henery's law constant at 30°C , $m = 0.85$ (Ref: 1).

(a) Minimum amount of solvent

(b) Number of theoretical stages at 1.2 times the minimum acid rate.

(c) The number of overall gas transfer units, N_{OG}

(d) H_{OG} based on K_{GA} ¹⁴.

$$K_{GA} = 78.1 \text{ kmol}/(\text{h} \cdot \text{m}^2 \cdot \text{atm}) \quad \text{for } 0.1 \text{ N H}_2\text{SO}_4 \text{ solution}$$

$$K_{GA} = 0.481 \text{ kmol}/(\text{h} \cdot \text{m}^2 \cdot \text{atm}) \quad \text{for water}$$

(e) Column height

REFERENCES

1. Treybal, R. E., *Mass Transfer Operations*, 3rd Ed., McGraw-Hill, 1980
2. Ludwig, E. E., *Applied Process Design for Chemical and Petrochemical Plants*, 3rd Ed. Vol. 2, Gulf Publishing Co., USA, 1997.
3. Perry, R. H. and Green D., *Perry's Chemical Engineers' Handbook*, McGraw-Hill, 6th edition 1984, Ch – 14 and Ch – 18.
4. Sinnott, R. K., *Coulson and Richardson's Chemical Engineering*, Vol. 6, Revised 2nd Ed., Asian Books Private Limited, New Delhi, 1988.
5. Timmermans, J., *Physico-Chemical Constants of Pure Organic Compounds*, Elsvier Publishing Co., USA, 1950.
6. Edwards, W. M. and P. Huang, *Chem. Engg. Progr.*, **73**(8), (1977) p. 64.
7. Niessen, W. R., *Combustion and Incineration Processes*, 3rd Ed., Marcel Dekker, Inc., USA, 2002.
8. Norman, W. S., *Absorption, Distillation and Cooling Towers*, Longmans, USA, 1961
9. Kister, H. Z., G. Balekjian, J. F. Litchfield, J. P. Damm and D. R. Merchant, *Chem. Engg. Progr.*, **88**(6), (1992) p. 41.
10. Bhatt, B. I. and S. M. Vora, *Stoichiometry*, 4th Ed., Tata McGraw-Hill Publishing Company Limited., 2004.
11. Kern, D. Q., *Process Heat Transfer*, McGraw-Hill, 1950.
12. Mcketta, J. J. and W. A. Cunningham (Ed.). *Encyclopedia of Chemical Processing and Design*, Marcel Dekker INC., Vol. 23, 1985, p. 353.
13. Wild, N. H., *Chem. Engg.*, **86**(4), Feb. 12, 1979, p. 103 to 113.
14. Henley, E. J. and J. D. Seader, *Equilibrium-stage Separation Operations in Chemical Engineering*, John Wiley & Sons, USA, 1981.
15. Sherwood, T. K., Pigford, R.L. and Wilke, C.R., *Mass Transfer*, McGraw-Hill, 1975

Chapter**10*****Process Design of Reactors*****10.1 INTRODUCTION**

Reactor is the most important equipment of any chemical plant. Minor change or improvement in the reactor design or in its performance greatly affects the process and mechanical design of other equipments. Sometimes it may even change the entire flow sheet. For example, in the process of manufacturing of monochloroacetic acid, reaction is carried out between acetic acid and chlorine. Conversion of acetic acid is about 90%. Product mass contains a mixture of monochloroacetic acid, dichloroacetic acid and unconverted acetic acid from which monochloroacetic acid is separated by crystallization. But, if in the same reactor, conversion of acetic acid is restricted to 50%, then dichloroacetic acid is not formed in noticeable quantity and the product mixture contains mainly acetic acid and monochloroacetic acid from which monochloroacetic acid can be separated by vacuum distillation. Thus, change in conversion of reactant in the reactor changes the entire flow sheet. Hence, for any chemical process, development in the reaction kinetics or reactor design is important in research. Major area of the research is the development of catalyst for the reaction. By developing a new catalyst, entirely a new process route can be developed. Final goal of the research for all the reactions and reactors is to carry out the reaction at atmospheric pressure and at room temperature with sufficiently high rate of reaction, 100% conversion and 100% yield. At the same time heating or cooling medium requirement to run the reaction should be minimum. To achieve this goal one can take inspiration from natural reactor like human body. In the human body thousands of reactions are running at about 37°C temperature and at a pressure close to one atmosphere. For the process design of the reactor, knowledge of various subjects of chemical engineering is required like of chemical reaction engineering, thermodynamics, stoichiometry, heat transfer operation, mass transfer operation, fluid flow operation, chemistry, etc. Process design of various types of reactors are outlined in the subsequent sections.

10.2 DIFFERENT TYPES OF REACTORS

Several types of reactors are in use in industry. Basically they fall in two categories; batch and continuous. In each category, different designs are available. In fact,

development of a reactor can be an innovative idea and a process engineer can use his skills for such a development.

In Fig. 10.1, different types of reactors are represented schematically. Designs date back to a simple batch reactor to modern continuous catalytic reactors such as ammonia converter. Designs also vary with the type of reactions; such as homogenous, heterogeneous, run-away reactions, etc.

In many cases, a number of reactors are used in series. Continuous stirred tank reactors. [Fig. 10.1(c)] can be cited as an example. In some cases, number of parallel reactors are used. For example, in reforming of hydrocarbons (such as naphtha or natural gas), several hundred tubular reactors are used in parallel in which catalyst is packed in different shapes and sizes. Selection is made on the basis of degree of conversion, ease of heat removal/addition, safety and other considerations.

10.3 BATCH REACTOR^{1, 2, 3}

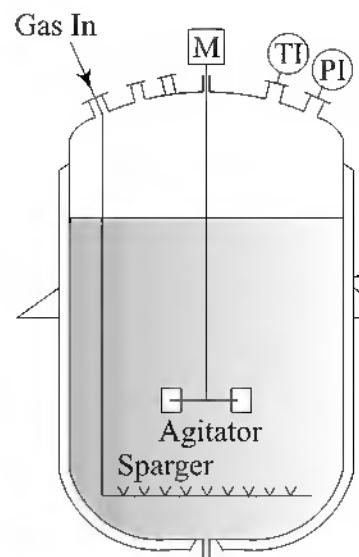
In a batch reactor all the reactants are initially charged into the reactor. In most cases, then they are allowed to react for certain period of time. The resultant mixture is then discharged. In any batch reactor, composition of reaction mass changes with time, hence it is called unsteady state reactor.

In an ideal batch reactor at any instant, composition throughout the reactor is uniform while the same is not true for a non-ideal batch reactor. Size of batch reactor does not directly depend on the reaction time but it depends on volume of material charged to the reactor. For small capacity plant having production capacity less than 5 tonnes per day, batch process with a batch reactor is more economical than continuous process (continuous reactor).

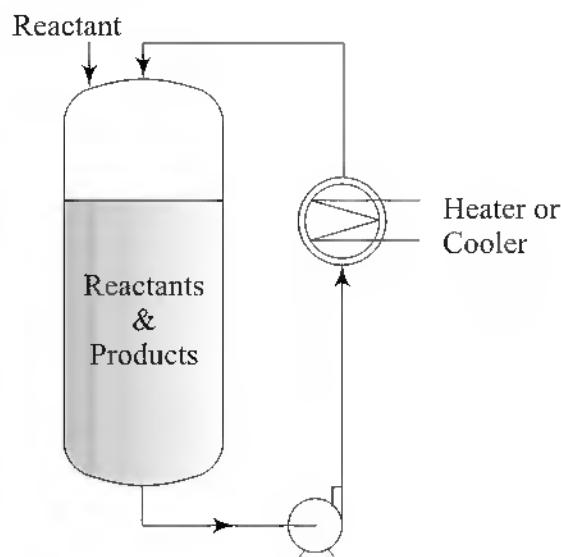
In a batch reactor, variety of agitators are used. Each one has its specific advantages and limitations. Figure 10.2 gives different types of mixing equipment, used in chemical industry. Selection of an agitator plays an important role in achieving desired conversion in the reactors. In majority of reactors, turbine type agitators are used. While for simple mixing, flat blade turbine agitator is used, pitched blade turbine agitator is preferred for a slurry. For viscous liquids (such as manufacture of amino/alkyl resins), anchor/paddle type agitators of different designs are used. For homogenizing applications, such as milk powder dissolution in water, degumming of edible oils (with phosphoric acid), etc., shear mixer is an ideal choice but is a costly equipment. For gas-liquid reactions gas induction type agitator and jet loop reactor are preferred. Design of agitation equipment is discussed at length in literature. Reference (3) gives a good treatise on the subject.

10.3.1 Industrial Examples of Batch Reactor

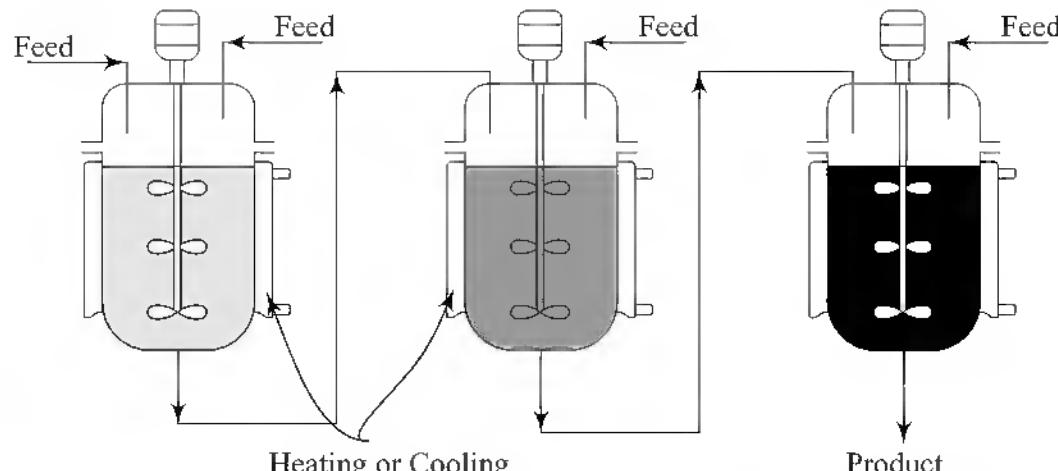
- (a) Synthesis of dyes: Sulfonation of napthalene gives β -sulfonate
- (b) Benzene sulfonic acid from benzene
- (c) Nitrobenzene from benzene with mixed acid
- (d) Synthesis of polyurethan from toluene diisocyanate and polyglycol
- (e) Synthesis of cypermathrin
- (f) Batch fermenter: Fermentation of molasses to ethanol
- (g) Manufacture of active pharmaceutical ingredients (API)
- (h) Manufacture of amino and alkyd resins, etc.



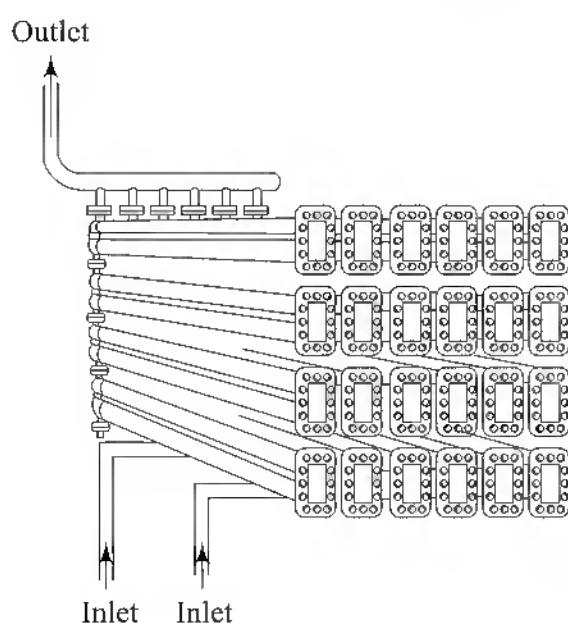
(a) Batch Reactor



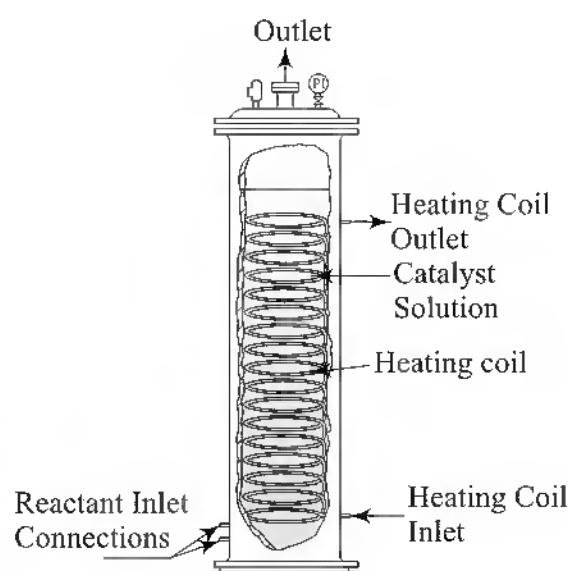
(b) Typical Batch Loop Reactor



(c) Battery of Stirred Tank Reactors



(d) Longitudinal Tubular Reactor



(e) Heterogeneous Continuous Reactor

Fig. 10.1 (Contd.)

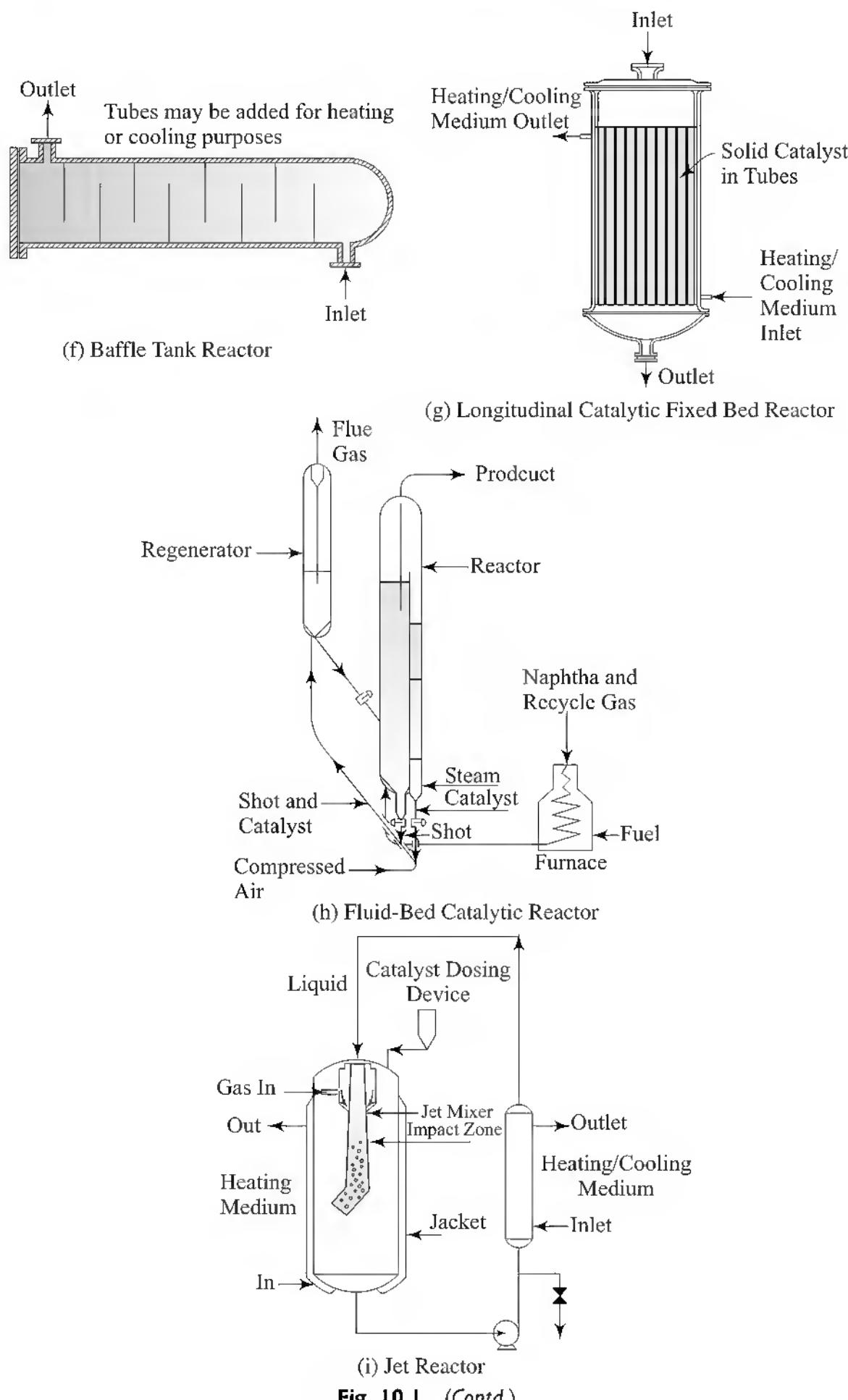
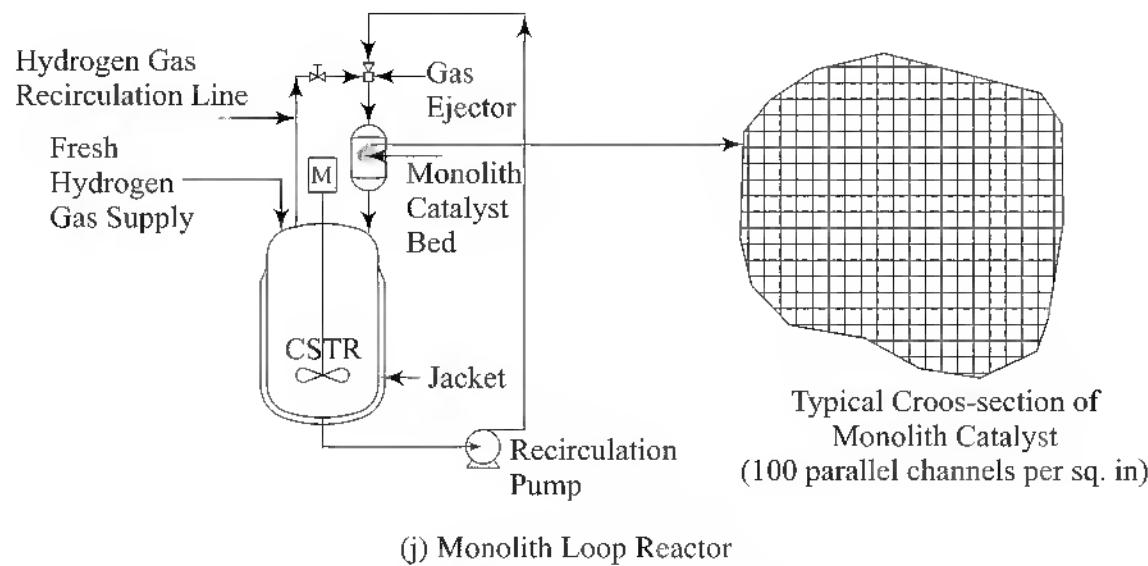
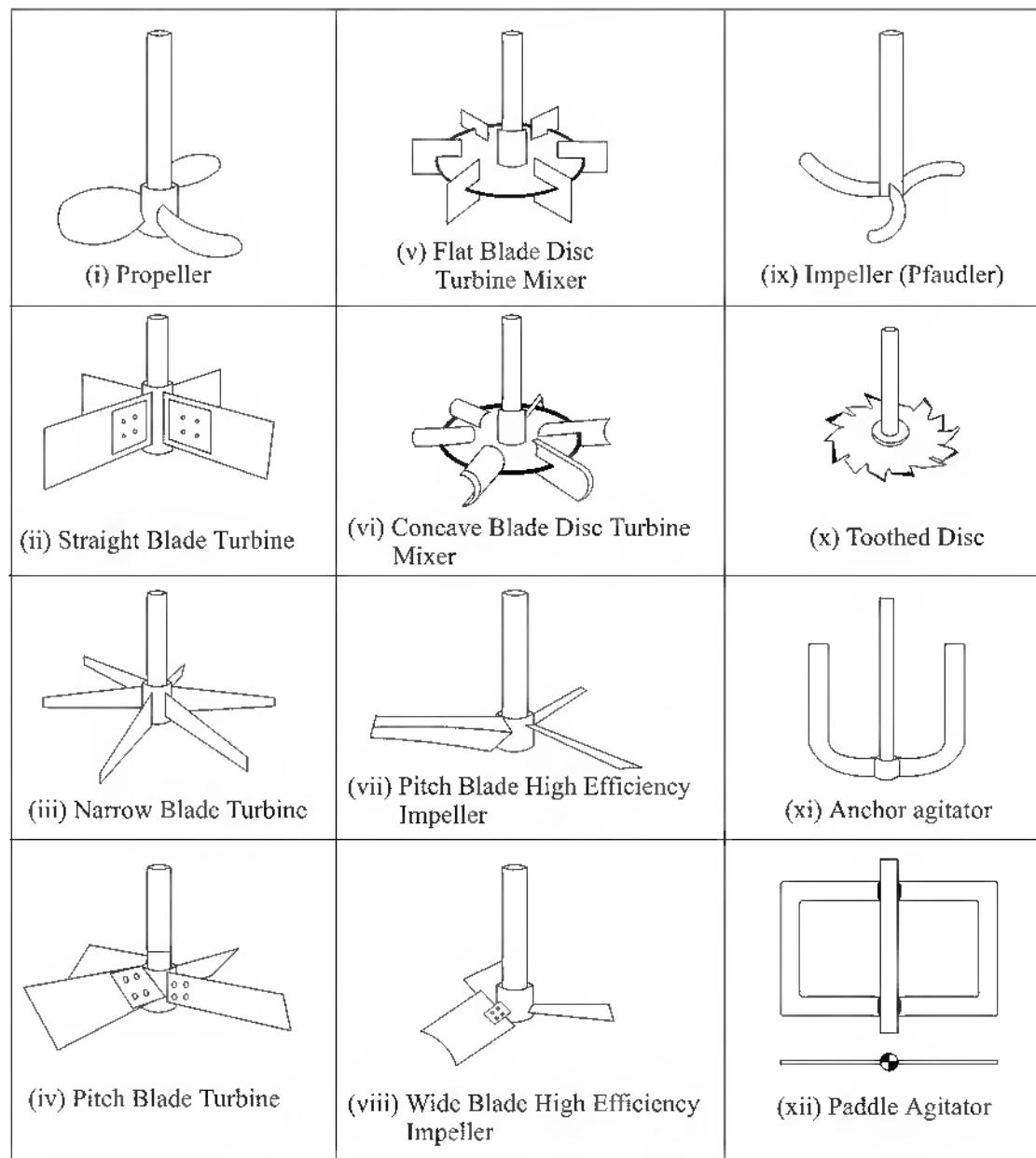


Fig. 10.1 (Contd.)

**Fig. 10.1 Different Types of Reactors****Fig. 10.2 (Contd.)**

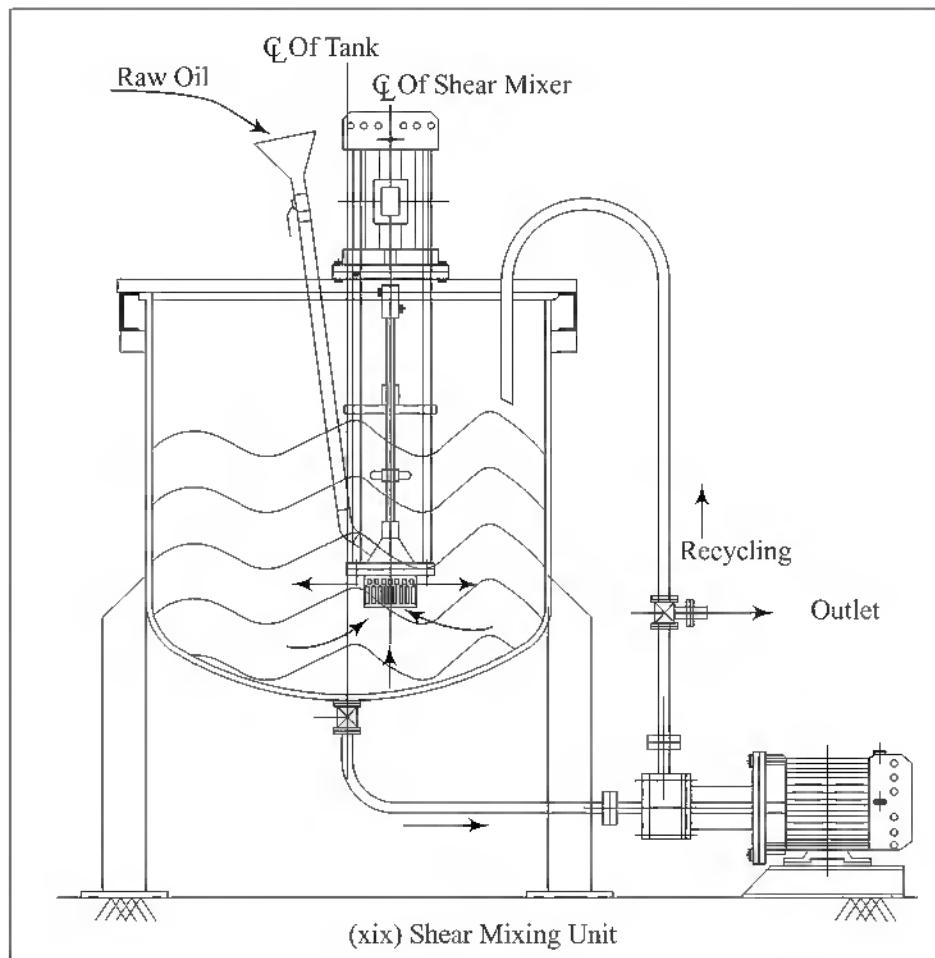
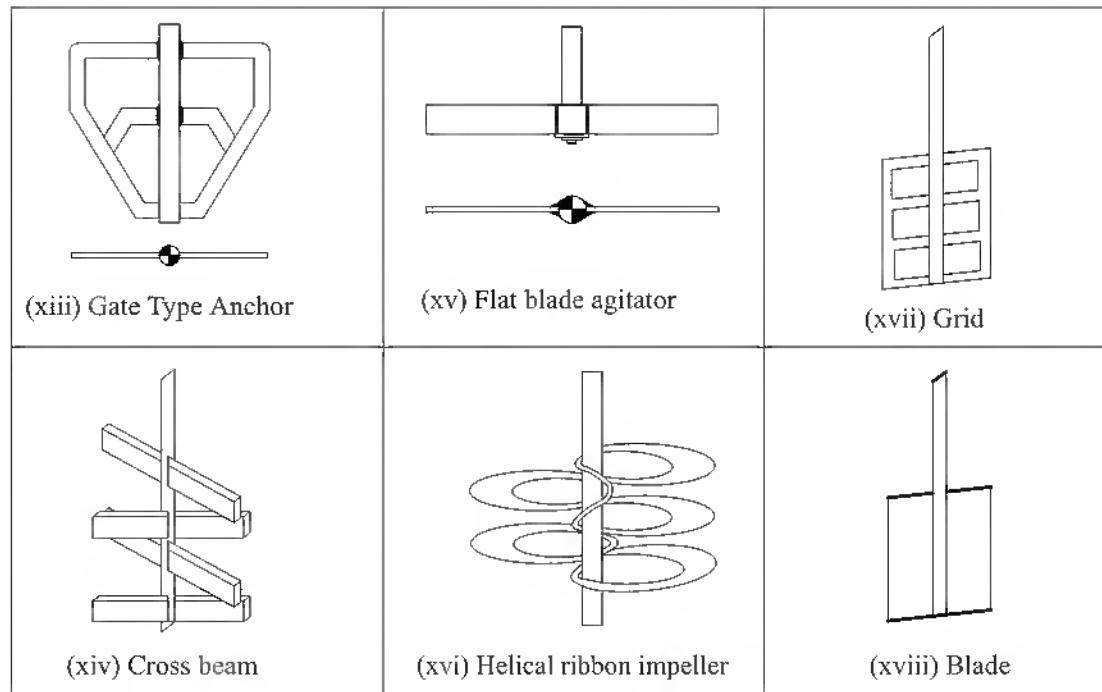


Fig. 10.2 Different Types of Agitators

10.3.2 Advantages of Batch Reactors Over Continuous Flow Reactors

- (a) For small scale production, batch process with a batch reactor is more economical than continuous process with continuous reactor.
- (b) Batch reactors provide more flexibility. For example, it is readily possible to increase the reaction time in the batch reactor while the same is difficult in continuous flow reactor without sacrificing the production rate.
- (c) Some process steps (upstream or downstream processing) of the reaction can also be performed in the reactor.
- (d) Batch reactors require less and relatively simple or cheaper instruments while continuous flow reactors require more sophisticated and costlier instruments.

10.3.3 Disadvantages of Batch Reactors Over Continuous Flow Reactors

- (a) High manpower requirement
- (b) Time is wasted in between two batches
- (c) Temperature control is poor, especially for highly endothermic or exothermic reaction. While starting reaction in a batch reactor, rate of reaction is high and hence heat duty is substantially high.
Some of these aspects were discussed in Chapter 3.
- (d) Quality of the product may vary from a batch to batch.

$$\text{Performance equation of ideal batch reactor is } \theta = N_{A_0} \int_o^{X_A} \frac{dX_A}{(-r_A)V} \quad (10.1)$$

where, θ = Time required to achieve the conversion X_A , s

X_A = Fractional conversion of limiting reactant A

$-r_A$ = Rate of reaction kmol/(m³ · s)

V = Working volume of reactor, m³

For the constant volume reaction system,

$$\theta = \frac{N_{A_0}}{V} \int_o^{X_A} \frac{dX_A}{-r_A} = C_{A_0} \int_o^{X_A} \frac{dX_A}{-r_A} \quad (10.2)$$

where, C_{A_0} = Initial concentration of limiting reactant A.

Example 10.1

Butyl acetate formation is carried out in a batch reactor at 90°C with sulphuric acid as a homogeneous catalyst. The feed contained 4.97 moles of *n*-butanol per mole of acetic acid and the catalyst concentration is 0.032% by mass as H₂SO₄.

Rate equation for this reaction is $-r_A = kC_A^2$.

where, C_A = Concentration of acetic acid in mol/cm³

$k = 17.4 \text{ cm}^3/(\text{mol} \cdot \text{min})$ (Ref. 4)

Density of the reaction mixture at 90°C can be assumed constant and equal to 0.75 g/cm³.

- Calculate the time required to obtain conversion of 50%.
- Determine the size of reactor and the mass of reactants that must be charged to the reactor in order to produce ester at the average rate of 100 kg/h. Conversion is 50%. Reactor will be shut down for 30 min between batches for removal of product, cleaning and start-up. Assume that batch reactor is ideal. Also decide height and diameter of the reactor.
- Determine the heat duty required and heat transfer area required for starting conditions of the reaction.

Solution:

- For ideal batch reactor, time required to obtain the conversion X_A

$$\theta = N_{Ao} \int_0^{X_A} \frac{dX_A}{(-r_A)V} \quad (10.1)$$

Given reaction is a liquid phase reaction in homogenous phase hence working volume can be assumed constant.

$$\theta = \frac{N_{Ao}}{V} \int_0^{X_A} \frac{dX_A}{kC_A^2} = C_{Ao} \int_0^{X_A} \frac{dX_A}{kC_{A0}^2 (1 - X_A)^2}$$

$$\theta = \frac{1}{kC_{Ao}} \left[\frac{1}{1 - X_A} \right]_0^{0.5} = \frac{1}{kC_{Ao}} \left(\frac{1}{(1 - 0.5)} - \frac{1}{(1 - 0)} \right)$$

$$\theta = \frac{1}{kC_{Ao}} (2 - 1) = \frac{1}{kC_{Ao}}$$

$$C_{Ao} = \frac{\text{Moles of acetic acid}}{\text{Total volume}}$$

$$= \frac{\text{Moles of acetic acid}}{\text{Total moles}} \times \frac{\text{Total moles}}{\text{Total volume}}$$

$$C_{Ao} = \frac{x_A^\circ \times \rho_{\text{mix}}}{M_{av}}$$

$$C_{Ao} = \frac{(1/5.97)}{\left(\frac{1}{5.97} \times 60 \right) + \left(\frac{4.97}{5.97} \times 74 \right)} \times 0.75 = 0.0018 \text{ mol/cm}^3$$

$$\text{Mole ratio in feed} = \frac{1 \text{ mole of acetic acid}}{4.97 \text{ mole of butyl alcohol}}$$

$$x_A^\circ = 1/5.97$$

$$M_{av} = \left(\frac{1}{5.97} \times 60 \right) + \left(\frac{4.97}{5.97} \times 74 \right) = 71.655$$

where molar mass of acetic acid = 60

and molar mass of butyl alcohol = 74

$$k = 17.4 \text{ cm}^3/(\text{mol} \cdot \text{min})$$

$$\theta = \frac{1}{17.4 \times 0.0018} = 32 \text{ min or } 0.53 \text{ h}$$

Time required to obtain 50% conversion is 32 min.

$$(b) \text{ Working volume of reactor} = \frac{\text{Mass of liquid (raw material) charged}}{\text{Density of liquid mixture charged}}$$

Assume that volume of liquid mass inside the reactor will remain same during the reaction (or change in liquid volume during the reaction is negligible).

For 100 kg/h ester production with 50% conversion,

Mass of acetic acid required per hour

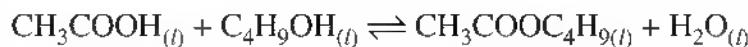
$$\begin{aligned} &= \frac{100 \text{ kg/h}}{\text{molar mass of ester}} \times \frac{\text{molar mass of acetic acid}}{0.5} \\ &= \frac{100}{116} \times \frac{60}{0.5} = 103.45 \text{ kg/h} \end{aligned}$$

(Molar mass of butyl acetate ($\text{CH}_3\text{COOC}_4\text{H}_9$) = $6 \times 12 + 2 \times 16 + 1 \times 12 = 116$)

Mass of acetic acid required per batch

$$\begin{aligned} m_A &= 103.45 \times \frac{(32 + 30)}{60} \quad (\text{Time between batches} = 30 \text{ min}) \\ &= 106.9 \text{ kg/batch} \end{aligned}$$

Reaction:



Reaction is carried out at 90°C and with 50% conversion.

Mass of butanol required per batch,

$$m_B = \frac{106.9}{60} \times 74 \times 4.97 = 655.26 \text{ kg/batch}$$

Mass of H_2SO_4 required per batch

$$m_s = \frac{106.9 + 655.26}{(1 - 0.00032)} \times 0.00032 = 0.244 \text{ kg/batch}$$

Total mass of raw materials = 762.4 kg

$$V_{\text{working}} = \frac{762.4}{0.75 \times 1000} = 1.0165 \text{ m}^3$$

$$\text{Let } \frac{h}{D_i} = 1,$$

where, h = Depth of liquid in reactor shell, m

D_i = Diameter of vessel (Inside diameter), m

Let the type of bottom head = Torispherical

Inside volume of Torispherical head,

$$V = 0.084\ 672\ D_i^3 + \frac{\pi}{4} D_i^2 S_F \quad (\text{Ref. 2}) \quad (10.3)$$

where, D_i = Inside diameter of reactor, m

V = Inside volume, m^3

S_F = Straight flange, m

$$V_{\text{working}} = \frac{\pi}{4} D_i^2 h + 0.084\ 672\ D_i^3 + \frac{\pi}{4} D_i^2 S_F = 1.0165\ \text{m}^3$$

Let $S_F = 1.5\ \text{in} = 0.0381\ \text{m}$ and $\frac{h}{D_i} = 1$

$$1.0165 = \frac{\pi}{4} D_i^3 + 0.084\ 672\ D_i^3 + 0.029\ 92 \times D_i^2$$

$$1.0165 = 0.870\ 007\ D_i^3 + 0.029\ 92\ D_i^2$$

$$D_i = 1.042\ \text{m}$$

$$h = \text{height of liquid in shell} = 1.042\ \text{m}$$

Consider provision of 20% extra space for vapour-liquid disengagement, then actual height of shell of reactor $H = 1.2\ \text{m}$.

- (c) To determine heat duty required at starting condition of reactor, heat of reaction ΔH_R is required.

Table 10.1 Standard Heat of Formation and Heat Capacity Data

Compound	ΔH_f° , kJ/mol at 25°C	C_{Li} , kJ/(kmol · °C) at 57.5°C
Acetic acid	-484.2	129.67
Butanol	-327.3	215.41
Water	-285.83	75.36
Butyl acetate	-522.766	267.12

$$\begin{aligned}\Delta H_R^\circ &= (-522.766 - 285.83) - (-327.3 - 484.2) \\ &= 2.904\ \text{kJ/mol (at 25°C)} \quad (\text{endothermic})\end{aligned}$$

In reactor, when reaction starts both reactants and products are at the reaction temperature
Heat of reaction at reaction temperature (90°C)

$$\Delta H_R = \Delta H_R^\circ + (\sum v_i C_{Li} \Delta t)/1000$$

where, v_i = Stoichiometric coefficient = 1 for all reactants

Assuming heat capacities to be nearly constant up to 90°C,

$$\Delta H_R = 2.904 + (267.12 + 75.26 - 215.41 - 129.67) \times \frac{(90 - 25)}{1000}$$

$$\Delta H_R = 2.904 - 0.1755 = 2.7285\ \text{kJ/mol}$$

Heat required at the starting of reaction

$$\phi = \Delta H_R \times 1000 \times \text{kmol/h of acetic acid consumed}$$

+ Heat required to remove the water continuously from the reaction mass of ternary azeotrope

$$\phi = \left(2.7285 \times 1000 \times \left(\frac{-dN_A}{dt} \right) + \phi_v \right) \times 1.05 \quad (\text{Considering 5% heat loss})$$

Water will leave the reaction mass either as binary azeotrope with butanol (normal boiling point = 92.2°C) or as ternary azeotrope with butyl acetate and butanol (normal boiling point = 89.4°C). Boiling point of ternary azeotrope is less than binary azeotrope.

Composition of ternary azeotrope:

60.86% butyl acetate, 10.47% butanol, 28.92% water (by mass).

$$\text{Ratio of } \frac{\text{Butyl acetate formed}}{\text{Water formed}} = \frac{116}{18} = 6.44 > \frac{60.86}{28.92} (= 2.1)$$

Hence it will be logical to assume that water will leave as ternary azeotrope.

Rate of reaction at the starting of reaction

$$-r_A = kC_{A_0}^2 \quad (C_A = C_{A_0} \text{ when } \theta = 0)$$

$$\text{or } -\frac{dC_A}{dt} = 17.4 \frac{\text{cm}^3}{\text{mol} \cdot \text{min}} \times (0.0018)^2 \left(\frac{\text{mol}}{\text{cm}^3} \right)^2$$

Moles of acetic acid consumed per unit volume of reactor

$$= \frac{-dC_A}{dt} = 5.6376 \times 10^{-5} \frac{\text{mol}}{\text{cm}^3 \cdot \text{min}}$$

$$\text{Moles of acetic acid consumed} = \frac{-dN_A}{dt} = 5.6376 \times 10^{-5} \frac{\text{mol}}{\text{cm}^3 \cdot \text{min}} \times V$$

$$\frac{-dN_A}{dt} = 5.6376 \times 10^{-5} \times 1.0165 \times 10^6 \times 60 \times \frac{1}{10^3}$$

$$\frac{-dN_A}{dt} = 3.43837 \text{ kmol/h} \quad (\text{when } \theta = 0 \text{ or at starting of reaction})$$

Water formation rate = 3.43837 kmol/h = 61.89 kg/h

Total flow rate of ternary azeotrope in starting of reaction

$$= \frac{61.89}{0.2892} = 214 \text{ kg/h}$$

It will contain 130.24 kg/h of butyl acetate, 22.4 kg/h of butanol and 61.89 kg/h of water
 ϕ_v at starting of reaction = $\sum n_i \lambda_i$

Table 10.2 Latent of Vaporization Data²

Component	Latent heat of vaporization at 89.4°C(λ), kJ/kg
Butanol	625.39
Butyl acetate	330.2
Water	2284

$$\phi_v = 130.24 \times 330.2 + 22.4 \times 625.39 + 61.89 \times 2284$$

$$\phi_v = 198370.7 \text{ kJ/h} \equiv 55.103 \text{ kW}$$

$$\phi = (2.7285 \times 1000 \times 3.43837 + \phi_v) \times 1.05 = 218139.9 \text{ kJ/h}$$

$$\equiv 60.5944 \text{ kW}$$

As a heating medium saturated steam is used.

Let saturated steam pressure = 0.2 MPa a

Saturation temperature = 120.23°C

Latent heat of vaporization of steam, λ_s = 2201.6 kJ/kg

$$\text{Steam required } \dot{m}_s = \frac{\phi}{\lambda_s} = \frac{60.5944}{2201.6} = 0.0275228 \text{ kg/s} \equiv 99.08 \text{ kg/h}$$

Saturated steam side heat transfer coefficient h'_o = 6000 W/(m² · °C) (assumed)

Inside heat transfer coefficient, h_i :

It is difficult to find the suitable correlation for the reacting mass side heat transfer coefficient as the reaction is taking place. h_i can be governed by boiling coefficient or by convective film coefficient. If flat blade disc turbine agitator is used to improve the reaction rate and to improve the convective film heat transfer coefficient, then

$$h_i = h_{nB} \text{ or } h_{ic} \text{ whichever is less}$$

where, h_{ic} = Convective film coefficient, W/(m² · °C)

h_{nB} = Nucleate boiling coefficient, W/(m² · °C)

h_{ic}

To calculate the convective film coefficient let the tip velocity of turbine agitator

$$V = 200 \text{ m/min} = \pi D_a n$$

where, D_a = Diameter of agitator

n = Rotational speed of agitator in revolutions per min

For flat blade disc turbine

$$D_a = \frac{\text{shell ID}}{3} = \frac{1.042}{3} = 0.347 \text{ m}$$

$$n = \frac{200}{\pi \times 0.347} = 183.5 \equiv 184 \text{ rpm}$$

For flat blade disc turbine, baffled vessel for $Re > 400$ (Ref. (5))

$$\frac{h_i D_a}{k} = 0.74 Re^{0.67} p_r^{0.33} \left(\frac{\mu}{\mu_w} \right)^{0.14} \quad (10.4)$$

$$\text{where, } Re = \frac{n D_a^2 \rho}{\mu} \quad (10.5)$$

Table 10.3 Composition of Reaction Mass at the Starting of Reaction

Component	Mass fractions at $\theta = 0$
Acetic acid	0.1405
Butanol	0.8595
Total	1.0000

$$\rho_{\text{mix}} = 750 \text{ kg/m}^3$$

Viscosity of mixture

$$\frac{1}{\mu_{\text{mix}}} = \frac{w_1}{\mu_1} + \frac{w_2}{\mu_2} = \frac{0.1405}{\mu_{AA}} + \frac{0.8595}{\mu_{Bu}}$$

$$\mu_{Bu} = 0.65 \text{ mPa} \cdot \text{s at } 90^\circ\text{C}$$

$$\mu_{AA} = 0.53 \text{ mPa} \cdot \text{s at } 90^\circ\text{C}$$

$$\mu_{\text{mix}} = 0.63 \text{ mPa} \cdot \text{s}$$

$$\text{Reynolds number, } Re = \frac{nD_a^2 \rho_{\text{mix}}}{\mu_{\text{mix}}} \frac{(184/60) 0.347^2 \times 750}{0.63 \times 10^{-3}} = 439\,588$$

$$Pr = \frac{C_p \mu}{k} = \frac{C_p \text{ mix} \cdot \mu_{\text{mix}}}{k_{\text{mix}}} \quad (10.6)$$

Table 10.4 Thermal Properties of Acetic Acid and Butanol

Component	C_{pi} , kJ/(kg · °C)	k , W/(m · °C)
Acetic acid	2.1855	0.1557
Butanol	2.8763	0.164

$$C_{P\text{ mix}} = \sum C_{pi} w_i = 2.1855 \times 0.1405 + 2.8763 \times 0.8595 = 2.78 \text{ kJ/(kg · °C)}$$

Thermal conductivity of liquid mixture can be calculated by Li method. (Equation 3.127 to 3.130 of Ref. 2)

$$k_{\text{mix}} = \phi_1^2 k_{L1} + 2\phi_1\phi_2 k_{12} + \phi_2^2 k_{L2} \quad (10.7)$$

In Eq. (10.7), ϕ_1 and ϕ_2 are parameters and do not represent heat loads.

$$k_{L1} = 0.1557 \text{ W/(m · °C)} \quad k_{L2} = 0.164 \text{ W/(m · °C)}$$

$$k_{12} = 2(k_{L1}^{-1} + k_{L2}^{-1})^{-1} = 0.159\,74 \text{ W/(m · °C)}$$

$$\phi_1 = \frac{x_1 V_1}{x_1 V_1 + x_2 V_2} \quad \text{and} \quad \phi_2 = \frac{x_2 V_2}{x_1 V_1 + x_2 V_2} \quad (10.8)$$

$$x_1 = \text{mole fraction of acetic acid} = \frac{\left(\frac{0.1405}{60}\right)}{\left(\frac{0.1405}{60}\right) + \left(\frac{0.8595}{74}\right)} = 0.1678$$

$$x_2 = 0.8322 = \text{mole fraction of butanol}$$

$$V_1 = \text{molar volume of pure acetic acid} = \frac{M}{\rho} = \frac{60}{1048} = 0.057\,25 \text{ m}^3/\text{kmol}$$

$$V_2 = \text{molar volume of pure butanol}$$

$$V_2 = \frac{M}{\rho} = \frac{74}{859.5} = 0.086 \text{ m}^3/\text{kmol}$$

$$\phi_1 = \frac{0.1678 \times 0.057\,25}{0.1678 \times 0.057\,25 + 0.8322 \times 0.086} = 0.118\,34$$

$$\phi_2 = 0.881\,66$$

$$k_{\text{mix}} = 0.118\,34^2 \times 0.1557 + 2 \times 0.118\,34 \times 0.881\,66 \times 0.159\,74 \\ + 0.881\,66^2 \times 0.164 \\ = 0.163 \text{ W/(m · °C)}$$

$$Pr = \frac{2.78 \times 0.63 \times 10^{-3} \times 10^3}{0.163} = 10.744$$

$$h_i = 0.74 \frac{k}{D_a} Re^{0.67} Pr^{0.33} \left(\frac{\mu}{\mu_w} \right)^{0.14}$$

$$h_i = 0.74 \times \frac{0.163}{0.347} \times (439\,588)^{0.67} \times (10.744)^{0.33} \times 1$$

$$h_i = 4594.25 \text{ W}/(\text{m}^2 \cdot ^\circ\text{C})$$

Nucleate boiling coefficient for the boiling of azeotropic mixture can be determined by Mostinski's equation.

$$h_{nB} = 0.104 p_c^{0.69} \left(\frac{\phi}{A} \right)^{0.7} \left[1.8 \left(\frac{p}{p_c} \right)^{0.17} + 4 \left(\frac{p}{p_c} \right)^{1.2} + 10 \left(\frac{p}{p_c} \right)^{10} \right] \quad (6.56)$$

It is valid for single component boiling. But it can be used for close boiling range and azeotropic mixture.

$$\phi = 60.5944 \times 10^3 \text{ W}$$

For nucleate boiling $\Delta T_m = 30.83^\circ\text{C}$ (as decided earlier)

Heat transfer area provided with full height covered jacket,

$$A_{opr} \approx \pi D_o h \approx \pi \times 1.0547 \times 1.042 = 3.4526 \text{ m}^2$$

(Assuming shell thickness, $t_s = 6.35 \text{ mm}$)

$$p = \text{operating pressure bar} = 0.9869 \text{ bar}$$

$$p_c = \text{critical pressure, bar}$$

Critical pressure of mixture $p_{cm} = \sum p_{ci} y_i$ (Kay's rule)

Table 10.5 Critical Pressure of Azeotropic Mixture

Component of Azeotropic mixture	Mass fraction	Mole fraction	Molar mass	Critical pressure, p_c , bar
Butyl acetate	0.6086	0.23	116	31.4
Butanol	0.1047	0.06	74	44.2
Water	0.2892	0.71	18	220.5
Total	1.0000	1.00		

$$p_{cm} = 0.23 \times 31.4 + 0.06 \times 44.2 + 0.71 \times 220.5 = 166.43 \text{ bar}$$

(pseudo-critical pressure)

$$h_{nB} = 0.104 (166.43)^{0.69} \left(\frac{60.5944 \times 10^3}{3.4526} \right)^{0.7} (1.8p_r^{0.17} + 4p_r^{1.2} + 10p_r^{10})$$

where

$$p_r = \frac{p}{p_c} = \frac{0.9869}{166.43} = 5.9298 \times 10^{-3}$$

$$h_{nB} = 2524.95 \text{ W}/(\text{m}^2 \cdot ^\circ\text{C})$$

Convective film coefficient, $h_i = 4594.25 \text{ W}/(\text{m}^2 \cdot ^\circ\text{C}) > h_{nB}$

Since nucleate boiling coefficient is less than the convective coefficient, it is taken for heat transfer area calculations.

$$h_i = 2524.95 \text{ W}/(\text{m}^2 \cdot ^\circ\text{C})$$

Jacket side or saturated steam side heat transfer coefficient, $h'_o = 6000 \text{ W}/(\text{m}^2 \cdot \text{°C})$

Thermal conductivity of reactor shell material (SS-304) = $16 \text{ W}/(\text{m} \cdot \text{°C})$

Let thickness of reactor shell = 6.35 mm (Assumed but it should be decided based on mechanical design)

Overall heat transfer coefficient:

$$\frac{1}{U_o} = \frac{1}{h_o} + \frac{1}{h_{od}} + \frac{D_o \ln(D_o/D_i)}{2k_w} + \frac{D_o}{D_i} \times \frac{1}{h_i} + \frac{D_o}{D_i} \times \frac{1}{h_{id}} \quad (6.42)$$

$$\frac{1}{U_o} = \frac{1}{h'_o} + \frac{D_o \ln\left(\frac{D_o}{D_i}\right)}{2k_w} + \frac{D_o}{D_i} \times \frac{1}{h_i} + \frac{D_o}{D_i} \times \frac{1}{h_{id}}$$

$$D_i = 1.042 \text{ m}, \quad D_o = D_i + 2t_s = 1.042 + 2 \times 0.00635 = 1.0547 \text{ m}$$

$$\frac{1}{U_o} = \frac{1}{6000} + \frac{1.547 \ln\left(\frac{1.0547}{1.042}\right)}{2 \times 16} + \frac{1.0547}{1.042} \times \frac{1}{2524.95} + \frac{1.0547}{1.042} \times \frac{1}{5000}$$

$$U_o = 737.66 \text{ W}/(\text{m}^2 \cdot \text{°C})$$

Heat duty, $\phi_t = 60.5944 \times 10^3 \text{ W}$, $\Delta T_m = 30.23 \text{ °C}$

Heat transfer area required

$$A = \frac{\phi_t}{U_o \Delta T_m} = \frac{60.5944 \times 10^3}{737.66 \times 30.23} = 2.7173 \text{ m}^2$$

Outside heat transfer area provided with full height covered jacket

$$A_{ov} \cong 3.4526 \text{ m}^2$$

% Excess heat transfer area $\cong 27.06\%$

Thus available heat transfer area is sufficient.

10.4 CONTINUOUS FLOW REACTORS

All continuous flow reactors are steady state flow reactors. In a steady state flow reactor, composition at any point is unchanged with time.

For continuous flow reactors, in-line mixers are used. In-line shear mixers are developed for continuous degumming of edible oils. In-line static mixers are effective in achieving desired mixing in continuous systems. In continuous nitration, mixing of a reactant with mixed acid is very effective through a static mixer. Similarly mixing of natural gas/naphtha vapour with steam prior to reforming can be carried out in an in-line static mixer. For removal of free fatty acids from edible/non-edible oils with caustic soda solution, a static mixer is used in a continuous plant. In Fig. 10.3, different designs, of static mixers are shown.

Continuous flow reactors are used for achieving high throughputs. Also quality of product can be maintained at desired level once the operating conditions are stabilised.

There are two ideal steady state flow reactors.

(a) Ideal plug flow reactor (b) Ideal mixed flow reactor

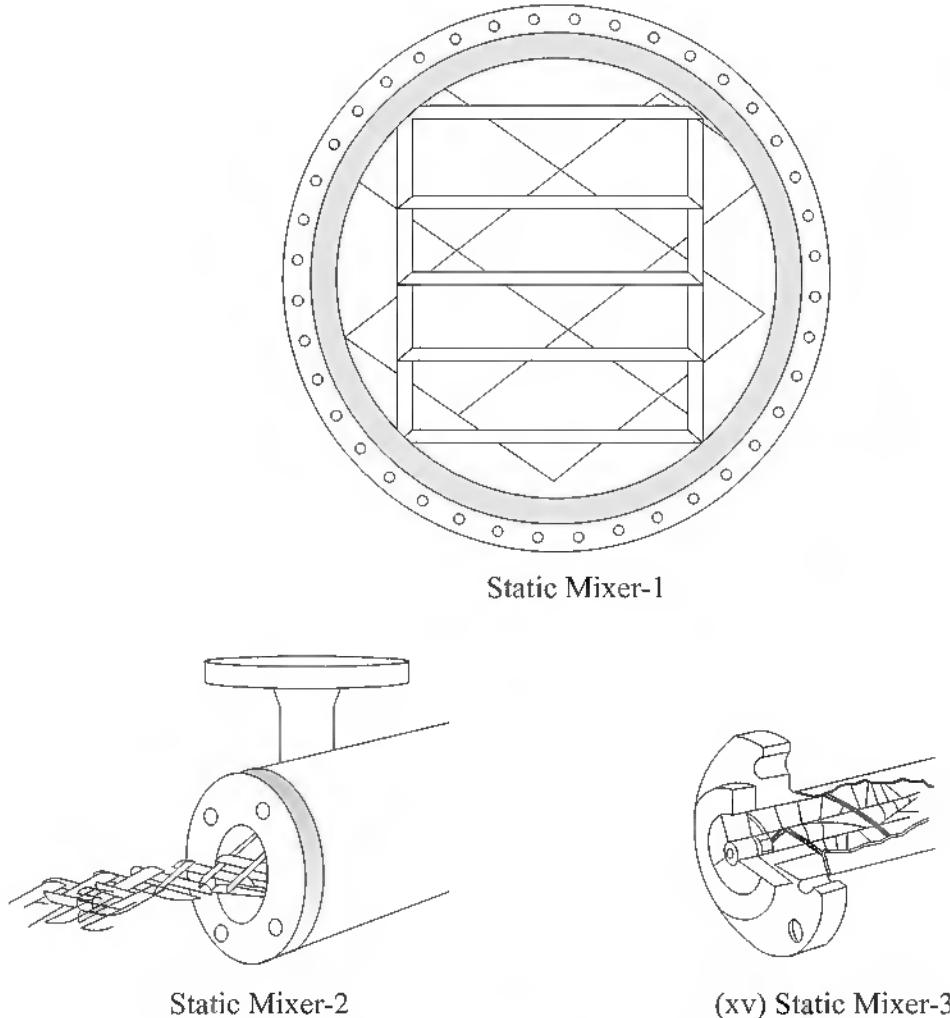


Fig. 10.3 Different Types of Static Mixers

10.4.1 Ideal Plug Flow Reactor^{1, 2, 3}

It is also known as ideal tubular, unmixed flow, slug flow or piston flow reactor. Ideal plug flow reactor has flat velocity profile. In this type of reactor, lateral or radial mixing is permitted, but axial mixing is not permitted. Hence in ideal PFR, the residence time in the reactor is same for all elements or particles of fluid.

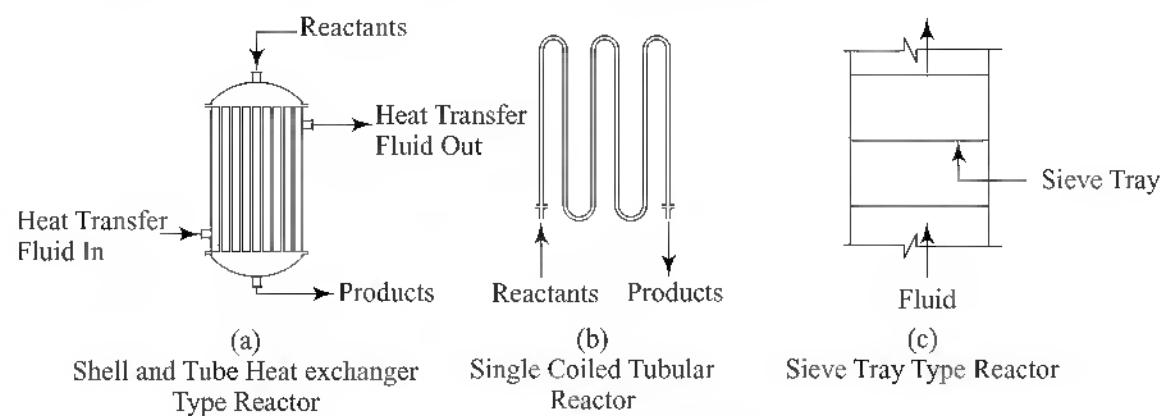


Fig. 10.4 Plug Flow Reactors

Ideal PFR is defined by two criteria.

- Over any cross section of ideal PFR, the mass flow rate, velocity and the fluid properties like pressure, temperature and composition are uniform and the same do not change with time.
- There is negligible diffusion relative to bulk flow or no element of fluid overtaking or mixing with any other element ahead or behind.

In ideal PFR, composition at any point is not changing with time but it changes with length or axial distance in reactor.

Industrial plug flow reactors can be fixed bed catalytic reactor, packed tower type reactor, tubular reactor with very high length to diameter ratio and zigzag path, shell and tube heat exchanger, double pipe heat exchanger, sieve tray tower, etc. Working volume of ideal PFR can be determined by using following equation.

$$\frac{V}{F_{Ao}} = \int_0^{X_A} \frac{dX_A}{-r_A} \quad (10.9)$$

where, V = Working volume of ideal PFR, m^3

F_{Ao} = Molar flow rate of limiting reactant A at inlet, kmol/s

X_A = Fractional conversion of liming reactant required

$$-r_A = \frac{dC_A}{dt} = \text{Rate of chemical reaction in } \text{kmol}/(\text{m}^3 \cdot \text{s})$$

Residence time required in ideal PFR for the desired extent of given reaction can be determined by following equation.

$$\theta = C_{Ao} \frac{V}{F_{Ao}} = \frac{V}{V_o} = C_{Ao} \int_0^{X_A} \frac{dX_A}{-r_A} \quad (10.10)$$

θ = Residence time required, s

V_o = Volumetric flow rate of reacting fluid at inlet, m^3/s

10.4.1.1 Advantages and Disadvantages of Plug Flow Reactors over Mixed Flow Reactors

(a) Advantages:

- For any positive order reaction, plug flow reactor require minimum volume among all types of continuous flow reactors. Volume required by ideal PFR is very much less than the same by ideal mixed flow reactor, particularly for a higher order reaction with higher desired conversion.
- Plug flow reactor does not use moving mechanical parts and hence it is suitable for high pressure reaction system, corrosive reaction system and for the reaction system which involves the use of a toxic gas.

(b) Disadvantages:

- Cost per unit volume of plug flow reactors is higher than the same of mixed flow reactor.
- It requires high investment cost for lower conversion and lower order reaction.

10.4.1.2 Few Industrial Examples of Plug Flow Reactors

- (i) Conversion of ethylene to low density polyethylene (LDPE)
- (ii) Hydrolysis of ethylene oxide to ethylene glycol.
- (iii) Fixed bed catalytic reaction.

Some examples are as follows:

- (a) Conversion of NH_3 from the mixture of N_2 and H_2
- (b) Methanol synthesis reaction
- (c) Steam reforming reaction.
- (d) Conversion of synthesis gas ($\text{H}_2 + \text{CO}$) to methanol
- (e) Air oxidation of methanol to formaldehyde
- (iv) Olefin production by steam cracking, e.g. ethylene
- (v) Conversion of ammonium carbamate to urea in an autoclave.

10.4.2 Ideal Mixed Flow Reactor

It is also known as the back mix reactor (BMR), ideal continuous stirred tank reactor or as constant flow stirred tank reactor. In ideal mixed flow reactor (MFR), composition within the reactor is uniform throughout and does not change with time. Hence, in ideal MFR (or in ideal BMR or CSTR) exit stream from the reactor has the same composition as the fluid within the reactor.

Among all types of continuous flow reactors, ideal mixed flow reactor requires maximum volume for the given positive order reaction. So, volume required by ideal mixed flow reactor (V_m) is greater than volume required by ideal plug flow reactor (V_p). Ratio V_m/V_p increases with increase in order of reaction and also with increase in required conversion.

In an ideal CSTR, once the reactants enter into the reaction mass, they immediately (practically within no time) achieve final required composition. This is not due to very fast reaction but this is merely due to the dilution of reactants by the products. Actually, the rate of reaction is minimum in ideal CSTR, compared to all types of continuous flow reactors, as it provides minimum concentration of reactants during reaction.

In an ideal CSTR, not only composition but temperature is also uniform throughout. Temperature of product stream is same as that within the reactor.

Industrial mixed flow reactors can be:

- (a) Agitated vessel type reactor
- (b) Sparged vessel (including gas induced agitator type) or bubble column type reactor
- (c) Loop reactor (with recirculation, recirculating pump and external heat exchanger)
- (d) Jet reactor with recirculation through an ejector

Volume required by ideal mixed flow reactor is determined by following the equation.

$$\frac{V}{F_{Ao}} = \frac{X_A}{-r_A} \quad (10.11)$$

Residence time required in ideal mixed flow reactor for the desired extent of given reaction is determined by the following equation.

$$\theta = C_{Ao} \frac{V}{F_{Ao}} = \frac{V}{V_o} = C_{Ao} \frac{X_A}{-r_A} \quad (10.12)$$

where,

θ = Residence time required, s

C_{Ao} = Initial concentration of limiting reactant A, kmol/m³

V = Working volume of reactor, m³

V_o = Volumetric flow rate of reacting fluid at inlet, m³/s

X_A = Fractional conversion of limiting reactant A

10.4.2.1 Advantages of Ideal Mixed Flow Reactor

- (i) Temperature is easier to control. Good mixing and lower rate of reaction (lower rate of heat generation in case of exothermic reaction) makes the temperature control easier. In the entrance portion of plug flow reactor and in starting period of batch reaction, rate of reaction is very high and hence temperature control is difficult. Agitated vessel type ideal mixed flow reactor also provides higher heat transfer coefficient compared to packed tower type or sieve tray type plug flow reactor which are normally operated in adiabatic manner.
- (ii) Ideal mixed flow reactor may require less fixed cost compared to ideal plug flow reactor for lower order reaction with lower equilibrium conversion because cost per unit volume of mixed flow reactor is less than the same of plug flow reactor.
- (iii) Polymerization reactions can be nicely controlled in ideal CSTR compared to the same in ideal PFR. (e.g. desired molar mass of polymer can be easily achieved.)
- (iv) Fine catalyst particles can be effectively suspended throughout the liquid reaction system with agitated vessel type mixed flow reactor.
- (v) For highly viscous fluid or slurry solution, agitated vessel type mixed flow reactor provides higher heat transfer coefficient and mass transfer coefficient, compared to plug flow reactor.

10.4.2.2 Disadvantages of Ideal Mixed Flow Reactor

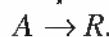
- (i) Ideal mixed flow reactor provides the minimum rate of reaction among all types of continuous flow reactors. Hence, it requires maximum volume among all types of continuous reactors for any given positive order reaction and required conversion.
- (ii) Change over to other product is difficult compared to stirred tank batch reactor.

10.4.2.3 Industrial Examples of Mixed Flow Reactors

- (i) Carbonylation of methanol gives acetic acid in presence of liquid phase catalyst
- (ii) Oxidation of acetaldehyde to acetic acid in presence of fine KMnO₄ (potassium permanganate) particles as catalyst
- (iii) Styrene to polystyrene by solution polymerization.
- (iv) Slurry polymerization of propylene to polypropylene
- (v) Chlorination of benzene to chlorobenzene by continuous process
- (vi) Oxidation of toluene to benzoic acid.
- (vii) Vinyl chloride to polyvinyl chloride by emulsion and suspension polymerization

Example 10.2

A homogeneous liquid phase reaction



takes place with 50% conversion in a mixed flow reactor. Its rate equation is

$$-r_A = \frac{dC_A}{dt} = kC_A^2$$

- (a) What will be the conversion if this reactor is replaced by the one, 6 times larger reactor and all else remaining unchanged ?
- (b) What will be the conversion if the original reactor is replaced by a plug flow reactor of equal size and all else remaining unchanged ?

Solution:

Performance equation of ideal mixed flow reactor is

$$\frac{V}{F_{Ao}} = \frac{X_A}{-r_A} \quad (10.11)$$

$$\frac{V}{F_{Ao}} = \frac{X_A}{kC_A^2} = \frac{X_A}{kC_{Ao}^2 (1-X_A)^2} = \frac{0.5}{kC_{Ao}^2 (1-0.5)^2} = \frac{2}{kC_{Ao}^2}$$

- (a) For $V' = 6V$ (where V' = volume of larger reactor)

$$\frac{V}{F_{Ao}} = \frac{X_A}{-r_A} = \frac{X_A}{kC_A^2} = \frac{X_A}{kC_{Ao}^2 (1-X_A)^2} \quad (1)$$

$$\text{Also } \frac{V'}{F_{Ao}} = 6 \frac{V}{F_{Ao}} = 6 \times \frac{2}{kC_{Ao}^2} = \frac{12}{kC_{Ao}^2} \quad (2)$$

$$\text{From Eq. (1) and (2), } \frac{X_A}{kC_{Ao}^2 (1-X_A)^2} = \frac{12}{kC_{Ao}^2}$$

$$12(1-X_A)^2 = X_A$$

$$12(1-2X_A+X_A^2) = X_A$$

$$12 - 24X_A + 12X_A^2 - X_A = 0$$

$$12X_A^2 - 25X_A + 12 = 0$$

$$X_A^2 - 2.08333X_A + 1 = 0$$

$$X_A = 1.333 \text{ or } X_A = 0.75$$

X_A can not be greater than 1; hence $X_A = 0.75$

Hence, six times larger ideal mixed flow reactor will give 75% conversion.

- (b) $V_{\text{mixed flow}} = V_{\text{plug flow}} = V$

Performance equation of ideal plug flow reactor

$$\frac{V}{F_{Ao}} = \int_0^{X_A} \frac{dX_A}{-r_A} = \int_0^{X_{AP}} \frac{dX_A}{kC_{Ao}^2 (1-X_{AP})^2} \quad (10.9)$$

where, X_{AP} = Fractional conversion of reactant A in ideal plug flow reactor

$$\text{Let } p = 1 - X_{AP}, \text{ hence } \frac{dp}{dX_{AP}} = -1$$

$$\frac{V}{F_{Ao}} = \frac{1}{kC_{Ao}^2} \int_1^p \frac{dp}{p^2} = -\frac{1}{kC_{Ao}^2} \left[\frac{p^{-2+1}}{-2+1} \right]_1^p = \frac{1}{kC_{Ao}^2} \times \frac{1-p}{p}$$

$$\frac{V}{F_{Ao}} = \frac{1}{kC_{Ao}^2} \frac{X_{AP}}{1-X_{AP}}$$

For ideal mixed flow reactor $\frac{V}{F_{Ao}} = \frac{2}{kC_{Ao}^2}$

$$V = \frac{2F_{Ao}}{kC_{Ao}^2} = V_{\text{mixed}} = V_{\text{plug}}$$

$$\frac{2F_{Ao}}{kC_{Ao}^2} \times \frac{1}{F_{Ao}} = \frac{1}{kC_{Ao}^2} \frac{X_{AP}}{1-X_{AP}}$$

$$2 - 2X_{AP} = X_{AP}$$

$$X_{AP} = 2/3 = 0.6667$$

Hence, if the original ideal mixed flow reactor (giving 50% conversion) is replaced by an ideal plug flow reactor of equal size, then conversion will be 66.67%.

Example 10.3

The following data have been obtained on the decomposition of gaseous reactant A in a constant volume batch reactor at 100°C.

Table 10.6 Time vs Pressure Data of Decomposition of A

θ, s	$p_A, \text{ atm}$	θ, s	$p_A, \text{ atm}$
0	1	140	0.25
20	0.8	200	0.14
40	0.68	260	0.08
60	0.56	330	0.04
80	0.45	420	0.02
100	0.37		

The stoichiometry of the reaction is $2A \rightarrow R + S$.

- What size of plug flow reactor (in liters) operating at 100°C and 1 atm can treat 100 moles of A/h in a feed consisting of 20% inert to obtain 95% conversion of A?
- What conversion of A can be expected in mixed flow reactor of volume $V = 208 \text{ L}$, for the same identical feed and identical operating conditions?

Solution:

First rate equation, based on constant volume batch reactor data, must be determined.

From the data, at $\theta = 420 \text{ s}$, p_A is 0.02 atm means it is reduced to a value very close to zero. Hence, given reaction is irreversible reaction,

Assuming first order irreversible reaction,

$$-r_A = \frac{-dC_A}{d\theta} = kC_A$$

$$p_A = C_A RT$$

$$-\frac{1}{RT} \frac{dp_A}{d\theta} = k \frac{p_A}{RT}$$

$$-\frac{dp_A}{d\theta} = kp_A$$

$$\int_{p_{Ao}}^{p_A} -\frac{dp_A}{p_A} = k \int_0^\theta d\theta$$

$$-\ln \left(\frac{p_A}{p_{Ao}} \right) = k\theta$$

From the given data, following table can be prepared.

Table 10.7 Tabulation of Calculations of Integral

θ, s	$p_A, \text{ atm}$	$-\ln \left(\frac{p_A}{p_{Ao}} \right)$
0	1.00	0.0000
20	0.80	0.2230
40	0.68	0.3856
60	0.56	0.5800
80	0.45	0.7980
100	0.37	0.9940
140	0.25	1.3860
200	0.14	1.9660
260	0.08	2.5260
330	0.04	3.2200
420	0.02	3.9100

The graph of $-\ln \left(\frac{p_A}{p_{Ao}} \right)$ vs θ (Fig. 10.5) is a straight line passing through origin.

Hence, given reaction is irreversible reaction and is of 1st order.

$$k = 0.00976 \text{ s}^{-1}$$

$$-r_A = 0.00976 C_A \text{ mol/(L} \cdot \text{s}) \equiv 35.136 C_A \text{ mol/(L} \cdot \text{h})$$

(a) Performance equation of ideal plug flow reactor,

$$\theta = C_{Ao} \int_0^{X_A} \frac{dX_A}{-r_A}$$

Reaction $2A \rightarrow R + S$, with 20% inert

$$\varepsilon_A = \frac{V_{X_A=1} - V_{X_A=0}}{V_{X_A=0}}$$

$\varepsilon_A = 0$, as total number of moles of reaction system are not changing

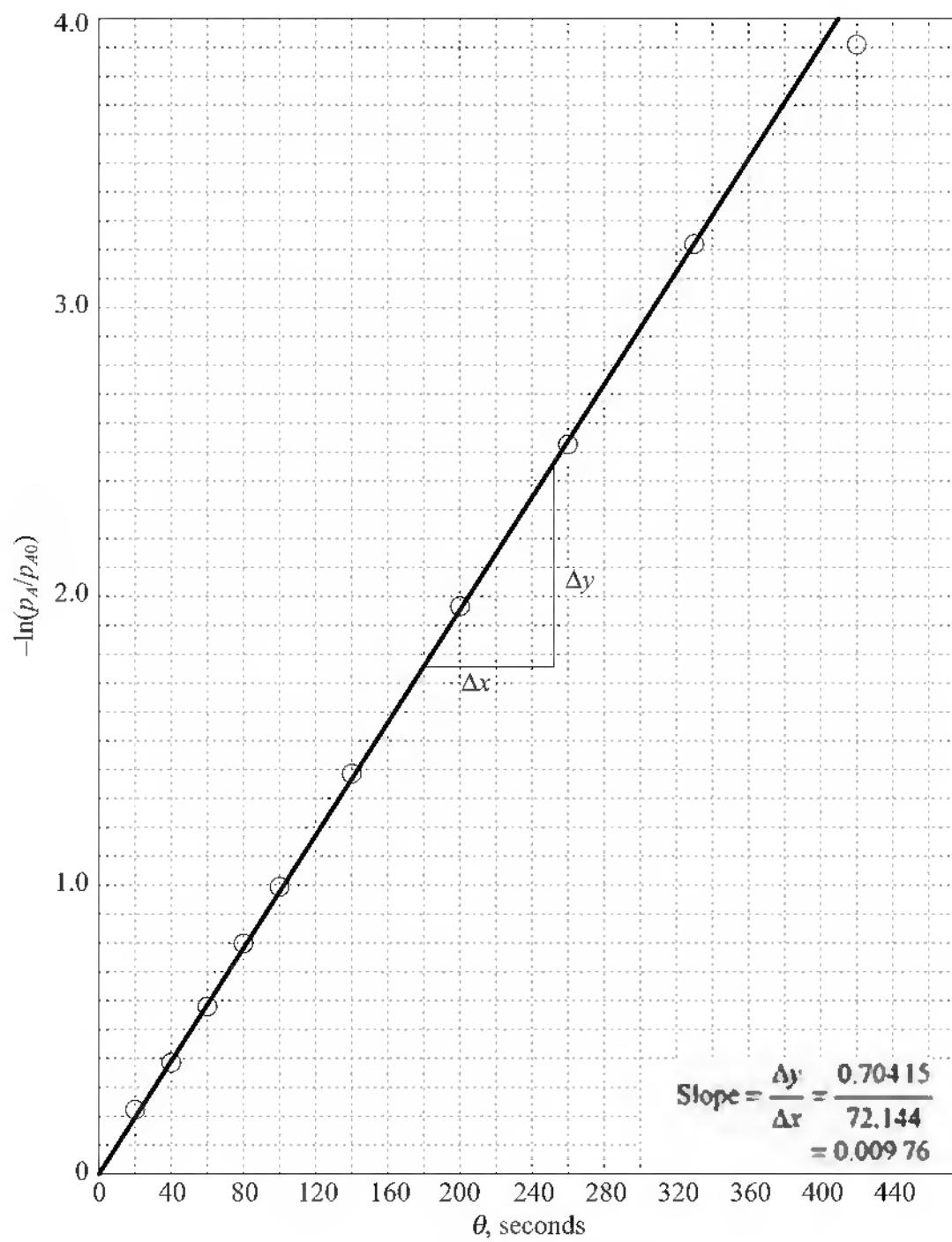


Fig. 10.5 Determination of Order of Reaction

$$\theta = C_{Ao} \int_o^{X_A} \frac{dX_A}{kC_{Ao} (1-X_A)} = \frac{1}{k} \int_o^{X_A} \frac{dX_A}{(1-X_A)}$$

$$-\ln (1-X_A) = k\theta$$

$$\theta = -\frac{1}{k} \ln(1-X_A) = -\frac{1}{0.00976} \ln (1-0.95) = 307 \text{ s}$$

$$\theta = C_{Ao} \frac{V}{F_{Ao}} = 307 \text{ s}$$

$$\text{Working volume of reactor, } V = \theta \frac{F_{Ao}}{C_{Ao}} = 307 \frac{F_{Ao}}{C_{Ao}}$$

$$C_{Ao} = \frac{P_{Ao}}{RT}, p_{Ao} = p_t \times 0.8 = 0.8 \text{ atm (20% inert)}$$

$$C_{Ao} = \frac{0.8}{(273 + 100) \times 0.082} = 0.02616 \text{ mol/L}$$

$$F_{Ao} = 100 \text{ mol/h}$$

$$V = \frac{307 \times 100 \times (1/3600)}{0.02616} = 326 \text{ L}$$

(b) For ideal mixed flow reactor,

$$\frac{V}{F_{Ao}} = \frac{X_A}{-r_A} = \frac{X_A}{kC_{Ao}(1-X_A)}$$

$$\frac{X_A}{1-X_A} = \frac{kC_{Ao}V}{F_{Ao}} = \frac{0.00976 \times 0.02616 \times 208}{100 \times \left(\frac{1}{3600}\right)}$$

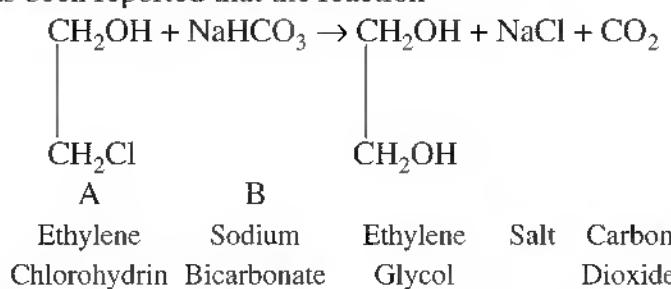
$$\frac{X}{1-X_A} = 1.912$$

$$X_A = 0.65$$

65.65% conversion of reactant A can be expected in ideal mixed flow reactor.

Example 10.4

It has been reported that the reaction



is elementary with rate constant $k = 5.2 \text{ L}/(\text{mol} \cdot \text{h})$ at 82°C . On the basis of this information it is intended to construct a pilot plant to determine the economical feasibility of producing ethylene glycol from two available feeds, a 15 mass % aqueous solution of sodium bicarbonate and a 30 mass % aqueous solution of ethylene chlorhydrin.

- What volume of tubular (plug flow) reactor will produce 20 kg/h ethylene glycol at 95% conversion of an equimolar feed produced by intimately mixing appropriate quantities of the two feed streams?
- What size of mixed reactor is needed for the same feed, conversion and production rate as in Part (a)?

Assume all operations at 82°C at which specific gravity of the mixed reacting fluid is 1.02.

Solution:

- (a) Performance equation of ideal tubular reactor

$$\frac{V_p}{F_{Ao}} = \int_o^{X_A} \frac{dX_A}{-r_A} \quad (10.9)$$

Here given reaction is elementary reaction. Hence rate equation is corresponding to stoichiometric equation.

$$-r_A = kC_A C_B$$

for equimolar feed, $C_{Ao} = C_{Bo}$ and $C_A = C_B$

$$-r_A = kC_A^2$$

$$\frac{V_p}{F_{Ao}} = \int_o^{X_A} \frac{dX_A}{kC_A^2} = \int_o^{X_A} \frac{dX_A}{kC_{Ao}^2 (1-X_A)^2}$$

$$kC_{Ao}^2 \frac{V_p}{F_{Ao}} = \int_o^{X_A} \frac{dX_A}{(1-X_A)^2} = \left[\frac{1}{1-X_A} \right]_0^{X_A} = \frac{1}{1-X_A} - 1 = \frac{X_A}{1-X_A}$$

$$5.2 \times C_{Ao}^2 \frac{V_p}{F_{Ao}} = \frac{0.95}{1-0.95}$$

For equimolar feed and based on stoichiometric equation,

$F_{Ao} \times 0.95$ = Moles of ethylene glycol produced (for 95% conversion)

$$F_{Ao} = \frac{20 \text{ kg/h}}{0.95 \times \text{Molar mass of ethylene glycol}}$$

$$F_{Ao} = \frac{20}{0.95 \times 62} = 0.3396 \text{ kmol/h}$$

Total Mass of Feed:

\dot{m}_F = Mass flow rate of ethylene chlorohydrin solution + mass flow rate of sodium bicarbonate solution.

\dot{m}_F = $(0.3396 \times M_{EC} + \text{mass of associated water}) + (0.3396 \times M_{SB} + \text{mass of associated water})$

where M_{EC} = Molar mass of ethylene chlorohydrin = 80.5 kg/kmol

M_{SB} = Molar mass of sodium bicarbonate = 84 kg/kmol

$$\begin{aligned} \dot{m}_F &= \left(0.3396 \times 80.5 + \frac{0.3396 \times 80.5}{0.3} \times 0.7 \right) \\ &\quad + \left(0.3396 \times 84 + \frac{(0.3396 \times 84)}{0.15} \times 0.85 \right) \\ &= (27.3378 + 63.7882) + (28.5264 + 161.6496) \\ &= 281.302 \text{ kg/h} \end{aligned}$$

Density of reacting fluid = 1020 kg/m³

$$V_o = \frac{281.302}{1020} = 0.2758 \text{ m}^3/\text{h}$$

$$C_{Ao} = \frac{F_{Ao}}{V_o} = \frac{0.3396}{0.2758} = 1.2313 \text{ kg/m}^3$$

$$k = 5.2 \text{ L/(mol} \cdot \text{h}) = 5.2 \text{ m}^3/(\text{kmol} \cdot \text{h})$$

$$5.2 \times C_{Ao}^2 \times \frac{V}{F_{Ao}} = \frac{0.95}{1-0.95}$$

$$5.2 \times \frac{(1.2313)^2}{0.3396} \times V_p = \frac{0.95}{1-0.95}$$

$$V_p = 0.81845 \text{ m}^3 \equiv 818.45 \text{ L}$$

(b) For ideal mixed flow reactor

$$\frac{V_m}{F_{Ao}} = \frac{X_A}{-r_A} = \frac{X_A}{kC_{Ao}^2 (1-X_A)^2}$$

$$V_m = \frac{F_{Ao}}{kC_{Ao}^2} \frac{X_A}{(1-X_A)^2}$$

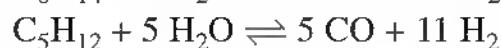
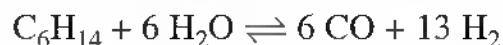
$$V_m = \frac{0.3396}{5.2 \times (1.2313)^2} \times \frac{0.95}{(1-0.95)^2}$$

$$V_m = 16.369 \text{ m}^3 \equiv 16369 \text{ L}$$

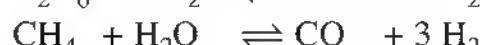
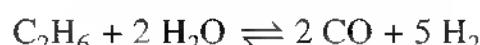
10.5 DEGREE OF COMPLETION OF REACTION

Chemical engineers are familiar with degree of conversion of a reaction which is well defined as the ratio of moles of limiting reactant consumed to that in the feed. However, it is not possible to define the degree of conversion of reaction(s) in all cases. Chief reason for not able to define the conversion clearly is composition of feed in which more than one reactant react simultaneously (i.e. parallel reactions) and degree of completion of each reaction is unknown. Under these circumstances, degree of completion of reaction is defined in several ways.

Consider a classical reforming reaction of hydrocarbons with steam. There are a number of hydrocarbons present in the feed. They react simultaneously with steam and produce carbon monoxide, carbon dioxide and hydrogen.



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Study of equilibrium conversions of competing reactions reveal that while most hydrocarbons reform to hydrogen, methane is not always fully reformed. Thus, degree of completion of the reforming reaction is judged by methane slip (i.e. mole % methane in outgoing gas mixture).

Edible oils are hydrogenated to produce hydrogenated fat. Basically edible oil is a mixture of (mostly) triglycerides of fatty acids. Each edible oil has different composition in terms of variety of fatty acids. It is not possible to know the exact composition in terms of fatty acids for each batch of oil. These fatty acids may be saturated or unsaturated. The degree of unsaturation is measured by titration with potassium iodide and is known as iodine number. Higher the iodine number, higher is the unsaturation. When hydrogenated, double and triple bonds open up and saturation takes place. For this hydrogenation reaction, degree of completion is measured by reduction in the iodine value of the oil.

Alkyd resins are produced by reacting an organic acid such as phthalic anhydride, benzoic acid, etc. with glycerine or other polyols. During the course of esterification reaction, acid is consumed. Degree of completion of this process is measured by the final acid value of the product.

In demineralization of water, cations are removed in the cation exchanger while anions are removed in the anion exchanger. While bi- and tri-valents are removed on a priority, monovalent ions are not easily removed. Hence, degree of demineralization (i.e. degree of completion of each ion exchanger) is measured by sodium and chloride slips, respectively.

Several other examples can be cited for the degree of completion.

It can, therefore, be visualized that the degree of completion of reaction is defined in terms of a measurable parameter.

These reactors are designed for desired completion of reaction. For such reactions, rate of reaction need not be defined as for pure chemical reaction (see Example 10.5). It is normally expressed in terms of measurable index with respect to other parameters.

Rate of reaction of manufacture of alkyd resin can be expressed in terms of disappearance of acid value. Typical kinetic expression could be

$$AV = X\theta + Y$$

where, AV = Acid value after time θ

θ = Time

X and Y are constants. These could be determined by laboratory and pilot plant studies.

In case of hydrocarbon reforming, methane slip is dependent of pressure and temperature. It can be defined in an empirical form in terms of pressure and temperature.

$$C_{CH_4} = X p^a T^b + Y$$

where, C_{CH_4} = Concentration of methane (mole %) at exit

p = Operating pressure

T = Operating absolute temperature

X, a, b and Y are constants.

Alternately, degree of approach is used to define completion of reforming reaction. Based on actual methane slip and pressure, equilibrium temperature is found from the standard chart. Difference between actual operating temperature and equilibrium temperature (read from the graph) is defined as the approach.

While designing a chemical reaction system, degree of conversion is used for well defined reaction, if known. For all other systems, desired degree of completion is defined and the system is designed accordingly. In case of ammonia manufacture, conversion per pass is commonly expressed as the reaction is well defined.

10.6 MIXING FOR THE DIFFERENT TYPE OF REACTION SYSTEMS⁶

For the better mixing or better mass transfer rate, an agitator, a circulating pump, etc. are used as mixing tools. Some new techniques are also available for the same. Selection of agitator for the given reaction system depends on the viscosity of reaction mixture, homogeneity of the reaction system, desired duty, etc.

Following are the guidelines for the selection of agitator (or other technique) for the different type of reaction systems.

(i) Homogeneous Liquid Phase Reaction System:

If the viscosity of homogeneous liquid phase system is less than $500 \text{ mPa} \cdot \text{s}$ then flat blade turbine stirrer (with four or six flat blades on disc as shown in Fig. 10.2(v)) or Pfaudler impeller (as shown in Fig. 10.2(ix)) can be selected. They are used with side baffles. These agitators create the currents mainly in radial and tangential directions. Use of side baffles are required to avoid the vortex formation.

If the viscosity is in the range of 500 to $5000 \text{ mPa} \cdot \text{s}$, cross beam, grid and blade stirrers (Ref. Fig. 10.2(xiv), (xvii) and (xviii)) are suitable for homogeneous liquid. For the lower viscosity (nearer to $500 \text{ mPa} \cdot \text{s}$), side baffles are used with these types of agitators. For the higher viscosity (nearer to $5000 \text{ mPa} \cdot \text{s}$), side baffles are not required.

For highly viscous liquid (viscosity, $\mu > 5000 \text{ mPa} \cdot \text{s}$), anchor stirrer (Ref. Fig. 10.2(xi)) is selected. Normally the diameter of anchor agitator is 90% or more of the inside diameter of tank. It rotates with keeping a close clearance with inside surface of shell. It removes the sticky material from the heat transfer surface and thereby improves heat transfer coefficient. But this agitator provides poor mixing. Anchor agitator can be used in conjunction with a higher speed paddle or other agitator to improve the mixing. Other agitator is usually turning in the opposite direction. Miscible liquids are often simply mixed in pipelines by using a static mixer. The deflection elements in static mixer divide the stream into two streams and turn each through 180° , so after passing through N elements the stream has been blended 2^N times.

(ii) Gas-Liquid Reaction:

- (a) Gas itself creates the axial currents. Hence for the gas-liquid reaction system suitable agitator is that which creates the current in tangential and radial directions. Hence, conventional flat blade turbine agitator (as shown in Fig. 10.2(ii) and 10.2(v)) is used for such applications in which separate sparger is provided.
- (b) Gas induction type hollow agitator is new innovation for this application. Special type of impeller (as shown in Fig. 10.12) is attached with hollow shaft. In the upper part of the hollow shaft, windows are provided for gas suction. Gas enters from these windows and discharges through the lowest part of the impeller. The agitator operates on the principle of water jet ejector. The suction so generated blows the stirrer edges during the rotation and hence gas enters through windows and discharges from

the bottom of impeller to liquid pool. A specially designed impeller vigorously disperses the gas bubbles and creates a mixture akin to a boiling liquid. Gas bubbles react with liquid as they rise. Unreacted gas is reinduced into the liquid through windows. Recirculation of gas is important because bubbling of gas only once through the liquid does not use it up completely. It offers the following advantages.

- (i) It provides vigorous gas liquid mixing.
- (ii) It substantially increases gas-liquid interfacial area of contact and enhances gas-liquid mass transfer rate.
- (iii) It reduces reaction time considerably for the gas-liquid reaction in which overall reaction rate is governed by rate of mass transfer.
- (iv) It provides very high vessel side (i.e. inside) coefficient which approaches a boiling coefficient.
- (v) It is also the best choice for the gas-liquid reaction with suspended solid catalyst. Example: hydrogenation in presence of suspended Reiny Ni catalyst.

It is used for hydrogenation, alkylation, ozonization, oxidation, amination, etc. reactions.

- (c) Jet reactor is a new design of reactor which can be used to achieve the excellent gas-liquid mixing. As shown in Fig. 10.1 (i). Jet reactor consists of a reaction autoclave, a circulation pump, an external heat exchanger and a venturi type ejector. Jet reactors are available in the capacities from 0.02 m^3 to 100 m^3 , operating pressure up to 200 bar, operating temperature up to 350°C and in variety of materials of constructions like stainless steel, Hastelloy, Monel, etc. This reactor can be used for the viscosity of reaction mixture up to $500 \text{ mPa} \cdot \text{s}$ and for gas-liquid reaction with suspended solid particles (solid catalyst load should be less than 10% by mass).

Following are the advantages of jet reactor over agitated vessel type reactor.

- (i) Length to diameter ratio of jet reactor is higher than the same of agitated vessel. Hence, jet reactor requires less cost particularly for high pressure reactions.
- (ii) The external heat exchanger (instead of internal coil or jacket) can be built as large as needed and is not limited by the reactor geometry. Sufficient heat transfer area could be made available for accurate temperature control even if the reactor is operated with reduced working volumes.
- (iii) The maximum power input per unit volume is often a limiting factor, especially for large reactors with an agitator. Since there is no agitator in the jet reactor, this limitation does not exist.

The circulation pump can provide very high power per m^3 of working volume if it is required to achieve the desired mass transfer rate.

- (iv) The down flow jet ejector forms fine gas bubbles in the liquid and creates high mass transfer rates.
Jet reactor is used for hydrogenation, alkylation, carbonylation oxidation, halogenation, amination, phosgenation, etc. reactions. Jet reactor is a type of loop reactor.
- (d) Another loop reactor is a monolith catalytic reactor [Fig. 10.1 (j)]. Monolithic structure of the base carrier is impregnated with a noble metal (such as platinum, palladium, etc.). Liquid is circulated through a pump and passed through the catalyst with induced gas. Originally developed for emission control from auto vehicles (for catalytic oxidation of carbon monoxide), monolithic structure is found equally effective for chemical reactors. Monolithic structure provides large surface area and hence low concentration of catalyst (0.5 to 1%) on the structure is sufficient for accelerating the reaction. The design is claimed to be highly effective in hydrogenating a nitro compound to an amine.
- (e) Recent innovation for gas-liquid reaction is to convert the heterogeneous gas-liquid reaction into single homogeneous phase; supercritical phase by changing the operating conditions. Foreign substance (such as carbon dioxide, propane, etc.) is added to the reaction system to get the homogeneous supercritical phase.

Examples:

- (A) Hydrogenation of oleochemicals at supercritical single phase conditions:
In this case, propane is added to reaction system. Supercritical propane dissolves both hydrogen and oil and creates the single homogeneous supercritical phase. This supercritical phase is contacted with catalyst and it gives very high overall rate of reaction and higher selectivity. To create the necessary single phase (supercritical phase) conditions, operating pressure is kept near 150 bar, temperature of 280°C and propane is added to the extent of 5 to 6 times product mass. Hydrogenated product will have less of *trans*-isomer and less free fatty acids.
- (B) Oxidation of waste water at supercritical phase conditions⁷:
It is carried out at 374°C and 22.1 MPa a pressure. It is carried out between supercritical wastewater and oxygen. Enhanced solubility of oxygen in supercritical water eliminates mass transfer resistance and provides very high mass transfer rate. Residence time required in reactor is only 60 seconds for 99.98% COD reduction.
- (iii) For gas-liquid reaction with suspended solid particles: Simple loop reactor or jet reactor are also valid for gas-liquid reaction with suspended solid particles, except with static mixer. Oxidation of acetaldehyde to acetic acid in presence of potassium permanganate can be classified in this category.
- (iv) For liquid - liquid reaction: Such type of reaction mixture is a mixture of two immiscible liquids; e.g. nitration of benzene using mixed acid, reaction of phenol with an alkali, etc.

For the better mixing of two immiscible liquids, axial currents are more important. If the viscosity of liquid-liquid mixture is less than $500 \text{ mPa} \cdot \text{s}$, then pitched blade turbine or propeller are more effective. For smaller vessel propeller (Fig. 10.2(i)) and for larger vessel pitched blade turbine (Fig. 10.2(iv)) is suitable. For higher viscosity ($\mu > 400 \text{ mPa} \cdot \text{s}$), agitator which contains cross beam with inclined blades (Fig. 10.2(xiv)), can be selected for liquid-liquid reaction system.

For nitration reaction, a loop reactor with a static mixer can be used. Mixed acid can be fed into the static mixer (at a controlled rate.) Heat exchanger, located on downstream of the static mixer, can remove exothermic heat of reaction effectively. It is claimed that in such a system chances of side reaction (such as dinitro formation) are minimal.

(v) For liquid-solid reaction:

In this case entire surface of solid particles must be accessible to the liquid. Hence, all solid particles must be suspended in the liquid. For the suspension of solids, axial currents are important. Hence, for the lower viscosity of slurry, 45° pitched blade turbine or propeller can be selected. For more viscous slurry ($500 \text{ mPa} \cdot \text{s} < \mu < 5000 \text{ mPa} \cdot \text{s}$) cross beam with inclined blades and for highly viscous slurry, helical ribbon (Fig. 10.2(xvi)) can be selected. If solids are sticky and heat transfer by jacket is important, then anchor agitator in conjunction with off-center propeller or pitched blade turbine is preferred.

Example 10.5

Hydrogenation of edible oil is carried out to produce ‘Vanaspati’ (hydrogenated fat) in presence of nickel catalyst in a batch reactor. In the standard age old process, edible oil is hydrogenated at about 2 bar g and $160\text{--}175^\circ\text{C}$ in 8 to 10 hours (excluding heating/cooling). During this period, iodine value of the mass is reduced from 128 to 68. Final mass has a melting (slip) point of 39°C . The batch reactor [Fig. 10.1(a)] has a jacket for heating the initial charge with circulating hot oil. Cooling requirements are met by passing cooling water in internal coils.

In a newly developed Jet Reactor [Fig. 10.1(i)], it is planned to complete the reaction in 5 hours by improving mass transfer in the reactor and cooling the mass in external heat exchanger, thereby maintaining near isothermal conditions. Figure 10.6 shows the suggested scheme.

Soybean oil, having iodine value (IV) of 128 is to be hydrogenated in the jet reactor at 5 bar g and 165°C . Initially the charge is heated from 30°C to 140°C with the circulating hot oil in external heat exchanger. Hydrogen is introduced in hot soybean oil and pressure is maintained in the reactor at 5 bar g. Reaction is exothermic and the temperature of mass increases. Cold oil flow in the external heat exchanger controls the temperature at 165°C as per the requirement, IV reduction is desired up to 68 when the reaction is considered over. Thereafter hydrogenated mass is cooled to 60°C in about 1.5 h before it is discharged to filter. 150 kg spent nickel catalyst is charged with soybean oil while fresh 5 to 10 kg nickel catalyst is charged at intervals in the reactor under pressure. A bleed is maintained from the system to purge out water vapour and non-condensables. Design the jet reactor for the following duty.

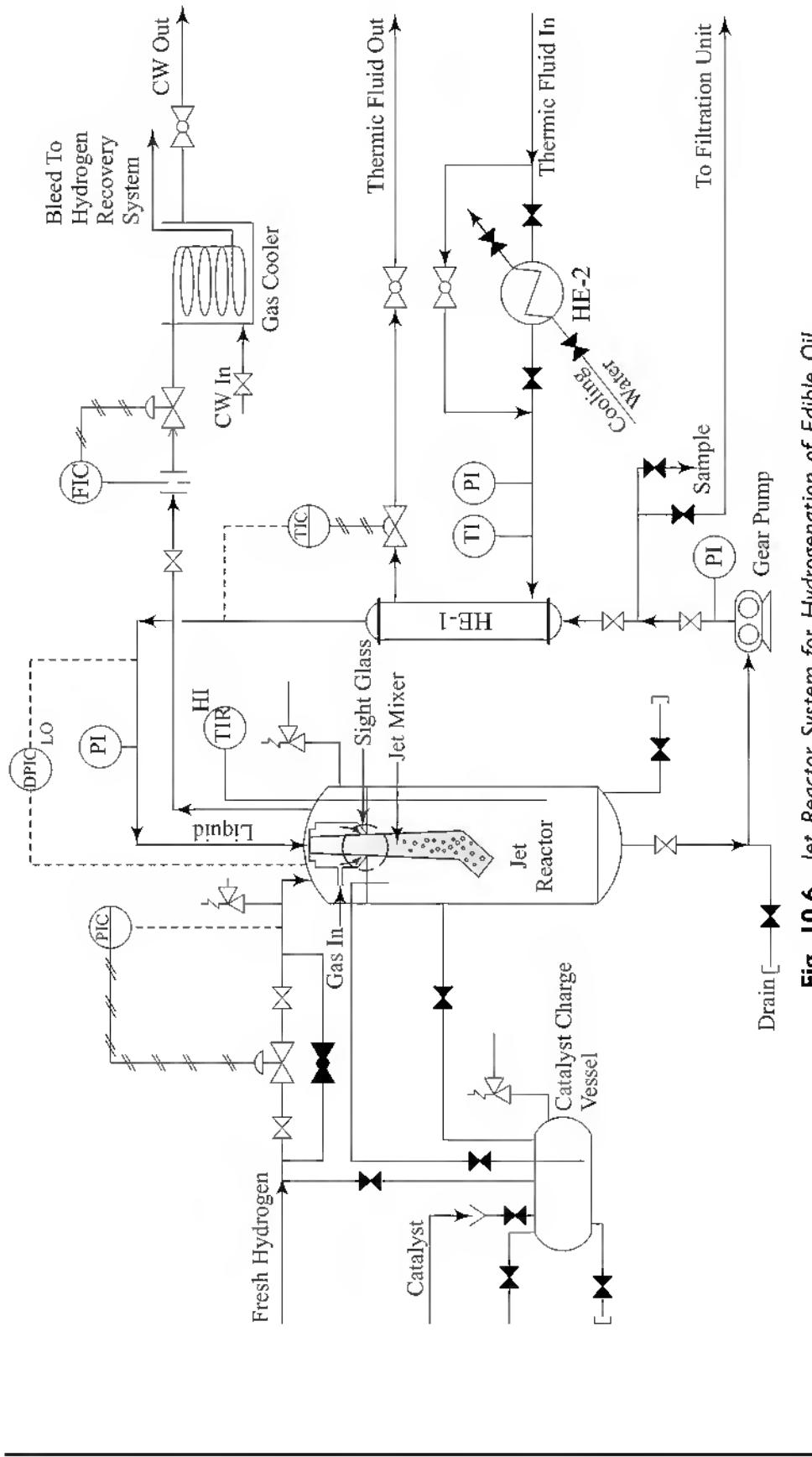


Fig. 10.6 Jet Reactor System for Hydrogenation of Edible Oil

- (i) Charge = 10 t soybean oil with 128 IV
- (ii) Average molar mass of soybean oil = 278.0
- (iii) Average chain length of fatty acids = 17.78
- (iv) Product specifications: 68 IV, 39°C melting point (max.)
Assume linear drop of IV in 5 hours.
- (v) Average exothermic heat of reaction = 7.1 kJ/(kg IV reduction)
- (vi) Hydrogen feed rate = 110 to 125 Nm³/h
Bleed rate = 1 to 2 Nm³/h
- (vii) Thermic fluid or oil is used as both, heating medium in starting of reaction and cooling medium in running of reaction.
- (viii) Cooling water is available at 2 bar g and 32°C. A rise of 5°C is permitted. Cooling water is used for cooling the oil from 80°C to 70°C in oil cooler (HE-2) of oil cycle.
- (ix) Assume following properties of fluids for the design.

Table 10.8 Average Properties of Edible Oil and Circulating Oil

Properties	Soybean oil or Hardened fat	Circulating oil (thermic fluid)
Density, kg/L	0.825	0.71
Specific heat, kJ/(kg · °C)	2.56	2.95
Viscosity, mPa · s	2.0	0.5
Thermal conductivity, W/(m · °C)	0.16	0.1

Solution:

$$\text{Volume of liquid inside the jet reactor, } V_L = \frac{10\ 000}{0.825} = 12\ 121.2 \text{ L} \equiv 12.12 \text{ m}^3$$

$$V_L = \frac{\pi}{4} D_i^2 h_L + \text{inside volume of bottom head}$$

where, D_i = Inside diameter of jet reactor, m
 h_L = Height of liquid inside the shell of jet reactor, m
Let $h_L = 1.5 D_i$
Type of bottom head = Torispherical

$$\text{Inside volume of torispherical head} = 0.084\ 672 D_i^3 + \frac{\pi}{4} D_i^2 S_F$$

$$\text{Let } S_F = 1.5 \text{ in} = 0.0381 \text{ m}$$

$$12.12 = \frac{\pi}{4} D_i^2 (1.5 D_i) + 0.084\ 672 D_i^3 + \frac{\pi}{4} \times 0.0381 D_i^2$$

$$12.12 = 1.26\ 28 D_i^3 + 0.029\ 92 D_i^2$$

$$D_i = 2.115 \text{ m}$$

$$h_L = 1.5 \times 2.115 = 3.1725 \text{ m}$$

$$\text{Let total height of shell of reactor, } H = 2 \times 2.115 = 4.23 \text{ m}$$

Design of external heat exchanger:

Type of heat exchanger: BEM type shell and tube heat exchanger

Heat duty of HE-1 for cooling period:

$$\phi_C = \text{Average heat of reaction} \times \text{IV reduction} \times \frac{\text{kg of reaction mass}}{\text{Reaction time}}$$

$$\phi_C = 7.1 \times (128 - 68) \times \frac{10\ 000}{5} = 852\ 000 \text{ kJ/h} \equiv 236.67 \text{ kW}$$

Let circulation rate of soybean oil = $71 \text{ m}^3/\text{h} \equiv 58\ 575 \text{ kg/h}$

$$\dot{m} = 58\ 575/3600 = 16.27 \text{ kg/s}, C_L = 2.56 \text{ kJ/(kg} \cdot ^\circ\text{C)}$$

$$\Delta T = 5.682^\circ\text{C}$$

Hence, in heat exchanger, temperature of circulating stream of reacting mass is to be reduced from 165°C to 159.318°C . It is cooled by thermic fluid entering at 70°C and leaving at 80°C .

Mass flow rate of cooling oil \dot{m}_o :

$$\dot{m}_o = \frac{236.67}{2.95(80 - 70)} = 8.023 \text{ kg/s} \equiv 28\ 883 \text{ kg/h}$$

Mean temperature difference:

$$\Delta T_m = \text{LMTD} \times F_t$$

$$\text{LMTD} = \frac{(165 - 80) - (159.318 - 70)}{\ln [(165 - 80)/(159.318 - 70)]}$$

$$\text{LMTD} = 87.141^\circ\text{C}$$

For 1-1 heat exchanger, $F_t = 1$.

Hence, $\Delta T_m = 87.141^\circ\text{C}$

Allocating reacting oil stream on tube side and thermic fluid (cooling medium) on shell side.

Calculations of h_i : Let tube OD, $d_o = 25.4 \text{ mm}$

For 16 BWG tube, tube ID, $d_i = 22.098 \text{ mm}$

(Table 11–2, of Ref. 2)

Let tube side velocity, $u_t = 1.5 \text{ m/s}$

$$u_t = G_t / \rho$$

$$G_t = u_t \rho = 1.5 \times 0.825 \times 10^3 = 1237.5 \text{ kg/(m}^2 \cdot \text{s})$$

$$G_t = \frac{\dot{m}}{a_t}$$

$$a_t = \frac{\dot{m}}{G_t} = \frac{58\ 575/3600}{1237.5} = 0.013\ 148 \text{ m}^2$$

$$a_t = \frac{N_t}{N_p} \times \frac{\pi}{4} d_i^2$$

where,

N_t = Total number of tubes, N_p = number of tube side passes = 1

$$N_t = \frac{0.013\ 148}{\frac{\pi}{4} \times (0.022\ 098)^2} \equiv 34$$

$$Re_t = \frac{d_t G_t}{\mu} = \frac{0.022\ 098 \times 1237.5}{2 \times 10^{-3}} = 13\ 673.14$$

$$Pr = \frac{C_p \mu}{k} = \frac{2.56 \times 2 \times 10^{-3} \times 10^3}{0.16} = 32$$

Dittus–Boelter's equation

$$\frac{h_i d_i}{k_f} = 0.023 Re^{0.8} Pr^{0.33} \left(\frac{\mu}{\mu_w} \right)^{0.14} \quad (6.19)$$

$$h_i = 0.023 \times \frac{0.16}{0.022098} \times (13\ 673.14)^{0.8} \times (32)^{0.33}$$

$$h_i = 1063.87 \text{ W}/(\text{m}^2 \cdot ^\circ\text{C})$$

Calculations of h_o :

For 25.4 mm (1 in) OD and 31.75 (1.25 in.) triangular pitch, from Table 6.1 (f), for

$N_t = 24$, BEM type 1.1 heat exchanger.

Shell ID, $D_s = 203 \text{ mm}$

Let baffle spacing, $B_s = 150 \text{ mm}$

Type of baffle = 25 % cut segmental

Shell side flow area

$$A_s = \frac{p_t - d_o}{p_t} \times D_s \times B_s \quad (6.29)$$

$$A_s = \frac{31.75 - 25.4}{31.75} \times 0.254 \times 0.15 = 7.62 \times 10^{-3} \text{ m}^2$$

$$\text{Shell side mass velocity, } G_s = \frac{\dot{m}_s}{A_s} \quad (6.30)$$

$$G_s = \frac{8.023}{7.62 \times 10^{-5}} = 1052.89 \text{ kg}(\text{m}^2 \cdot \text{s})$$

$$u_s = \frac{G_s}{\rho_o} = \frac{1052.89}{710} = 1.483 \text{ m/s}$$

Shell side equivalent diameter, d_e :

$$d_e = \frac{1.1}{d_o} (p_t^2 - 0.907 d_o^2) \quad (6.32)$$

$$= \frac{1.1}{25.4} (31.75^2 - 0.907 \times 25.4^2) = 18.3147 \text{ mm}$$

Shell side Reynolds number:

$$Re = \frac{d_e G_s}{\mu_o} = \frac{0.018\ 3147 \times 1052.89}{0.5 \times 10^{-3}} = 38\ 566.7$$

Prandtl number:

$$Pr = \frac{C_{po} \mu_o}{k_o} = \frac{2.95 \times 0.5 \times 10^{-3} \times 10^3}{0.1} = 14.75$$

$$\frac{h_o d_e}{K_o} = 0.36 Re^{0.55} Pr^{0.33} \left(\frac{\mu}{\mu_w} \right)^{0.14} \quad (6.35)$$

$$h_o = \frac{0.36 \times 0.1}{0.018\ 3147} \times (38\ 566.7)^{0.55} \times (14.75)^{0.33}$$

$$h_o = 1590.82 \text{ W}/(\text{m}^2 \cdot ^\circ\text{C})$$

Calculations of overall heat transfer coefficient, U_o :

$$\frac{1}{U_o} = \frac{1}{h_o} + \frac{1}{h_{od}} + \frac{d_o \ln(d_o/d_i)}{2k_w} + \frac{d_o}{d_i} \frac{1}{h_{id}} + \frac{d_o}{d_i} \frac{1}{h_i} \quad (6.42)$$

Take thermic fluid (oil) side fouling coefficient, $h_{od} = 5000 \text{ W}/(\text{m}^2 \cdot ^\circ\text{C})$ and soybean oil side fouling coefficient, $h_{id} = 3000 \text{ W}/(\text{m}^2 \cdot ^\circ\text{C})$ (Ref.: Table 6.9)

Tube material = SS 316

Thermal conductivity of tube material, $k_w = 16.26 \text{ W}/(\text{m}^2 \cdot ^\circ\text{C})$

(Table 3–322, of Ref. 2)

$$\begin{aligned} \frac{1}{U_o} &= \frac{1}{1590.82} + \frac{1}{5000} + \frac{0.0254 \ln(25.4/22.098)}{2 \times 16.26} + \frac{25.4}{22.098} \times \frac{1}{3000} \\ &\quad + \frac{25.4}{22.098} \times \frac{1}{1063.87} \end{aligned}$$

$$U_o = 416.5 \text{ W}/(\text{m}^2 \cdot ^\circ\text{C})$$

Heat transfer area:

$$\text{Heat transfer area required, } A_r = \frac{\phi_c}{U_o \Delta T_m} = \frac{236.67 \times 10^3}{416.5 \times 87.141} = 6.52 \text{ m}^2$$

$$\text{Length of tubes required, } L_r = \frac{A_r}{N_t \pi d_o} = \frac{6.52}{34 \times \pi \times 0.0254} = 2.403 \text{ m}$$

Let tube length $L = 3 \text{ m}$

Heat transfer area provided, $A = N_t \pi d_o L$

$$A = 34 \times \pi \times 0.0254 \times 3 = 8.139 \text{ m}^2$$

$$\% \text{ Excess heat transfer area} = \left(\frac{8.139 - 6.52}{6.52} \right) \times 100 = 24.83 \% \text{ (satisfactory)}$$

Tube side pressure drop, Δp_t :

$$\Delta p_t = N_p \left(8 j_f (L/d_i) \left(\frac{\mu}{\mu_w} \right)^{-0.14} + 2.5 \right) \frac{\rho u_t^2}{2} \quad (6.27)$$

For $Re = 13\,673.14, J_f = 0.0045$ (from Fig. (6.13))

$$\begin{aligned} \Delta p_t &= 1 \left(8 \times 0.0045 \left(\frac{3}{0.022\,098} \right) \times 1 + 2.5 \right) \left(\frac{825 \times 1.5^2}{2} \right) \\ &= 6856.4 \text{ Pa} \equiv 6.856 \text{ kPa (adequate)} \end{aligned}$$

Shell side pressure drop, Δp_s :

$$\Delta p_s = 8 J_f \left(\frac{D_s}{d_e} \right) \left(\frac{L}{B_s} \right) \frac{\rho_o u_s^2}{2} \left(\frac{\mu}{\mu_w} \right)^{-0.14} \quad (6.40)$$

For $R_e = 38\,566.73$ and 25 % cut segmental baffles

$J_f = 0.04$ (From Fig. 6.15)

$$\Delta p_s = 8 \times 0.04 \times \left(\frac{254}{18.3147} \right) \left(\frac{3}{0.15} \right) \left(\frac{710 \times 1.483^2}{2} \right) = 69\,299 \text{ Pa} = 69.3 \text{ kPa}$$

$\Delta p_s = 69.3 \text{ kPa}$ which is (optimum) pressure drop, $\Delta p_{s \max} = 70 \text{ kPa}$

(Ref. Table 6.8)

Resulting data for cooling:

Reacting soybean oil flow rate = $70 \text{ m}^3/\text{h} \equiv 58\ 575 \text{ kg/h}$
 Inlet temperature of soybean oil = 165°C
 Outlet temperature of soybean oil = 159.318°C
 Flow rate of thermic fluid = $40.68 \text{ m}^3/\text{h} \equiv 28\ 883 \text{ kg/h} \equiv 8.023 \text{ kg/s}$
 Inlet temperature of thermic fluid = 70°C
 Outlet temperature of thermic fluid = 80°C
 Heat duty of cooler (HE-1), $\phi_C = 236.67 \text{ kW}$
 Overall heat transfer coefficient, $U_o = 416.5 \text{ W}/(\text{m}^2 \cdot {}^\circ\text{C})$
 Type of heat exchanger (HE-1) = BEM Type, 1-1 shell and tube
 Tube $OD = 25.4 \text{ mm}$, 16 BWG thick
 Number of tubes = 34, Δ pitch, $p_t = 31.75 \text{ mm}$
 Tube length = 3 m
 Heat transfer area = 8.139 m^2 (provided)
 % Excess heat transfer area = 24.83%
 Shell ID = 254 mm
 Baffle type = 25% cut segmental
 Baffle spacing = 150 mm
 Tube side pressure drop, $\Delta p_t = 6.856 \text{ kPa}$
 Shell side pressure drop, $\Delta p_s = 69.3 \text{ kPa}$

Heating before starting of reaction:

Let time required for heating the soybean oil from 30°C to 140°C , $\theta = 2 \text{ h} = 3600 \times 2 = 7200 \text{ s}$

The same thermic fluid (oil) will be used as heating medium. Let T_1 is the inlet temperature of hot oil (thermic fluid) to heat exchanger in heating period. Both side flow rates (shell side and tube side) for heating period can be kept same as that for cooling period.

For heating in batch reactor by circulation through external exchanger.

$$\ln \left(\frac{T_1 - t_1}{T_1 - t_2} \right) = \frac{\dot{m} C_L}{M C_L} \left(\frac{K_2 - 1}{K_2} \right) \theta \quad (10.13)$$

(Eq. (10-147d) of Ref. 2)

T_1 = temperature of heating medium at inlet, ${}^\circ\text{C}$
 t_1 = temperature of cold fluid at the beginning of heating period, ${}^\circ\text{C}$
 t_2 = temperature of cold fluid at the end of heating period, ${}^\circ\text{C}$
 \dot{m} = flow rate of cold fluid through external heat exchanger, kg/s
 θ = time of heating period, s

$$K_2 = e^{(UA/\dot{m}C_L)} \quad (10.14)$$

U = Overall heat transfer coefficient, $\text{W}/(\text{m}^2 \cdot {}^\circ\text{C})$

A = heat transfer area, m^2

C_L = specific heat of cold fluid, $\text{kJ}/(\text{kg} \cdot {}^\circ\text{C})$

$t_1 = 30^\circ\text{C}$, $t_2 = 140^\circ\text{C}$, $\dot{m} = 16.27 \text{ kg/s}$, $C_L = 2560 \text{ J}/(\text{kg} \cdot {}^\circ\text{C})$

$A = 8.139 \text{ m}^2$, $M = 10\ 000 \text{ kg}$, $\theta = 7200 \text{ s}$

$U = 400 \text{ W}/(\text{m}^2 \cdot {}^\circ\text{C})$ (approximately same as that for cooling period)

Here, flow rates of oils are same as that for cooling period. However, small change in the value of U can be expected due to the change in the values of thermal properties of both oils with temperature.

$$\frac{UA}{\dot{m}C_L} = \frac{400 \times 8.139}{16.27 \times 2560} = 0.078163$$

$$K_2 = e^{(UA/\dot{m}C_L)} = 1.0813$$

$$\ln\left(\frac{T_1 - 30}{T_1 - 140}\right) = \frac{16.27 \times 2560}{10000 \times 2560} \left(\frac{1.0813 - 1}{1.0813}\right) \times 7200 = 0.881$$

$$\frac{T_1 - 30}{T_1 - 140} = e^{0.881} = 2.4133$$

$$T_1 = 217.83^\circ\text{C}$$

Let inlet temperature of hot oil, $T_1 = 230^\circ\text{C}$

Outlet temperature of hot oil, $T_2 = 220^\circ\text{C}$

Design of cooler (HE-2) of oil cycle:

Type of heat exchanger: BEM type, Fixed tube sheet

Tube side fluid: Cooling water

Shell side fluid: Oil (thermic fluid)

Cooling water inlet temperature = 32°C

Cooling water outlet temperature = 37°C

Heat duty, ϕ = heat duty of external heat exchanger during cooling period = 236.67 kW

Cooling water flow rate,

$$236.67 \times 10^3 = \dot{m}C_L \Delta t = \dot{m} \times (4.1868 \times 10^3) (37 - 32)$$

$$\dot{m} = 11.3055 \text{ kg/s} \equiv 40700 \text{ kg/h}$$

Mean temperature difference:

$$\Delta T_m = \text{LMTD} \times F_t$$

$$\text{LMTD} = \frac{(80 - 37) - (70 - 32)}{\ln \frac{(80 - 37)}{(70 - 32)}} = 40.4485^\circ\text{C}$$

Let number of the side passes = two

$$R = \frac{T_1 - T_2}{t_2 - t_1}, \quad S = \frac{t_2 - t_1}{T_1 - t_1} \quad (6.15)$$

$$R = \frac{80 - 70}{37 - 32} = 2, \quad S = \frac{37 - 32}{80 - 32} = 0.1042$$

$$F_t = 0.99 \quad (\text{From Fig. 6.11})$$

$$\Delta T_m' = 40.044^\circ\text{C}$$

Evaluation of tube side heat transfer coefficient, h_t :

Let tube side velocity, $u_t = 1.5 \text{ m/s}$

Density of water at $34.5^\circ\text{C} = 994.202 \text{ kg/m}^3$

$$\text{Volumetric flow rate of water} = \frac{40700/3600}{994.202} = 0.01137 \text{ m}^3/\text{s}$$

$$u_t = \frac{0.01137}{a_t} = 1.5$$

Hence, tube side flow area, $a_t = 7.58 \times 10^{-3} \text{ m}^2 = \frac{N_t}{N_p} \times \frac{\pi}{4} d_i^2$

$$N_p = 2$$

Let $d_0 = 19.05 \text{ mm}$, $d_i = 15.748 \text{ mm}$

$$7.58 \times 10^{-3} = \frac{N_t}{2} \times \frac{\pi}{4} (0.015748)^2$$

$$N_t = 78$$

From Table 6.1 (d), for 25.4 mm triangular pitch, $N_p = 2$, Shell ID = 305 mm

$$Re = \frac{d_i u_t \rho}{\mu} = \frac{0.015748 \times 1.5 \times 994.202}{0.73 \times 10^{-3}}$$

$$Re = 32171.3$$

$$Pr = \frac{C_L \mu}{k} = \frac{4.1868 \times 0.73 \times 10^{-3} \times 10^3}{0.628} = 4.867$$

(Viscosity of water, $\mu = 0.73 \text{ cP}$, Thermal conductivity of water, $k = 0.628 \text{ W/(m} \cdot ^\circ\text{C)}$

$$\frac{h_i d_i}{k} = 0.023 Re^{0.8} Pr^{0.33} \left(\frac{\mu}{\mu_w} \right)^{0.14} \quad (6.19)$$

$$h_i = 0.023 \times \frac{0.628}{0.015748} \times 32171.3^{0.8} \times 4.867^{0.33}$$

$$h_i = 6240.7 \text{ W/(m}^2 \cdot ^\circ\text{C)}$$

Evaluation of shell side heat transfer coefficient, h_o :

Shell side flow area,

$$A_s = \frac{P_t - d_o}{P_t} \times D_s \times B_s \quad (6.29)$$

$$P_t = 25.4 \text{ mm}, \quad d_o = 19.05 \text{ mm}, \quad D_s = 305 \text{ mm}$$

Let $B_s = 125 \text{ mm}$

$$A_s = \frac{25.4 - 19.05}{25.4} \times 0.305 \times 0.125 = 9.53125 \times 10^{-3} \text{ m}^2$$

$$\text{Shell side mass velocity, } G_s = \frac{\dot{m}_o}{A_s} \quad (6.30)$$

$$G_s = \frac{8.023}{9.53125 \times 10^{-3}} = 841.757 \text{ kg/(m}^2 \cdot \text{s})$$

$$u_s = (G_s / \rho_o) = (841.757 / 710) = 1.1856 \text{ m/s}$$

Shell side equivalent diameter,

$$d_e = \frac{1.1}{d_o} (P_t^2 - 0.907 d_o^2) \quad (6.32)$$

$$d_e = \frac{1.1}{19.05} (25.4^2 - 0.907 \times 19.05^2)$$

$$d_e = 18.25 \text{ mm}$$

$$Re = \frac{d_e G_s}{\mu} = \frac{0.01825 \times 841.757}{0.5 \times 10^{-3}} = 30724$$

$$Pr = 14.75$$

$$\frac{h_o d_e}{k} = 0.36 R_e^{0.55} Pr^{0.33} \left(\frac{\mu}{\mu_w} \right)^{0.14} \quad (6.35)$$

$$h_o = \frac{0.36 \times 0.1}{0.01825} \times (30724)^{0.55} \times (14.75)^{0.33}$$

$$h_o = 1408.8 \text{ W/(m}^2 \cdot ^\circ\text{C)}$$

Evaluation of overall heat transfer coefficient, U_o :

$$\frac{1}{U_o} = \frac{1}{h_o} + \frac{1}{h_{od}} + \frac{d_o \ln(d_o/d_i)}{2 k_w} + \frac{d_o}{d_i} \frac{1}{h_{id}} + \frac{d_o}{d_i} \frac{1}{h_i} \quad (6.42)$$

Thermic fluid (oil) side fouling coefficient, $h_{od} = 5000 \text{ W/(m}^2 \cdot ^\circ\text{C)}$

Tube material = mild steel

$$k_w = 50 \text{ W/(m} \cdot ^\circ\text{C)}$$

$$\frac{1}{U_o} = \frac{1}{1408.8} + \frac{1}{5000} + \frac{0.01905 \ln(19.05/15.748)}{2 \times 50}$$

$$+ \frac{19.05}{15.748} \times \frac{1}{5000} + \frac{19.05}{15.748} \times \frac{1}{6240.7}$$

$$U_o = 723.66 \text{ W/(m}^2 \cdot ^\circ\text{C)}$$

Heat transfer area required,

$$A_r = \frac{\phi}{U_o \Delta T_m} = \frac{236.67 \times 10^3}{723.66 \times 40.044} = 8.167 \text{ m}^2$$

$$\text{Tube length required, } L_r = \frac{8.167}{78 \times \pi \times 0.01905} = 1.7495 \text{ m}$$

Let tube length, $L = 2 \text{ m}$

Heat transfer area, $A = N_t \pi d_o L = 78 \times \pi \times 0.01905 \times 2 = 9.336 \text{ m}^2$

$$\% \text{ Excess heat transfer area} = \frac{9.336 - 8.167}{8.167} \times 100 = 14.31 \% \text{ (adequate)}$$

Tube side pressure drop, Δp_t :

$$\Delta p_t = N_p \left(8 j_f (L/d_i) \left(\frac{\mu}{\mu_w} \right)^{-m} + 2.5 \right) \left(\frac{\rho u_t^2}{2} \right) \quad (6.27)$$

For $Re = 32171.3, J_f = 3.5 \times 10^{-3}$ from Fig. 6.13

$$\Delta p_t = 2 \times \left(8 \times 3.5 \times 10^{-3} \left(\frac{2000}{15.748} \right) \times 1 + 2.5 \right) \left(\frac{994.202 \times 1.5^2}{2} \right)$$

$$\Delta p_t = 13547 \text{ Pa} \equiv 13.547 \text{ kPa (adequate)}$$

Shell side pressure drop, ΔP_s :

$$\Delta P_s = 8 J_f \left(\frac{D_s}{d_e} \right) \left(\frac{L}{B_s} \right) \frac{\rho_s u_s^2}{2} \left(\frac{\mu}{\mu_w} \right)^{-0.14} \quad (6.40)$$

For $Re = 30724, J_f = 0.041$ from Fig. 6.15 for 25% baffle cut

$$\begin{aligned} \Delta p_s &= 8 \times 0.041 \left(\frac{305}{18.25} \right) \left(\frac{2000}{125} \right) \frac{710 \times 1.203^2}{2} \times 1 \\ &= 45060 \text{ Pa} \equiv 45.06 \text{ kPa (adequate)} \end{aligned}$$

Resulting data for oil cooler (HE-2) of oil cycle:

Shell side fluid = Oil (thermic fluid), In = 80°C, Out = 70°C

Tube side fluid = Cooling water, In = 32°C, Out = 37°C

Heat duty = 236.67 kW

Mean temperature difference = 40.044°C

Overall heat transfer coefficient = 723.66 W/(m² · °C)

Heat transfer area required = 6.873 m²

Heat transfer area provided = 9.336 m²

% Excess heat transfer area = 14.31 %

Tube OD = 19.05 mm, Tube ID = 15.748 mm, Tube nos. = 78

Tube length = 2 m, nos. of tube side passes = 2

Heat exchanger type = BEM as per TEMA

MOC of heat exchanger = Mild steel

Shell ID = 254 mm

Tubeside pressure drop = 13.547 kPa

Shell side pressure drop = 37.525 kPa

Note: Circulation rate of soybean oil is fixed at 71 m³/h. However, differential pressure (DPIC) is unknown. It will depend on desired hydrogen circulation rate in the reactor. To begin with DP = 3 bar may be fixed and by actual experience this can be optimized. Reactor pressure and differential pressure together will decide discharge pressure and power requirement of the gear pump.

10.7 BUBBLE COLUMN REACTOR^{8, 9}

In a bubble column reactor, gas is dispersed in liquid phase at the bottom of column by a suitable distributor. In most of the cases this distributor is a sparger. Bubble column can be operated in a semibatch, countercurrent or cocurrent manner. Bubble columns are used as reactors, absorbers and strippers.

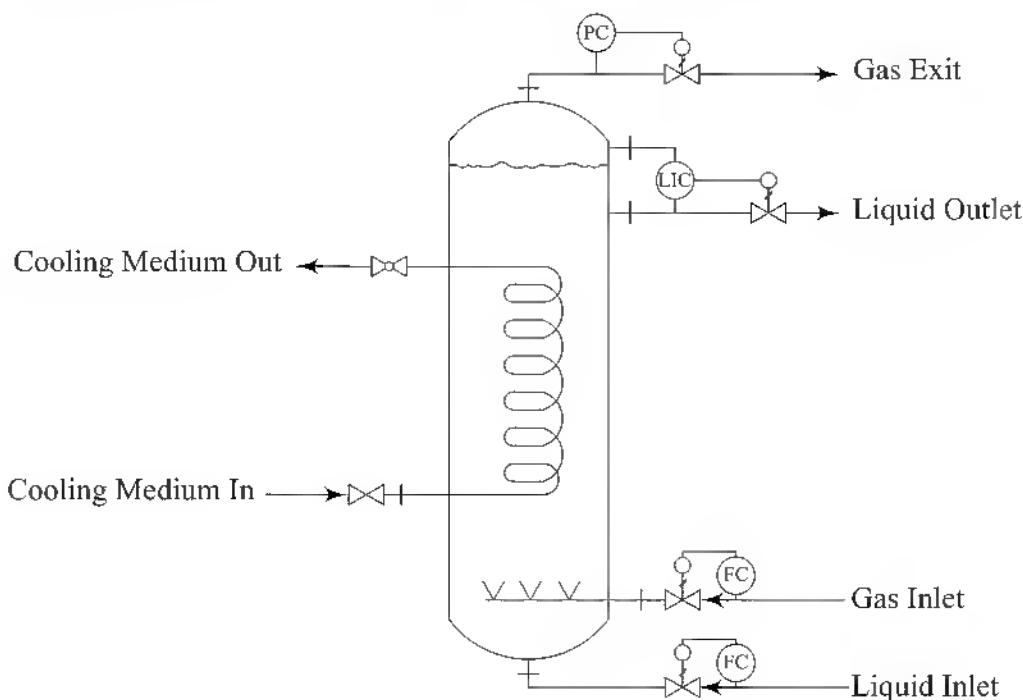


Fig. 10.7 Bubble Column Reactor

Bubble column reactor is preferred for slow or very slow gas–liquid reactions. As overall rate of this reaction is governed by rate of chemical reaction, the rate of consumption of limiting reactant in chemical reaction ($-dN_A/dt$) is directly proportional to V_l (volume of liquid phase inside the reactor or liquid holdup). Rate of consumption of limiting reactant in reaction ($-dN_A/dt$), in case of slow or very slow reaction, does not depend on interfacial surface area of contact between gas and liquid phase.

Bubble column reactor does not use any moving part like agitator. Construction of this reactor is simple and inexpensive.

[10.7.1] Various Factors Affecting the Performance of Bubble Column Reactor

(i) Superficial Gas Velocity

Increase in superficial gas velocity increases gas holdup, the effective interfacial area and the overall mass transfer rate. Increase in superficial gas velocity decreases the size of bubbles hence increases the interfacial area of contact between gas and liquid. Effect of superficial gas velocity is negligible on mass transfer coefficient (K_L) but significant on mass transfer area (a). For chemical reaction controlled gas–liquid reaction (slow or very slow gas–liquid reaction) also, certain minimum $K_L a$ must be achieved to overcome the effect of mass transfer. It is recommended to keep superficial velocity of gas less than or equal to 10 m/s.

(ii) Properties of Gas Phase and Liquid Phase

Physical properties of gas phase have no effect on the performance of the column. However, the physical properties of liquid phase like surface tension, viscosity, etc., have a profound effect on the performance of column. Increase in the viscosity of liquid phase or decrease in the surface tension increases the effective interfacial area and hence increases mass transfer rate. Presence of electrolyte in the liquid phase effects greatly on performance. The electrolyte solution gives smaller bubble size and consequently higher effective interfacial area and higher rate of mass transfer.

(iii) Back Mixing

Based on a study, it is found that gas flows in bubble column operates in plug flow manner without any back mixing while considerable amount of back mixing in the liquid phase is observed.

Back mixing in liquid phase decreases the concentration of liquid reactant and hence the rate of chemical reaction. Consequently it decreases the overall rate of reaction. Use of packings, trays or baffles reduces liquid back mixing and thereby reduces dilution of reactants by products. Hence, they provide the higher concentration of reactant and hence the higher rate of chemical reaction, $-r_A = (-1/V_l)(-dN_A/d\theta)$. But they decrease the liquid holdup (V_l). Hence, combined effect on rate of consumption of limiting reactant in reaction ($-dN_A/d\theta$) must be checked before using any internal in the bubble column. In case of highly exothermic gas–liquid reaction, back mixing in liquid phase is desirable to control the exothermicity of reaction or to control the temperature of reaction. In such a

case placing packings to improve the overall rate makes the temperature control very difficult because it increases the heat duty required and decreases heat transfer coefficient.

(iv) Mode of Operation

For very slow gas–liquid reaction, semibatch operation is preferred in which liquid is charged to the reactor in batchwise manner while gas is continuously passed through the column. For moderately fast reaction, continuous operation is preferred. Both counter current and cocurrent operation is used. But cocurrent is more common. In counter current contact, velocities of gas and liquid through the tower are limited by flooding conditions while cocurrent contact permits the higher velocities of gas and liquid through the tower compared to counter current contact. Also, concentration of reactants, rate of chemical reaction and overall rate of reaction do not depend on the mode of operation (whether it is cocurrent or counter current) because bubble column reactors are ideal mixed flow reactors.

If superficial liquid velocity is greater than 30 cm/s and superficial gas velocity is less than 1 to 3 cm/s then it is better to use cocurrent down flow column. (Ref.: 8)

(v) Gas Expansion and Shrinkage

Inside the bubble column, there may be a dramatic increase in superficial velocity of gas from gas inlet to outlet and is called gas expansion. Decrease in superficial velocity of gas from inlet to outlet is called gas shrinkage.

Example of gas expansion:

Chlorination of benzene gives chlorobenzene and hydrogen chloride gas. In this case theoretically total number of moles of gas phase remains constant and hence flow rate of gas and superficial velocity of gas remains unchanged. But actually this reaction is exothermic, resulting in vaporization of organic liquids. Hydrogen chloride and unconverted chlorine, leaving from the top, are saturated with organic vapours at the outlet conditions. In addition to that hydrostatic head of liquid decreases in upper part of column. Because of these combined effects, there is a considerable increase in superficial velocity of gas from inlet to outlet.

Example of gas shrinkage:

Alkylation of benzene with ethylene in presence of AlCl_3 (aluminium chloride) solution as catalyst. Because of the consumption of gas (ethylene) in the reaction, there is a substantial decrease in superficial velocity of gas in upper part of column.

Since superficial gas velocity affects overall reaction rate, mass transfer rate and heat transfer rate, this factor is important and should be carefully considered in designing of bubble column reactor.

(vi) Pulsation

The performance of bubble column can be improved by pulsations. For very low superficial velocity of gas (0.8 to 2.4 cm/s) the value of $K_L a$ can be increased by factor as much as 3 by pulsation.

(vii) Addition of Packing or Packed Bubble Column

Adding packing reduces axial mixing of liquid. Hence, packed bubble column is used where liquid back mixing is undesirable. For gas–liquid reaction in which substantial gas shrinkage is taking place in the reactor due to reaction, this results in very low superficial gas velocity which in turn results in poor rate of mass transfer in the upper part of reactor. Hence, in such a case placing of packings in upper part is beneficial. Packing increases effective interfacial area and gas hold-up to an extent. Hence, it provides higher rate of mass transfer. Packed bubble column is not used if fine solid particles are present in the system as catalyst or as reactant. Packing is also not preferred for highly exothermic or endothermic reaction as packed tower provides poor heat transfer coefficient.

10.7.2 Industrial Examples of Bubble Column Reactor

- (i) Production of protein from methanol is carried out in a fermentor which are bubble column reactors. Largest size of bubble column reactor having capacity of 3000 m^3 is used for this application. (Ref: 3)
- (ii) Absorption of carbon dioxide in ammoniated brine for the manufacture of soda ash.
- (iii) Liquid phase air oxidation of a variety of organic compounds.
Ex. (a) Air oxidation of acetaldehyde in presence of fine KMnO_4 (potassium permanganate) particles as catalyst (b) Air oxidation of *p*-nitrotoluene sulphonic acid
- (iv) Air oxidation of black liquor containing Na_2S (sodium sulphide) in pulp (in a paper mill)
- (v) Air oxidation of ammonium sulphide $[(\text{NH}_4)_2\text{S}]$
- (vi) Liquid phase chlorination of variety of organic compounds. Ex. (a) Chlorination of benzene to chlorobenzene (b) Chlorination of acetic acid to monochloroacetic acid, etc.
- (vii) (a) Carbonylation of methanol to acetic acid.
(b) Carbonylation of ethanol to propionic acid
- (viii) Liquid phase oxychlorination of ethylene for the production of vinyl chloride
- (ix) Hydration of propylene with sulphuric acid for the production of vinyl chloride
- (x) Reaction between ethylene (C_2H_2) and liquid hydrogen fluoride for the manufacture of difluoroethane.

10.7.3 Advantages and Disadvantages of Bubble Column Reactor Over Stirred Tank Reactor (Agitated Vessel Type Reactor)

(a) Advantages

- (i) Sealing problem is negligible in bubble column while the same is severe in agitated vessel type reactor. Sealing of the shaft of an agitator for high pressure reactor is not only an initial design problem but also a continuing maintenance problem. This problem is very important for highly toxic, high

pressure reaction system, e.g. ozonation reaction. Above 40 atm operating pressure, agitated vessel type reactor is not recommended for use.

- (ii) Bubble column reactor provides more liquid hold-up than agitated vessel type reactor. Hence, it requires less volume for chemical reaction controlled gas–liquid reaction (for slow and very slow gas–liquid reaction).
- (iii) Bubble column reactor requires less maintenance as compared to agitated vessel type reactor.
- (iv) Bubble column reactor requires less power consumption. Because of the agitator, power consumption is higher with agitated vessel type reactor.
- (v) It requires lesser floor space.

(b) Disadvantages

- (i) Agitated vessel type reactor provides higher mass transfer coefficient. Hence, for mass transfer controlled gas – liquid reaction, agitated vessel type reactor requires lesser volume for the given extent of reaction. It may require lesser fixed capital investment than bubble column reactor for such applications.
- (ii) Agitated vessel type reactor provides higher heat transfer coefficient compared to bubble column reactor. Hence temperature control is easier with agitated vessel type reactor than the same for bubble column reactor.
- (iii) Higher interfacial area is obtained with agitated vessel type reactor. Agitator cuts the bubbles in smaller sizes and thereby increases interfacial area of contact. Higher interfacial area provides higher mass transfer rate.
- (iv) Bubble column reactor requires more height.

10.7.4 Criteria of Selection for Different Types of Gas–Liquid Reactors

Table 10.9 Typical Ratios for Gas–Liquid Reactors (Ref: 1)

Type of reactor	S/V_l	S/V_R	V_l/V_R	V_l/V_{film}
Packed column	1200	100	0.08	10 to 100
Tray tower	1000	150	0.15	40 to 100
Agitated vessel	200	200	0.9	150 to 800
Bubble Column	20	20	0.98	4000 to 100 00

where, S = Interfacial surface area, m^2

V_l = Volume of liquid, m^3

V_R = Volume of reactor, m^3

V_{film} = Volume of liquid film, m^3

Overall rate of any gas – liquid reaction is a function of rate of chemical reaction and rate of mass transfer. [Overall rate = f (Rate of chemical reaction, rate of mass transfer)].

For very fast reaction overall rate of reaction is governed by rate of mass transfer, i.e. rate of chemical reaction \gg rate of mass transfer or

Overall rate of reaction \equiv Rate of mass transfer.

To get the higher rate of mass transfer, packed tower is the best choice. Packed tower provides higher mass transfer coefficient as well as higher interfacial area of contact. But packed bed provides poor heat transfer coefficient.

For slow and very slow gas liquid reaction overall rate is governed by rate of chemical reaction. Rate of consumption of reactant in a chemical reaction does not depend on interfacial area of contact (S), but it depends on liquid holdup V_l ,

$$\left(\frac{-dN_A}{d\theta} \right) = V_l k f(C). \text{ Bubble column reactor provides maximum liquid holdup.}$$

V_l/V_R ratio is maximum for bubble column reactor while the same is very low for packed bed reactor. Bubble column reactor provides low heat transfer coefficient.

For the gas–liquid reactions having intermediate rates, overall rate of reaction depends on both; rate of chemical reaction and rate of mass transfer. Hence, for these reactions agitated vessel type reactor is the best choice, because it provides higher S/V_R ratio and higher V_l/V_R ratio. It provides higher heat transfer coefficient but it consumes more power. For high pressure gas–liquid reaction if gaseous reactant is toxic then bubble column reactor is preferred against agitated vessel type reactor because of the sealing problem. Example: Carbonylation of methanol gives acetic acid. It is an intermediate gas–liquid reaction. Agitated vessel type reactor is the best choice. But, operating pressure of reactor is 50 atm and carbon monoxide is a toxic gas. So, to avoid the use of agitator shaft sealing, for the same reaction, bubble column reactor is preferred.

For highly exothermic gas–liquid reaction, loop reactor or jet reactor (with recirculation, recirculating pump and external heat exchanger) can be considered. Loop reactor provides higher heat transfer coefficient and hence better temperature control than bubble column reactor. But, it consumes more power. For hydrogenation of edible and non-edible oils, loop reactor or jet reactor or gas induced agitated reactor is found to be an excellent choice as it gives desired product pattern in less time than an agitated type batch reactor with sparger mechanism.

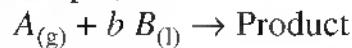
10.7.5 Process Design of Bubble Column Reactor

It can be divided in following steps.

- (i) Find the working volume of reactor. Working volume of reactor means volume of liquid in reactor in running condition which also includes gas holdup.

Bubble column reactors are selected for slow or very slow gas–liquid reaction for which overall rate of reaction is totally controlled by the rate of chemical reaction. Hence, with most of the bubble column reactors (bubble column reactors are used for different applications), it is very easy to overcome the effect of mass transfer. Superficial velocity of gas in bubble column reactor is fixed in such a way that effect of $K_L a$ or mass transfer is eliminated. If the superficial velocity of the gas is fixed above the limiting value S_{gm} then overall rate of gas–liquid reaction is equal to the rate of chemical reaction. S_{gm} is the minimum superficial velocity of gas required to overcome the effect of mass transfer.

For example,



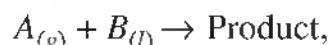
$$\text{If } S_g \gg S_{gm} \text{ Overall rate} = -r_{Al} = \frac{1}{V_l} \frac{dN_A}{d\theta} = k C_A C_B$$

In bubble column reactor, liquid is flowing in mixed flow manner. Hence, composition of reactants in liquid phase is almost uniform throughout and working volume of reactor can be determined by using the performance equation of ideal mixed flow reactor.

$$\frac{V_l}{F_{Ao}} = \frac{X_A}{-r_{Al}} \quad (10.15)$$

If for the given gas-liquid reaction it is not possible to overcome the effect of mass transfer, then form of overall rate of gas-liquid reaction must be developed. It can be developed by laboratory scale and pilot plant scale experiments. Different forms of rate equation are assumed and then verified by experimental data. In such a case overall rate is the function of both mass transfer coefficient ($K_L a$) and reaction rate constant k .

For example, for the gas-liquid reaction



following form of rate equation can be assumed for verification.

$$-r_A = \frac{1}{V_l} \frac{dN_A}{dt} = \frac{p_A}{\left[\frac{H_A}{K_L a} + \frac{H_A}{kC_B} \right]} = \frac{C_A}{\left[\frac{1}{K_L a} + \frac{1}{kC_B} \right]} \quad (\text{Typical})$$

where, $K_L a$ = Mass transfer coefficient

a = Interfacial area per unit volume

k = Reaction rate constant

H_A = Henry's law constant

p_A = Partial pressure of reactant A in gas phase

C_A = Concentration of reactant A in liquid phase

In such a case also, since in bubble column reactor liquid phase is flowing in mixed flow manner, working volume V_l can be determined by following equation.

$$\frac{V_l}{F_{Ao}} = \frac{X_A}{-r_A} \quad (10.16)$$

where, $-r_A$ = Overall rate of reaction = $f(K_L a, k)$

- (ii) Find or fix the value of superficial velocity of gas. Superficial velocity of gas in bubble column reactor should be such that overall rate of reaction becomes independent of mass transfer coefficient $K_L a$. Minimum superficial velocity of gas required to overcome the effect of mass transfer should be determined by actual experiment in small scale (lab scale or pilot plant

reactor). Many different correlations are available which relate the superficial velocity of gas and mass transfer coefficients; $K_L a$. However, they are not reliable and change from system to system. If it is not possible to overcome the effect of mass transfer, then correlation of $K_L a$ must be developed in small scale reactor for the given system and the same can be used to determine the diameter of commercial reactor. In scale – up, influence of wall effect (which is significant in small scale reactor while it is negligible in commercial scale reactor) must be considered.

$$\frac{\pi}{4} D_i^2 = \frac{Q_v}{S_g} \quad (10.17)$$

where, Q_v = Volumetric flow rate of gas, m^3/s

D_i = Inside diameter of reactor, m

S_g = Superficial velocity of gas, m/s

Based on the value of D_i , height of reactor H can be determined.

- (iii) Calculate the heat transfer area required:

Heat duty required for bubble column reactor can be determined from the heat of reaction ΔH_R at reaction temperature.

$$\phi_t = \Delta H_R \text{ kJ/mol} \times \frac{1000 \text{ mol}}{1 \text{ kmol}} \times \frac{\text{kmol}}{\text{h}} \text{ of limiting reactant}$$

consumed $\pm \phi'$

where, ϕ' = Heat utilized for other purpose

$$h_i = 21\ 766.5 \ S_g^{0.22} \left(\frac{Pr_w}{Pr_L} \right)^{1/2} \quad (10.18)^9$$

where, h_i = Reacting fluid side heat transfer coefficient, $\text{W}/(\text{m}^2 \cdot \text{C})$

S_g = Superficial velocity of gas, m/s

Pr_w = Prandtl number of water at room temperature

Pr_L = Prandtl number of liquid phase of reactor at reaction condition.

- (iv) Sparger design is not important if superficial gas velocity is more than or equal to 10 cm/s.

Example 10.6

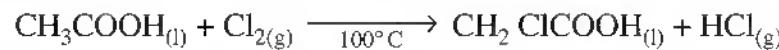
In the continuous process for the manufacturing of monochloroacetic acid (MCA) conversion of acetic acid is restricted to 50% to avoid the formation of dichloroacetic acid. Reaction is carried out in a bubble column reactor.

Determine the following.

- Working volume of reactor
- Diameter of reactor
- Height of liquid inside the reactor during reaction
- Heat duty of over head condenser in which exist gas–vapour mixture is cooled down to 40°C by cooling with water
- Heat transfer area required for bubble column reactor

Data

(i) Reaction



(ii) Heat of reaction at reaction temperature, i.e. at 100°C

$$\Delta H_R = -87.92 \text{ kJ/mol (Exothermic)}$$

(iii) 20% excess chlorine is used.

(iv) Cooling water is available in plant at 32°C.

(v) Mass transfer coefficient data

$$S_g = 1 \text{ to } 30 \text{ cm/s} \quad K_L a = 0.25 \times 10^{-2} \text{ to } 0.4 \text{ s}^{-1}$$

(vi) Rate of chemical reaction

$$-r_A = \frac{1}{V_l} \frac{dN_A}{dt} = k C_A, \quad k = 2.777 \times 10^{-5} \text{ s}^{-1}$$

Density of acetic acid, $\rho = 1048 \text{ kg/m}^3$

(vii) Production rate of monochloroacetic acid = 1 t/h

(viii) Operating pressure in reactor = 0.1 atm g

Solution:

(a) Working volume of reactor

Bubble column reactor can be assumed as ideal mixed flow reactor.

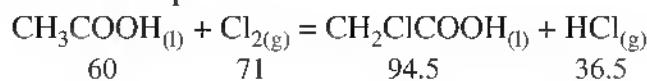
$$\frac{V_l}{F_{Ao}} = \frac{X_A}{-r_A} \quad (10.15)$$

$$\frac{V_l}{F_{Ao}} = \frac{X_A}{K C_A} = \frac{X_A}{k C_{Ao} (1 - X_A)} = \frac{1}{k C_{Ao}} \times \frac{0.5}{(1 - 0.5)}$$

$$V_l = \left(\frac{F_{Ao}}{C_{Ao}} \right) \cdot \frac{1}{k} = \frac{V_o}{k}$$

where, V_o = Volumetric flow rate of acetic acid at inlet

Stoichiometric equation:



Conversion of acetic acid is restricted to 50% to avoid the DCA formation.

Acetic acid required to produce 1 t/h of MCA:

$$\dot{m}_{AA} = 1000 \times \frac{60}{94.5} \times \frac{1}{0.5}$$

$$\dot{m}_{AA} = 1269.84 \text{ kg/h}$$

$$V_o = \frac{1269.84}{1048} = 1.2117 \text{ m}^3/\text{h}$$

$$V_o = 3.3658 \times 10^{-4} \text{ m}^3/\text{s}$$

$$V_l = \frac{V_o}{k} = \frac{3.3658 \times 10^{-4}}{2.777 \times 10^{-5}} = 12.12 \text{ m}^3$$

Working volume of reactor, $V_l = 12.12 \text{ m}^3$

Ans. (a)

(b) for superficial velocity of gas

$$s_g \in \{1 \text{ cm/s}, \dots, 30 \text{ cm/s}\}, K_L a \gg k \quad (\text{as given in data})$$

$$\text{Let } s_g = 10 \text{ cm/s}, K_L a \gg k$$

Overall rate of reaction = Rate of chemical reaction

Mass flow rate of chlorine at inlet

$$\dot{m}_{\text{Cl}_2} = 1.2 \times \frac{71}{94.5} \times 1000$$

$$\dot{m}_{\text{Cl}_2} = 901.59 \text{ kg/h}$$

Density of chlorine gas

$$\rho_{\text{Cl}_2} = \frac{pM}{RT} = \frac{1.1 \times 71}{(273 + 100)} \times \frac{273}{1 \times 22.414} = 2.55 \text{ kg/m}^3$$

$$\text{Volumetric flow rate of chlorine} = \frac{901.59}{2.55} = 353.56 \text{ m}^3/\text{h} \equiv 0.09821 \text{ m}^3/\text{s}$$

$$\frac{\pi}{4} D_i^2 = \frac{Q_v}{s_g} \quad (10.17)$$

$$\frac{\pi}{4} D_i^2 = \frac{0.09821}{10 \times \left(\frac{1}{100}\right)} = 0.9821 \text{ m}^2$$

$$D_i = 1.118 \text{ m}$$

Ans. (b)

(c) Let h_L = Height of liquid during reaction

$$V_l = \frac{\pi}{4} D_i^2 h_L$$

$$12.12 = \frac{\pi}{4} (1.118)^2 \times h_L$$

$$h_L = 12.346 \text{ m}$$

Minimum distance required to facilitate gas-liquid separation is recommended to be D_i .

Let H = height of bubble column reactor = $h_L + D_i$

$$H \cong 12.346 + 1.118 = 13.464 \text{ m}$$

Let $H = 14 \text{ m}$

Ans. (c)

(d) Let ϕ_c = Heat duty of overhead condenser

In overhead condenser gas-vapour mixture is cooled down to 40°C.

Let \dot{n}_t = Total molar flow rate of gas-vapour mixture leaving the reactor.

Exist gas from the reactor is saturated with liquid vapour at outlet conditions of the reactor. Molar flow rate of chlorine at outlet of reactor

$$\dot{n}_{\text{Cl}_2} = 0.2 \times \frac{1000}{94.5} = 2.116 \text{ kmol/h}$$

Molar flow rate of hydrogen chloride gas at outlet of reactor

$$\dot{n}_{\text{HCl}} = \frac{1000}{94.5} = 10.582 \text{ kmol/h}$$

$$\begin{aligned} \dot{n}_t &= \dot{n}_{\text{Cl}_2} + \dot{n}_{\text{HCl}} + \dot{n}_{\text{AA}} + \dot{n}_{\text{MCA}} \\ \text{and} \quad p_t &= \bar{p}_{\text{Cl}_2} + \bar{p}_{\text{HCl}} + \bar{p}_{\text{AA}} + \bar{p}_{\text{MCA}} \\ p_t &= \bar{p}_{\text{Cl}_2} + \bar{p}_{\text{HCl}} + p_{\text{VAA}} \times x_{\text{AA}} + x_{\text{MCA}} \times p_{\text{VMCA}} \end{aligned}$$

At 100°C vapour pressure of acetic acid $p_{\text{VAA}} = 400$ torr

At 100°C vapour pressure of MCA, $p_{\text{VMCA}} = 30$ torr

$$p_t = 1.1 \times 760 = \bar{p}_{\text{Cl}_2} + \bar{p}_{\text{HCl}} + 400 \times 0.5 + 30 \times 0.5 = 836 \text{ torr}$$

$$\bar{p}_{\text{Cl}_2} + \bar{p}_{\text{HCl}} = 621 \text{ torr}$$

$$(\bar{p}_{\text{Cl}_2} + \bar{p}_{\text{HCl}})/p_t = (\dot{n}_{\text{HCl}} + \dot{n}_{\text{Cl}_2})/\dot{n}_t$$

$$\frac{621}{836} = \frac{10.582 + 2.1164}{\dot{n}_t}$$

$$\dot{n}_t = 17.095 \text{ kmol/h}$$

At 100°C or in the exist gas – vapour mixture from the reactor

$$\dot{n}_{\text{AA}} = \frac{400 \times 0.5}{836} \times 17.095 = 4.09 \text{ kmol/h}$$

$$\dot{n}_{\text{MCA}} = \frac{30 \times 0.5}{836} \times 17.095 = 0.307 \text{ kmol/h}$$

This gas–vapour mixture is cooled to 40°C by cooling water in the overhead condenser. At outlet of the overhead condenser, gas mixture is saturated with acetic acid and MCA vapour.

To find out the composition of gas–vapour mixture at the outlet of overhead condenser, for the 1st trial calculations assume that almost total condensation is taking place in condenser.

Condensate composition at outlet of condenser

$$x_{\text{AA}} = \frac{4.09}{4.09 + 0.307} = 0.93$$

$$x_{\text{MCA}} = 0.07$$

At 40°C, $p'_t = p_t - \Delta p_{\text{HE}}$

$$p'_t = 1.1 - 0.04 = 1.06 \text{ atm}$$

The pressure drop in heat exchanger for gas–vapour mixture is assumed to be 0.04 atm.
At 40°C or at outlet of overhead condenser

$$p'_t = 1.06 \times 760 = \bar{p}_{\text{HCl}} + \bar{p}_{\text{Cl}_2} + 0.93 \times p_{\text{VAA}} + 0.07 \times p_{\text{VMCA}}$$

At 40°C, $p_{\text{VAA}} = 38$ torr, $p_{\text{VMCA}} = 1$ torr

$$805.6 = \bar{p}_{\text{HCl}} + \bar{p}_{\text{Cl}_2} + 0.93 \times 38 + 0.07 \times 1$$

$$\bar{p}_{\text{HCl}} + \bar{p}_{\text{Cl}_2} = 770.19 \text{ torr}$$

$$\frac{\bar{p}_{\text{HCl}} + \bar{p}_{\text{Cl}_2}}{p'_t} = \frac{10.582 + 2.1164}{\dot{n}_t} = \frac{770.19}{805.6}$$

$$\dot{n}_t = 13.28 \text{ kmol/h}$$

At 40°C, $P_{\text{VAA}} = 38$ torr, $P_{\text{VMCA}} = 1$ torr

$$\dot{n}_{AA} = \frac{0.93 \times 38 \times 13.28}{805.6} = 0.5826 \text{ kmol/h}$$

$$\dot{n}_{MCA} = \frac{0.07 \times 1}{805.6} \times 13.28 = 1.154 \times 10^{-3} \text{ kmol/h}$$

For 2nd trial calculations, composition of condensate at outlet of overhead condenser is calculated.

$$x_{AA} = \frac{4.09 - 0.5826}{(4.09 - 0.5826) + (0.307 - 1.154 \times 10^{-3})} = 0.9198$$

$$x_{MCA} = 1 - x_{AA} = 0.0802$$

$$p_t = 805.6 = \bar{p}_{HCl} + \bar{p}_{Cl_2} + 0.9198 \times p_{VAA} + 0.0802 \times p_{vMCA}$$

$$805.6 = \bar{p}_{HCl} + \bar{p}_{Cl_2} + 0.9198 \times 38 + 0.0802 \times 1$$

$$\bar{p}_{HCl} + \bar{p}_{Cl_2} = 770.5674 \text{ torr}$$

$$\frac{\bar{p}_{HCl} + \bar{p}_{Cl_2}}{p_t} = 0.9565 = \frac{10.582 + 2.1164}{\dot{n}_t}$$

$$\dot{n}_t = 13.276 \text{ kmol/h}$$

$$\dot{n}_{AA} = \frac{0.9198 \times 38 \times 13.276}{805.6} = 0.576 \text{ kmol/h}$$

$$\dot{n}_{MCA} = \frac{0.0802 \times 1}{805.6} \times 13.276 = 1.32 \times 10^{-3} \text{ kmol/h}$$

For the new values of \dot{n}_{AA} and \dot{n}_{MCA}

$$x_{AA} = 0.91997, \quad x_{MCA} = 0.08 \text{ or } x_{AA} = 0.92, \quad x_{MCA} = 0.08$$

These values are very close to previous values. Hence, third trial is not required.

Mass of acetic acid condensed = $(4.09 - 0.576) \times 60 = 210.84 \text{ kg/h}$

Mass of MCA condensed = $(0.307 - 1.32 \times 10^{-3}) \times 94.5 = 28.887 \text{ kg/h}$

Heat duty of overhead condenser

ϕ_{tc} = Sensible heat transfer of gas-vapour mixture + Latent heat transfer for condensation of vapours + Subcooling of condensate
(Subcooling of condensate from inlet to outlet temperature is necessary in multicomponent condensation.)

$$\phi_{tc} = (\dot{m}_{Cl_2} C_{p, Cl_2} + \dot{m}_{HCl} C_{p, HCl} + \dot{m}'_{AA} C_{p, AA} + \dot{m}'_{MCA} C_{p, MCA}) \Delta T + \dot{m}_{AA} \lambda_{AA} + \dot{m}_{MCA} \lambda_{MCA} + (\dot{m}''_{AA} C_{LAA} + \dot{m}''_{MCA} C_{LMCA}) \Delta T$$

where, $\dot{m}_{Cl_2} = 2.1164 \times 71 = 150.26 \text{ kg/h}$

$$\dot{m}_{HCl} = 10.582 \times 36.5 = 386.24 \text{ kg/h}$$

$$\dot{m}'_{AA} = \frac{4.09 + 0.576}{2} \times 60 = 139.98 \text{ kg/h}$$

$$\dot{m}'_{MCA} = \frac{0.307 + 1.32 \times 10^{-3}}{2} \times 94.5 = 14.568 \text{ kg/h}$$

Table 10.10 Specific Heat of Gases/Vapours Vapours and Liquids at 70°C

Component	C_{pi} , kJ/(kg · °C)	C_{Li} , kJ/(kg · °C)
Chlorine	0.5024	—
Hydrogen chloride	0.7955	—
Acetic acid	1.2267	2.22
MCA	1.0467	1.9

$$\dot{m}_{AA} = 210.84 \text{ kg/h}, \quad \dot{m}_{MCA} = 28.887 \text{ kg/h}$$

$$\lambda_{AA} = 110 \text{ kcal/kg} = 460.55 \text{ kJ/kg at } 70^\circ\text{C temperature}$$

$$\lambda_{MCA} = 85 \text{ kcal/kg} = 355.88 \text{ kJ/kg at } 70^\circ\text{C temperature}$$

$$\dot{m}_{AA}'' = \frac{0 + 210.84}{2} = 105.42 \text{ kg/h}$$

$$\dot{m}_{MCA}'' = \frac{0 + 28.887}{2} = 14.44 \text{ kg/h}$$

$$\begin{aligned} \phi_{tc} &= (150.26 \times 0.5024 + 386.24 \times 0.7955 + 139.98 \times 1.2267 + 14.568 \\ &\quad \times 1.0467) \times (100 - 40) + 210.84 \times 460.55 + 28.887 \times 355.88 \\ &\quad + (105.42 \times 2.22 + 14.44 \times 1.9) \times (100 - 40) \end{aligned}$$

$$\phi_{tc} = 157\ 253.15 \text{ kJ/h} \equiv 43.6814 \text{ kW} \quad (\text{Ans. (d)})$$

(e) Heat transfer area required for bubble column reactor:

Energy balance around reactor

Heat must be removed by cooling medium circulated through the jacket around reactor

ϕ_t = Heat produced during reaction – Heat removed in overhead condenser

$$= 87.92 \frac{\text{kJ}}{\text{mol}} \times 1000 \frac{\text{mol}}{\text{kmol}} \times \frac{\text{kmol}}{\text{h}} \text{ of Acetic acid consumed} - 157\ 253.15$$

$$= 87.92 \times 1000 \times 10.582\ 011 - 157\ 253.15$$

$$= 773\ 117.2 \text{ kJ/h} \equiv 214.75 \text{ kW}$$

Let outlet temperature of cooling water from heat exchanger = 40°C

$$\text{Mass flow rate of cooling water, } \dot{m}_w = \frac{773\ 117.22}{4.1868 \times 8}$$

$$\dot{m}_w = 23\ 082 \text{ kg/h} \equiv 23.1 \text{ m}^3/\text{h}$$

If cooling water is circulated through plain jacket then jacket side heat transfer coefficient can be calculated by considering plain jacket as outside pipe of double pipe heat exchanger.

h_o :

$$\frac{h_0 d_e}{K} = 0.023 Re^{0.8} Pr^{1/3} \left(\frac{\mu}{\mu_w} \right)^{0.14} \quad (10.19)$$

$$\text{where, } d_e = \text{Equivalent diameter} = 4 \times r_H \quad (10.20)$$

$$r_H = \frac{\text{Cross sectional area}}{\text{Wetted perimeter}}$$

$$r_H = \frac{(\pi/4)(d_2^2 - d_1^2)}{\pi(d_2 + d_1)} \quad (10.21)$$

where,

d_2 = Inside diameter of jacket, m

d_1 = Outside diameter of reactor shell, m

$$r_H = \frac{d_2 - d_1}{4}$$

Let

$$d_2 - d_1 = 100 \text{ mm}$$

$$d_e = 4r_H = d_2 - d_1 = 0.10 \text{ m}$$

$$Re = \frac{d_e G}{\mu}$$

$$G = \frac{23100/3600}{(\pi/4)(d_2^2 - d_1^2)}$$

d_1 = Reactor inside diameter + 2 × thickness of shell (t_s)

Let thickness of reactor shell, $t_s = 8 \text{ mm}$ (assumption)

This thickness is actually determined based on mechanical design of shell.

$$d_1 = 1118 + 2 \times 8 = 1134$$

$$d_2 = 1134 + 100 = 1234 \text{ mm}$$

$$G = \frac{23100/3600}{(\pi/4)(1.234^2 - 1.134^2)} = 34.50 \text{ kg}/(\text{m}^2 \cdot \text{s})$$

$$Re = \frac{0.1 \times 34.50}{0.72 \times 10^{-3}} = 4792 \text{ (Viscosity of water at } 36^\circ\text{C} = 0.72 \text{ mPa} \cdot \text{s})$$

$$\begin{aligned} Pr &= \frac{C_L \mu}{k} = \frac{4.1868 \times (0.72 \times 10^{-3}) 10^3}{0.6228} \\ &= 4.84 \end{aligned}$$

$$h_o = 0.023 \times \frac{0.6228}{0.1} \times (4792)^{0.8} \times (4.84)^{1/3}$$

$$h_o = 213.19 \text{ W}/(\text{m}^2 \cdot {}^\circ\text{C})$$

Value of h_o is very low. To improve or to increase its value, let outlet temperature of cooling water = 34°C

$$\dot{m}_w = \frac{773117}{4.1868 \times 2} = 92328 \text{ kg/h}$$

$$G = 34.5 \times \frac{92328}{23100} = 137.9 \text{ kg}/(\text{m}^2 \cdot \text{s})$$

$$Re = \frac{d_e G}{\mu} = 4792 \times \frac{92328}{23100} = 19153$$

$$h_o = 213.18 \times \left(\frac{19153}{4792} \right)^{0.8} = 645.84 \text{ W/(m}^2 \cdot ^\circ\text{C)}$$

 h_i :

Reacting fluid side heat transfer coefficient

$$h_i = 21766.5 S_g^{0.22} \left(\frac{Pr_w}{Pr_L} \right)^{\frac{1}{2}} \quad (10.18)$$

where,

 s_g = Superficial velocity of gas, m/s $s_g = 10 \text{ cm/s} = 0.1 \text{ m/s}$

$$Pr_L = \frac{C_L \mu_L}{k_L}$$

Where C_L , μ_L and k_L are the properties of liquid phase of reactor at 100°C, temperature (Reaction temperature).**Table 10.11 Properties at 100°C**

	Acetic acid	MCA	Water
C_L , kJ/(kg.°C)	2.3	1.884	4.1868
μ , mPa · s	0.49	0.55	0.28
k , W/(m · °C)	0.173	0.143	0.68

$$C_{L_{\text{mix}}} = \sum w_i C_{Li}$$

$$w_{AA} = \frac{0.5 \times 60}{(0.5 \times 60) + (0.5 \times 94.5)} = 0.3883$$

$$w_{MCA} = 1 - w_{AA} = 0.6117$$

$$C_L = \sum C_{Li} w_i = 0.6117 \times 1.884 + 0.3883 \times 2.3 = 2.046 \text{ kJ/(kg} \cdot ^\circ\text{C)}$$

$$\frac{1}{\mu_L} = \frac{1}{\mu_{\text{mix}}} = \frac{w_{AA}}{\mu_{AA}} + \frac{w_{MCA}}{\mu_{MCA}} = \frac{0.6117}{0.55} + \frac{0.3883}{0.49}$$

$$\mu_L = 0.525 \text{ mPa} \cdot \text{s}$$

$$k_L = \sum k_i w_i = 0.3883 \times 0.173 + 0.6117 \times 0.143$$

$$k_L = 0.1546 \text{ W/(m} \cdot ^\circ\text{C)}$$

$$Pr_L = \frac{2.046 \times 0.525 \times 10^{-3}}{0.1546} \times \frac{1000}{1}$$

$$Pr_L = 6.9479$$

Prandtl number of water at 100°C:

$$Pr_W = \frac{4.1868 \times 0.28 \times 10^{-3}}{0.68} \times \frac{10^3}{1}$$

$$Pr_W = 1.724$$

$$h_i = 21766.5 \times (0.1)^{0.22} \times \left(\frac{1.724}{6.9479} \right)^{1/2}$$

$$h_i = 6533.27 \text{ W/(m}^2 \cdot ^\circ\text{C)}$$

For MCA reactor, lead lined vessel is normally used. Let thickness of lead lining = 5 mm

$$d'_o = \text{OD of lead lining} = 1.118 \text{ m}$$

$$d'_i = \text{ID of lead lining} = 1.108 \text{ m}$$

At 100°C, $k_{\text{lead}} = 34 \text{ W/(m} \cdot ^\circ\text{C)}$,

$$k_{\text{steel}} = 45 \text{ W/(m} \cdot ^\circ\text{C)}$$

Conductive resistance offered by lead lining

$$\begin{aligned} &= \frac{d_o}{d'_{om}} \times \frac{\Delta x}{k_{\text{lead}}} = \frac{d_o}{(d'_o - d'_i)} \times \ln \left(\frac{d'_o}{d'_i} \right) \times \frac{(d'_o - d'_i)/2}{k_{\text{lead}}} \\ &= \frac{d_o \ln (d'_o/d'_i)}{2 k_{\text{lead}}} \end{aligned}$$

Overall heat transfer coefficient U_o is calculated by following equation.

$$\frac{1}{U_o} = \frac{1}{h_o} + \frac{1}{h_{od}} + \frac{d_o \ln (d_o/d_i)}{2 k_{ws}} + \frac{d_o \ln (d'_o/d'_i)}{2 k_{\text{lead}}} + \frac{d_o}{d_i} \frac{1}{h_i} + \frac{d_o}{d_i} \frac{1}{h_{id}} \quad (6.42)$$

Let fouling coefficients

$$h_{od} = h_{id} = 5000 \text{ W/(m}^2 \cdot ^\circ\text{C)}$$

$$\begin{aligned} \frac{1}{U_o} &= \frac{1}{645.84} + \frac{1}{5000} + \frac{1.134 \ln \left(\frac{1.134}{1.118} \right)}{2 \times 45} + \frac{1.134 \ln \left(\frac{1.118}{1.108} \right)}{2 \times 34} \\ &\quad + \frac{1.134}{1.108} \times \frac{1}{6533.27} + \frac{1.134}{1.108} \times \frac{1}{5000} = 410.07 \text{ W/(m}^2 \cdot ^\circ\text{C)} \end{aligned}$$

$$\Delta T_m = \Delta T_{\ln} = \frac{(100 - 32) - (100 - 34)}{\ln \left(\frac{100 - 32}{100 - 34} \right)} = 66.995 \text{ }^\circ\text{C}$$

$$A_{\text{oreq}} = \frac{\phi_t}{U_o \Delta T_m} = \frac{800000 \times \left(\frac{1}{3600} \right) \times \left(\frac{1000}{1} \right)}{410.07 \times 66.995} = 8.089 \text{ m}^2$$

Let $A_{\text{oavai}} = 8.089 \times 1.2 = 9.707 \text{ m}^2$ (Area provided)

$$A_{\text{oavai}} = 9.707 \text{ m}^2 = \pi d o L' = \pi \times (1.134) L'$$

where, L' = Length of cylinder that must be covered by jacket = 2.725 m

However, entire height of liquid pool ($h_L = 12.346 \text{ m}$) must be covered by plain jacket as shown in Fig. 10.8 to keep the uniform temperature of entire liquid pool. Reaction temperature can be controlled by controlling the flow rate of cooling water as shown in Fig. 10.8.

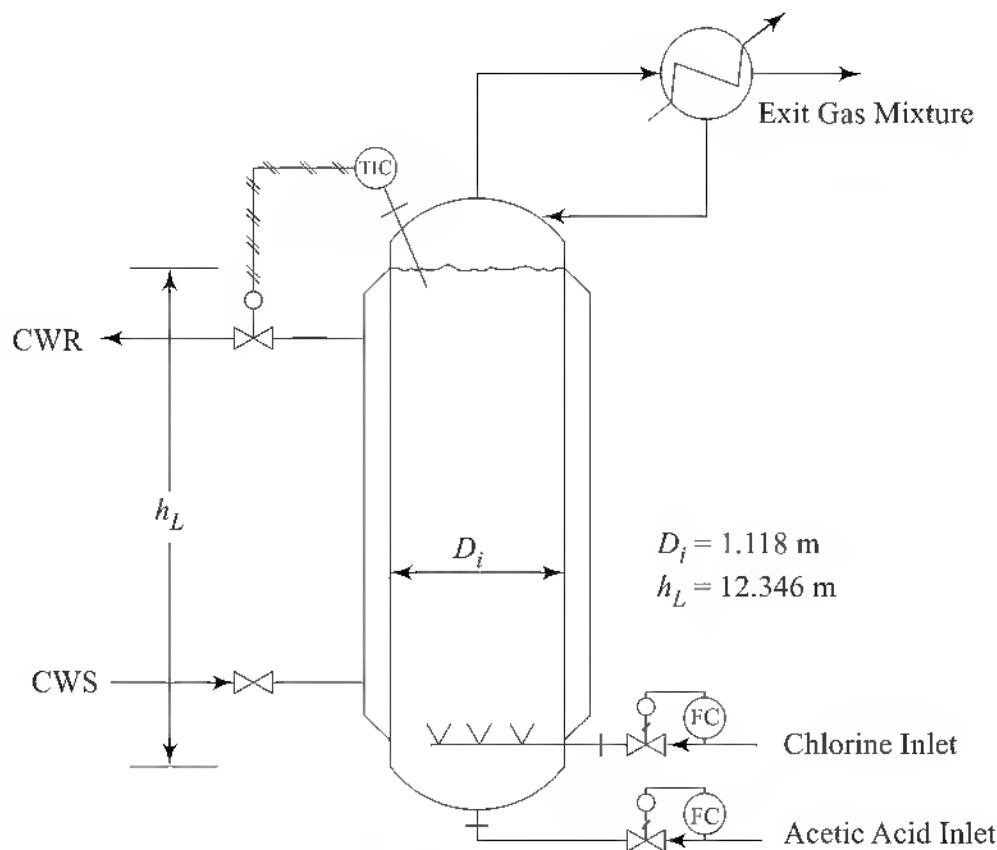


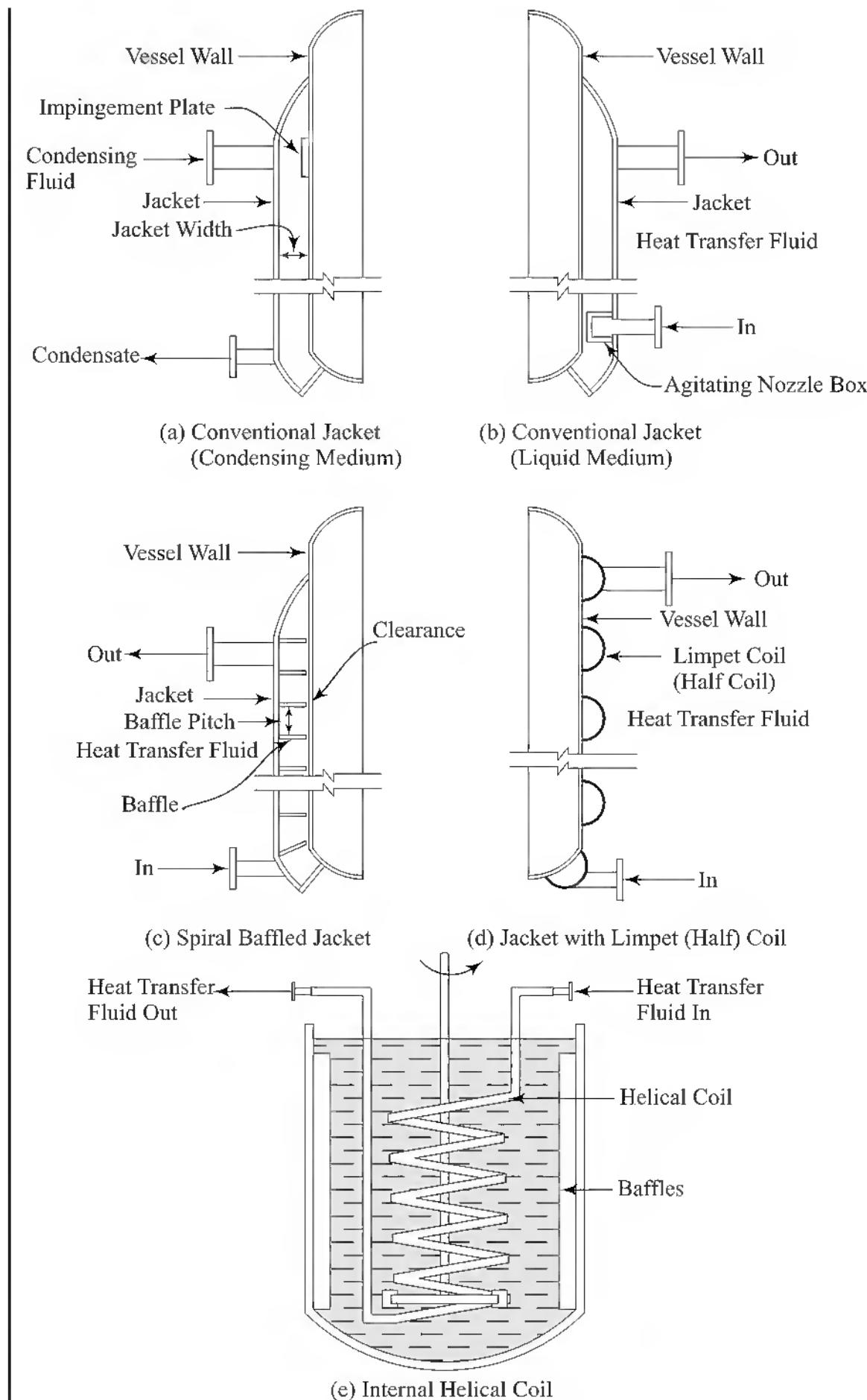
Fig. 10.8 Proposed MCA Reactor Design

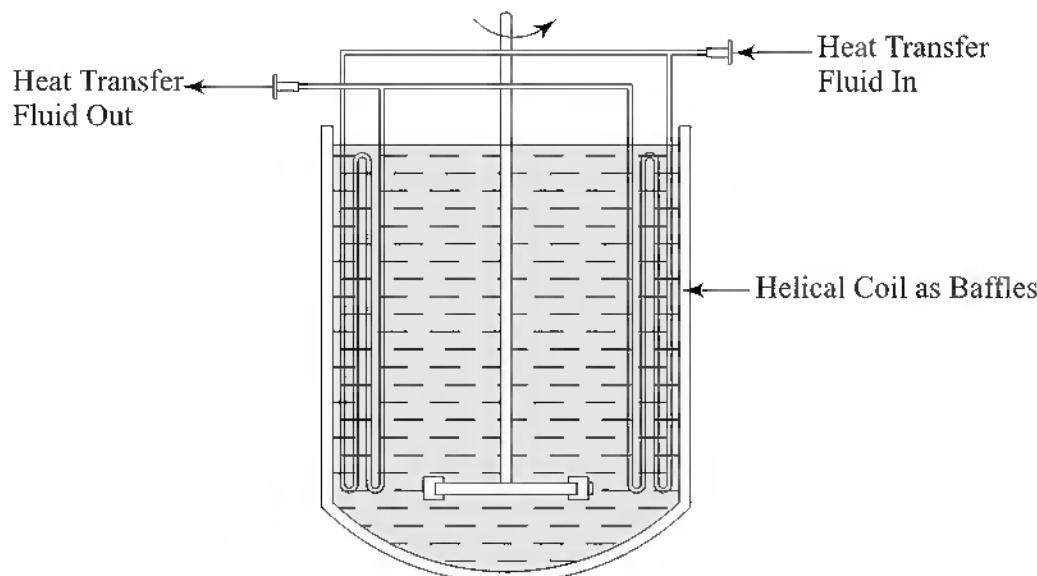
Use of spirals in the plain jacket decreases flow area considerably, increases velocity and Re of jacket side fluid and consequently increases jacket side heat transfer coefficient considerably. In this case flow area for jacket side fluid is area between two successive metal strips which forms the spiral. (Refer Fig. 10.9(c))

For the exothermic reaction (e.g. chlorination of acetic acid) saturated steam is fed into the jacket to start the reaction but then after cooling water is circulated through the same jacket to remove the heat of reaction. For such a case plain jacket is selected as it is preferred for saturated steam. For cooling water circulation or hot oil circulation spiral jacket, channel jacket, limpet coil, etc. provide considerably higher heat transfer coefficient. Refer Fig. 10.9 for common arrangement of jackets, limpet coil and internal coils.

10.8 DESIGN OF FIXED CATALYST BED REACTORS FOR GASEOUS REACTIONS^{10, 11, 12, 2}

In many syntheses, fixed bed catalytic reactors are used. Water shift reaction to produce hydrogen by reaction of carbon monoxide with steam is carried out in a

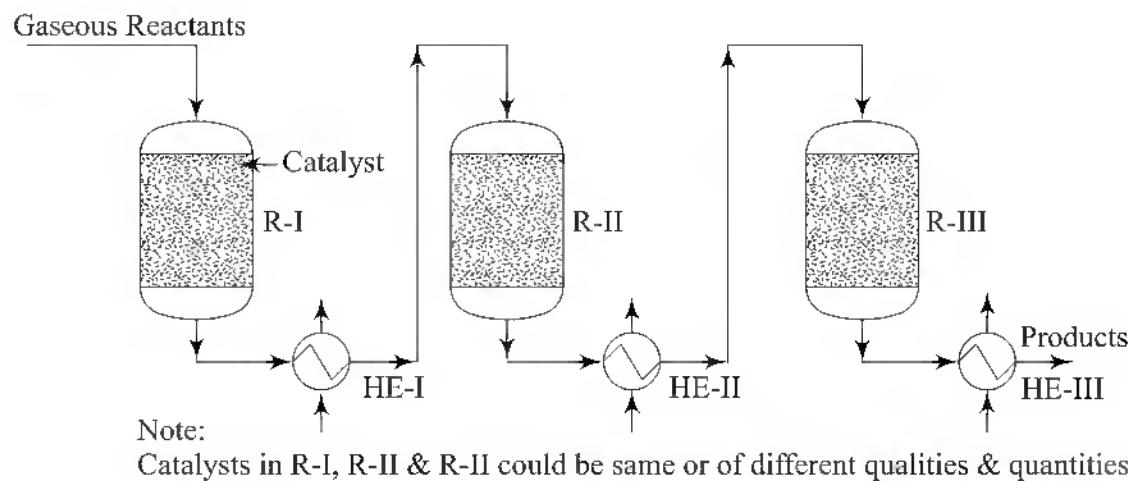
**Fig. 10.9 (Contd.)**



(f) Vessel with Helical Coils as Baffles

Fig. 10.9 Common Arrangements of Heat Transfer Jackets and Internal Coils

fixed catalyst bed. However, this reaction is exothermic with practically no effect of pressure on the reaction rate. At high temperature, the rate of reaction is relatively high but equilibrium conversion is low. In such a situation, a battery of reactors in series is used. High temperature, medium temperature and low temperature shift converters are common in syngas production from hydrocarbons. Each reactor is packed with different types of catalysts. Exit gas from each converter is passed through a heat exchanger to recover the heat and to lower the gas temperature, entering the next reactor. In this way, advantages of high heat of reaction at higher temperature and higher equilibrium conversion at low temperature are achieved. In a typical natural gas based plant, carbon monoxide content in the reformer outlet gas is about 15% (v/v) which is reduced to 0.2% (v/v) in two or three different axial flow converters. Generally, these converters are designed for low to moderate pressures.

**Fig. 10.10** Multiple Catalytic Reactors with External Heat Exchangers

For certain exothermic gaseous reactions, rate of reaction at low temperature is so low that required volume of catalyst works out to be very large. Cost of such a large reactor could be prohibitive, more so for high pressure reaction. Hence certain exothermic gaseous reactions are carried out at high temperature. Examples are ammonia synthesis, methanol synthesis, dissociation of nitrous oxide to nitrogen, etc. While designing such converters, process engineer can consider different options. In one option, reaction is carried out in multiple reactors having the same or different catalysts. Exit gas from each reactor is cooled in an external heat exchanger. Partially converted gas mixture from the heat exchanger enters the next reactor and so on as shown in Fig. 10.10.

Cost of number of reactors and heat exchangers is becoming prohibitive in many cases. Also heat recovery from the exit gas mixture from a reactor may not be possible in each case.

Quench bed reactors are developed for carrying out exothermic reactions at high temperature. Figures 10.11(a) and 10.11(b) depict such designs. Entire reactor is one vessel in which there are number of catalyst beds (usually 3 or 4.)

A known portion of incoming gas mixture (stream F_1 which is more than 50%) is introduced at the bottom at relatively low temperature. It passes through the annulus around catalyst baskets, thereby keeping the shell of the reactor cool and also gets some heat from the reacting gas mixture. F_1 stream enters the shell and tube heat exchanger, located at top and gets heated by outgoing gas mixture. Hot F_1 stream enters 1st catalyst bed and partial conversion takes place thereby the gas mixture is heated up. Portion of the main gas stream is so controlled that temperature of 1st bed exit gas stream is controlled at predefined value. Partially reacted gas mixture from 1st bed is cooled by quenching by a definite proportion of the fresh gas mixture (1st quench). Mixing of both gas mixtures results in lowering of the temperature of the gas mixture, entering 2nd catalyst bed. In this manner, temperature of gas mixture, entering other beds is controlled by quenching, thereby, conversion in each bed is increased. Gas mixture from final catalyst bed is taken out through a central pipe and taken to the heat exchanger for exchanging heat with incoming gas mixture. Overall effect is to get definite conversion per pass through the converter without exchanging heat in external heat exchangers.

Since number of moles reduce during syntheses of ammonia and methanol, high pressure is employed. Quench bed reactor for such syntheses are multi-layer wall vessel. As explained earlier, wall temperature of the reactor is kept low by the flow of ingoing gas mixture in the annulus. This is advantageous as at low temperature, lower wall thickness is required.

In Fig. 10.11(a), gas flow through the catalyst bed is axial while in Fig. 10.11(b), it is radial. Design, shown in Fig. 10.11(b), results in lower pressure drop in the gas mixture, resulting in energy saving. Volume of catalyst in each bed differs. Volume of 4th bed is highest while that of 1st bed is lowest. Volumes are decided by reaction rate, catalyst activity and safety margin against possible deactivation due to catalyst poisons.

While dissociating nitrous oxide to nitrogen and oxygen, increase in moles takes place. According to Le Chatelier's principle, low pressure favours the reac-

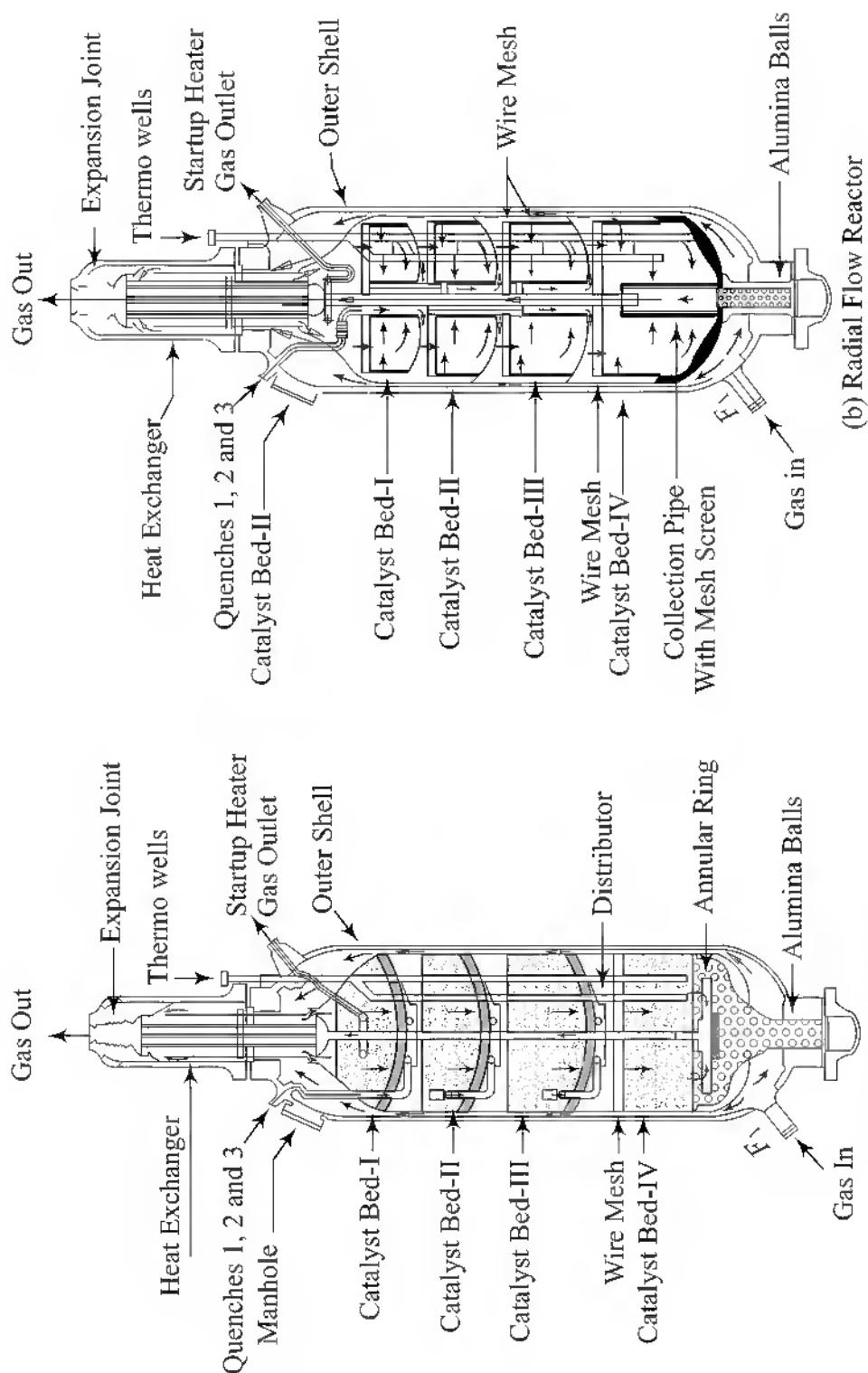


Fig. 10.11 *Multiple Quench Bed Reactor*

tion. Therefore, a quench bed reactor with multiple catalyst bed for dissociation of nitrous oxide is designed for low pressure.

Design of fixed catalyst bed reactors for gaseous reactions thus can be innovative. Process engineer can design an efficient system with best heat recovery options. Some other designs of industrial fixed bed catalytic reactors are:

(i) packed bed reactor (ii) shell and tube heat exchangers in which catalysts are placed inside the tubes (iii) annulus flow type reactors in which catalyst are placed inside the inner tube. Feed gas first pass through the annulus and then enter the inner tube. Product gases are taken out from the bottom of inner tube, (iv) catalysts can be in form of mesh. Mesh of catalyst may be placed inside tower. Feed gas mixture is introduced from the top of tower. Product gases leave from the bottom of catalyst mesh. (Example: ammonia oxidation to nitric acid). In some cases catalyst mesh may be wound around the tubes of shell and tube heat exchanger. (Example: reactor of acetaldehyde plant. In partial oxidation of ethanol vapour, silver catalyst in the form of mesh is wrapped around the tubes of shell and tube heat exchanger.) Section 10.8.1 shows the industrially important examples of fixed bed catalytic reactor.

10.8.1 Industrial Example of Fixed Bed Catalytic Reactor

<i>Product</i>	<i>Major reaction step</i>	<i>Catalyst</i>
1. Ammonia	$N_2 + 3 H_2 \rightleftharpoons 2 NH_3$	FeO/Fe ₂ O ₃ , promoted by Al ₂ O ₃ and K ₂ O
2. Nitric acid	$4 NH_3 + 5 O_2 \rightarrow 4 NO + 5 H_2O$ $2 NO + O_2 \rightarrow 2 NO_2$ $3 NO_2 + H_2O \rightarrow 2 HNO_3 + NO$	Pt and Rh
3. Sulphur trioxide (Sulphuric acid plant)	$SO_2 + 1/2 O_2 \rightarrow SO_3$	V ₂ O ₅ + K ₂ O
4. Methanol	$CO + 2 H_2 \rightarrow CH_3OH$	CuO
5. Formaldehyde	$CH_3OH + 1/2 O_2 \rightarrow HCHO + H_2O$	Ag
6. Aniline	$C_6H_5OH + NH_3 \rightarrow C_6H_5NH_2 + CO_2 + H_2O$	Ni Supported on Al ₂ O ₃
7. Disintegration of nitrous oxide	$N_2O \rightarrow N_2 + 1/2 O_2$	Transition metal oxides
8. Ethyl benzene	$C_6H_6 + C_2H_4 \rightarrow C_6H_5CH_2CH_3$	Zeolite (Refer Fig. 2.2)

Fixed bed catalytic reactors are operated in both adiabatic and nonadiabatic manner. If the heat of reaction is small then adiabatic packed bed type reactor is used as fixed bed reactor. In such type of reactor there is no provision for heat transfer.

But the reactions with a large heat of reaction as well as reactions that are extremely temperature sensitive are carried out in nonadiabatic reactors in which

indirect heat exchange takes place by circulating heat transfer medium integrated in the fixed bed. With large heat of reaction, shell and tube type design is preferred in which catalyst is inside the tubes and heat transfer medium is circulated on shell side. Alternatively, multiple quench bed reactor can be used (e.g. ammonia synthesis). When high pressure and high temperature reaction is carried out with indirect heat exchangers, conversion per pass can be near equilibrium. However, in multiple quench bed reactor, temperature of final exit stream controls the conversion per pass.

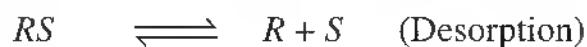
The heart of a fixed-bed reactor and the site of the chemical reaction is the catalyst. The overall reaction taking place on catalyst surface can be theoretically divided into the following five separate steps.

- (i) Diffusion of the reactants from the gas space through the outer gas – particle boundary larger, macropores and micropores.
- (ii) Chemisorption on active centers
- (iii) Surface reactions
- (iv) Desorption of the products
- (v) Back-diffusion of the products into the gas space. Also deactivation of catalyst due to fouling, poisoning and elevation in temperature must be considered. In the development of rate equation only a few of these five steps are considered which control the overall rate; other steps may be either neglected or combined. These steps do not necessarily proceed in series or in parallel, frequently making it impossible to combine them by simple means.

For different type of solid catalyzed reactions and for different mechanisms, rate equations are given in Table 4.8 of Ref. 2.

For example, if the chemical reaction is $A \rightleftharpoons R$

Assumed mechanism for this reaction is



If desorption of R controls the overall rate, then rate equation is

$$r_R = \frac{k \left(C_A - \frac{C_R}{K} \right)}{1 + k_A C_A} \quad (10.22)$$

where,

r_R = Rate of formation of product R (overall)

k = Overall reaction rate constant

k_A = Reaction rate constant for forward reaction

K = Overall equilibrium constant for the chemical reaction

C_A, C_R = Concentrations of A and R , respectively.

All fixed bed catalytic reactors are assumed to behave like ideal plug flow reactor. Equation used for the design or sizing of fixed bed reactor is

$$\frac{W}{F_{Ao}} = \int_0^{X_A} \frac{dX_A}{-r'_A} \quad (10.23)$$

where, W = Mass of catalyst, kg

F_{Ao} = Molar flow rate of limiting reactant in feed kmol/s

X_A = Fractional conversion of limiting reactant A

or $X_A = \frac{\text{Moles of } A \text{ reacted}}{\text{Moles of } A \text{ in feed}}$

$-r'_A$ = Overall rate of reaction in $\frac{\text{Moles of } A \text{ reacted}}{S \cdot \text{kg of catalyst}}$

S = Surface area of catalyst, m^2/kg

Most difficult part is the development of reliable expression of overall rate of reaction. It also requires detailed kinetic data.

10.8.2 Scale up Method

Any type of commercial scale reactor cannot be designed totally based on theoretical equations. It requires at least laboratory and/or pilot plant data of the reactions involved. A satisfactory scale up procedure requires a stepwise empirical approach in which the size of the reactor is increased successively. Basically the rate of chemical reaction is independent of the size and structure of a reactor. But the overall rate of reaction is influenced by rate of mass transfer and rate of heat transfer. Rate of mass transfer and heat transfer are usually controlled by the size and structure of the reactor. For example, due to significant wall effect smaller size reactor provides higher value of mass transfer coefficient compared to larger size reactor. Shell and tube type structure provides higher heat transfer coefficient, compared to jacketted tower type structure. Thus a chemical reaction is indirectly affected by reactor type and scale. Based on only lab scale data it is not possible to predict the reliable values of overall rate of reaction, reaction time and product composition for commercial scale reactor.

Example 10.7

Design the fixed bed catalytic reactor for 1000 t/a diethylbenzene plant based on the following data obtained on a pilot plant reactor.

Data of pilot plant reactor¹³

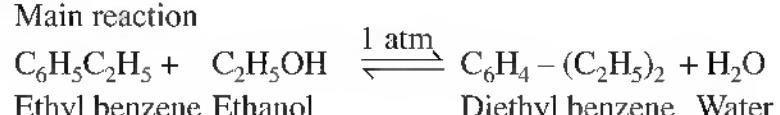
(i) Feed composition

Component	Mass %
Ethyl benzene	89
Ethanol	11

Feed flow rate in pilot plant reactor = 100 kg/h, Feed temperature = 375°C

(ii) Chemical reaction

(a) Main reaction



Reaction temperature = 375° C

(b) Side reactions are taking place in the reactor.

(iii) Product composition:

Component	Mass %
Diethyl benzene	16
Benzene	6
Ethyl benzene	74
Water	4

Product rate is approximately equal to the feed rate.

(iv) Catalyst: Zeolite with binders

Density of catalyst particle: 630 kg/m³

Catalyst porosity: 0.3

Form of catalyst: 1.5 mm extrudates of 4 to 6 mm length

(v) Type of reactor: Shell and tube heat exchanger type in which catalyst is placed inside the tube.

(vi) Reaction is endothermic. It is carried out in isothermal manner. Dowtherm A is to be circulated in liquid form on shell side to maintain the isothermal condition.

$$(vii) \frac{W}{F_{Ao}} = 17.3 \frac{\text{kg of catalyst}}{\text{kg of ethanol in feed/hour}}$$

(viii) Superficial mass velocity of feed gas

$$G = 0.55 \text{ kg}/(\text{m}^2 \cdot \text{s})$$

Feed composition, feed temperature, reaction temperature, operating pressure, catalyst, type of reactor etc. are same in pilot plant and commercial reactor.

Solution:

Capacity of plant = 1000 t/a of diethyl benzene (DEB)

Let number of working days per annum = 330 days

$$\text{Production rate of DEB} = \frac{1000 \times 1000}{330 \times 24} = 126.26 \text{ kg/h}$$

Let design production rate of DEB = 150 kg/h

Table 10.12 Product Composition for Commercial Scale Plant

Component	Mass %	kg/h
DEB	16	150.00
Benzene	6	56.25
Ethyl benzene	74	693.75
Water	4	37.50
	100	937.50

Feed rate ≡ Product rate

Table 10.13 Feed Composition for Commercial Scale Plant

Component	Mass %	kg/h
Ethyl benzene	89	834.375
Ethanol	11	103.125
	100	937.500

Ethanol is the limiting reactant. It does not appear in the product mixture which means conversion based on ethanol is 100%

Mass of catalyst required in commercial scale plant = $17.3 \times 103.125 = 1784 \text{ kg (min.)}$

$$\text{Volume of solid catalyst} = \frac{\text{Mass of catalyst}}{\text{Density of catalyst}}$$

$$= \frac{1784}{630} = 2.8317 \text{ m}^3$$

Porosity of catalyst bed = 0.3

Bulk volume occupied by the catalyst = $2.8317/0.7 = 4.045 \text{ m}^3$

Superficial mass velocity of feed gas

$$G = 0.55 \text{ kg/(m}^2 \cdot \text{s)}$$

Total cross sectional area of catalyst or of tubes

$$= \frac{(937.5/3600)}{0.55} = 0.4735 \text{ m}^2$$

Select 50 mm NB tubes of 14 BWG (3.76 mm) thickness

MOC of tube = SS 316

Tube OD = 50.8 mm

Tube ID = 43.28 mm

Total number of tubes required

$$n_t = \frac{0.4735}{(\pi/4)(0.04328)^2} = 322$$

Length of tubes required

$$L = \frac{\text{Net volume of catalyst}}{n_t \times (\pi/4) d_i^2} = \frac{4.045}{322 \times (\pi/4)(0.04328)^2}$$

$$L = 8.539 \text{ m}$$

Select the tubes of 10 m length. Above calculations have fixed the value of heat transfer area available for heat transfer. Temperatures and flow rate of Dowtherm A should be fixed to satisfy the heat transfer requirement.

$$\begin{aligned} A_{av} &= N_t \pi d_o L \\ &= 322 \times \pi \times 0.0508 \times 10 \\ &= 513.89 \text{ m}^2 \end{aligned}$$

$$\begin{aligned} \text{Heat duty, } \theta_t &= \Delta H_R \times \text{kmol/h of ethanol consumption} = 29 \times 1000 \times \frac{103.125}{46} \\ &= 65\,013.6 \text{ kJ/h} \equiv 18.06 \text{ kW} \end{aligned}$$

Calculations of fixed bed side film coefficient, h_i :

d_p = equivalent diameter of catalyst cylindrical particle, m

$$\frac{\pi d_p^3}{6} = \frac{\pi}{4}(1.5)^2 \times 5 \times 10^{-9}$$

$$d_p = 0.002\ 565\ \text{m}$$

$$\frac{d_p}{d_t} = \frac{0.002\ 565}{0.043\ 28} = 0.059 < 0.35$$

$$\frac{h_i d_t}{k} = 0.813 e^{(-6d_p/d_t)} (d_p G/\mu)^{0.9} \quad (10.24)$$

$$G = 0.55\ \text{kg}/(\text{m}^2 \cdot \text{s})$$

$$k = 0.04\ \text{W}/(\text{m} \cdot \text{K})$$

$$\mu = 0.015\ \text{mPa} \cdot \text{s} = 0.015 \times 10^{-3}\ \text{Pa} \cdot \text{s}$$

$$h_i = 0.813 \times \left(\frac{0.04}{0.043\ 28} \right) e^{(-6 \times 0.059)} \times \left(\frac{0.002\ 565 \times 0.55}{0.015 \times 10^{-3}} \right)^{0.9}$$

$$= 31.49\ \text{W}/(\text{m}^2 \cdot \text{°C})$$

$$\frac{h_i d_p}{k} = 3.6 \left(\frac{d_p G}{\mu} \right)^{0.365} \quad (10.25)$$

$$\frac{h_i \times 0.002\ 565}{0.04} = 3.6 \left(\frac{0.002\ 565 \times 0.55}{0.015 \times 10^{-3} \times 0.3} \right)^{0.365}$$

$$h_i = 457.51\ \text{W}/(\text{m}^2 \cdot \text{°C})$$

Let $h_i = 31.49\ \text{W}/(\text{m}^2 \cdot \text{°C})$ (Lesser of two values)

Shell side heat transfer coefficient, h_o :

Let tube pitch, $P_t = 1.25 d_o = 1.25 \times 0.0508 = 0.0635\ \text{m}$

Type of tube arrangement = Equilateral triangular

$$\text{Equivalent diameter, } d_e = \frac{1.1}{d_o} (P_t^2 - 0.907 d_o^2) \quad (6.32)$$

$$= \frac{1.1}{0.0508} (0.0635^2 - 0.907 \times 0.0508^2) = 0.036\ 63\ \text{m}$$

Shell side mass flow rate, \dot{m} :

$$\theta_t = \dot{m} C_L \Delta t$$

Table 10.14 Properties of Dowtherm A at 380°C¹⁴

Property	Value
C_L	2.5832 kJ/(kg · °C)
μ	0.135 mPa · s
k	0.098 W/(m · °C)

Let $\Delta t = 2^\circ\text{C}$ for Dowtherm-A

$$\dot{m} = \frac{18.06}{(2.5832 \times 2)} = 3.4957 \text{ kg/s}$$

Density of Dowtherm A at 380°C , $\rho_l = 0.709 \text{ kg/L}$

$$\text{Circulation rate, } q_v = \frac{3.4957}{0.709} \times \frac{3600}{1000} = 17.775 \text{ m}^3/\text{h}$$

Shell side flow area, A_s :

$$A_s = \frac{(P_t - d_o) D_s B_s}{P_t} \quad (6.29)$$

Shell inside diameter, D_b :

$$D_b = d_o \left(\frac{N_t}{K_1} \right)^{\frac{1}{n_1}} = 50.8 \left(\frac{322}{0.319} \right)^{\frac{1}{2.285}} \quad (6.1)$$

($k_1 = 0.319$ and $n_1 = 2.285$ from Table 6.2)

$$D_b = 1048.46 \text{ mm}$$

Let $D_S = 1065 \text{ mm}$

Baffle spacing, $B_S = 0.4 D_S = 426 \text{ mm}$

$$A_s = \frac{(0.0635 - 0.0508)}{0.0635} \times 1.04846 \times 0.426 = 0.0893 \text{ m}^2$$

$$G_S = \frac{\dot{m}}{A_s} = 39.1456 \text{ kg/(m}^2 \cdot \text{s)}$$

Reynolds number, Re :

$$Re = \frac{d_e G_S}{\mu} = \frac{0.03663 \times 39.1456}{0.135 \times 10^{-3}} = 10621.5$$

Prandtl number, Pr :

$$Pr = \frac{C_L \mu}{k} = \frac{2.5832 \times 0.135 \times 10^{-3} \times 10^3}{0.098} = 3.558$$

$$\frac{h_o d_e}{k} = J_h Re Pr^{0.33} \left(\frac{\mu}{\mu_w} \right)^{0.14} \quad (6.36)$$

From Fig. 6.14, $J_h = 0.0058$ for 25% baffle cut

$$\frac{h_o \times 0.03663}{0.098} = 0.0058 \times 10621.5 \times 3.558^{0.33}$$

$$h_o = 250.55 \text{ W/(m}^2 \cdot ^\circ\text{C)}$$

Overall heat transfer coefficient, U_o :

$$\frac{1}{U_o} = \frac{1}{h_o} + \frac{1}{h_{od}} + \frac{d_o \ln(d_o/d_i)}{2 k_w} + \frac{d_o l}{d_i h_{id}} + \frac{d_o l}{d_i h_i} \quad (6.42)$$

$$h_{od} = h_{id} = 5000 \text{ W}/(\text{m}^2 \cdot ^\circ\text{C})$$

$$\frac{1}{U_o} = \frac{1}{250.55} + \frac{1}{5000} + \frac{0.0508 \ln(50.8/43.28)}{2 \times 16} + \frac{50.8}{43.28}$$

$$\times \frac{1}{5000} + \frac{50.8}{43.28} \times \frac{1}{31.49}$$

$$U_o = 23.8356 \text{ W}/(\text{m}^2 \cdot ^\circ\text{C})$$

$$\theta_f = U_o \cdot A \Delta T_{in} = 18.06 \times 10^3 = 23.8356 \times 513.89 \times \Delta T_{in}$$

$$\Delta T_{in} = 1.474^\circ\text{C}$$

Very low temperature difference is required for the required duty of heat transfer. Hence, area available is adequate for heat transfer. Dowtherm—A will be circulated at pressure above its saturation pressure (8.21 bar a) corresponding to operating temperature of 380°C. This means shell will be under pressure.

Note: Empty volume of catalyst tubes works out to be 4.7272 m³ (based on 10 m tube length) which is 17.11% in excess over the bulk volume of the catalyst (4.045 m³). In reality, manufacturing tolerance of tubes ($\pm 12.5\%$) and support arrangement at the bottom of the tubes will call for sufficient excess volume so that required volume of the catalyst can be packed.

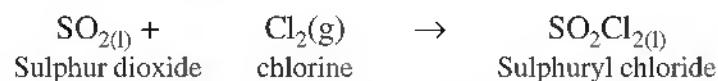
Catalysts are available in different forms; such as extrudates, Raschig rings, partition rings, monolith structure, etc. Bulk volume of catalyst in each shape will be different. Also pressure drop of the catalyst packed tubes is usually much higher than that of clean tubes. Manufacturers of catalysts provide data relating to pressure drop in the catalyst bed and suggest right size of the tube (i.e. diameter).

Over a period of operation some catalyst may settle or crumble and pressure drop may increase. All these aspects will have to be borne in mind while designing a reactor with the use of catalyst.

EXERCISES

- 10.1 Sulphuryl chloride (SO₂Cl₂) formation is carried out in a batch reactor at 0°C and 330 kPa a. Reaction time is 6 h.

Reaction:



At the reaction conditions, sulphur dioxide is in liquid state and it is a limiting reactant. % conversion of SO₂ at the end of reaction (at equilibrium) is 60% when 10% excess of chlorine is used. Reaction is carried out in glass lined reactor. Heat of reaction at 0°C is -394 kJ/mol (exothermic). For the production of 1000 kg of SO₂Cl₂ per batch,

- Decide the diameter and height of batch reactor.
- Select the suitable agitator for this gas–liquid reaction and calculate the rpm and power required by the agitator.
- Calculate the heat duty of overhead condenser. Overhead condenser will recycle almost all excess SO₂ back to the reactor.
- Calculate the heat duty of jacket.

- 10.2 In Example 10.5, a jet reactor is used for hydrogenation of edible oil. Instead of the jet reactor, a reactor with gas induction type stirrer⁶ and spiral jacket is to be designed as shown in Fig. 10.12. About 200 kg catalyst (1 to 2 μm particle size) is used with 10 t soybean oil. Use spiral baffled jacket (Fig. 10.9(c)) for heat transfer. Following equation⁵ can be used to calculate jacket side heat transfer coefficient. For $Re > 10,000$

$$\frac{h_j D_e}{k} = 0.027 Re^{0.8} Pr^{0.33} \left[\left(\frac{\mu}{\mu_w} \right)^{0.14} \left(1 + 3.5 \left(\frac{D_e}{D_j} \right) \right) \right] \quad (10.26)$$

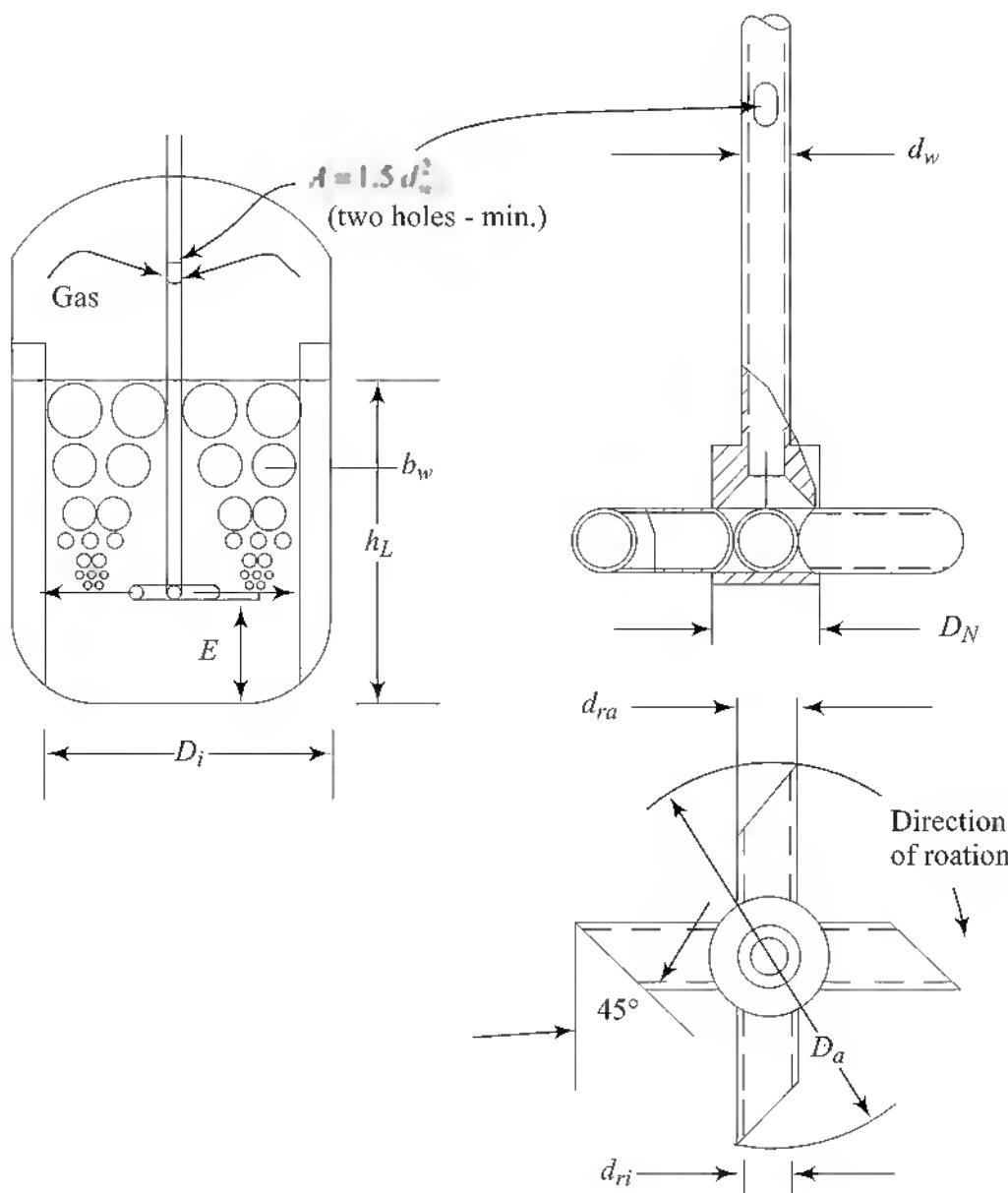


Fig. 10.12 Gas Induction Type Tube Stirrer for Hydrogenation

where, D_e = Equivalent diameter for cross section, m

$$D_e = 4 W$$

W = Width of jacket, m

$$Re = \frac{D_e v \rho}{\mu}$$

$$U = \frac{\dot{m}'\rho}{PW}$$

p = pitch of baffle spiral, m

\dot{m}' = Effective mass flow rate through spiral, kg/s

$\dot{m}' = 0.6 \dot{m}$

\dot{m} = Actual mass flow rate through spiral jacket, kg/s.

$$D_j = \frac{D_{jo} + D_{ji}}{2}, \text{ mean diameter of jacket, m}$$

Inside heat transfer coefficient can be safely calculated by following equation⁶.

$$\frac{h_i D_i}{k} = 0.36 Re^{2/3} Pr^{1/3} \left(\frac{\mu}{\mu_w} \right)^{0.14} \quad (10.25)$$

where, D_i = Inside diameter of shell of reactor, m

If heat transfer area provided by spiral jacket is not sufficient for the given heat duty, then use internal helical coil. Coil side heat transfer coefficient can be calculated by Eq. (10.24).

For coil, $D_e = d_{ci}$, where, d_{ci} = Inside diameter of coil. Also velocity of fluid through coil, $v_c = \frac{\dot{m}/\rho}{\frac{\pi}{4} d_{ci}^2}$.

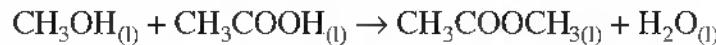
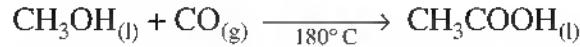
- 10.3 Acetic acid is manufactured via carbonylation of methanol. Reaction is carried out at 180°C and 50 atm in presence of liquid phase rhodium promoted methyl iodide as catalyst. Reaction is carried out in bubble column reactor. Acetic acid, itself is used as solvent.

Determine the followings:

- (a) Working volume of reactor.
- (b) Diameter of reactor.
- (c) Height of liquid inside the reactor.
- (d) Heat duty of overhead condenser in which exist gas-vapour mixture is cooled down to 40°C.
- (e) Design a spiral baffled jacket around bubble column reactor.

Data:

- (i) Reaction:



- (ii) Bubble column reactor can be approximated as ideal mixed flow reactor. Its uniform composition is 80% acetic acid, 10% methanol, 5% methyl iodide, 2.5% methyl acetate, and 2.5% water (by mass).
- (iii) 10% excess of carbon monoxide is used.
- (iv) Cooling water is available in the plant at 32°C.
- (v) Overall rate of reaction does not depend on $K_L a$, if $S_g > 2 \text{ cm/s}$.
- (vi) Rate of chemical reaction

$$-r_A = \frac{1}{V_l} \frac{dN_A}{dt} = k C_A, k = 89.26 \times 10^{-5} \text{ s}^{-1}$$

C_A = Concentration of methanol, kmol/m³

- (vii) Production rate of acetic acid = 50 tpd
 (ix) Heat of reaction at reaction temperature

$$\Delta H_R = -137 \text{ kJ/mole}$$

- (x) Conversion per pass = 70%

- 10.4 Design the fixed bed catalytic reactor for 1000 t/a ethyl chloride plant based on the following data obtained on a pilot plant reactor.¹⁵

Data of pilot plant reactor:

- (i) Feed flow rate = 0.025 53 kmol/h.

- (ii) Feed composition

Component	mole %
Methane	86.55
Ethylene	7.40
Hydrogen chloride	6.05

- (iii) Feed temperature = 171.1°C

- (iv) Reaction is taking place at 171.1°C and 2859.22 kPa g.



- (v) Product composition

Component	mole %
CH ₄	90.77
C ₂ H ₄	2.88
HCl	1.47
C ₂ H ₅ Cl	4.88

- (vi) Catalyst: Zirconium oxychloride supported on silica gel

Bulk density = 650 kg/m³

Porosity of catalyst bed = 0.32

$$(vii) \frac{W}{F} = 177.62 \frac{\text{kg catalyst}}{\text{kmol of feed/hour}}$$

- (viii) Superficial mass velocity of feed gas, $G = 58.93 \text{ kg}/(\text{m}^2 \cdot \text{h})$

- (ix) Rate of reaction

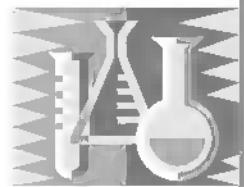
$$-r_A = 2.62 \times 10^{-4} \frac{\text{kg mol of ethylene converted}}{(\text{h} \cdot \text{kg catalyst})}$$

- (x) Reaction is endothermic. It can be carried out in a shell and tube heat exchanger type reactor. Saturated steam can be used on shell side to maintain the isothermal conditions. Feed composition, feed temperature, reaction temperature, operating pressure, catalyst, etc. are assumed same in pilot plant and commercial scale reactors.

REFERENCES

- Levenspiel, O., *Chemical Reaction Engineering*, 2nd Ed., John Wiley & Sons, Inc., USA, 1972.
- Perry, R. H. and D. Green, *Perry's Chemical Engineers' Handbook*, 6th Ed., McGraw-Hill Book Co., 1984.

3. Elvers, B., Hawking, S. and Schulz, G. (Editors), *Ullmann's Encyclopedia of Industrial Chemistry*, Vol. B4, 5th Ed. VCH Verlagsgesellschaft, GmbH, Germany, 1992
4. Smith, J.M., *Chemical Engineering Kinetics*, 3rd Ed., McGraw-Hill Book Co., USA, 1981.
5. Bondy, F. and S. Lippa, *Chem. Engg.*, **90** (7), 1983, p. 62.
6. Elvers, B., Hawking, S. and Schulz, G. (Ed.), *Ullmann's Encyclopedia of Industrial Chemistry*, Vol. B2, 5th Ed., Germany VCH Verlagsgesellschaft GmbH, Germany 1988, p. 25-15, p. 25-17 and 25-21 to 25-22.
7. Moore, S and S. Samdani, *Chem. Engg.*, **101** (3), March, 1994, p. 32.
8. Doraiswamy, L. K. and Sharma, M. M., *Heterogeneous Reactions; Analysis, Examples and Reactor Design*, Vol.: 1 & 2, John Wiley & Sons, USA, 1983.
9. Rase, H. F., *Chemical Reactor Design for Process Plants*, Vol. 1 & 2, John Wiley & Sons, Inc., USA, 1977.
10. Denbich, K.G. and Turner, T. C., *Chemical Reactor Theory—An Introduction*, 3rd Ed., Cambridge University Press, 1984.
11. Van den Hark S. M, Harrod, *Applied Catalysis A: General*, **210**, 2001, p . 207.
12. Fogler, H.S., *Elements of Chemical Reaction Engineering*, 3rd Ed., Prentice-Hall of India Pvt. Ltd., New Delhi, 2003.
13. Bokade, V. V. and R. Joshi, *Chemical Engineering World*, **37** (6) June, 2002, p. 59.
14. *Dowtherm A, Product Technical Data*, The Dow Chemical, Co., USA 1991.
15. Thodos, G. and Stutzman, L., *Ind. Engg. Chem.*, **50** (3), March, 1958, p. 413.

Appendix**1****Conversion Tables****Table AI. 1 Length Units**

	Metres (m)	Centimetres (cm)	Inches (in)	Feet (ft)	Yards (yd)
m	1	100	39.370 08	3.280 84	1.093 613
cm	0.01	1	0.393 701	$3.280\ 84 \times 10^{-2}$	$1.093\ 613 \times 10^{-2}$
inch	0.0254	2.54	1	$8.333\ 333 \times 10^{-2}$	$2.777\ 78 \times 10^{-2}$
ft	0.3048	30.48	12	1	0.333 333
yd	0.9144	91.44	36	3	1

Table AI. 2 Area Units

	Square Metres (m ²)	Square centimetres (cm ²)	Square inches (in ²)	Square feet (ft ²)	Square yards (yd ²)
m ²	1	10 ⁴	1550.003	10.763 91	1.195 99
cm ²	10 ⁻⁴	1	0.155	$1.076\ 391 \times 10^{-3}$	$1.195\ 99 \times 10^{-4}$
inch ²	6.4516×10^{-4}	6.4516	1	$6.944\ 444 \times 10^{-3}$	$7.716\ 049 \times 10^{-4}$
ft ²	0.092 903	929.0304	144	1	0.111 111
yd ²	0.836 127	8361.273	1296	9	1

Table AI. 3 Density and Concentration Units

	Kilograms per cubic meter (kg/m ³)	Grams per cubic centimetre (g/cm ³)	Pound per cubic foot (lb/ft ³)	Pound per UK gallon (lb/UK gal)	Pound per US gallon (lb/US gal)
kg/m ³	1	10 ⁻³	$6.242\ 795 \times 10^{-2}$	$1.002\ 2 \times 10^{-2}$	$8.345\ 402 \times 10^{-3}$
g/cm ³	1000	1	62.427 95	10.0224	8.345 402
Ib/ft ³	16.018 462	$1.601\ 846 \times 10^{-2}$	1	0.160 54	0.133 681
Ib/UKgal	99.776 5	9.978×10^{-2}	6.228 842	1	0.832 675
Ib/USgal	119.8264	0.119 826	7.480 519	1.200 95	1

* $1\text{ g/cm}^3 = 1\text{ t/m}^3 = 1.000\ 028\ \text{g/mL} = 1.000\ 028\ \text{kg/L}$

Table A1.4 Volume and Capacity Units

	Cubic Metres (m ³)	Cubic Centimetres (cm ³)	Cubic Inches (in ³)	Cubic Feet (ft ³)	UK (Imperial) gallons (UK gal)	US gallons (US gal)
m ³	1	10 ⁶	6.102 374 × 10 ⁴	35.314 67	219.9625	264.172
cm ³	10 ⁻⁶	1	6.102 374 × 10 ⁻²	3.531 467 × 10 ⁻⁵	2.199 69 × 10 ⁻⁴	2.641 722 × 10 ⁻⁴
inch ³	1.638 706 × 10 ⁻⁵	16.387 06	1	5.787 037 × 10 ⁻⁴	3.604 651 × 10 ⁻³	4.329 007 × 10 ⁻³
ft ³	2.831 685 × 10 ⁻²	28.316 85 × 10 ³	1728	1	6.2289	7.480 52
UK gal	4.546 095 × 10 ⁻³	4.546 095 × 10 ³	2.774 194 × 10 ²	0.160 54	1	1.200 95
UK gal	3.785 412 × 10 ⁻³	37.854 12 × 10 ²	231	0.133 681	0.832 674	1

Table A1.5 Mass Units

	Kilograms (kg)	Grams (g)	Tonnes (t)	Pounds (av.) (lb)	Tons (T)	Tons (short) (Ts)
kg	1	1000	10 ⁻³	2.204 623	9.8421 × 10 ⁻⁴	1.102 312 × 10 ⁻³
g	10 ⁻³	1	10 ⁻⁶	2.204 623 × 10 ⁻³	9.8421 × 10 ⁻⁷	1.102 312 × 10 ⁻⁶
t	1000	10 ⁶	1	2204.623	0.984 21	1.102 312
lb	453.592 37 × 10 ⁻³	453.592 37	4.535 924 × 10 ⁻⁴	1	4.4643 × 10 ⁻⁴	5 × 10 ⁻⁴
T	1016.047	1.016 047 × 10 ⁶	1.016 271	2240	1	1.12
Ts	907.185	9.071 847 × 10 ⁵	0.907 185	2000	0.892 86	1

Table AI. 6 Force Units

	Newton (N)	Kilograms force (kgf)	Dynes (dyn)	Pounds force (lbf)	Poundals (pdl)
N	1	0.101 972	10^5	0.224 809	7.223
kgf	9.806 65	1	$9.806 65 \times 10^5$	2.204 62	70.932
dyn	10^{-5}	$1.019 72 \times 10^{-6}$	1	2.2×10^{-6}	7.233×10^{-5}
Ibf	4.448 22	0.453 592	$4.448 22 \times 10^5$	1	32.174
pdl	0.138 256	1.4098×10^{-2}	$1.382 25 \times 10^4$	3.1081×10^{-2}	1

Table AI. 7.1 Pressure Units

	Newton per square metre N/m ² (Pa)	Bars (bar)	Standard atmospheres (atm)	Kilograms-force per square centimetre (kgf/cm ² or at)	Dynes per square centimetre (dyn/cm ²)
N/m ² (Pa)	1	10^{-5}	$9.869 223 \times 10^{-6}$	$1.019 716 \times 10^{-5}$	10
bar	10^5	1	0.986 923	1.019 716	10^6
atm	101.325×10^3	1.013 25	1	1.033 227	101.325×10^4
kgf/cm ²	908.665×10^2	0.980 665	0.967 841	1	$9.806 65 \times 10^5$
dyn/cm ²	0.1	10^{-6}	$9.869 233 \times 10^{-7}$	$1.019 716 \times 10^{-6}$	1
torr	133.3224	$1.333 224 \times 10^{-3}$	$1.315 79 \times 10^{-3}$	$1.359 51 \times 10^{-3}$	1333.224
in Hg	3386.388	$3.386 395 \times 10^{-2}$	$3.342 105 \times 10^{-2}$	$3.453 155 \times 10^{-2}$	$33.863 886 \times 10^3$
m H ₂ O	9806.65	9.8067×10^{-2}	9.6784×10^{-2}	0.1	$9.806 65 \times 10^4$
ft H ₂ O	2989.063	2.989×10^{-2}	$2.949 89 \times 10^{-2}$	3.048×10^{-2}	$2.989 067 \times 10^4$
Ibf/in ²	6894.757	$6.894 757 \times 10^{-2}$	$6.804 596 \times 10^{-2}$	$7.030 696 \times 10^{-2}$	$6.894 757 \times 10^4$

Table AI. 7.2 Pressure Units (Contd.)

	Torr or Barometric millimetres of mercury (torr or mmHg)	Barometric inches of mercury (in Hg)	Head of water		Pounds-force per square inch (Ibf/in ²)
			m H ₂ O	ft H ₂ O	
N/m^2					
(Pa)	$7.500 616 \times 10^{-3}$	$2.952 999 \times 10^{-4}$	$1.019 72 \times 10^{-4}$	$3.345 53 \times 10^{-4}$	$1.450 377 \times 10^{-4}$
bar	750.061 6	29.529 99	10.1972	33.4554	14.503 77
atm	760	29.921 26	10.332 27	33.8985	14.695 95
kgf/cm ²	735.5592	28.959 03	10	32.8084	14.223 34
dyn/cm ²	$7.500 617 \times 10^{-4}$	$2.952 999 \times 10^{-5}$	$1.019 72 \times 10^{-5}$	$3.345 53 \times 10^{-5}$	$1.450 377 \times 10^{-5}$
torr	1	$3.937 008 \times 10^{-2}$	$1.359 15 \times 10^{-2}$	$4.460 351 \times 10^{-2}$	$1.933 678 \times 10^{-2}$
in Hg	25.4	1	0.345 316	1.132 92	0.491 154
m H ₂ O	73.5556	2.8959	1	3.280 84	1.422 334
ft H ₂ O	22.4198	0.882 67	0.3048	1	0.433 526
Ibf/in ²	51.714 93	2.036 021	0.703 072	2.306 67	1

Table AI. 8.1 Energy and Heat Units

	Joules (J)	Kilowatt hours (kWh)	Kilocalories (kcal _{IT})	Kilogram-force metres (kgf · m)
J	1	$2.777\ 778 \times 10^{-7}$	$2.388\ 459 \times 10^{-4}$	0.101 972
kWh	3.6×10^6	1	859.845 2	$3.670\ 98 \times 10^5$
kcal _{IT}	4186.8	1.163×10^{-3}	1	426.935
kgf · m	9.806 65	$2.724\ 07 \times 10^{-6}$	2.3423×10^{-3}	1
L · atm	101.325	$2.814\ 583 \times 10^{-5}$	$2.420\ 107 \times 10^{-2}$	10.332 31
Btu _{IT}	1055.056	$2.930\ 711 \times 10^{-4}$	0.251 996	107.586
Ibf · ft	1.355 818	$3.766\ 161 \times 10^{-7}$	$3.238\ 315 \times 10^{-4}$	0.138 255

Table AI. 8.2 Energy and Heat Units (Contd.)

	Litre atmospheres (L · atm)	British thermal units (Btu _{IT})	Pound-force feet (lbf · ft)
J	$9.869\ 233 \times 10^{-3}$	$9.478\ 172 \times 10^{-4}$	0.737 562
kWh	$3.552\ 924 \times 10^4$	$3.412\ 142 \times 10^3$	$2.655\ 224 \times 10^6$
kcal _{IT}	41.3205	3.968 321	3088.96
kgf · m	$9.678\ 38 \times 10^{-2}$	$9.294\ 88 \times 10^{-3}$	7.233
L · atm	1	$9.603\ 759 \times 10^{-2}$	74.733 51
Btu _{IT}	10.412 59	1	778.1693
Ibf · ft	$1.338\ 088 \times 10^{-2}$	$1.285\ 067 \times 10^{-3}$	1

Table AI. 9 Specific Energy Units

	Joules per kilogram (J/kg)	Kilocalories per kilogram (kcal _{IT} /kg)	British thermal unit per pound (Btu _{IT} /lb)
J/kg	1	$2.388\ 459 \times 10^{-4}$	$4.299\ 226 \times 10^{-4}$
kcal _{IT} /kg	4186.8	1	1.8
Btu _{IT} /lb	2326	0.555 556	1

Table AI. 10 Power Units

	Kilowatts (kW)	Kilogram force metre per second (kgf · m/s)	Metric horse- powers (mhp)	Pound- force feet per second (lbf · ft/s)	Horsepowers (hp)
kW	1	101.971 44	1.359 62	737.562 2	1.341 022
kgf · m/s	$9.806\ 6 \times 10^{-3}$	1	1.3333×10^{-2}	7.233	1.3151×10^{-2}
mhp	0.7355	75	1	542.4764	0.986 32
Ibf · ft/s	$1.355\ 82 \times 10^{-3}$	0.138 25	1.8434×10^{-3}	1	1.8182×10^{-3}
hp	0.7457	75.0395	1.013 87	550	1

Table A I. 11 Temperature Units

	Degrees Kelvin (K)	Degrees Celsius (°C)	Degrees Fahrenheit (°F)	Degrees Rankine (°R)
K	T	$t + 273.15$	$5/9 (t' + 459.67)$	$5/9 T'$
°C	$T - 273.15$	t	$5/9 (t' - 32)$	$5/9 (T' - 459.67)$
°F	$9/5 T - 459.67$	$9/5 t + 32$	t'	$T' - 459.67$
°R	$9/5 T$	$9/5 t + 491.67$	$t' + 459.67$	T'

Table A I.12 Absolute Viscosity Units

	kg/(m · s) or N · s/m ²	Poise (P) or g/(cm · s) or dyn · s/cm ²	lb/(ft · s)
kg/(m · s)	1	10	0.671 97
Poise	0.1	1	0.067 197
Ib/(ft · s)	1.488 162	14.881 62	1

1 Poise = 100 centipoises (cP) = 100 mPa · s

Table A I.13 Kinematic Viscosity Units

	m ² /s	Stoke (St)	ft ² /s
m ² /s	1	10 ⁴	10.763 91
Stoke	10 ⁻⁴	1	1.076 391 × 10 ⁻³
ft ² /s	0.092 903	929.03	1

1 St = 100 cSt

Table A I.14 Thermal Conductivity Units

	W/(m · K)	kcal _{IT} /(h · m · °C)	Btu _{IT} /(h · ft · °F)
W/(m · K)	1	0.859 854	0.577 789
kcal _{IT} /(h · m · °C)	1.163	1	0.671 969
Btu _{IT} /(h · ft · °F)	1.730 735	1.488 164	1

Table A I.15 Heat Transfer Coefficient Units

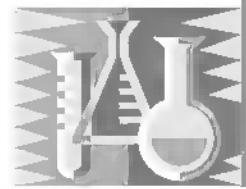
	W/(m ² · °C)	kcal _{IT} /(h · m ² · °C)	Btu _{IT} /(h · ft ² · °F)
W/(m ² · C)	1	0.859 854	0.176 11
kcal _{IT} /(h · m ² · °C)	1.163	1	0.204 816
Btu _{IT} /(h · ft ² · °F)	5.678 264	4.882 432	1

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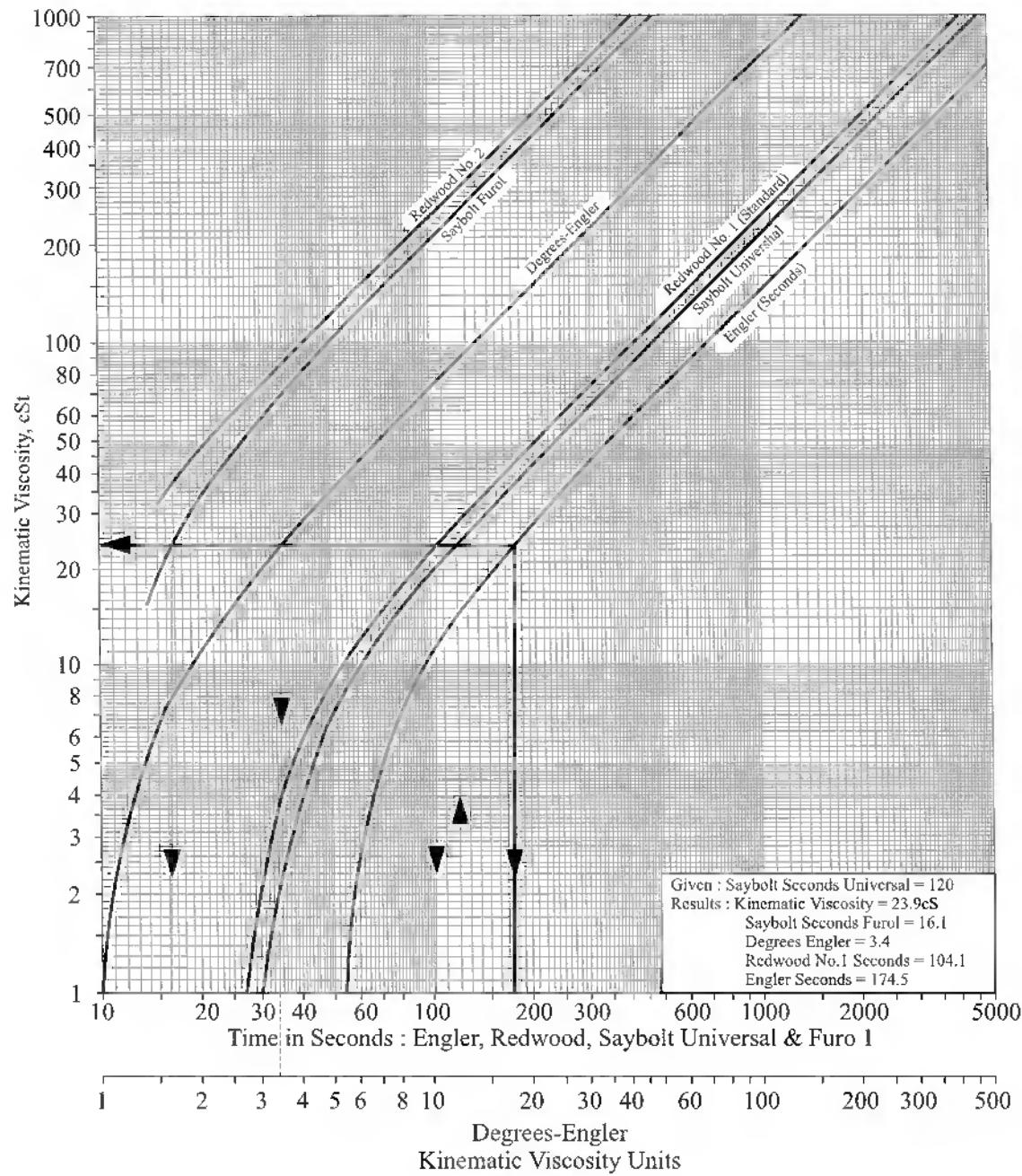
- (i) *Thermodynamics Research Centre (TRC)*, USA from its Publication: *Reprint of the Introduction to the TRC Thermodynamic Tables: Non-Hydrocarbons*, December 31, 1991 and
- (ii) *International Union of Pure and Applied Chemistry (IUPAC)*, UK from its Publication: *Quantities, Units and Symbols in Physical Chemistry*, Edited by I. Mills, T. Cvitas, K. Homann, N. Kallay and K. Kuchitsu, 2nd Ed., Blackwell Science Ltd., UK, 1993.

Appendix

2



Viscometer Conversion Chart





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