

Rajiv Gandhi University of Knowledge Technologies



Chemical Engineering Department  
Mass Transfer Operations Lab  
(CH3701)

### **Course Objectives:**

- This lab gives an overall idea of various mass transfer operations used in the industry.

### **List of Experiments**

S.No	Name of the experiment
1.	Cooling tower
2.	Gaseous diffusion coefficient apparatus
3.	Ion exchange
4.	Liquid-liquid extraction in packed bed column
5.	Packed bed distillation column
6.	Rotary dryer
7.	Simple batch distillation
8.	Simple steam distillation
9.	Solid –liquid extractor

### **Course Outcomes:**

- To demonstrate an understanding of heat and mass transfer modes and models.
- To formulate the idea of the different types of interface reactions.
- To apply principles of heat and mass transfer phenomena to chemical process industries.
- To enable solving the problems on process and materials related combined heat and mass transfer phenomena.

## **CL 332/334-UG LAB**

### **Cooling Tower- MT 305**

#### **Objective:**

1. To study the operation of cooling tower
2. To determine average heat and mass transfer coefficients and to estimate the tower height
3. To examine the effect of various parameters such as feed flow rate, air flow rate, inlet water temperature etc. on the performance of the cooling tower.

#### **Theory:**

When a component is transferred from a gas phase to solution in a liquid phase, the latent heat associated with the condensation is evolved. In the operations where mass transfer proceeds by equimolar counter diffusion, as in distillation or in operations where latent heat effect are small, as in liquid –liquid extraction, gas absorption in dilute solutions, and leaching, heat transfer is of minor importance as a rate controlling mechanism. In others, particularly where there is a net transfer of mass from gas phase to condensed phase or vice versa, the heat transfer rate is important. In these cases, heat transfer significantly limits the rate at which mass can be transferred.

Of those operations where both heat transfer and mass transfer affect the rate, humidification and dehumidification involve two components and two phases. The liquid phase, most often water, is single component, and the gas phase consists of a non-condensable gas, usually air, in which some vapor from the liquid phase is present.

#### **HUMIDIFICATION: INDUSTRIAL APPLICATIONS**

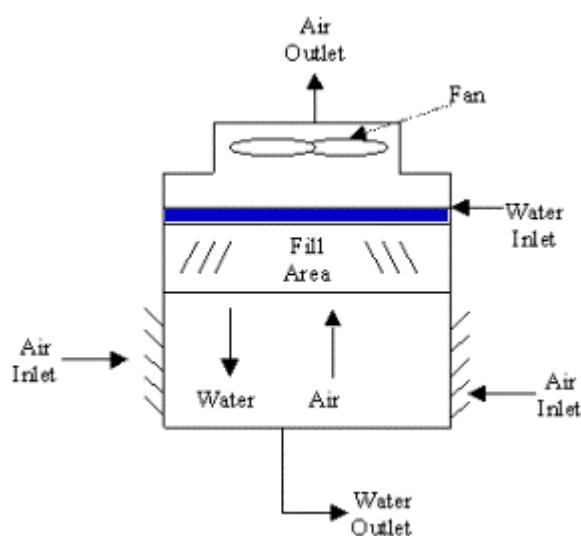
Humidification process may be carried out to control the humidity of a space or, more usually, to cool and recover water by contacting it with low humidity air. The water that has lost heat to the atmosphere can then be reused in heat exchanger throughout a plant. Alternatively, the water could be cooled in the surface heat exchanger. The choice is one of economics, with the designer balancing the loss of cooling water inherent in the air- water contact cooler against the cost of supplying and handling the cooling source to the surface cooler and the higher cost of surface units.

## HUMIDIFICATION EQUIPMENT

The most obvious form of humidification equipment is the spray chamber. Here, the contacting liquid is sprayed as a mist into the gas stream. Gas velocity is kept low so that the contact time is high so that there will be only small amount of liquid physically entrained in the gas stream. These units are usually restricted to the small-scale operations and are frequently used in humidity control of a room or plant where either humidification or dehumidification of the inlet air is required.

### Types of cooling towers

Cooling towers are generally used for humidification operations. Cooling towers fall into two main sub-divisions: natural draft and mechanical draft. Natural draft designs use very large concrete chimneys to introduce air through the media. Due to the tremendous size of these towers (500 ft high and 400 ft in diameter at the base) they are generally used for water flow rates above 200,000 gal/min. Usually these types of towers are only used by utility power stations. Mechanical draft cooling towers are much more widely used. These towers utilize large fans to force air through circulated water. The water falls downward over fill surfaces that help increase the contact time between the water and the air. This helps maximize heat transfer between the two. The experimental setup for UG lab uses mechanical draft cooling tower, which is shown in Fig.1.



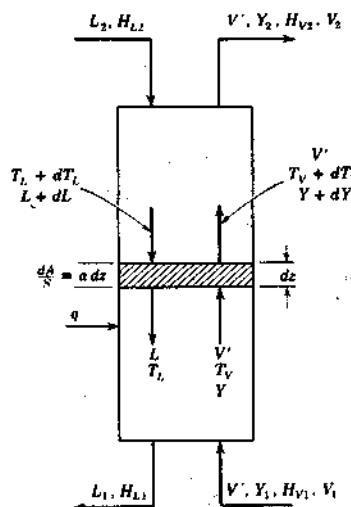
**Figure 1:** Mechanical Draft Counterflow Tower

## CALCULATIONS FOR HUMIDIFICATION AND DEHUMIDIFICATION OPERATIONS

The design of equipment to carry out humidification and dehumidification operations depends upon the concepts of the direction of the transfer processes and their rates (determined by writing enthalpy) and material balance equations and rate equations by combining these equations into the applicable design equations. In this section, these concepts will be applied to the special conditions surrounding the operation of humidification and dehumidification equipment.

In a dehumidification process, a warm vapor gas mixture is contacted with a cool liquid. Vapor condenses from the gas phase, the gas phase cools, and the liquid is warmed. Both sensible and latent heat are transferred toward the liquid phase. In the normal convention, the gas phase is chosen as the system, and heat and mass-transfer rates and quantities are positive when transfer is toward the gas phase. This sign convention is equivalent to measuring distance in the direction toward the gas phase. Then, heat- and mass-transfer quantities for this physical situation will be negative.

In a water-cooling process, warm water is contacted by a cooler gas-vapor mixture. Mass and heat are transferred toward the gas phase. As a result, the signs of the driving-force terms and transfer rates are positive based upon the convention described above.



**Figure 2:** Representation of the general humidification process

The physical arrangement is shown in Fig.2. The subscripts 1 and 2 refer to the bottom and top of the column.

On the basis, for a tower of constant cross section, an overall material balance gives

$$L_1 - L_2 = V_1 - V_2 \quad \dots(1)$$

a material balance for the condensable component gives

$$V^1 (Y_2 - Y_1) = L_2 - L_1 \quad \dots(2)$$

and an enthalpy balance gives

$$L_2 H_{L2} + V^1 H_{V1} + Q = L_1 H_{L1} + V^1 H_{V2} \quad \dots(3)$$

Most commonly, the column will operate nearly adiabatically with  $q \rightarrow 0$ . The approach to adiabatic operation will be closer the larger the column diameter. For this situation, similar balances for the differential height ( $dz$ ) will be written. The condensable component balance becomes

$$V^1 dY = dL \quad \dots(4)$$

The corresponding enthalpy balance is

$$V^1 dH_V = d(Lh_L) \quad \dots(5)$$

If the rate of solute transfer between phases is small compared to the total flow stream, an average value of  $L$  may be used, and the change in enthalpy of the liquid phase may be expressed as if it resulted solely from the change in temperature at constant specific heat. Thus,

$$d(LH_L) = L_{av} c_L dT_L \quad \dots(6)$$

where

$$L_{av} = (L_1 + L_2)/2 \quad \dots(7)$$

For the change in gas-phase enthalpy, the expression in terms of temperature is rigorous if  $c_h$  is constant.

$$V^1 dH_V = V^1 d[ c_h (T_V - T_0) + Y\lambda_0 ] = V^1 c_h dT_V + V^1 \lambda_0 dY \quad \dots(8)$$

Rate equations for heat and mass transfer can also be written. Here, however, complications arise because of the fact that heat is transferred from the bulk of the liquid phase to the liquid-gas interface entirely as a result of the temperature potential, but from the interface to the bulk of the gas phase, heat is transferred as the result by two mechanisms. On the gas-phase side of the interface, heat is transferred as the result of a temperature potential, and the latent heat

associated with the mass transfer is transferred by these two mechanisms are separated as the two terms on the right side of the equation (8).

With these ideas, equations for the heat-transfer processes indicated in equations (6) and (8) can be written separately. For the liquid phase transfer

$$\frac{L_{av}}{S} c_L dT_L = h L a (T_L - T_i) dz \quad \dots(9)$$

where  $T_i$  = the interfacial temperature,  $^{\circ}\text{F}$

For the gas-phase sensible-heat transfer,

$$V^1 c_h dT_V = h_c a (T_i - T_V) dz \quad \dots(10)$$

And, for the gas-phase latent heat transfer,

$$\frac{V^1}{S} \lambda_0 dY = \lambda_0 k_y a (Y_i - Y) dz \quad \dots(11)$$

where

$Y_i$  = the gas-phase mole ratio of solute to solvent at the interface

### Development of the Design Equation

The enthalpy-balance equations given above can be combined to give a design equation. These equations will relate the change in gas-phase temperature and molal humidity to the rates of heat and of mass transfer to, or from, the gas-phase. Thus, combining equation (8) with equations (10) and (11).

$$\frac{V^1}{S} dH_V = h_c a (T_i - T_V) dz + \lambda_0 k_y a (Y_i - Y) dz \quad \dots(12)$$

for the gas phase. Separating  $k_y a$  from the right side of the equation and designating  $h_c/k_y c_h$  as  $r$ , the psychrometric ratio, give

$$\frac{V^1}{S} dH_V = k_y a [(c_h r T_i + \lambda_0 Y_i) - (c_h r T_V + \lambda_0 Y)] \quad \dots(13)$$

In putting  $r$  into this equation for  $h_c a / k_y c_h$ , the assumption is made that  $a$ , the area per unit volume of tower, is the same for heat transfer as it is for mass transfer. This will be true only at such high liquid rates that the tower packing is completely wet. If  $r$  is equal to 1, as it is for the air-water system under normal conditions, the terms within parentheses in equation (13) are enthalpies as defined by equation (13a).

$$H = C_b(T - T_0) + Y[\lambda_0 + C_a(T - T_0)] \quad \dots(13a)$$

$$\frac{V^1}{S} dH_V = k_y a (H_i - H_V) dz \quad \dots(14)$$

$$\int_{H_{v1}}^{H_{v2}} \frac{V^1 dH_v}{Sk_y A (\mathbf{H}_i - \mathbf{H}_v)} = \int_0^z dz = z \quad ... (15)$$

Equation (15) is a design equation; the driving force is expressed as an enthalpy difference. Enthalpy is an extensive thermodynamic property. As such, it cannot be a driving force for any transfer operation. Thus, the mathematical treatment that leads to equation (15) should be examined. First, in the energy balances, the total flow per unit time is fixed. The enthalpy terms are then “specific enthalpy”, based upon a fixed mass of material. Second, in the basic design equation, Equation (13), the driving force is a function of  $T$  and  $Y$ , the quantities that would be expected to control rates of heat and of mass transfer. Only in the fortuitous case that  $r=1$  can  $H$  be substituted for these  $T$  and  $Y$  functions. In all other cases, Equation (15) would be written as

$$\int \frac{V^1 d(c_h r T_v + \lambda_0 Y)}{Sk_y a [(c_h r T_i + \lambda_0 Y_i) - (c_h r T_v + \lambda_0 Y)]} = \int dz \quad ... (16)$$

### Integration of the Design Equation

Integration indicated by equation (15) is usually performed by values of  $V^1$  and  $k_y a$  averaged over the column height. This introduces small error in light of the low concentration of water vapor in the gas stream. Beyond this, knowledge of the relation between the enthalpy in the main gas phase and that at the gas-liquid interface is necessary. Such a relation can be obtained by now considering the transfer process on the liquid side of the interface. Combining the enthalpy balance (Equation (5)) with the liquid transfer rate (Equation (9)) gives

$$\frac{V^1}{S} dH_V = h_L a (T_L - T_i) dz \quad ... (17)$$

and combining this equation with Equation (14) gives

$$-\frac{h_L a}{k_y a} = \frac{H_v - H_i}{T_L - T_i} \quad ... (18)$$

Equation (18) applies at any point in an air-water contacting device. From it, the temperature and the enthalpy of the interface can be determined at any point for which the liquid temperature ( $T_L$ ), the gas enthalpy ( $H_V$ ), and the ratio of the liquid-phase heat transfer coefficient to the gas-

phase heat transfer coefficient to the gas-phase mass transfer coefficient based upon mole-ratio driving forces are known.

The interface conditions can be obtained through equation (18) using a graphical method.

A plot is drawn with coordinates of liquid-phase temperature versus enthalpy of the gas phase. On it, the locus of interface  $H_i$  and  $T_i$  values can be plotted by realizing that at the interface the vapor phase will be saturated at the interface temperature if the assumption that equilibrium exists at a phase boundary is tenable. From the saturation curve on the air-water humidity chart, the saturation molal humidity can be obtained for any desired temperature. The saturation, or interface, enthalpy can then be calculated or read from the humidity chart.

On the same plot, an “operating line” of  $H_V$  versus  $T_L$  can be plotted by combining equations (5) and (6) and integrating. This curve represents the path of bulk-phase conditions as the fluids pass through the unit. Thus,

$$\int_{H_{V1}}^{H_{V2}} V^1 dH_V = \int_{T_{L1}}^{T_{L2}} L_{av} c_L dT_L \quad \dots(19)$$

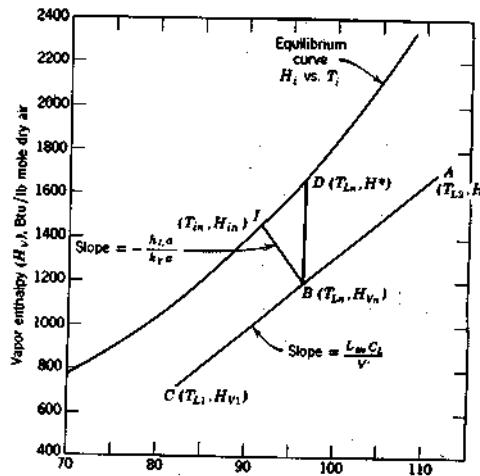
where the limits again refer to the bottom and top of the column. Integrating,

$$V^1 (H_{V2} - H_{V1}) = L_{av} C_L (T_{L2} - T_{L1}) \quad \dots(20)$$

And rearranging,

$$\frac{H_{V1} - H_{V2}}{T_{L2} - T_{L1}} = \frac{L_{av} c_L}{V^1} \quad \dots(21)$$

This equation gives the slope of the  $H_V$  versus  $T_L$  “operating” line as  $L_{av}C_L/V^1$ . For the air-water system and for most other dilute gas-solution systems, this ratio is constant over a moderate humidity range, and the straight  $H_V$  versus  $T_L$  line can be determined from knowledge of the liquid- and gas-phase flow rates and the conditions of both streams at one end of the column, or alternatively the line can be located from the conditions at both ends of the column.



**Figure 3:** Graphical representation of adiabatic gas-liquid contacting operation

Fig.3 shows graphical representation of adiabatic gas-liquid contacting operation (humidification operation). The equilibrium curve represented on it was obtained from data on the air-water psychometric chart. On this curve are located all possible conditions of  $T_i$ ,  $H_i$  throughout the column. Line ABC is the operating line containing all values of  $H_V$  corresponding to liquid temperature ( $T_L$ ) throughout the column. This line could have been obtained from knowledge of the two end conditions,  $(T_{L1}, H_{V1})$  and  $(T_{L2}, H_{V2})$ , or from either one of these two plus the slope  $(L_{av}C_L/V^l)$ . On this line, point B represents an arbitrary point in the column at which the liquid temperature and gas enthalpy have the values of  $T_{Ln}$  and  $H_{Vn}$ . The interface conditions at this point can be found by using Equation (18). Thus, a tie line starting at point B and having a slope equal to  $-h_{La} / k_{ya}$  will intersect the equilibrium curve at the interface conditions corresponding to point B. Point I represents the interphase conditions designated by  $(T_{in}, H_{in})$ . In this way, interphase conditions can be found corresponding to any point between A and C on the operating line. Of course, values of  $k_{ya}$  and  $h_{La}$  applicable to the column conditions are necessary. Usually, these values must be obtained experimentally.

From this construction, Equation (15) can now be integrated graphically. The necessary driving force in terms of enthalpy is determined for representative points throughout the column. For point B on Fig.3, this would be  $(H_{in} - H_{Vn})$ . Values of  $V^l/SK_{ya}(H_i - H_V)$  are then plotted against  $H_V$  and the area under this curve determined between the limits of  $H_{V1}$  and  $H_{V2}$  in order to find the column height (z).

Psychrometric chart is shown in appendix at the end of this report. From the wet bulb and dry bulb temperature of the air, other air properties like absolute humidity, relative humidity, enthalpy of saturated and unsaturated air can be found out from the psychrometric chart and are used for estimating the tower height.

**Procedure:**

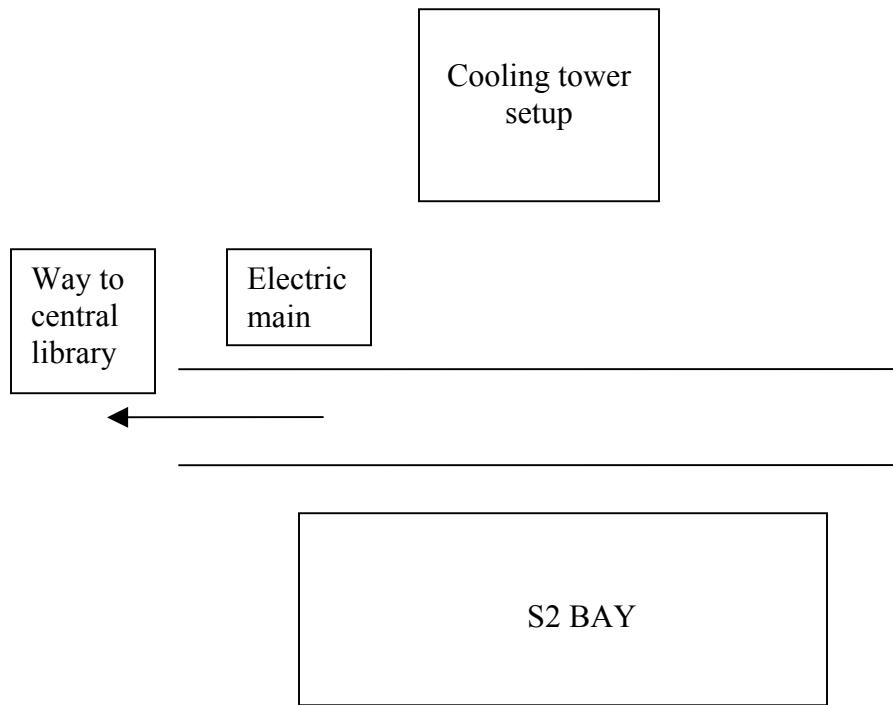
1. Start the fan.
2. Start water pump and adjust the flow rate to a desired value.
3. Start the water heater and set the inlet temperature to a desired value.
4. Measure wet bulb temperature and dry bulb temperature of outlet water.
5. Wait till the steady state is obtained. The attainment of the steady state is confirmed by the constancy in the outlet temperature.
6. Measure air velocity by anemometer.
7. Measure inlet temperature and outlet temperature of air.
8. Repeat steps 2 to 7 by changing water flow rate or inlet temperature of water.

**Calculations:**

1. Verify the overall material and energy balance
2. Calculate heat and mass transfer coefficients by a graphical method described above.

## Safety precautions:

1. Ensure the valve V<sub>1</sub>(cooling tower inlet water valve) in partly open position and remaining valves in fully open position.
2. Restrict the flow rate of inlet water to cooling tower to 100 LPM.
3. Don't touch any electrical connections.
4. Don't put hands in any rotating equipment like fan, pump shaft etc.
5. If any equipment malfunctions, turn off electric mains immediately.



**Figure 4. Location of cooling tower**

## Contact phone numbers:

S2 Bay: 4213

Main Security : 1122

Prof. Chandra Venkataraman: 7224

## **Nomenclature:**

$L_2$  = liquid flow rate into the top of the column, lb mole/hr (or mol/s)

$V_1$  = flow rate of gas phase entering the column, lb mole/hr (or mol/s)

$V^1$  = flow rate of solvent, or "dry" gas, lb mole/hr (or mol/s)

$Y_2$  = mole ratio of solute to solvent gas at the top of the column

$H_{V1}$  = enthalpy of gas phase entering the column, Btu/lb mole of "dry" gas  
(or joule/mol)

$H_{L2}$  = enthalpy of liquid phase entering the top of the column, Btu/lb mole of liquid  
(or joule/mol)

$q$  = heat transferred to the column from its surroundings, Btu/hr (or joule/s)

$T_L, T_v$  = temperature of liquid and gas phases, respectively

$dz$  = a differential height of column packing, ft (or m)

$A$  = interfacial surface,  $\text{ft}^2$  (or  $\text{m}^2$ )

$a$  = interface area,  $\text{ft}^2/\text{ft}^3$  of column volume (or  $\text{m}^2/\text{m}^3$  of column volume)

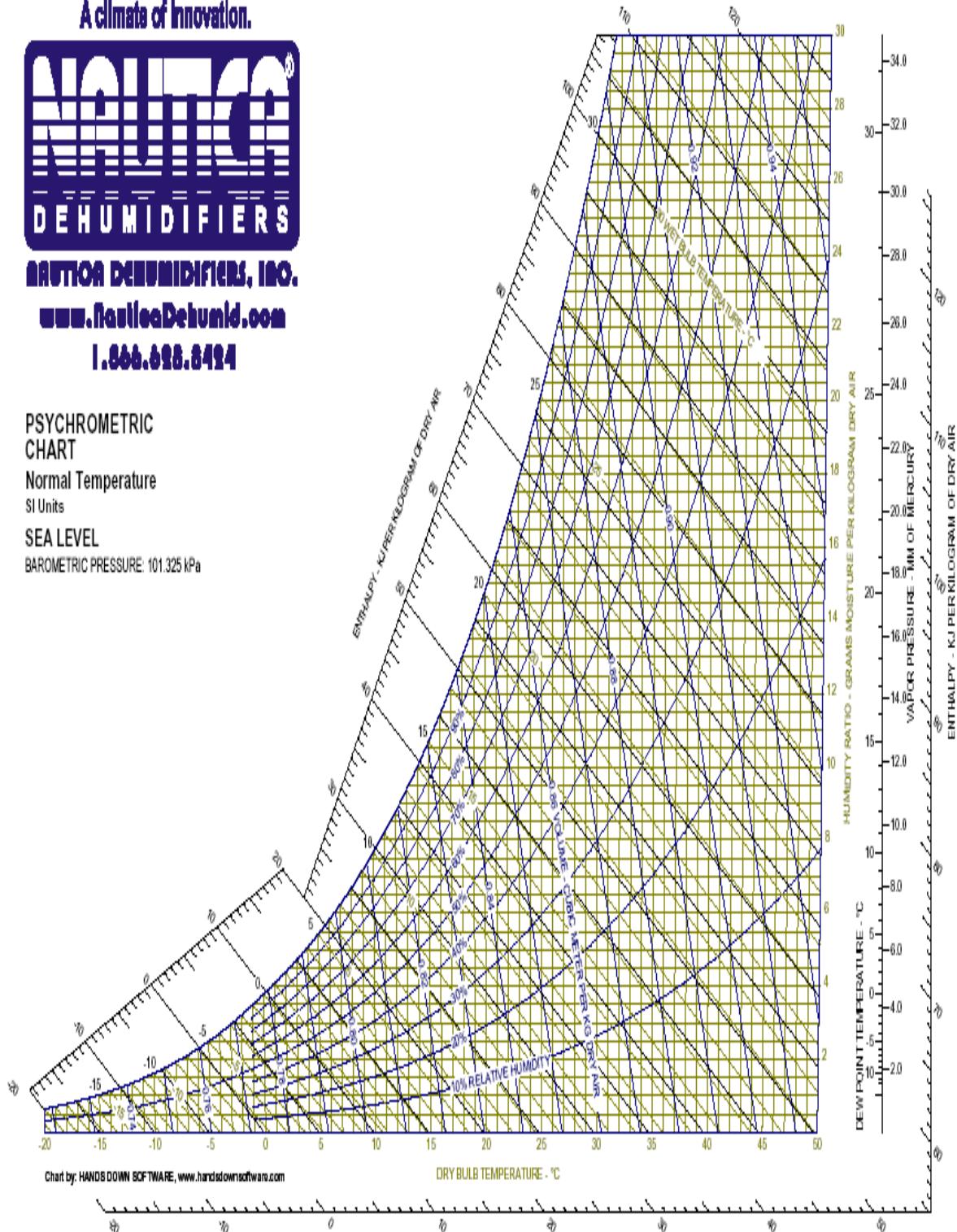
$S$  = tower cross section,  $\text{ft}^2$  (or  $\text{m}^2$ )

## Appendix

### Psychrometric chart



PSYCHROMETRIC  
CHART  
Normal Temperature  
SI Units  
SEA LEVEL  
BAROMETRIC PRESSURE: 101.325 kPa





**ROTARY DRYER**  
**(With Data Logging Facility)**  
**(MT-943 RH)**

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# ROTARY DRYER

## 1. OBJECTIVE:

To study the operation of rotary dryer.

## 2. AIM:

- 2.1 To study the drying characteristics of a solid material in a rotary dryer.
- 2.2 To plot the rate of drying curve.

## 3. INTRODUCTION:

In many cases, the drying of materials is the final operation in the manufacturing process, carried out immediately prior to packaging or dispatch. Drying refers to final removal of water, & the operation often follows evaporation, filtration or crystallization. Drying is carried out for one or more of the following reasons:

- To reduce the transportation cost.
- To make a material more suitable for handling, for example soap powders, dyestuffs, fertilizers.
- To provide definite properties, such as maintaining free flowing nature of salt.
- To remove moisture; which may otherwise lead to corrosion, for example, the drying of coal gas or benzene prior to chlorination.

Almost all drying processes involve the removal of water by vaporization, & thus require the addition of heat.

The rotary dryer is a type of industrial dryer employed to reduce or minimize the liquid moisture content of the material by bringing it in direct contact with a heated gas .The dryer is made up of large rotating cylinder which slopes slightly so that the discharge end is lower than the material feed end in order to convey the material under gravity. Material to be dried enters the dryer and as the dryer rotates, the material is lifted up by a series of internal fins lining the inner wall of the dryer. Heating options include steam, gas, oil, thermal oil, and auxiliary biomass burner system.

#### 4. THEORY:

Consider a rotary continuous counter-current dryer fed with a nonporous material having all moisture as unbound moisture. As this material enters the dryer, it is first heated to the drying temperature. It will then pass through the length of dryer at nearly the wet-bulb temperature and theoretically at the end of the dryer, the material shall be discharged as dry material nearly at the wet bulb temperature.

Assumptions:

- No heat losses from the dryer.
- Heat is applied to the material only from the air, not by conduction from the dryer shell.
- All the moisture present is free moisture.
- There is no evaporation of moisture in the preliminary heating period.

Drying proceeds at a constant wet bulb temperature until desired amount of water has been removed. The entering air is assumed to be 100% saturated, so its temperature needs to be raised so as to decrease the relative saturation. This would enable the air to absorb moisture from the wet solid feed. In turn the exiting air is more saturated than the entering one.

For continuous dryer at steady state operating conditions,

$$F(X_1 - X_2) = G(Y_1 - Y_2) \quad \dots \quad (1)$$

This assume that the dry gas flow  $G$  and dry solids flow  $F$  do not change between dryer inlet and outlet. Mass balances can also be performed on the overall gas and solids entrainment in the exhaust gas stream.

The required solids flow rate, inlet moisture content  $X_1$  and outlet moisture  $X_0$  are normally specified, and the evaporation rate and outlet gas flow are calculated.

For batch dryer with a dry mass  $m$  of solids, a mass balance only gives a snapshot at one point during the drying cycle and an instantaneous drying rate given by:

$$m \frac{dX}{dt} = G(Y_1 - Y_2) \quad \dots \quad (2)$$

$$\frac{dX}{dt} = \frac{G(Y_1 - Y_2)}{m} \quad \dots \quad (3)$$

Sometimes  $\frac{dX}{dt}$  is called rate of drying.

## **5. DESCRIPTION:**

The set-up consists of a long resolving cylindrical shell slightly inclined toward the outlet. The shell is fitted with two brackets for support. Air from a blower passes through a heating chamber serves the purpose of drying agent. An arrangement is provided for rotating the dryer shell connected with electric motor and a reduction gear box. Flow control valve and by-pass valve are fitted to regulate the airflow.

The present set up has a facility to interface the system with computer which enables to log the experimental data using computer. The educational software and data-logging package has been developed per unit. This software is capable to tabulate the sample readings according to the requirement of experiment under study and results obtained can be compared.

## **6. UTILITIES REQUIRED:**

- 6.1 Electricity Supply: Single Phase, 220 V AC, 50 Hz, 32 Amp MCB with earth connection.
- 6.2 Floor Area Required: 3 m x 1.5 m.
- 6.3 Computer system: Pentium IV with DVD drive, Windows 7 MS-office pre loaded, One USB slot required in PC for DATA ACQUISITION CARD.
- 6.4 Solid material (Seeds 3-5 mm): 2 kg

## **7. INSTALLATION PROCEDURE OF SOFTWARE:**

Two DVDs are provided with the equipment. One DVD is of Device Driver and other DVD of Operating Software. First, place the device driver DVD in DVD-ROM and wait for 5 to 10 seconds. A blue-colored window appears on the screen. Install the software as per directions. When installation is finished, a shortcut would be created on the desktop showing name as “NI-Max”. Restart the computer system. Fix USB Card in empty USB slots. This ends the completion of loading of DAQ Card Driver. Now, take Operating Software DVD and place in DVD-ROM. Open DVD and open folder “Rotary Dryer.” and then double click on “Setup.exe”. Follow the instructions and do the installation.

Go to desktop and double click on “NI-Max”. A window opens and looks at left side. Follow the path as “My System” → “Devices and Interfaces” → Click on USB-6008:

"Dev1", Right click and rename it as "050" and press "enter". Now, it appears as USB-6008: "050". Close the window. After completion of installation "restart" the computer system.

## **8. METHOD OF OPERATING SOFTWARE:**

Open the EXE of selected condenser by clicking it from program Menu of main Menu bar. Enter Password "kce" for the software and press "enter" or click "OK". Click the "Interface" button to open interface of the software. In the entire software, a "NEXT" button is provided, at left front, which enables to go to next step. Another button named as "BACK" is present. This facilitates to retrieve last step taken. An "EXIT" button, at right bottom end, is present there. It may be used to exit at any session and finally, stopping the software.

**Note: In the absence of external hardware (USB-6008), the interfacing section may not able to work.**

## **9. EXPERIMENTAL PROCEDURE:**

### **9.1 STARTING PROCEDURE:**

- 9.1.1 Take known amount of the solid material and weigh it.
- 9.1.2 Now mix known amount of water in the solid and weigh the wet solid.
- 9.1.3 Ensure that switches given on the panel are at OFF position.
- 9.1.4 Set the preheating temperature for air.
- 9.1.5 Switch ON the main power supply.
- 9.1.6 Switch ON the heater & wait till the set point temperature is arrived.
- 9.1.7 Run the blower and set the flow rate of air by adjust the flow control valve for air.
- 9.1.8 Fill the feed hopper with wet solid.
- 9.1.9 Start the dryer in rotary motion.
- 9.1.10 Allow the wet solid to flow through the dryer by starting the screw conveyor and rotating hand wheel manually.
- 9.1.11 At steady state record the air temperature at inlet and outlet and velocity of air in computer interface.

9.1.12 Repeat the experiment for different air velocities.

9.1.13 Repeat the experiment for different air inlet temperature.

## 9.2 CLOSING PROCEDURE:

- 9.2.1 When experiment is over switch OFF the blower, heater and dryer.
- 9.2.2 Switch OFF the main power supply.
- 9.2.3 Clean the dryer properly.

## 10. OBSERVATION & CALCULATION:

### 10.1 DATA:

Area of the duct A	= 0.011 m <sup>2</sup>
Density of air $\rho_a$	= 1.21 kg/m <sup>3</sup>
Molecular weight of air $M_A$	= 29 g/mole
Molecular weight of water $M_W$	= 18 g/mole

### OBSERVATIONS:

### 10.2 OBSERVATION TABLE: 1

S.No.	T <sub>1</sub> (°C)	T <sub>2</sub> (°C)	T <sub>3</sub> (°C)	T <sub>4</sub> (°C)	V (m/sec)

### 10.3 CALCULATIONS:

$$G = V \times \rho_a \times A \quad (\text{kg/sec})$$

To calculate the humidity ( $y_1$  &  $y_2$ ) of air at temperature (T<sub>1</sub> & T<sub>2</sub>) and (T<sub>3</sub> & T<sub>4</sub>) respectively by psychometric chart.

$$y_1 = \underline{\hspace{10mm}}$$

$$y_2 = \underline{\hspace{10mm}}$$

$$Y_1 = y_1 \frac{M_w}{M_A} \text{ (kg of moisture/kg dry air)}$$

$$Y_2 = y_2 \frac{M_w}{M_A} \text{ (kg of moisture/kg dry air)}$$

$$N = G \times (Y_1 - Y_2) \text{ (kg of moisture/sec)}$$

**CALCULATION TABLE: 2**

S.No.	G (Kg/sec)	Y <sub>1</sub> (kg moisture/sec)	Y <sub>2</sub> (kg moisture/sec)	N (kg moisture/sec)

## 11. NOMENCLATURE:

Nom	Column Heading	Units	Type
A	Area of the dryer	m <sup>2</sup>	Given
G	Mass flow rate of air	kg/sec	Calculated
V	Velocity	m/sec	Measured
M <sub>A</sub>	Molecular weight of air	g/mole	Given
M <sub>w</sub>	Molecular weight of water	g/mole	Given
N	Rate of drying	kg moisture/sec	Calculated
T <sub>1</sub>	Inlet dry bulb temperature of air	°C	Measured
T <sub>3</sub>	Outlet dry bulb temperature of air	°C	Measured
T <sub>2</sub>	Inlet wet bulb temperature of air	°C	Measured
T <sub>4</sub>	Outlet wet bulb temperature of air	°C	Measured
Y	Absolute Humidity Difference	(kg of moisture / kg dry air)	Calculated
Y <sub>1</sub>	Absolute Humidity of air at the inlet of dryer	(kg of moisture / kg dry air)	Calculated
Y <sub>2</sub>	Absolute Humidity of air at the outlet of dryer	(kg of moisture / kg dry air)	Calculated

$y_1$	Moisture content of air at the inlet of dryer	kg of moisture / kg dry air)	Calculated
$y_2$	Moisture content of air at the outlet of dryer	kg of moisture / kg dry air)	Calculated
$\rho_a$	Density of air	kg/m <sup>3</sup>	Given

\* Symbols represent unitless quantity.

## 12. PRECAUTION & MAINTENANCE INSTRUCTIONS:

- 12.1 Proper oiling should be necessary for moving parts like roller.
- 12.2 Feed should be granular.
- 12.3 Before switching ON heater; switch ON the blower.

## 13. TROUBLESHOOTING:

- 13.1 If D.T.I display '1' on display board it means sensors connection are not OK tight it properly.
- 13.2 If switch of the heater is ON but temperature can't rise but panel LED is ON it means heater had burned replace it

## 14. REFERENCES:

- 14.1 Coulson, J M & Richardson, J F (1991). *Chemical Engineering Vol-2.* 4<sup>th</sup> Ed. ND: Asian Books Pvt. Ltd. pp 693-695, 707-708.
- 14.2 McCabe, Warren L. Smith, Julian C. Harriott, Peter (2005). *Unit Operations of Chemical Engineering.* 7<sup>th</sup> Ed. NY: McGraw-Hill. pp 804-812, 818-812.
- 14.3 Green, D.W. Perry, R.H. (2008). *Perry's Chemical Engineers' Handbook.* 8<sup>th</sup> Ed. NY: McGraw-Hill. p 12.27.

## PSYCHROMETRIC CHART

- 14.4 Green, D.W. Perry, R.H. (2008). *Perry's Chemical Engineers' Handbook.* 8<sup>th</sup> Ed. NY: McGraw-Hill. p 12.7.

# **ION EXCHANGE**

## **(MT-270RH)**

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*Our products are easy to understand and operate. They are excellent for students who are trying to gain practical knowledge through experiments.*

*However your comfort and safety are important to us, so we want you have an understanding of proper procedure to use the equipment. For the purpose, we urge you to read and follow the step-by-step operating instructions and safety precautions in this manual. It will ensure that your favourite product delivers reliable, superior performance year after year.*

*This manual includes information for all options available on this model. Therefore, you may find some information that does not apply to your equipment.*

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# ION EXCHANGE

## 1. OBJECTIVE:

To study the demineralization of water using two bed system.

## 2. AIM:

- 2.1 To deionise water to make it soft.
- 2.2 To determine the exchange rate and saturation point.

## 3. INTRODUCTION:

A typical ion exchange cycle used in water-treatment applications involves (a)backwash- used to remove accumulated solids obtained by an upflow of water to expand and fluidize the exchanger bed; (b)regeneration- a regenerant is pass slowly through the used to restore the original ionic form of the exchanger; (c)rinse- water is passed through the bed to remove regenerant from the void volume and, in the case of porous exchangers, from the resin pores;(d)loading- the fresh solution to be treated is passed through the bed until leakage begins to occur. Water softening is practiced in this way with a cation exchange column in sodium form. At the low ionic strength used in the loading step, calcium and magnesium are strongly preferred over sodium, allowing nearly complete removal.

Complete deionization with ion-exchange columns is the classical method of producing ultrapure water for boiler feed in electronics manufacture, and for other general uses in the chemical and allied industries. Deionization requires use of two exchangers with opposite functionality to remove both cations and anions. These can be separate columns; packed in adjacent layers in the same column, or more frequently, in a mixed bed.

## 4. THEORY:

The most usual ion exchange material employed in water softening is a sulphonated styrene-based resin, supplied by the makers in the sodium form. This resin has a strong affinity for calcium and magnesium ions, and will also remove ferrous ions after the more or less compete removal of calcium and magnesium.

Softening can be carried out as a batch process by stirring a suspension of the resin in the water for a period until equilibrium, or an acceptable level of hardness is reached

It is more convenient to operate a continuous flow process by passing the water slowly downwards through a column of resin beads. The exchange reaction takes place rapidly enough for the upper layers of the bed to approach exhaustion before the lower layers of the bed are able to exchange ions. There is thus a zone of active exchange which moves down the column until the resin at all depths becomes exhausted.

## **5. DESCRIPTION:**

The set-up consists of two glass column packed with anion and cation exchanger resin upto suitable height. The layer of glass wool is placed on bottom and top of the resin bed. The feed is allowed to enter from the bottom of first column and comes out of the top, which is again passed through the second column in same fashion. A digital conductivity meter measures the conductivity of liquids. A peristaltic pump is provided to measure the flow of water and circulation of feed. The flow rate can be adjusted by operating the speed variation knob of pump.

## **6. UTILITIES REQUIRED:**

- 6.1 Electricity Supply: Single Phase, 220 V AC, 50 Hz, 5-15 Amp combined socket with earth connection.
- 6.2 Water Supply: (Initial fill).
- 6.3 Floor Drain Required.
- 6.4 Bench Area Required: 1.2m x 1m.

## **7. EXPERIMENTAL PROCEDURE:**

### **7.1 STARTING PROCEDURE:**

- 7.1.1 Close all the valves.
- 7.1.2 Fill the sump tank with raw water 3/4<sup>th</sup> of its capacity.
- 7.1.3 Connect electric supply to the set-up.
- 7.1.4 Switch ON the mains ON/OFF switch.
- 7.1.5 Switch on the peristaltic pump.
- 7.1.6 By operating Auto/Manual switch and speed variation pot water passed into the column1.

- 7.1.7 Measure the conductivity of inlet water by dipping conductivity meter prob in the sump tank.
- 7.1.8 Open the outlet valve of column 1.
- 7.1.9 Put the conductivity meter prob at the outlet point of column-2.
- 7.1.10 Run the water at least half an hour and then measure the conductivity of treated water.
- 7.1.11 Repeat the experiment for different RPM.

## 7.2 CLOSING PROCEDURE:

- 7.2.1 When experiment is over switch OFF the pump.
- 7.2.2 Switch OFF the electric supply to the set up.

## 8. OBSERVATION & CALCULATION:

8.1 OBSERVATION TABLE:			
S. No.	N (RPM)	C <sub>I</sub> (mS)	C <sub>F</sub> (mS)

## 8.2 CALCULATIONS:

$$F = N \times 0.15 \text{ (LPH)}$$

If the conductivity decreases means deionization occurs.

## 9. NOMENCLATURE:

Nom	Column Heading	Units	Type
F	Flow rate of water	LPH	Calculated
C <sub>I</sub>	Initial conductivity of the water	mS	Measured
C <sub>F</sub>	Final conductivity of the water	mS	Measured
N	Speed of peristaltic pump	RPM	Measured

## **10. PRECAUTION & MAINTENANCE INSTRUCTIONS:**

- 10.1 Never run the apparatus if power supply is less than 200 volts and more than 230 volts.
- 10.2 Always keep the apparatus free from dust.

## **11. TROUBLESHOOTING:**

- 11.1 If pump gets jam, open the back cover of pump and rotate the shaft manually.

## **12. REFERENCES:**

- 12.1 Coulson, J M & Richardson, J F (1998). *Chemical Engineering Vol-2.* 4<sup>th</sup> Ed. ND: Asian Books Pvt.Ltd. pp 822, 835-837.
- 12.2 R.H. Perry (1983). *Perry's Chemical Engineers' Handbook.* 8<sup>th</sup> Ed. NY: McGraw Hill. pp 16-54, 16-67.





Engineering **Growth** Connection

## **LIQUID-LIQUID EXTRACTION IN A PACKED BED (MT-901RH)**

## ***Foreword***

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# LIQUID-LIQUID EXTRACTION IN A PACKED BED

## 1. OBJECTIVE:

To study the extraction of benzoic acid from toluene (dispersed phase) by water (continuous phase) in a packed bed column.

## 2. AIM:

- 2.1 To determine the overall mass transfer coefficient ( $K_{Wa}$ ) based on continuous phase.
- 2.2 To determine the overall mass transfer coefficient ( $K_{Ta}$ ) based on dispersed phase.
- 2.3 To determine the overall height of transfer unit ( $H_{OW}$ ) based on continuous phase.
- 2.4 To determine the overall height of transfer unit ( $H_{OT}$ ) based on dispersed phase.
- 2.5 To determine the individual height of transfer unit ( $H_w$ ) based on continuous phase.
- 2.6 To determine the individual height of transfer unit ( $H_t$ ) based on dispersed phase.

## 3. INTRODUCTION:

Liquid- liquid extraction, sometimes called solvent extraction, is the separation of the constituents of a liquid solution by contact with another insoluble liquid. If the substances constituting the original solution distribute themselves differently between the two liquid phases, a certain degree of separation will result, and this can be enhanced by use of multiple contacts or their equivalent in the manner of gas absorption and distillation.

## 4. THEORY:

Packed towers filled with some random packing (say rasching rings) are widely used for extraction of valuable chemicals from dilute solutions by liquid-liquid extraction. The packing provides a larger extract area for mass transfer and also reduces the axial mixing to some extent. The extraction rate in such columns depends on:

- (a) Choice of either continuous or dispersed phase.
- (b) Packing and column variables.

(c) Velocity of the liquid phases in the tower.

For dilute solutions and where the equilibrium relation is straight line, a simple expression can be obtained for determining the required height of a column (Z).

$$L_T dC_T = K_T a(C_T - C_T^*) dZ \quad \dots \quad (1)$$

Where  $L_T$ ,  $C_T$ ,  $C_T^*$  and  $K_T a$  are volumetric flow rate, concentration of extract material, concentration of extract material at equilibrium and overall mass transfer coefficient in dispersed phase.

$$\Delta C_{T1} = (C_T - C_T^*)_1$$

$$\Delta C_{T2} = (C_T - C_T^*)_2$$

$$(\Delta C_T)_{LM} = \frac{\Delta C_{T1} - \Delta C_{T2}}{\ln\left(\frac{\Delta C_{T1}}{\Delta C_{T2}}\right)}$$

On solving equation (1) became:

$$L_T (C_{T1} - C_{T2}) = K_T a (\Delta C_T)_{LM} Z \quad \dots \quad (2)$$

Where  $C_{T1}$ ,  $C_{T2}$ ,  $\Delta C_{T1}$ ,  $\Delta C_{T2}$ ,  $(\Delta C_T)_{LM}$  are inlet, outlet concentration, concentration difference at inlet, concentration at outlet and log mean concentration difference in dispersed phase.

The overall mass transfer coefficient based on dispersed phase (toluene),  $K_T a$  is defined as:

$$K_T a = \frac{L'_T}{V (\Delta C_T)_{LM}} \quad \dots \quad (3)$$

Where  $L'_T$  is molar flow rate of toluene and benzoic acid mixture,  $V$  is volume of packing.

Similarly overall mass transfer coefficient based on continuous phase (water),  $K_w a$  is defined as:

$$K_w a = \frac{L'_w}{V (\Delta C_w)_{LM}} \quad \dots \quad (4)$$

Where  $L'_w$  is Molar flow rate of water.

The tower performance is generally based on extraction rate data and evaluated in terms of over all height of transfer unit, based on continuous phase,  $H_{OW}$  and the extraction factor expressed in terms of flow rate ratios of the liquid phases,  $\frac{mQ_w}{Q_T}$ .

Where, m is the slope of equilibrium curve,  $m = dC_w/dC_T$

Under certain conditions (e.g. low concentrations) m is a constant

As per chilton and colburn, the over all height of transfer unit based on continuous phase (water),  $H_{OW}$  is defined as:

$$H_{OW} = \frac{L_w}{K_w a} \quad \dots \dots \dots \quad (5)$$

And the over all height of transfer unit based on dispersed phase (toluene),  $H_{OT}$  is defined as:

$$H_{OT} = \frac{L_T}{K_T a} \quad \dots \dots \dots \quad (6)$$

These two variables are plotted against each other on cartesian co-ordinates. As suggested by colburn, the slope and intercept of such a plot would represent the resistance of the individual films as:

$$H_{OW} = H_w + H_T \left[ \frac{mQ_w}{Q_T} \right] \quad \dots \dots \dots \quad (7)$$

$$H_{OT} = H_T + H_w \left[ \frac{Q_T}{mQ_w} \right] \quad \dots \dots \dots \quad (8)$$

Plot the graph of  $H_{OW}$  vs  $\left( m \times \frac{Q_w}{Q_T} \right)$  on a simple graph and calculate the slope  $H_T$  and intercept  $H_w$ .

Where  $H_w$  and  $H_T$  are the individual height of transfer unit in continuous and dispersed phase.

## 5. DESCRIPTION:

The set up consists of a glass column packed with rasching rings. Peristaltic pumps are provided for measured flow rate of solvent and solute that can be adjusted by varying RPM. Two feed tanks are given with peristaltic pumps for feed supply. Two receiving tank are provided to collect extract and raffinate phase product. Three way valves are

provided for continuous and discontinuous flow of dispersed and continuous phase. Provision is provided for changing the height of interface zone.

## **6. UTILITIES REQUIRED:**

- 6.1 Water Supply: (Initial fill).
- 6.2 Floor Drain Required.
- 6.3 Floor Area Required: 1 m x 1 m.
- 6.4 Weighing Balance 2 kg (least count 1 gm)
- 6.5 Laboratory glassware.

Conical flask (500 ml) : 02 No.

Measuring cylinder (50 ml) : 02 No.

Burette (50 ml) : 02 No.

- 6.6 Chemicals:

Toluene : 20 Lit.

Benzoic acid : 500 gm

Distilled water : 20 Lit.

N/5 NaOH in alcohol : 500 ml

N/20 NaOH in water : 500 ml

Phenolphthalein : 10 ml

## **7. EXPERIMENTAL PROCEDURE:**

### **7.1 STARTING PROCEDURE:**

- 7.1.1 Prepare a mixture of toluene and benzoic acid by mixing 488 gm of benzoic acid in 20 liter of toluene.
- 7.1.2 Take 20 liter of distilled water.
- 7.1.3 Fill one burette by N/20 sodium hydroxide solution.
- 7.1.4 Fill other burette by N/5 sodium hydroxide in alcohol solution.
- 7.1.5 Close all the valves .

- 7.1.6 Fill the one feed tank with prepared solution (toluene and benzoic acid) and other with water.
- 7.1.7 Adjust the RPM of peristaltic pumps for dispersed (toluene+benzoic acid) and continuous (water) phase to desired values and set the continuous and discontinuous flow for dispersed and continuous phase by operating three way valve.
- 7.1.8 Wait till steady state achieves (approx 20 to 30 minute).
- 7.1.9 Collect the known amount of sample in measuring cylinder from both the outlet and note down the volume of sample.
- 7.1.10 Titrate the sample solution of dispersed phase, against N/5 sodium hydroxide in alcohol (add NaOH from burette) by using phenolphthalein as an indicator.
- 7.1.11 Titrate the sample solution of continuous phase, against N/20 sodium hydroxide in water (add NaOH from burette) by using phenolphthalein as an indicator.
- 7.1.12 Repeat the experiment for different RPM of feed mixture and water .

## 7.2 CLOSING PROCEDURE:

- 7.2.1 When experiment is over stop the flow of feed by switch off pumps.
- 7.2.2 Drain the packed column and feed tanks by opening the drain valves.

## 8. OBSERVATION & CALCULATION:

8.1.a DATA:	
EQUIPMENT DATA	
Diameter of column $D_c$	= 0.048 m
Packed height of column Z	= 1.16 m
Weight of benzoic acid in feed solution $W_B$	= _____ gm
Molecular weight of benzoic acid $M_B$	= 122 g/mole
Volume of toluene in feed solution V	= _____ Lit
Normality of NaOH used to titrate the sample of dispersed phase $N_2$	= 0.05 g eq/L
Normality of NaOH used to titrate the sample of continuous phase $N_1$	= 0.2 g eq/L

Inlet concentration of benzoic acid in continuous phase $C_{W_2}$	$= 0 \text{ k mole/m}^3$
---	--------------------------

### 8.1.b DATA:

#### EQUILIBRIUM DATA FOR BENZOIC ACID - TOLUENE - WATER SYSTEM

$C_w \text{ (g mole/L)}$	$C_T \text{ (g mole/L)}$
0.0000	0.0000
0.0016	0.0080
0.0064	0.0336
0.0080	0.0673
0.00961	0.08811
0.011211	0.12015
0.012816	0.14097
0.01442	0.17301

### 8.2 OBSERVATION TABLE:

S.No.	RPM $T$	RPM $w$	$V_1 \text{ (ml)}$	$V_2 \text{ (ml)}$	$V_T \text{ (ml)}$	$V_w \text{ (ml)}$

### 8.3 CALCULATIONS:

Plot the graph of  $C_w$  vs  $C_T$  of equilibrium data and find the slope 'm'.

$$m = \underline{\hspace{2cm}}$$

$$Q_T = RPM_T \times 2.5 \times 0.06 \text{ LPH}$$

$$Q_w = RPM_w \times 2.5 \times 0.06 \text{ LPH}$$

$$A_c = \frac{\pi}{4} D_c^2 \text{ (m}^2\text{)}$$

$$V_c = A_c Z \text{ (m}^3\text{)}$$

$$C_{T1} = \frac{W_B}{V \times M_B} \text{ (Kmole/m}^3\text{)}$$

$$N_T = \frac{N_1 V_1}{V_T} \text{ (g eq/L)}$$

$$C_{W1} = N_T \text{ (Kmole/m3)}$$

$$N_W = \frac{N_2 V_2}{V_W} \text{ (g eq/L)}$$

$$C_{T2} = N_W \text{ (Kmole/m3)}$$

$$C_{T1}^* = \frac{C_{W1}}{m} \text{ (kmole/m}^3\text{)}$$

$$C_{T2}^* = \frac{C_{W2}}{m} \text{ (kmole/m}^3\text{)}$$

$$\Delta C_{T1} = (C_{T1} - C_{T1}^*) \text{ (kmole/m}^3\text{)}$$

$$\Delta C_{T2} = (C_{T2} - C_{T2}^*) \text{ (kmole/m}^3\text{)}$$

$$\Delta C_{Tlm} = \frac{(\Delta C_{T1} - \Delta C_{T2})}{\ln\left(\frac{\Delta C_{T1}}{\Delta C_{T2}}\right)} \text{ (kmole/m}^3\text{)}$$

$$L_T = \frac{Q_T \times 10^{-3}}{3600} (C_{T2} - C_{T1}) \text{ (kmole/sec)}$$

$$K_T a = \frac{L_T}{V_c \times \Delta C_{Tlm}} \text{ (kmole/m}^3\text{sec) (kmole/m}^3\text{)}$$

$$H_{OT} = \frac{Q_T \times 10^{-3}}{K_T a \times A_c \times 3600} \text{ (m)}$$

$$C_{W1}^* = m \times C_{T1} \text{ (kmole/m}^3\text{)}$$

$$C_{W2}^* = m \times C_{T2} \text{ (kmole/m}^3\text{)}$$

$$\Delta C_{W1} = (C_{W1}^* - C_{W1}) \text{ (kmole/m}^3\text{)}$$

$$\Delta C_{W2} = (C_{W2}^* - C_{W2}) \text{ (kmole/m}^3\text{)}$$

$$\Delta C_{Wlm} = \frac{(\Delta C_{W1} - \Delta C_{W2})}{\ln\left(\frac{\Delta C_{W1}}{\Delta C_{W2}}\right)} \text{ (kmole/m}^3\text{)}$$

$$L_w = \frac{Q_w \times 10^{-3}}{3600} (C_{w1} - C_{w2}) \text{ (kmole/sec)}$$

$$K_w a = \frac{L_w}{V_c \times \Delta C_{wlm}} \text{ (kmole/m}^3\text{sec (kmole/m}^3\text{))}$$

$$H_{ow} = \frac{Q_w \times 10^{-3}}{K_w a \times A_c \times 3600} \text{ (m)}$$

<b>CALCULATION TABLE:</b>							
S. No	Q <sub>T</sub> (LPH)	Q <sub>w</sub> (LPH)	m (Q <sub>w</sub> /Q <sub>T</sub> )	K <sub>Ta</sub> (kmole/m <sup>3</sup> sec (kmole/m <sup>3</sup> ))	K <sub>wa</sub> (kmole/m <sup>3</sup> sec (kmole/m <sup>3</sup> ))	H <sub>OT</sub> (m)	H <sub>ow</sub> (m)

Plot the graph of H<sub>ow</sub> vs  $\left( m \times \frac{Q_w}{Q_T} \right)$  on a simple graph and calculate the slope H<sub>T</sub> and intercept H<sub>w</sub>.

$$H_T = \text{_____ (m)}$$

$$H_w = \text{_____ (m)}$$

## 9. NOMENCLATURE:

Nom	Column Heading	Units	Type
A <sub>c</sub>	Area of the column	m <sup>2</sup>	Calculated
C <sub>T</sub>	Concentration of benzoic acid in dispersed phase	gmole/L	Given
C <sub>T1</sub>	Inlet concentration of benzoic acid in dispersed phase	Kmole/m <sup>3</sup>	Calculated
C <sup>*</sup> <sub>T1</sub>	Inlet concentration of benzoic acid in dispersed phase at equilibrium	Kmole/m <sup>3</sup>	Calculated
C <sub>T2</sub>	Outlet concentration of benzoic acid in dispersed phase	Kmole/m <sup>3</sup>	Calculated
C <sup>*</sup> <sub>T2</sub>	Outlet concentration of benzoic acid in dispersed phase at equilibrium	Kmole/m <sup>3</sup>	Calculated

$C_w$	Concentration of benzoic acid in continuous phase	gmole/L	Given
$C_{w1}$	Outlet concentration of benzoic acid in continuous phase	Kmole/m <sup>3</sup>	Calculated
$C^{*}_{w1}$	Outlet concentration of benzoic acid in continuous phase at equilibrium	Kmole/m <sup>3</sup>	Calculated
$C_{w2}$	Inlet concentration of benzoic acid in continuous phase	Kmole/m <sup>3</sup>	Given
$C^{*}_{w2}$	Inlet concentration of benzoic acid in continuous phase at equilibrium	Kmole/m <sup>3</sup>	Calculated
$D_c$	Diameter of column	m	Given
$H_{OT}$	Overall height of transfer unit based on dispersed phase	m	Calculated
$H_{OW}$	Overall height of transfer unit based on continuous phase	m	Calculated
$H_T$	Individual height of transfer unit based on dispersed phase	m	Calculated
$H_w$	Individual height of transfer unit based on continuous phase	m	Calculated
$K_{Ta}$	Overall mass transfer coefficient based on dispersed phase	(kmole/m <sup>3</sup> sec) c (kmole/m <sup>3</sup> )	Calculated
$K_{Wa}$	Overall mass transfer coefficient based on continuous phase	(kmole/m <sup>3</sup> sec) c (kmole/m <sup>3</sup> )	Calculated
$L_T$	Molar flow rate of dispersed phase	Kmole/sec	Calculated
$RPM_T$	RPM for dispersed phase(Toluene+Benzoic acid)	RPM	Observed
$RPM_w$	RPM for continuous phase water	RPM	Observed
$L_w$	Molar flow rate of continuous phase	Kmole/sec	Calculated
$M_B$	Molecular weight of benzoic acid	g/mole	Given
$m$	Slope of the graph $C_w$ vs $C_T$	*	Calculated
$N_1$	Normality of sodium hydroxide used to titrate the sample of continuous phase	g eq/L	Given
$N_2$	Normality of sodium hydroxide used to titrate the sample of dispersed phase	g eq/L	Given
$N_T$	Normality of benzoic acid in dispersed phase	g eq/L	Calculated
$N_w$	Normality of benzoic acid in continuous phase	g eq/L	Calculated
$Q_T$	Volumetric flow rate of toluene (dispersed phase)	LPH	Measured

$Q_w$	Volumetric flow rate of water (continuous phase)	LPH	Measured
$V$	Volume of toluene in feed solution	Lit	Given
$V_1$	Volume of sample taken from continuous phase	ml	Measured
$V_2$	Volume of sample taken from dispersed phase	ml	Measured
$V_c$	Volume of the column	$m^3$	Calculated
$V_T$	Volume of sodium hydroxide used to titrate the sample of dispersed phase	ml	Measured
$V_W$	Volume of sodium hydroxide used to titrate the sample of continuous phase	ml	Measured
$W_B$	Weight of benzoic acid in feed solution	gm	Given
$Z$	Packed height	m	Given
$\Delta C_{T1}$	Inlet concentration difference of benzoic acid in dispersed phase	Kmole/m <sup>3</sup>	Calculated
$\Delta C_{T2}$	Outlet concentration difference of benzoic acid in dispersed phase	Kmole/m <sup>3</sup>	Calculated
$(\Delta C_T)_{IM}$	Log mean concentration difference of benzoic acid in dispersed phase	Kmole/m <sup>3</sup>	Calculated
$\Delta C_{W1}$	Outlet concentration difference of benzoic acid in continuous phase	Kmole/m <sup>3</sup>	Calculated
$\Delta C_{W2}$	Inlet concentration difference of benzoic acid in continuous phase	Kmole/m <sup>3</sup>	Calculated
$(\Delta C_T)_{IM}$	Log mean concentration difference of benzoic acid in continuous phase	kmole/m <sup>3</sup>	Calculated

\* Symbols are unit less.

## 10. PRECAUTION & MAINTENANCE INSTRUCTIONS:

- 10.1 Interface should not be disturbed during the experiment.
- 10.2 Don't exceed the rpm more than 100.
- 10.3 Water outlet should be toluene free.
- 10.4 Always use clean water & ensure that there are no foreign particles in it.
- 10.5 Always clean the column feed tanks & receiving tanks properly after experiment.
- 10.6 Never use feed tanks to store the chemicals used in the experiment.

- 10.7 Always drain the column once before running the experiment on desired second flow rates.

## **11. TROUBLESHOOTING:**

- 11.1 If there is any leakage tight that part or fix it again after wrapping teflon tape.

## **12. REFERENCES:**

- 12.1 Coulson, J M & Richardson, J F (1991). *Chemical Engineering Vol-2. 4<sup>th</sup> Ed.* ND: Asian Books Pvt. Ltd. pp 519-520, 584-591, 597-601.
- 12.2 Dutta K. Binay (2007). *Principles of Mass Transfer and Separation Processes.* ND: Prentice Hall of India Pvt. Ltd. pp 128-132, 455-456.



## **PACKED BED DISTILLATION**

### **COLUMN (MT-913RH)**

## ***Foreword***

*Welcome to the fast growing family of K.C. product owners. We appreciate your interest in us and thank you for buying our product.*

*You have chosen the finest quality product in the market which is produced using latest techniques and has underwent strict quality control tests. It is a product that we are proud to build and you are proud to own it.*

*Our products are easy to understand and operate. They are excellent for students who are trying to gain practical knowledge through experiments.*

*However your comfort and safety are important to us, so we want you have an understanding of proper procedure to use the equipment. For the purpose, we urge you to read and follow the step-by-step operating instructions and safety precautions in this manual. It will ensure that your favourite product delivers reliable, superior performance year after year.*

*This manual includes information for all options available on this model. Therefore, you may find some information that does not apply to your equipment.*

*All information, specifications and illustrations in this manual are those in effect at the time of printing. We reserve the right to change specifications or design at any time without notice.*

*Customer satisfaction is our primary concern. Feel Free to contact us for any assistance. So what are you waiting for, roll up your sleeves and let us get down to work!*

***K.C. Engineers Pvt. Ltd.***

## *Important Information About This Manual*

### *Reminder for Safety*

#### ***Modification on Equipment:***

*This equipment should not be modified. Modification could affect its performance, safety or disturbance. In addition damage or performance problems resulting from modification may not be covered under warranties.*

#### ***Precautions and Maintenance:***

*This is used to indicate the presence of a hazard that could cause minor or moderate personal injury or damage to your equipment. To avoid or reduce the risk, the procedures must be followed carefully.*

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# PACKED BED DISTILLATION COLUMN

## 1. OBJECTIVE:

To study the operation of a packed bed distillation column.

## 2. AIM:

- 2.1 To verify the rayleigh equation.
- 2.2 To obtain the T-x data under total reflux condition at steady state and compare it with theoretical value.
- 2.3 To operate the column under total reflux condition and calculate the minimum number of theoretical stages using fenske's equation.
- 2.4 To operate the column under any desired reflux condition and calculate theoretical number of stages using Mc-Cabe Thiele's method.
- 2.5 To calculate the HETP for known packed height.
- 2.6 To estimate the batch distillation curves for a binary system and verify the binary batch distillation equation for a known packed height.
- 2.7 To operate the column under total reflux condition and estimate the HETP for the packing.

## 3. INTRODUCTION:

Batch distillation is often preferable to continuous distillation in case where relatively small quantities of material are to be handled at irregularly scheduled periods. The simplest case of batch distillation is one in which the material to be separated is charged to a heated kettle, fitted with a total condenser and product receiver. The material is distilled without reflux until a definite quantity of one of the components of the mixture has been recovered or until a definite change in composition of the still contents has been effected.

In all types of batch distillation, a quantity of feed is charged to a still pot, or kettle, and heat is applied to it. The vapor, which is usually passed through a fractionating column, is then condensed, giving the overhead product while a less volatile residue remains in the kettle at the end of distillation. Continuous distillation is a steady state process because

once equilibrium has been attained, conditions at any given point remains constant, whereas batch distillation is an unsteady state process, the concentration of the more volatile component decreasing continually so that the temperature and composition of the mixture at a point in the system must alter as the distillation proceeds

#### **4. THEORY:**

##### **PART-I**

Packed column are often used for distillation when the separation is relatively easy and the required column diameter is not very large. The column height is generally based on the number of theoretical plates and the height equivalent to theoretical plate (HETP).

For the calculation of height equivalent to theoretical plate (HETP):

$$HETP = \frac{Z}{N_t}$$

Where Z and N<sub>t</sub> are packed height and number of theoretical plates respectively.

For simple batch distillation process, initially the still is filled with F moles of feed with x<sub>F</sub> as the mole fraction of the more volatile component. Let at any moment the number of moles of liquid in the still be B having a mole fraction x of the more volatile component (i.e A) and let the moles of accumulated condensate be D. Concentration of the equilibrium vapour is y\*. Over a small time, the change in the amount of liquid in the still is dB and the amount of vapour withdrawn is dD. The following differential mass balance equations may be written:

Total material balance:

$$-dB = dD \quad \dots \dots \dots \quad (1)$$

Component A balance:

$$-d(Bx) = y^* dD \quad \dots \dots \dots \quad (2)$$

On solving the above equation:

$$-Bdx - xdB = y^* dD$$

Put  $-dB = dD$  from eq<sup>n</sup>(1):

$$-Bdx = y^* dD - x dD$$

$$-Bdx = (y^* - x)dD$$

Again put  $dD = -dB$

$$Bdx = (y^* - x)dB \quad \dots \dots \dots \quad (3)$$

By arranging the above equation:

$$\frac{dB}{B} = \frac{dx}{y^* - x} \quad \dots \dots \dots \quad (4)$$

Where  $y^*$  is the vapour composition in equilibrium with liquid composition  $x$

Integrating Eq<sup>n</sup> (4):

$$\int_{F}^{B} \frac{dB}{B} = \int_{x_F}^{x_B} \frac{dx}{y^* - x} \quad \dots \dots \dots \quad (5)$$

Eq. (5) is the rayleigh equation describes the variation of the liquid and vapour mole fractions,  $x, y$  in terms of the fraction remaining in the still.

$$\ln \frac{F}{B} = \int_{x_B}^{x_F} \frac{dx}{y^* - x} \quad \dots \dots \dots \quad (6)$$

We have:

$$y^* = \frac{\alpha x}{1 + (\alpha - 1)x}$$

For constant relative volatility,  $\alpha$  we can write the equation (6):

$$\begin{aligned} \ln \frac{F}{B} &= \int_{x_B}^{x_F} \frac{dx}{\frac{\alpha x}{1 + (\alpha - 1)x} - x} \\ \ln \frac{F}{B} &= \int_{x_B}^{x_F} \frac{dx}{(\alpha - 1)x(1 - x)} + \frac{dx}{(1 - x)} \quad \dots \dots \dots \quad (7) \end{aligned}$$

By partial differentiation of the above equation:

$$\ln \frac{F}{B} = \frac{1}{\alpha - 1} \left[ \ln \frac{x_F(1 - x_B)}{x_B(1 - x_F)} \right] + \ln \left[ \frac{(1 - x_B)}{(1 - x_F)} \right]$$

$$\ln \frac{F}{B} - \ln \left[ \frac{(1-x_B)}{(1-x_F)} \right] = \frac{1}{\alpha - 1} \left[ \ln \frac{x_F(1-x_B)}{x_B(1-x_F)} \right]$$

$$\ln \left[ \frac{F(1-x_F)}{B(1-x_B)} \right] = \frac{1}{\alpha - 1} \left[ \ln \frac{x_F(1-x_B)}{x_B(1-x_F)} \right]$$

$$\alpha \ln \left[ \frac{F(1-x_F)}{B(1-x_B)} \right] = \left[ \ln \frac{x_F(1-x_B)}{x_B(1-x_F)} \right] + \ln \left[ \frac{F(1-x_F)}{B(1-x_B)} \right]$$

$$\alpha \ln \left[ \frac{F(1-x_F)}{B(1-x_B)} \right] = \ln \left[ \frac{Fx_F}{Bx_B} \right] \quad \dots \dots \dots \quad (8)$$

The above equation is more convenient form of rayleigh equation.

Batch distillation with only a single still does not give a good separation unless the relative volatility is very high. To obtain product with a narrow composition range, a rectifying batch still is used that consist of a reboiler, a rectifying column, a condenser, some means of splitting of a portion of condensed vapor or distillate as reflux and the receiver. The operation of a batch still and column can be analyzed using the same operating line equation as for the rectifying section of the continuous distillation:

$$y_{n+1} = \frac{R}{R+1} x_n + \frac{x_D}{R+1}$$

for the binary system:

$$y_2 = 1 - y_1 \quad x_2 = 1 - x_1$$

Since the slope of the operating line is  $R / (R+1)$ , the slope increases as the reflux increases, until when reflux is infinite. Under total reflux slope is 1. The operating line then coincides with the diagonal. The number of plate is minimum at total reflux.

Minimum number of plates required can be calculated from the terminal concentration of  $x_B$  and  $x_D$  based on the relative volatility of the components  $\alpha$ , which is defined in terms of equilibrium concentrations:

Minimum number of theoretical plates required:

$$N_m = \frac{\log[x_D(1-x_B)/x_B(1-x_D)]}{\log \alpha} - 1 \quad \dots \dots \dots \quad (9)$$

The above equation is known as fenske's eq<sup>n</sup>.

The final ratio does not change much over the range of temperature encountered in a typical column, so the relative volatility is taken as constant.

## **PART-II**

### **METHOD TO CALCULATE NUMBER OF THEORETICAL PLATES (Mc-Cabe Thiele's Method):**

For the determination of number of theoretical plates at desired reflux ratio. We are using graphical method which is called Mc-Cabe Thiele's method.

In this method draw the equilibrium data on x-y plane. Locate the point  $x_D$ ,  $x_B$ , and  $x_F$  on diagonal. For the given reflux ratio R calculate the intercept on the rectifying section operating line on the y-axis.

$$I = \frac{x_D}{R + 1}$$

Now calculate the slope of the feed line:

$$S = \tan^{-1} \left( \frac{q}{q - 1} \right)$$

Where q is the fraction of liquid in the feed.

Follow the following procedure to calculate theoretical number of plates ( $N_t$ ):

- a) Plot the equilibrium data on x-y plane.
- b) Locate the point  $x_D$ ,  $x_B$ , and  $x_F$  on diagonal.
- c) Draw the rectifying line from point  $x_D$  with intercept I on the y axis.
- d) Also draw the feed line from the point  $x_F$  with slope S and intersect the rectifying line, that intersection point is M.
- e) Now draw the stripping line from point  $x_B$  to the intersection point M.
- f) Draw horizontal line from the point  $x_D$  to equilibrium curve.
- g) Draw vertical line from the point at equilibrium curve (where horizontal line is end) to the operating line.
- h) The above step made triangular construction.

- i) Again draw horizontal line from the point at operating line (where vertical line is end) to the equilibrium curve.
- j) Repeat the above three steps between equilibrium curve and operating line, till cover the point  $x_B$  on diagonal.
- k) Count the number of triangles made.
- l) Number of triangles is equal to number of theoretical stages including reboiler.
- m) Subtract one from the number of triangle, and then it is theoretical number of stages required at desired reflux conditions.

For the overall efficiency of the column can be use following formulae:

$$\eta_O = \frac{N_t}{N_A} \times 100 \text{ (%)}$$

Where  $N_A$  is number of actual plates in the column.

## 5. DESCRIPTION:

The set-up consists of packed column made up of borosilicate glass, which is packed with glass rasching rings. An electrically heated reboiler is installed at the bottom of the column. A tank is provided to collect the bottom product. Shell and tube type condenser is provided to condensate the vapour. A cold water tank with pump and rotameter is provided for circulation of cooling water. Cold water flowrate is adjusted by rotameter and bypass valve. Reflux drum is provided to collect the condensate. An automatic reflux divider is provided to divide condensate into reflux and distillate. Receiving tank is for collecting the distillate. Drain valves are provided for drainage purpose.

## 6. UTILITIES REQUIRED:

- 6.1 Electricity Supply: Single Phase, 220 V AC, 50 Hz, 32 Amp MCB with earth connection.
- 6.2 Water Supply: Initial fill.
- 6.3 Floor Drain Required.
- 6.4 Chemicals :

Water	:	15 Lit.
Methanol	:	4 Lit.

## 7. EXPERIMENTAL PROCEDURE:

### 7.1 STARTING PROCEDURE:

- 7.1.1 Prepare methanol water solution by mixing known amount of water and methanol.
- 7.1.2 The composition of methanol in solution should be in the range of 15 to 25 % by volume.
- 7.1.3 The total amount of solution should not be less than 15 liters.
- 7.1.4 Note down the volume of water and methanol.
- 7.1.5 Close all the valves.
- 7.1.6 Ensure that switches given on the panel are at OFF position.
- 7.1.7 Fill water in the cold water tank.
- 7.1.8 Fill the reboiler with methanol-water solution by open the feed supply valve of funnel and air vent valve .
- 7.1.9 Close the feed supply valve of funnel and air vent valve.
- 7.1.10 Connect electric supply to the set-up.
- 7.1.11 Set a process temperature for the process using the digital temperature controller (DTC). The temperature should be in the range of 85 to 95 °C.
- 7.1.12 Start the heater & cooling water pump.
- 7.1.13 Adjust the cold water flow rate by control valve and by pass valve to a moderate value.
- 7.1.14 Set the cyclic timer for total reflux.
- 7.1.15 Wait 25-30 minute for the system to achieve steady state.
- 7.1.16 Now take out the samples from the bottom & distillate stream (by closing the timer for short while).
- 7.1.17 Cool down the samples to room temperature & measure the refractive indices.
- 7.1.18 Also take samples at different temperatures and measure the refractive indices.

7.1.19 Note down the temperature and refractive indices.

7.1.20 Now adjust the cyclic timer to a desired reflux ratio & wait for 3-5 min.

7.1.21 Now take out the samples from both distillate & bottom product.

7.1.22 Cool down the samples & measure their refractive indices.

7.1.23 Also take samples at different temperatures and measure the refractive indices.

7.1.24 Note down the temperature and refractive indices.

7.1.25 Repeat the experiment for different set point temperatures & reflux ratios.

## **7.2 CLOSING PROCEDURE:**

7.2.1 When experiment is over stop the water supply.

7.2.2 Switch OFF the pump and heater.

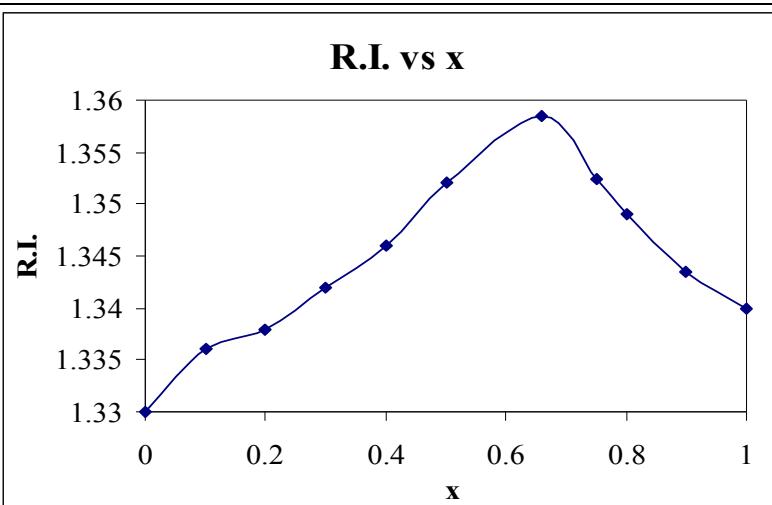
7.2.3 Switch OFF the main power supply.

7.2.4 Drain the column, reflux drum, receiving tank and water tank.

7.2.5 Drain the vessel after temperatures fall down to atmospheric values space by open the drain valve.

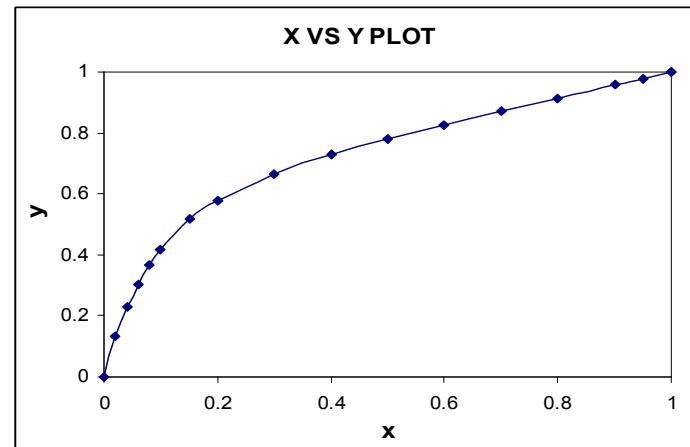
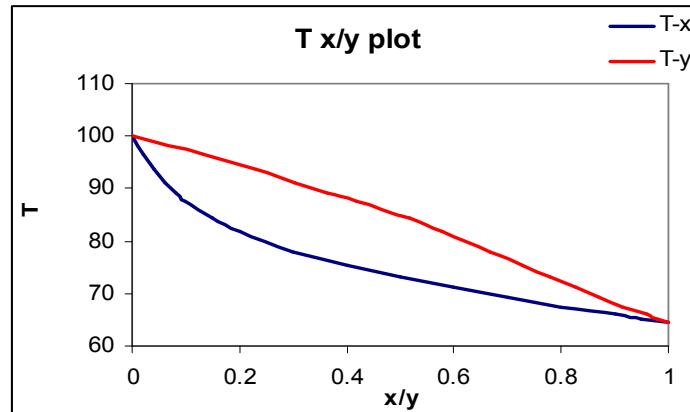
## **8. OBSERVATION & CALCULATION:**

<b>8.1.a DATA:1</b>	
Relative volatility $\alpha$	= 3.32
Molecular weight of methanol $M_M$	= 32 g/mole
Molecular weight of Water $M_w$	= 18 g/mole
Enthalpy of feed $H_F$	= 0 kJ/kmole
Packed height Z	= _____ m

**8.1.b DATA:2**


X <sub>the</sub>	R.I. (at 25°C)
0	1.33
0.1	1.3315
0.2	1.3325
0.3	1.3345
0.4	1.336
0.5	1.337
0.66	1.3375
0.75	1.337
0.8	1.3345
0.9	1.3305
1	1.3235

T-x-y data for methanol water system		
T <sub>the</sub> (°C)	X <sub>the</sub>	Y <sub>the</sub>
100	0	0
96.4	0.02	0.134
93.5	0.04	0.23
91.2	0.06	0.304
89.3	0.08	0.365
87.7	0.1	0.418
84.4	0.15	0.517
81.7	0.2	0.579
78.0	0.3	0.665
75.3	0.4	0.729
73.1	0.5	0.779
71.2	0.6	0.825
69.3	0.7	0.87
67.5	0.8	0.915
66.0	0.9	0.958
65.0	0.95	0.979
64.5	1	1



### OBSERVATIONS (AT TOTAL REFLUX CONDITION):

R = \_\_\_\_\_

V = \_\_\_\_\_ Lit.

T<sub>F</sub> = \_\_\_\_\_ °C

R.I<sub>F</sub> = \_\_\_\_\_

R.I<sub>B</sub> = \_\_\_\_\_

R.I<sub>D</sub> = \_\_\_\_\_

**8.2.a OBSERVATION TABLE (AT TOTAL REFLUX CONDITION):**

S.No.	T (°C)	R.I
1		
2		
3		
4		
5		
6		
7		

**OBSERVATIONS (AT DESIRED REFLUX CONDITION):**

$$R = \underline{\hspace{2cm}}$$

$$T_F = \underline{\hspace{2cm}} ^\circ C$$

$$R.I_F = \underline{\hspace{2cm}}$$

$$R.I_D = \underline{\hspace{2cm}}$$

**8.2.b OBSERVATION TABLE (AT DESIRED REFLUX CONDITION):**

S.No.	T (°C)	R.I
1		
2		
3		
4		
5		
6		
7		

**8.3.a CALCULATIONS (AT TOTAL REFLUX CONDITION):**

$$x = \underline{\hspace{2cm}} \quad [\text{From R.I vs } x \text{ graph}]$$

**CALCULATION TABLE: 1**

S.No.	T (°C)	x	x <sub>the</sub>
1			
2			
3			
4			
5			
6			
7			

$x_F = \underline{\hspace{2cm}}$  [From R.I vs x graph, value of x corresponding to R.I<sub>F</sub>]

$x_B = \underline{\hspace{2cm}}$  [From R.I vs x graph, value of x corresponding to R.I<sub>B</sub>]

$x_D = \underline{\hspace{2cm}}$  [From R.I vs x graph, value of x corresponding to R.I<sub>D</sub>]

To calculate the property of methanol ( $\rho$ ) at temperature T<sub>F</sub> (°C) from data book.

$$\rho = \underline{\hspace{2cm}} \text{ (kg/m}^3\text{)}$$

$$F = \frac{\rho V}{M_M \times 1000} \text{ (kmole)}$$

$$B = \frac{x_F - x_D}{x_B - x_D} \times F \text{ (kmole)}$$

$$\ln \left[ \frac{Fx_F}{Bx_B} \right] = \underline{\hspace{2cm}}$$

$$\alpha \ln \frac{F(1-x_F)}{B(1-x_B)} = \underline{\hspace{2cm}}$$

$$E = \ln \left[ \frac{Fx_F}{Bx_B} \right] - \alpha \ln \frac{F(1-x_F)}{B(1-x_B)}$$

$$N_m = \frac{\ln \left[ \frac{x_D(1-x_B)}{x_B(1-x_D)} \right]}{\ln \alpha} - 1$$

$$HETP = \frac{Z}{N_m} \text{ (m)}$$

### 8.3.b CALCULATIONS (AT DESIRED REFLUX CONDITION):

$x_F = \underline{\hspace{2cm}}$  [From R.I vs x graph, value of x corresponding to R.I<sub>F</sub>]

$x_D = \underline{\hspace{2cm}}$  [From R.I vs x graph, value of x corresponding to R.I<sub>D</sub>]

$x = \underline{\hspace{2cm}}$  [From R.I vs x graph]

**CALCULATION TABLE: 2**

S.No.	T (°C)	x
1		
2		
3		
4		
5		
6		
7		

$$y = \frac{\alpha x}{1 + (\alpha - 1)x}$$

**CALCULATION TABLE: 3**

S.No.	T (°C)	x	y
1			
2			
3			
4			
5			
6			
7			

Plot T vs (x,y) on a simple graph.

$T_D = \underline{\hspace{2cm}}$  (°C) [corresponding value of T with respect to x<sub>F</sub> from T-y curve]

$T_B = \underline{\hspace{2cm}}$  ( $^{\circ}\text{C}$ ) [corresponding value of  $T$  with respect to  $x_B$  from  $T-x$  curve]

To calculate the property of methanol ( $C_{PM}, \lambda_M$ ) and water ( $C_{PW}, \lambda_W$ ) at temperature  $T_B$  ( $^{\circ}\text{C}$ ) from data book.

$$C_{PM} = \underline{\hspace{2cm}} (\text{kJ/kg } ^{\circ}\text{C})$$

$$C_{PW} = \underline{\hspace{2cm}} (\text{kJ/kg } ^{\circ}\text{C})$$

$$\lambda_M = \underline{\hspace{2cm}} (\text{kJ/kg})$$

$$\lambda_W = \underline{\hspace{2cm}} (\text{kJ/kg})$$

$$C_{PF} = x_F C_{PM} + (1 - x_F) C_{PW} (\text{kJ/kg } ^{\circ}\text{C})$$

$$M_{av} = x_F M_M + (1 - x_F) M_W$$

$$H_L = C_{PF} M_{av} (T_B - T_F) (\text{kJ/kmole})$$

$$H_G = [(x_F)[C_{PM} M_M (T_D - T_F) + \lambda_M M_M] + (1 - x_F)[C_{PW} M_W (T_D - T_F) + \lambda_W M_W]] (\text{kJ/kmole})$$

$$q = \frac{H_G - H_F}{H_G - H_L}$$

$$S = \tan^{-1} \left( \frac{q}{q-1} \right)$$

$$I = \frac{x_D}{R+1}$$

$$N_t = \underline{\hspace{2cm}} \quad [\text{Refer theory PART-II}]$$

$$\eta_O = \frac{N_t}{N_A} \times 100 \text{ (%)}$$

## 9. NOMENCLATURE:

Nom	Column Heading	Units	Type
B	Moles of methanol in residue	kmole	Calculated
$C_{PF}$	Specific heat of feed solution	kJ/kg $^{\circ}\text{C}$	Calculated
$C_{PM}$	Specific heat of liquid methanol	kJ/kg $^{\circ}\text{C}$	Calculated
$C_{PW}$	Specific heat of liquid water	kJ/kg $^{\circ}\text{C}$	Calculated

E	Error	*	Calculated
F	Moles of methanol in feed	kmole	Calculated
HETP	Height equivalent to a theoretical plate	m	Calculated
$H_F$	Enthalpy of feed	kJ/kmole	Calculated
$H_G$	Enthalpy of saturated vapour	kJ/kmole	Calculated
$H_L$	Enthalpy of saturated liquid	kJ/kmole	Calculated
I	Intercept of rectifying line	*	Calculated
$M_{av}$	Average molecular weight of feed solution	g/mole	Calculated
$M_M$	Molecular weight of methanol	g/mole	Given
$M_W$	Molecular weight of water	g/mole	Given
$N_A$	Number of actual plates	*	Given
$N_m$	Minimum number of plates	*	Calculated
$N_t$	Number of theoretical plates	*	Calculated
q	Fraction of liquid in the feed	*	Calculated
R	Reflux ratio	*	Measured
R.I	Refractive index	*	Measured
R.I <sub>B</sub>	Refractive index of residue sample	*	Measured
R.I <sub>D</sub>	Refractive index of distillate sample	*	Measured
R.I <sub>F</sub>	Refractive index of feed sample	*	Measured
S	Slope of feed line	*	Calculated
T	Temperature	°C	Measured
$T_B$	Bubble point temperature	°C	Calculated
$T_D$	Dew point temperature	°C	Calculated
$T_F$	Temperature of feed	°C	Measured
$T_{the}$	Theoretical tempearture	°C	Given
V	Volume of methanol in feed	Lit	Measured
x	Experimental value of mole fraction of methanol in liquid phase	*	Calculated
$x_F$	Mole fraction of methanol in feed	*	Calculated
$x_B$	Mole fraction of methanol in residue	*	Calculated
$x_D$	Mole fraction of methanol in distillate	*	Calculated
$x_{the}$	Theoretical value of mole fraction of methanol in liquid phase	*	Given
y	Experimental value of mole fraction of methanol	*	Calculated

	in vapour phase		
$y_{\text{the}}$	Theoretical value of mole fraction of methanol in vapour phase	*	Given
$Z$	Packed height	m	Given
$\alpha$	Relative volatility	*	Given
$\rho$	Density of methanol	kg/m <sup>3</sup>	Calculated
$\lambda_M$	Latent heat of vaporization for methanol	kJ/kg	Calculated
$\lambda_W$	Latent heat of vaporization for water	kJ/kg	Calculated

\* Symbols represent unitless quantity.

## 10. PRECAUTION & MAINTENANCE INSTRUCTIONS:

- 10.1 Heater should not be switch ON before filling the feed into reboiler.
- 10.2 Always drain, tanks and reboiler, when experiment is over.
- 10.3 Always use clean water and ensure that there are no foreign particles in the flow streams.

## 11. TROUBLESHOOTING:

- 11.1 If electric panel is not showing the input on the mains light, check the mains supply.

## 12. REFERENCES:

- 12.1 McCabe, Warren L. Smith, Julian C. Harriott, Peter (2005). *Unit Operations of Chemical Engineering*. 7<sup>th</sup> Ed. NY: McGraw-Hill. pp 673-674,687-688.
- 12.2 Coulson, J M & Richardson, J F (1991). *Chemical Engineering Vol-2*. 4<sup>th</sup> Ed. ND: Asian Books Pvt. Ltd. pp 513-514
- 12.3 Dutta K. Binay (2007). *Principles of Mass Transfer and Separation Processes*. ND: Prentice Hall of India Pvt. Ltd. pp 353-355, 376, 391, 401.





**ROTARY DRYER**  
**(With Data Logging Facility)**  
**(MT-943 RH)**

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# ROTARY DRYER

## 1. OBJECTIVE:

To study the operation of rotary dryer.

## 2. AIM:

- 2.1 To study the drying characteristics of a solid material in a rotary dryer.
- 2.2 To plot the rate of drying curve.

## 3. INTRODUCTION:

In many cases, the drying of materials is the final operation in the manufacturing process, carried out immediately prior to packaging or dispatch. Drying refers to final removal of water, & the operation often follows evaporation, filtration or crystallization. Drying is carried out for one or more of the following reasons:

- To reduce the transportation cost.
- To make a material more suitable for handling, for example soap powders, dyestuffs, fertilizers.
- To provide definite properties, such as maintaining free flowing nature of salt.
- To remove moisture; which may otherwise lead to corrosion, for example, the drying of coal gas or benzene prior to chlorination.

Almost all drying processes involve the removal of water by vaporization, & thus require the addition of heat.

The rotary dryer is a type of industrial dryer employed to reduce or minimize the liquid moisture content of the material by bringing it in direct contact with a heated gas .The dryer is made up of large rotating cylinder which slopes slightly so that the discharge end is lower than the material feed end in order to convey the material under gravity. Material to be dried enters the dryer and as the dryer rotates, the material is lifted up by a series of internal fins lining the inner wall of the dryer. Heating options include steam, gas, oil, thermal oil, and auxiliary biomass burner system.

#### 4. THEORY:

Consider a rotary continuous counter-current dryer fed with a nonporous material having all moisture as unbound moisture. As this material enters the dryer, it is first heated to the drying temperature. It will then pass through the length of dryer at nearly the wet-bulb temperature and theoretically at the end of the dryer, the material shall be discharged as dry material nearly at the wet bulb temperature.

Assumptions:

- No heat losses from the dryer.
- Heat is applied to the material only from the air, not by conduction from the dryer shell.
- All the moisture present is free moisture.
- There is no evaporation of moisture in the preliminary heating period.

Drying proceeds at a constant wet bulb temperature until desired amount of water has been removed. The entering air is assumed to be 100% saturated, so its temperature needs to be raised so as to decrease the relative saturation. This would enable the air to absorb moisture from the wet solid feed. In turn the exiting air is more saturated than the entering one.

For continuous dryer at steady state operating conditions,

$$F(X_1 - X_2) = G(Y_1 - Y_2) \quad \dots \quad (1)$$

This assume that the dry gas flow  $G$  and dry solids flow  $F$  do not change between dryer inlet and outlet. Mass balances can also be performed on the overall gas and solids entrainment in the exhaust gas stream.

The required solids flow rate, inlet moisture content  $X_1$  and outlet moisture  $X_0$  are normally specified, and the evaporation rate and outlet gas flow are calculated.

For batch dryer with a dry mass  $m$  of solids, a mass balance only gives a snapshot at one point during the drying cycle and an instantaneous drying rate given by:

$$m \frac{dX}{dt} = G(Y_1 - Y_2) \quad \dots \quad (2)$$

$$\frac{dX}{dt} = \frac{G(Y_1 - Y_2)}{m} \quad \dots \quad (3)$$

Sometimes  $\frac{dX}{dt}$  is called rate of drying.

## **5. DESCRIPTION:**

The set-up consists of a long resolving cylindrical shell slightly inclined toward the outlet. The shell is fitted with two brackets for support. Air from a blower passes through a heating chamber serves the purpose of drying agent. An arrangement is provided for rotating the dryer shell connected with electric motor and a reduction gear box. Flow control valve and by-pass valve are fitted to regulate the airflow.

The present set up has a facility to interface the system with computer which enables to log the experimental data using computer. The educational software and data-logging package has been developed per unit. This software is capable to tabulate the sample readings according to the requirement of experiment under study and results obtained can be compared.

## **6. UTILITIES REQUIRED:**

- 6.1 Electricity Supply: Single Phase, 220 V AC, 50 Hz, 32 Amp MCB with earth connection.
- 6.2 Floor Area Required: 3 m x 1.5 m.
- 6.3 Computer system: Pentium IV with DVD drive, Windows 7 MS-office pre loaded, One USB slot required in PC for DATA ACQUISITION CARD.
- 6.4 Solid material (Seeds 3-5 mm): 2 kg

## **7. INSTALLATION PROCEDURE OF SOFTWARE:**

Two DVDs are provided with the equipment. One DVD is of Device Driver and other DVD of Operating Software. First, place the device driver DVD in DVD-ROM and wait for 5 to 10 seconds. A blue-colored window appears on the screen. Install the software as per directions. When installation is finished, a shortcut would be created on the desktop showing name as “NI-Max”. Restart the computer system. Fix USB Card in empty USB slots. This ends the completion of loading of DAQ Card Driver. Now, take Operating Software DVD and place in DVD-ROM. Open DVD and open folder “Rotary Dryer.” and then double click on “Setup.exe”. Follow the instructions and do the installation.

Go to desktop and double click on “NI-Max”. A window opens and looks at left side. Follow the path as “My System” → “Devices and Interfaces” → Click on USB-6008:

"Dev1", Right click and rename it as "050" and press "enter". Now, it appears as USB-6008: "050". Close the window. After completion of installation "restart" the computer system.

## **8. METHOD OF OPERATING SOFTWARE:**

Open the EXE of selected condenser by clicking it from program Menu of main Menu bar. Enter Password "kce" for the software and press "enter" or click "OK". Click the "Interface" button to open interface of the software. In the entire software, a "NEXT" button is provided, at left front, which enables to go to next step. Another button named as "BACK" is present. This facilitates to retrieve last step taken. An "EXIT" button, at right bottom end, is present there. It may be used to exit at any session and finally, stopping the software.

**Note: In the absence of external hardware (USB-6008), the interfacing section may not able to work.**

## **9. EXPERIMENTAL PROCEDURE:**

### **9.1 STARTING PROCEDURE:**

- 9.1.1 Take known amount of the solid material and weigh it.
- 9.1.2 Now mix known amount of water in the solid and weigh the wet solid.
- 9.1.3 Ensure that switches given on the panel are at OFF position.
- 9.1.4 Set the preheating temperature for air.
- 9.1.5 Switch ON the main power supply.
- 9.1.6 Switch ON the heater & wait till the set point temperature is arrived.
- 9.1.7 Run the blower and set the flow rate of air by adjust the flow control valve for air.
- 9.1.8 Fill the feed hopper with wet solid.
- 9.1.9 Start the dryer in rotary motion.
- 9.1.10 Allow the wet solid to flow through the dryer by starting the screw conveyor and rotating hand wheel manually.
- 9.1.11 At steady state record the air temperature at inlet and outlet and velocity of air in computer interface.

9.1.12 Repeat the experiment for different air velocities.

9.1.13 Repeat the experiment for different air inlet temperature.

## 9.2 CLOSING PROCEDURE:

- 9.2.1 When experiment is over switch OFF the blower, heater and dryer.
- 9.2.2 Switch OFF the main power supply.
- 9.2.3 Clean the dryer properly.

## 10. OBSERVATION & CALCULATION:

### 10.1 DATA:

Area of the duct A	= 0.011 m <sup>2</sup>
Density of air $\rho_a$	= 1.21 kg/m <sup>3</sup>
Molecular weight of air $M_A$	= 29 g/mole
Molecular weight of water $M_W$	= 18 g/mole

### OBSERVATIONS:

### 10.2 OBSERVATION TABLE: 1

S.No.	T <sub>1</sub> (°C)	T <sub>2</sub> (°C)	T <sub>3</sub> (°C)	T <sub>4</sub> (°C)	V (m/sec)

### 10.3 CALCULATIONS:

$$G = V \times \rho_a \times A \quad (\text{kg/sec})$$

To calculate the humidity ( $y_1$  &  $y_2$ ) of air at temperature (T<sub>1</sub> & T<sub>2</sub>) and (T<sub>3</sub> & T<sub>4</sub>) respectively by psychometric chart.

$$y_1 = \underline{\hspace{10mm}}$$

$$y_2 = \underline{\hspace{10mm}}$$

$$Y_1 = y_1 \frac{M_w}{M_A} \text{ (kg of moisture/kg dry air)}$$

$$Y_2 = y_2 \frac{M_w}{M_A} \text{ (kg of moisture/kg dry air)}$$

$$N = G \times (Y_1 - Y_2) \text{ (kg of moisture/sec)}$$

**CALCULATION TABLE: 2**

S.No.	G (Kg/sec)	Y <sub>1</sub> (kg moisture/sec)	Y <sub>2</sub> (kg moisture/sec)	N (kg moisture/sec)

## 11. NOMENCLATURE:

Nom	Column Heading	Units	Type
A	Area of the dryer	m <sup>2</sup>	Given
G	Mass flow rate of air	kg/sec	Calculated
V	Velocity	m/sec	Measured
M <sub>A</sub>	Molecular weight of air	g/mole	Given
M <sub>w</sub>	Molecular weight of water	g/mole	Given
N	Rate of drying	kg moisture/sec	Calculated
T <sub>1</sub>	Inlet dry bulb temperature of air	°C	Measured
T <sub>3</sub>	Outlet dry bulb temperature of air	°C	Measured
T <sub>2</sub>	Inlet wet bulb temperature of air	°C	Measured
T <sub>4</sub>	Outlet wet bulb temperature of air	°C	Measured
Y	Absolute Humidity Difference	(kg of moisture / kg dry air)	Calculated
Y <sub>1</sub>	Absolute Humidity of air at the inlet of dryer	(kg of moisture / kg dry air)	Calculated
Y <sub>2</sub>	Absolute Humidity of air at the outlet of dryer	(kg of moisture / kg dry air)	Calculated

$y_1$	Moisture content of air at the inlet of dryer	kg of moisture / kg dry air)	Calculated
$y_2$	Moisture content of air at the outlet of dryer	kg of moisture / kg dry air)	Calculated
$\rho_a$	Density of air	kg/m <sup>3</sup>	Given

\* Symbols represent unitless quantity.

## 12. PRECAUTION & MAINTENANCE INSTRUCTIONS:

- 12.1 Proper oiling should be necessary for moving parts like roller.
- 12.2 Feed should be granular.
- 12.3 Before switching ON heater; switch ON the blower.

## 13. TROUBLESHOOTING:

- 13.1 If D.T.I display '1' on display board it means sensors connection are not OK tight it properly.
- 13.2 If switch of the heater is ON but temperature can't rise but panel LED is ON it means heater had burned replace it

## 14. REFERENCES:

- 14.1 Coulson, J M & Richardson, J F (1991). *Chemical Engineering Vol-2.* 4<sup>th</sup> Ed. ND: Asian Books Pvt. Ltd. pp 693-695, 707-708.
- 14.2 McCabe, Warren L. Smith, Julian C. Harriott, Peter (2005). *Unit Operations of Chemical Engineering.* 7<sup>th</sup> Ed. NY: McGraw-Hill. pp 804-812, 818-812.
- 14.3 Green, D.W. Perry, R.H. (2008). *Perry's Chemical Engineers' Handbook.* 8<sup>th</sup> Ed. NY: McGraw-Hill. p 12.27.

## PSYCHROMETRIC CHART

- 14.4 Green, D.W. Perry, R.H. (2008). *Perry's Chemical Engineers' Handbook.* 8<sup>th</sup> Ed. NY: McGraw-Hill. p 12.7.

## SIMPLE BATCH DISTILLATION

### 1. OBJECTIVE:

To study the process of simple batch distillation.

### 2. AIM:

2.1 To verify Rayleigh equation for batch distillation.

2.2 To study the characteristics of simple distillation.

### 3. INTRODUCTION:

Batch distillation is a process by which a mixture of volatile components is partially separated by evaporation and condensation. The feed mixture is initially charged into the still (boiler), heated and the vapour emerging from the still is condensed and collected as distillate. As the distillation proceeds, the liquid in the still becomes more concentrated in the less volatile component while the distillate tends to have a higher concentration of the more volatile component. The distillate can be collected as a whole or several fractions can be taken.

### 4. THEORY:

For simple batch distillation process, initially the still is filled with  $F$  moles of feed with  $x_F$  as the mole fraction of the more volatile component. Let at any moment the number of moles of liquid in the still be  $B$  having a mole fraction  $x$  of the more volatile component (i.e. A) and let the moles of accumulated condensate be  $D$ . Concentration of the equilibrium vapour is  $y^*$ . Over a small time, the change in the amount of liquid in the still is  $dB$  and the amount of vapour withdrawn is  $dD$ . The following differential mass balance equations may be written:

Total material balance:

$$-dB = dD \quad \dots \quad (1)$$

Component A balance:

$$-d(Bx) = y^* dD \quad \dots \quad (2)$$

On solving the above equation:

$$-Bdx - xdB = y^* dD$$

Put  $-dB = dD$  from eq<sup>n</sup> (1):

$$-Bdx = y^* dD - x dD$$

$$-Bdx = (y^* - x)dD$$

Again put  $dD = -dB$

$$Bdx = (y^* - x)dB \quad \dots \dots \dots \quad (3)$$

By arranging the above equation:

$$\frac{dB}{B} = \frac{dx}{y^* - x} \quad \dots \dots \dots \quad (4)$$

Where  $y^*$  is the vapour composition in equilibrium with liquid composition  $x$

Integrating Eq<sup>n</sup> (4):

$$\int_F^B \frac{dB}{B} = \int_{x_B}^{x_F} \frac{dx}{y^* - x} \quad \dots \dots \dots \quad (5)$$

Eq. (5) is the rayleigh equation, which describes the variation of the liquid and vap mole fractions,  $x, y$  in terms of the fraction remaining in the still.

$$\ln \frac{F}{B} = \int_{x_B}^{x_F} \frac{dx}{y^* - x} \quad \dots \dots \dots \quad (6)$$

We have:

$$y^* = \frac{\alpha x}{1 + (\alpha - 1)x}$$

For constant relative volatility,  $\alpha$  we can write the equation (6):

$$\ln \frac{F}{B} = \int_{x_B}^{x_F} \frac{dx}{\frac{\alpha x}{1 + (\alpha - 1)x} - x}$$

$$\ln \frac{F}{B} = \int_{x_B}^{x_F} \frac{dx}{(\alpha - 1)x(1 - x)} + \frac{dx}{(1 - x)} \quad \dots \dots \dots \quad (7)$$

By partial differentiation of the above equation:

$$\begin{aligned} \ln \frac{F}{B} &= \frac{1}{\alpha - 1} \left[ \ln \frac{x_F(1-x_B)}{x_B(1-x_F)} \right] + \ln \left[ \frac{(1-x_B)}{(1-x_F)} \right] \ln \frac{F}{B} - \ln \left[ \frac{(1-x_B)}{(1-x_F)} \right] = \frac{1}{\alpha - 1} \left[ \ln \frac{x_F(1-x_B)}{x_B(1-x_F)} \right] \\ \ln \left[ \frac{F(1-x_F)}{B(1-x_B)} \right] &= \frac{1}{\alpha - 1} \left[ \ln \frac{x_F(1-x_B)}{x_B(1-x_F)} \right] \\ \alpha \ln \left[ \frac{F(1-x_F)}{B(1-x_B)} \right] &= \left[ \ln \frac{x_F(1-x_B)}{x_B(1-x_F)} \right] + \ln \left[ \frac{F(1-x_F)}{B(1-x_B)} \right] \\ \alpha \ln \left[ \frac{F(1-x_F)}{B(1-x_B)} \right] &= \ln \left[ \frac{Fx_F}{Bx_B} \right] \quad \dots \dots \dots \quad (8) \end{aligned}$$

The above eq<sup>n</sup> is more convenient form of rayleigh equation.

### DESCRIPTION:

The set-up consists of a distillation still made of borosilicate glass. Mental heater is provided to heat the feed. Concentric tube type Condenser is provided to condensate the vapour. Rotameter is given to measure the flow rate of cold water. Temperature is controlled by the digital temperature controller. Receiver is given to collect the product.

The present set up has a facility to interface the system with computer which enables to log the experimental data using computer. The educational software and data-logging package has been developed per unit. This software is capable to tabulate the sample readings according to the requirement of experiment under study and results obtained can be compared.

### UTILITIES REQUIRED:

- 6.1 Electrical Supply: Single Phase, 220 V AC, 50 Hz, 5-15 Amp combined socket with earth connection.
- 6.2 Water Supply: Continous @ 4 LPM at 1 bar.
- 6.3 Floor Drain Required.
- 6.4 Computer system : Pentium IV with DVD drive, Windows 7, Ms-office 7 version pre loaded, one USB slot required in PC for DATA ACQUITION CARD.
- 6.5 Chemicals:

Methanol	:	4 Lit
Distilled water	:	15 Lit

## 7. INSTALLATION PROCEDURE OF SOFTWARE:

Two DVDs are provided with the equipment. One DVD is of Device Driver and other DVD of Operating Software. First, place the device driver DVD in DVD-ROM and wait for 5 to 10 seconds. A blue-colored window appears on the screen. Install the software as per directions. When installation is finished, a shortcut would be created on the desktop showing name as "NI-Max". Restart the computer system. Fix USB Card in empty USB slots. This ends the completion of loading of DAQ Card Driver. Now, take Operating Software DVD and place in DVD-ROM. Open DVD and open folder "Simple Batch distillation" and then double click on "Setup.exe". Follow the instructions and do the installation.

Go to desktop and double click on "NI-Max". A window opens and looks at left side. Follow the path as "My System" → "Devices and Interfaces" → Click on USB-6008: "Dev1", Right click and rename it as "051" and press "enter". Now, it appears as USB-6008: "051". Close the window. After completion of installation "restart" the computer system.

## METHOD OF OPERATING SOFTWARE:

Open the EXE of selected condenser by clicking it from program Menu of main Menu bar. Enter Password "kce" for the software and press "enter" or click "OK". Click the "Interface" button to open interface of the software. In the entire software, a "NEXT" button is provided, at left front, which enables to go to next step. Another button named as "BACK" is present. This facilitates to retrieve last step taken. An "EXIT" button, at right bottom end, is present there. It may be used to exit at any session and finally, stopping the software.

**Note: In the absence of external hardware (USB-6008), the interfacing section may not able to work.**

## 9. EXPERIMENTAL PROCEDURE:

### 9.1 STARTING PROCEDURE:

- 9.1.1 Prepare methanol water solution by mixing known amount of water and methanol.
- 9.1.2 The composition of methanol in solution should be in the range of 15 to 25 % by volume.
- 9.1.3 Note down the volume of water and methanol.
- 9.1.4 Close all the valves.
- 9.1.5 Ensure that switches given on the panel is at OFF position.
- 9.1.6 Fill the vessel with methanol-water solution by open the feed supply valve of funnel and air vent valve.
- 9.1.7 Connect electric supply to the set-up.
- 9.1.8 Set a process temperature for the process using the digital temperature controller (DTC). The temperature should be in the range of 85 to 95 °C.
- 9.1.9 Start the heater & cooling water pump.
- 9.1.10 Adjust the cooling water flow rate by control valve to a moderate value.
- 9.1.11 Wait 25-30 minute for the system to achieve steady state.
- 9.1.12 Now take out the samples from the bottom & distillate stream by open the valve for distillate collection and open the air vent valve of condenser.
- 9.1.13 Cool down the samples to room temperature & measure the refractive indices.
- 9.1.14 Record the Refractive indices in computer interface.
- 9.1.15 Repeat the experiment for different set point temperatures.

### 9.2 CLOSING PROCEDURE:

- 9.2.1 When experiment is over stop the water supply.
- 9.2.2 Switch OFF the cooling water supply and heater.
- 9.2.3 Switch OFF the main power supply.

## 10.2 OBSERVATIONS:

$$V = \text{_____ Lit.}$$

$$R.I_F = \text{_____}$$

$$R.I_B = \text{_____}$$

$$R.I_D = \text{_____}$$

## 10.2 OBSERVATION TABLE: 1

S.No.	$T_1$ ( $^{\circ}\text{C}$ )	$T_2$ ( $^{\circ}\text{C}$ )	$T_3$ ( $^{\circ}\text{C}$ )	$T_4$ ( $^{\circ}\text{C}$ )

## 10.3 CALCULATIONS:

To calculate the property of methanol ( $\rho$  &  $\alpha$ ) at temperature  $T_2$  ( $^{\circ}\text{C}$ ) from data book.

$$\rho = \text{_____} (\text{kg/m}^3)$$

$$\alpha = \text{_____}$$

$$x_F = \text{_____} \quad [\text{From R.I vs x graph, value of x corresponding to R.I}_F]$$

$$x_B = \text{_____} \quad [\text{From R.I vs x graph, value of x corresponding to R.I}_B]$$

$$x_D = \text{_____} \quad [\text{From R.I vs x graph, value of x corresponding to R.I}_D]$$

$$F = \frac{\rho V}{M \times 1000} \text{ (kmole)}$$

$$B = \frac{x_F - x_D}{x_B - x_D} \times F \text{ (kmole)}$$

$$E = \ln \left[ \frac{Fx_F}{Bx_B} \right] - \alpha \ln \frac{F(1-x_F)}{B(1-x_B)}$$

9.2.4 Drain the vessel by open the drain valve.

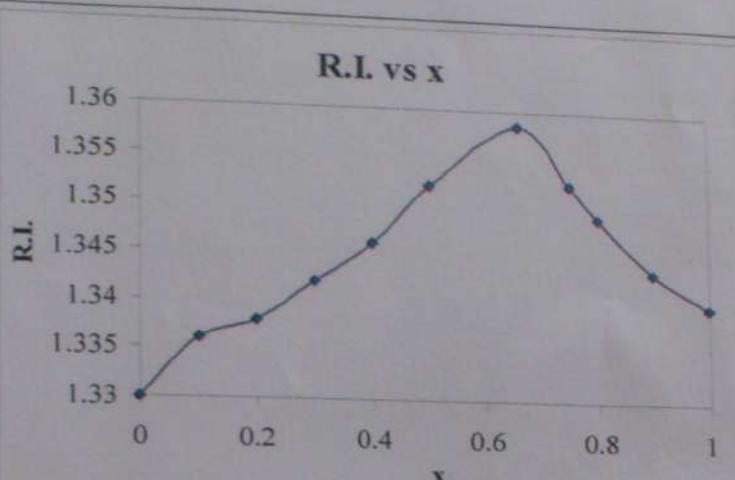
## 10. OBSERVATION & CALCULATION:

### 10.1.a DATA:1

Molecular weight of methanol M

= 32 g/mole

### 10.1.b DATA:2



$x_{\text{the}}$	R.I. (at 25°C)
0	1.33
0.1	1.3315
0.2	1.3325
0.3	1.3345
0.4	1.336
0.5	1.337
0.66	1.3375
0.75	1.337
0.8	1.3345
0.9	1.3305
1	1.3235

## 11. NOMENCLATURE:

Nom	Column Heading	Units	Type
B	Moles of methanol in residue	kmole	Calculated
E	Error	*	Calculated
F	Moles of methanol in feed	kmole	Calculated
M	Molecular weight of methanol	g/mole	Given
R.I <sub>B</sub>	Refractive index of residue sample	*	Measured
R.I <sub>D</sub>	Refractive index of distillate sample	*	Measured
R.I <sub>F</sub>	Refractive index of feed sample	*	Measured
T <sub>1</sub>	Temperature of vapours	°C	Measured
T <sub>2</sub>	Temperature of distillate	°C	Measured
T <sub>3</sub>	Temperature of cooling water inlet	°C	Measured
T <sub>4</sub>	Temperature of cooling water outlet	°C	Measured
V	Volume of methanol in feed	L	Measured
X <sub>F</sub>	Mole fraction of methanol in feed	*	Calculated
X <sub>B</sub>	Mole fraction of methanol in residue	*	Calculated
X <sub>D</sub>	Mole fraction of methanol in distillate	*	Calculated
α	Relative volatility	*	Calculated
ρ	Density of methanol	kg/m <sup>3</sup>	Calculated

\* Symbols are unitless.

## 12. PRECAUTION & MAINTENANCE INSTRUCTIONS:

- 12.1 Heater should not be switch ON before charging the solution in still.
- 12.2 Always drain the tanks and re-boiler after the end of experiment.
- 12.3 Always use clean water and ensure that there are no foreign particles in the flow stream.

## 13. TROUBLESHOOTING:

- 13.1 In case of any problem regarding operation of the apparatus; the apparatus should be quickly switch OFF and electric supply should be cut OFF.

- 13.2 Electric shock: It means that either earth wire has loose connection or there is no earth provided in the socket to which the equipment is plugged in. So make it sure that the equipment is earthed properly.
- 13.3 Leakage: The point of leakage should be detected & the concerned part is tightened properly. If the problem persists then that part should be remove & teflon tape is wrapped on the threads properly and refitted carefully.

#### 14. REFERENCES:

- 14.1 McCabe, Warren L. Smith, Julian C. Harriott, Peter (2005). *Unit Operations of Chemical Engineering*. 7<sup>th</sup> Ed. NY: McGraw-Hill. pp 724-727.
- 14.2 Dutta K. Binay (2007). *Principles of Mass Transfer and Separation Processes*. ND: Prentice Hall of India Pvt. Ltd. pp 353-355, 391.



**SIMPLE STEAM DISTILLATION  
(With Data Logging Facility)  
(MT-914 RH)**

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# SIMPLE STEAM DISTILLATION

## 1. OBJECTIVE:

To study the characteristics of steam distillation process using turpentine oil as a feed stock.

## 2. AIM:

- 2.1 To calculate the vaporizing efficiency of the steam distillation column.
- 2.2 To study the character sticks of steam distillation.

## 3. INTRODUCTION:

Steam distillation is a separation process in which live steam is blown through a liquid containing a component. When the component vaporizes slowly and leaves with the steam. The component is the target component which we want to recover reasonably pure state. The mixture to be separated may contain traces of a non-volatile impurity.

The feed is taken in the distillation vessel or still through which live steam is sparged at the bottom. The vapour containing the vaporized product as well as steam is led to a condenser. If the component has only little miscibility with water, the condensate forms two layers and can be easily separated to recover to product.

## 4. THEORY:

If the substance A is immiscible with water. The feed contains A and traces of non-volatile impurities. Let  $P_A^v$  be the vapour pressure of A at the operating temperature,  $P_B^v$  the vapour pressure of B (water), and P be the total pressure. Since A & B are essentially immiscible, the total pressure is the sum of the individual pressure.

$$P = P_A^v + P_B^v$$

If  $m_A$  moles of the substance are volatilized out by putting in  $m_B$  moles of water and if the system operates at equilibrium, we may write:

$$\frac{m_A}{m_B} = \frac{P_A^v}{P_B^v} = \frac{P_A^v}{P - P_A^v}$$



However, if the system does not operate at equilibrium, the partial pressure of A will be less than its vapour pressure. To take into account such a deviation, we define a factor called vaporizing efficiency E of the product such that the partial pressure of A in the steam phase is  $P_A = EP_A^v$ .

$$E = \frac{P}{P_A^v} \left( \frac{m_A}{m_A + m_B} \right)$$

## 5. DESCRIPTION:

The set-up consists of pressure vessel fitted with pressure sensor. Steam generator is provided to generate the steam. Condenser is given to condensate the vapour. Rotameter is provided to measure the flow rate. Steam trap is for releasing the pressure. Digital temperature indicator & digital temperature controller are provided to indicate and control the temperature. Digital level indicator is provided to indicate the level of distillate product.

The present set up has a facility to interface the system with computer which enables to log the experimental data using computer. The educational software and data-logging package has been developed per unit. This software is capable to tabulate the sample readings according to the requirement of experiment under study and results obtained can be compared.

## 6. UTILITIES REQUIRED:

- 6.1 Electrical Supply: Single Phase, 220 V AC, 50 Hz, 32 Amp MCB with earth connection.
- 6.2 Water Supply: Continuous @ 2 LPM at 1 bar.
- 6.3 Floor Drain Required.
- 6.4 Computer system: Pentium IV with DVD drive, Windows 7, MS-office 7 version pre loaded, One USB slot required in PC for DATA ACQUISITION CARD.
- 6.5 Chemicals:

Turpentine oil : 2 Lit.

Water : Known volume

## 7. INSTALLATION PROCEDURE OF SOFTWARE:

Two DVDs are provided with the equipment. One DVD is of Device Driver and other DVD of Operating Software. First, place the device driver DVD in DVD-ROM and wait for 5 to 10 seconds. A blue-colored window appears on the screen. Install the software as per directions. When installation is finished, a shortcut would be created on the desktop showing name as “NI-Max”. Restart the computer system. Fix USB Card in empty USB slots. This ends the completion of loading of DAQ Card Driver. Now, take Operating Software DVD and place in DVD-ROM. Open DVD and open folder “Simple steam distillation” and then double click on “Setup.exe”. Follow the instructions and do the installation.

Go to desktop and double click on “NI-Max”. A window opens and looks at left side. Follow the path as “My System” → “Devices and Interfaces” → Click on USB-6008: “Dev1”, Right click and rename it as “**052**” and press “enter”. Now, it appears as USB-6008: “052”. Close the window. After completion of installation “restart” the computer system.

## 8. METHOD OF OPERATING SOFTWARE:

Open the EXE of selected condenser by clicking it from program Menu of main Menu bar. Enter Password “kce” for the software and press “enter” or click “OK”. Click the “Interface” button to open interface of the software. In the entire software, a “NEXT” button is provided, at left front, which enables to go to next step. Another button named as “BACK” is present. This facilitates to retrieve last step taken. An “EXIT” button, at right bottom end, is present there. It may be used to exit at any session and finally, stopping the software.

**Note: In the absence of external hardware (USB-6008), the interfacing section may not able to work.**

## 9. EXPERIMENTAL PROCEDURE:

### 9.1 STARTING PROCEDURE:

- ✓ 9.1.1 Take known amount of turpentine oil and note down the volume of it.
- 9.1.2 Take known amount of water, note down the volume of it & mix it with turpentine oil.
- 9.1.3 Close all the valves.

- 9.1.4 Ensure that ON/OFF switches given on the panel are at OFF position.
- 9.1.5 Fill water in the steam generator by open the valve of funnel and air vent valve of steam generator and close them.
- 9.1.6 Charge the distillation still with turpentine oil & water mixture by open the valve for feed inlet to water.
- 9.1.7 Switch ON the main power supply.
- 9.1.8 Set the temperature of steam generator with the help of DTC (approx. 115°C).
- 9.1.9 Switch ON the heater.
- 9.1.10 Wait for the steam formation.
- 9.1.11 As steam formed, open the steam supply valve of steam generator.
- 9.1.12 Start the flow of live steam by open the steam supply valve for vessel.
- 9.1.13 Connect cold water supply to condenser.
- 9.1.14 Note down the live steam pressure.
- 9.1.15 Collect the condensed vapors in glass separating chamber.
- 9.1.16 Continue the distillation process for sufficient time (approx 1 hr).
- 9.1.17 Stop the steam supply and collect the distillate in separate chamber.
- 9.1.18 Wait for the formation of two layers.
- 9.1.19 Record the total level of two layers formed in computer interface.
- 9.1.20 Collect the separate layer of turpentine oil in measuring cylinder.
- 9.1.21 Note down the level of remaining water in separate chamber.

## 9.2 CLOSING PROCEDURE:

- 9.2.1 When experiment is over stop the water supply.
- 9.2.2 Switch OFF the heater.
- 9.2.3 Switch OFF the main power supply.
- 9.2.4 Drain the vessel by open the drain valve of vessel.

9.2.5 Drain the steam generator by open the drain valve.

## 10. OBSERVATION & CALCULATION:

10.1 DATA:	
Molecular weight of turpentine oil $M_A$	= 136 g/mole
Molecular weight of water $M_B$	= 18 g/mole
Density of Turpentine oil $\rho_A$	= 871 Kg/m <sup>3</sup>
Density of water $\rho_B$	= 1000 Kg/m <sup>3</sup>

### OBSERVATIONS:

$$V_A = \underline{\hspace{2cm}} \text{ Lit.}$$

$$V_B = \underline{\hspace{2cm}} \text{ Lit.}$$

10.2 OBSERVATION TABLE: 1					
S.No.	T <sub>1</sub> (°C)	T <sub>2</sub> (°C)	T <sub>3</sub> (°C)	P°(kg/cm <sup>2</sup> )	W <sub>AD</sub> (Kg)

### 10.3 CALCULATIONS:

$$W_A = \frac{\rho_A \times V_A}{1000} \text{ (kg)}$$

$$m_A = \frac{W_A}{M_A} \text{ (kmole)}$$

$$W_B = \frac{\rho_B \times V_B}{1000} \text{ (kg)}$$

$$m_B = \frac{W_B}{M_B} \text{ (kmole)}$$

$$P_A = 13.84793 - \frac{3377.754}{62.817 + (T_1 + 273.15)} \text{ (mm of Hg)}$$

$$E = \frac{(P^o + 1.01325)}{P_A \times 0.00133} \left[ \frac{m_A}{m_A + m_B} \right] \times 100 \text{ (%)}$$

## 11. NOMENCLATURE:

Nom	Column Heading	Units	Type
E	Vaporization efficiency	%	Calculated
M <sub>A</sub>	Molecular weight of turpentine oil	g/mole	Given
M <sub>B</sub>	Molecular weight of water	g/mole	Given
m <sub>A</sub>	Mole of turpentine oil	kmole	Calculated
m <sub>B</sub>	Mole of water	kmole	Calculated
ρ <sub>A</sub>	Density of turpentine oil	kg/m <sup>3</sup>	Calculated
P <sub>A</sub>	Vapour pressure of turpentine oil	mm of Hg	Calculated
ρ <sub>B</sub>	Density of water	kg/m <sup>3</sup>	Calculated
P <sup>o</sup>	Pressure of steam in still	kg/cm <sup>2</sup>	Measured
T <sub>1</sub>	Temperature of solution	°C	Measured
T <sub>2</sub>	Temperature of cooling water inlet	°C	Measured
T <sub>3</sub>	Temperature of cooling water outlet	°C	Measured
V <sub>A</sub>	Volume of turpentine oil	L	Measured
V <sub>B</sub>	Volume of water	L	Measured
W <sub>A</sub>	Weight of turpentine oil	kg	Calculated
W <sub>AD</sub>	Weight of turpentine oil in distillate	kg	Calculated
W <sub>B</sub>	Weight of water	kg	Calculated

## 12. PRECAUTION & MAINTENANCE INSTRUCTIONS:

- 12.1 Never run the apparatus if power supply is less than 180 volts & more than 230 volts.
- 12.2 Don't switch ON the heater before filling water in the steam generator.
- 12.3 For condenser the cold water supply should be constant.
- 12.4 Feed should be fill-up in the vessel at least up-to the mark.

### 13. TROUBLESHOOTING:

- 13.1 If any type of suspended particles is come in the rotameter, remove the rotameter, clean the tube and fit it at its place.
- 13.2 If there is any leakage tight that part or fix it again after wrapping teflon tape.
- 13.3 If rotameter fluctuating more than average tight the control knob properly.
- 13.4 If D.T.C display '1' on display board it means sensors connection are not proper tight it properly.
- 13.5 If heater is switch ON, but temperature can't rise but panel LED is ON it means bath heater had burned replace it.

### 14. REFERENCES:

- 14.1 Dutta K. Binay (2007). *Principles of Mass Transfer and Separation Processes*. ND: Prentice Hall of India Pvt. Ltd. pp 327-329, 350-353, 391.

## **SOLID-LIQUID EXTRACTION (BONNOTTO TYPE) (MT-104)**

## ***Foreword***

*Welcome to the fast growing family of K.C. product owners. We appreciate your interest in us and thank you for buying our product.*

*You have chosen the finest quality product in the market which is produced using latest techniques and has underwent strict quality control tests. It is a product that we are proud to build and you are proud to own it.*

*Our products are easy to understand and operate. They are excellent for students who are trying to gain practical knowledge through experiments.*

*However your comfort and safety are important to us, so we want you have an understanding of proper procedure to use the equipment. For the purpose, we urge you to read and follow the step-by-step operating instructions and safety precautions in this manual. It will ensure that your favourite product delivers reliable, superior performance year after year.*

*This manual includes information for all options available on this model. Therefore, you may find some information that does not apply to your equipment.*

*All information, specifications and illustrations in this manual are those in effect at the time of printing. We reserve the right to change specifications or design at any time without notice.*

*Customer satisfaction is our primary concern. Feel Free to contact us for any assistance. So what are you waiting for, roll up your sleeves and let us get down to work!*

**K.C. Engineers Pvt. Ltd.**

## *Important Information About This Manual*

### *Reminder for Safety*

#### *Modification on Equipment:*

*This equipment should not be modified. Modification could affect its performance, safety or disturbance. In addition damage or performance problems resulting from modification may not be covered under warranties.*

#### *Precautions and Maintenance:*

*This is used to indicate the presence of a hazard that could cause minor or moderate personal injury or damage to your equipment. To avoid or reduce the risk, the procedures must be followed carefully.*

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# **SOLID-LIQUID EXTRACTION UNIT**

## **(BONNOTTO TYPE)**

### **1. OBJECTIVE:**

To study the solid liquid extraction in a column with solid in batch & liquid in continuous recirculation

### **2. AIM:**

- 2.1 To calculate the percentage recovery of oil from oil seed.
- 2.2 To show the effect of solvent temperature and solvent rate on the percentage recovery of oil from oil seeds.

### **3. INTRODUCTION:**

Leaching is a method of extracting a desired chemical that is dispersed or dissolved in a solid. The solid is ground up into small pieces and "washed" with a solvent in which the desired chemical is soluble. Sometimes the solid and solvent form slurry that can be conveyed. In any event the desired chemical is transferred from the solid to the liquid phase. The resultant liquid, which is rich in the desired chemical, is filtered to remove fine suspended grains of the solid. The liquid can then be distilled or otherwise purified.

### **4. THEORY:**

Leaching is a common industrial process, but it also occurs in natural systems, such as in the motion of water through soil. In this case, the solid phase is soil particles, and the leaching solvent is water, presumably rainwater that percolates through the soil. Some chemicals attached to the soil particles are water-soluble. These chemicals are transferred to the water as it flows through the soil. Such a process is sometimes called in-site leaching, because it occurs with the solid phase fixed in place. This laboratory experiment uses simple equipment to explore a leaching operation that is familiar to all of us the extraction of oil from oil seeds using hexane as the leaching solvent.

We will examine quantitatively a few of the variables that affect the leaching process.

- a) Size of particle: Smaller size particles provides greater interfacial area between the solid and liquid which in turn results in higher rate of transfer of material. However the particle size should not be too small, otherwise separation of solid from liquid and drainage of solid residue will become more difficult.

- b) Temperature: High temperature favours the leaching operation.
- c) Solvent flow rate: It should be not too less or high for effective extraction.

## **5. DESCRIPTION:**

The column consists of slotted circular plates arranged one above the other in opposite radial opening. The solids are introduced at the top through a hopper spread over the top plate by a rack and pushed to slot to fall to the next plate below and so on to the bottom of the column. The solvent is introduced near the bottom of the column and it flows upward counter-currently to the solids. A pump supplies the solvent and flow rate is monitored by means of a rotameter. At the bottom of column a screw conveyor arrangement is done to remove the extracted solids driven by means of an electric motor. Solvent feed tank is kept in a bath fitted with heater and digital temperature controller is used to maintain the constant temperature.

## **6. UTILITIES REQUIRED:**

- 6.1 Electricity Supply: Single Phase, 220 V AC, 50 Hz, 5-15 Amp combined socket with earth connection.
- 6.2 Floor Drain Required.
- 6.3 Oven for drying solid.
- 6.4 Chemicals:-

Hexane : 50 lit.

Expeller waste/Oil seeds : 10 kg

## **7. EXPERIMENTAL PROCEDURE:**

### **7.1 STARTING PROCEDURE:**

- 7.1.1 Prepare mixture of 100 gm of expeller waste and 1 liter of hexane.
- 7.1.2 After one hour filter the expeller waste, dry and weigh it.
- 7.1.3 The loss in the weight is the initial oil content.
- 7.1.4 Fill known amount of solid in the feed hopper.
- 7.1.5 Fill hexane in the solvent tank.

- 7.1.6 Fill water in the water tank for heating.
- 7.1.7 Set desired temperature of hexane by DTC.
- 7.1.8 Note down the temperature.
- 7.1.9 Connect electric supply to the set-up.
- 7.1.10 Rotate hand wheel manually and pass solid into column.
- 7.1.11 Switch ON the heater.
- 7.1.12 After required temperature achieved switch ON the pump.
- 7.1.13 Allow hexane to flow through column at particular flow rate by adjust control valve  $V_1$  and by pass valve  $V_2$ .
- 7.1.14 Note down the flow rate.
- 7.1.15 Run the experiment for three hour and then switch OFF the pump.
- 7.1.16 Collect the wet solids in the tray, dry it in the air first and then in oven till constant weight and measure the weight of dry solid.
- 7.1.17 Repeat the experiment for different flow rate of hexane.
- 7.1.18 Repeat the experiment for different desired temperature.

## 7.2 CLOSING PROCEDURE:

- 7.2.1 When experiment is over switch OFF the heater.
- 7.2.2 Switch OFF the pump.
- 7.2.3 Switch OFF the main power supply.
- 7.2.4 Drain the solvent tank by open the valve  $V_3$ .
- 7.2.5 Drain the water from water tank by open the valve  $V_4$ .

## 8. OBSERVATION & CALCULATION:

### 8.1 DATA:

Initial weight of expeller waste $W_i$	= _____ gm
Weight of expeller waste in column $W_o$	= _____ gm

## 8.2 OBSERVATIONS:

T = \_\_\_\_\_ °C

Q = \_\_\_\_\_ LPH

W<sub>f</sub> = \_\_\_\_\_ gm

W = \_\_\_\_\_ gm

## 8.3 CALCULATIONS:

$$X_o = \frac{W_i - W_f}{W_i}$$

$$O_e = W_o - W \text{ (gm)}$$

$$O_{\max} = W_o X_o \text{ (gm)}$$

$$R = \frac{O_e}{O_{\max}} \times 100 \text{ (%)}$$

<b>CALCULATION TABLE:</b>			
S.No.	T (°C)	Q (LPH)	R (%)

## 9. NOMENCLATURE:

Nom	Column Heading	Units	Type
O <sub>e</sub>	Oil recovered	gm	Calculated
O <sub>max</sub>	Maximum oil content in oil seed	gm	Calculated
Q	Flow rate of hexane	LPH	Measured
R	Percentage recovery	%	Calculated
T	Temperature	°C	Measured
W	Weight of exhausted oil seeds	gm	Measured

$W_f$	Weight of expeller waste after drying	gm	Measured
$W_i$	Initial weight of expeller waste	gm	Given
$W_o$	Weight of expeller waste in column	gm	Given
$X_o$	Initial concentration of oil in oil seeds	*	Calculated

\* Symbols represent unitless quantity.

## 10. PRECAUTION & MAINTENANCE INSTRUCTIONS:

- 10.1 Always use the fresh oil seed or expeller waste that should be contain some oil.
- 10.2 Hexane should be oil free or not used.
- 10.3 Don't switch ON the heater before filling water in the water tank.
- 10.4 Proper cleaning should be necessary for the set-up.

## 11. TROUBLESHOOTING:

- 11.1 If temperature is not rising, but heater is ON, it means there is some loose connection, tight it properly.

## 12. REFERENCES:

- 12.1 Coulson, J M & Richardson, J F (1991). *Chemical Engineering Vol-2. 4<sup>th</sup> Ed.* ND: Asian Books Pvt. Ltd. pp 395-399.
- 12.2 Dutta K. Binay (2007). *Principles of Mass Transfer and Separation Processes.* ND: Prentice Hall of India Pvt. Ltd. p 485.

### 13. BLOCK DIAGRAM:

