



FEDERAL POLYTECHNIC NASARAWA
DEPARTMENT OF CHEMICAL ENGINEERING TECHNOLOGY



**CHEMICAL
ENGINEERING**
LABORATORY V

HIGHER NATIONAL DIPLOMA (HND) II

2024/2025 ACADEMIC SESSION

ADDRESS:

Department of Chemical Engineering Technology
School of Engineering Technology
Federal Polytechnic Nasarawa
PMB 001, Nasarawa
Nasarawa State
www.fpn.edu.ng/chet
www.fpn.edu.ng/set
engineeringtech@fpn.edu.ng
chetfpn@fpn.edu.ng
Tel: 081 40976963



MISSION STATEMENT

"Molding students to become disciplined, skilled and innovative for self-actualization and national development"

Goals of Chemical Engineering Department

A Department that is known for academic excellence, highly dynamic staff and a corporate outlook that serves as a world-class centre for the training of Chemical Engineering professionals and academics for Nigerian establishments and beyond.

CHEMICAL ENGINEERING LABORATORY PERSONNEL



Dr. SALIHU S. MAIWALIMA
(MNSChE, MNSE, MSPE, GRADIGEM(UK), COREN)

Head of Department

Tel: 07030699569

Email: ssalihum@gmail.com
suleimansalihum@fedpolynas.edu.ng



ABDULAZEEZ A. ABDULAZEEZ
(Sub Head of Department)

CHIEF LABORATORY COORDINATOR

Tel: 08140976963

Email: azbinbaz@gmail.com
abdulazeezaabdulazeez@fedpolynas.edu.ng



ALIYU ABDULQADIR
MNSChE, MNSE

Assistant Lab Coordinator

Tel: 08069153662

Email:
abdulqadiraliyu@fedpolynas.edu.ng
aliyuabdulqadir@gmail.com



ALIYU S. FATIMA

MNSChE, MNSE,
Assistant Lab Coordinator

Tel: 07062639198

Email: Fatynnnhamza@gmail.com



JIBRIL ABUBAKAR AHMAD
Assistant Lab Coordinator

Tel: 08054543261

Email: jibrila02@gmail.com
jibrilabubakarahmad@fedpolynas.edu.ng



MUHAMMAD YASIR
Assistant Lab Coordinator

Tel: 08068684298

Email: yaakzu@gmail.com



SADIQ JOSEPH
Assistant Lab Coordinator

Tel: 08069123122

Email: saddypa247@gmail.com
sadiqjoseph@fedpolynas.edu.ng



IBRAHIM D IBRAHIM
Technician

Tel: 08038331333

Email: ibrahimd.ibrahim65@gmail.com
ibrahimdibrahim@fedpolynas.edu.ng

LABORATORY PERSONNEL

SN	PERSONNEL	DESIGNATION	PR. CODE	PHONE NUMBER
1	ABDULAZEEZ A. ABDULAZEEZ	Chief Coordinator	LAB01	08140976963
2	ALIYU ABDULQADIR	Asst. Coordinator	LAB02	08069153662
3	FATIMA S. ALIYU	Asst. Coordinator	LAB03	07062639198
4	JIBRIL A. AHMAD	Asst. Coordinator	LAB04	08054543261
5	MUHAMMAD YASIR	Asst. Coordinator	LAB05	08035965974
6	SADEEQ JOSEPH	Asst. Coordinator	LAB06	08068684298
	IBRAHIM D IBRAHIM	Technician	LAB07	08038331333
7	ALIYU IBRAHIM	Lab Attendant	LAB08	
8	CORP MEMBER(s)		LAB09	

DEPARTMENTAL LABORATORIES

- Physical Transport Phenomenal (PTP) Laboratory (Fluid Mechanics/ Heat and Mass Transfer
- Unit Operation Laboratory (UOP)/ Chemical Reaction Engineering Laboratory
- Solid Minerals Process Laboratory/Strength of Materials Lab.
- Computer Process Simulation Laboratory

Laboratories according to courses

Name of Laboratory: Mass Transfer Operations

S/ No	Name of Equipments
1	Wetted wall column
2	Experimental cooling water tower system
3	Fluid bed dryer
4	Liquid-liquid extraction unit
5	Gas absorption in sieve plate column
6	Packed bed distillation column
7	Gas Chromatograph
8	UV-Visible Spectrophotometer
9	Ultrasonic Probe
10	Liquid Diffusion Apparatus
11	Gas Diffusion Apparatus
12	Solid-liquid Extraction Unit
13	Sedimentation Tank
14	Solid conveying system
15	Soxhlet apparatus
16	Batch distillation column
17	Fluid Mixing Apparatus
18	Packed absorption system

Name of Laboratory: Heat Transfer Operations

S/N	Name of Equipment
1	Tray Dryer
2	Heat conduction Apparatus and its service unit
3	Parallel & Counter Current Heat Exchanger
4	Shell and Tube Heat exchanger
6	Fixed and Fluidized bed system
7	Centrifugal Pump Test Rig
8	Convective Heat Transfer Apparatus
9	Radiative Heat Transfer Apparatus

Name of Laboratory: Fluid and Particles Mechanics

S/N	Name of Equipment
1	Basic Hydraulics Bench
2	Bernoulli's Theorem Apparatus
3	Reynolds Apparatus
4	Thermos Fluid tutor
5	Fluid Friction Apparatus
6	Fluid Mixing Apparatus
7	Model Sedimentation Tank

Name of Laboratory: Instrumentation and Process Control

Sr. N	Name of Equipment
1	Multi Process Control System Pressure Control Trainer Temperature Control Trainer Level Control Trainer Flow Control Trainer

2	Thermocouple
3	PID controller
	Batch Reactor

Name of the Laboratory: Chemical Reaction Engineering

SN	Name of the Equipment
1.	Gas Chromatograph (Thermo Scientific)
2.	UV-Vis Spectrophotometer (Thermo Scientific)
3.	UV-Vis Spectrophotometer (Hach)
4.	Enzyme catalytic reactor
5.	Continuous Stir tank Reactor
6.	Kinematic Viscometer Bath
7.	Incubator
8.	Laboratory Centrifuge

Name of Laboratory: Corrosion and Material Science

Sr. No	Name of the Equipment
1.	Corrosion studies Kit

Name of Laboratory: Computer Simulation Lab

Software available:

- ASPEN HYSYS
- DWSIM
- ProSIM
- CHEMCAD
- MATHCAD
- MATHLAB
- SPRSS Analysis Package
- VISIO
- AutoCAD
- Microsoft Office SUITE

COURSE TITLE: CHEMICAL ENGINEERING LABORATORY V

COURSE CODE: CHE 409

LEVEL: 400 SEMESTERS: First semester

General Objective 1.0: Know how to carry out experiments on instrumentation and process control, simultaneous heat and mass transfer, chemical reaction engineering and unit operations using engineering equipment and pilot plants

HEAT TRANSFER II AND UNIT OPERATION

EXPERIMENT 1: Determination of drying characteristics of a solid material under batch drying condition.

COOLING TOWER

EXPERIMENT 2: Study of the heat & mass transfer in Water Cooling Tower for different flow & thermodynamic conditions.

CHEMICAL REACTION ENGINEERING II

ISOTHERMAL OPERATION

EXPERIMENT 3: Determination of Reaction rate constant in a stirred batch reactor

EXPERIMENT 4: Study of the effect of reactant concentration on the reaction rate

ADIABATIC OPERATION

EXPERIMENT 5: To determine the rate equation for the hydrolysis of acetic anhydride to acetic acid in an adiabatic reactor.

CONTINUOUS STIRRED TANK REACTOR (CSTR)

EXPERIMENT 6: Determination of reaction rate constant for saponification of ethyl-acetate with NaOH at ambient conditions.

MULTI-FUNCTIONAL PLATE DISTILLATION COLUMN

EXPERIMENT 7: Study of basic principles of SIEVE PLATE Distillation

pH MEASUREMENT AND ITS APPLICATIONS

Experiment 8:

A: To measure the pH of various solutions using pH indicators and meter. B: To determine the value of K_a for an unknown acid. And

C: To perform a pH titration (OPTIONAL, if time permits)

PROCESS CONTROL AND INSTRUMENTATION

- PRESSURE PROCESS CONTROLLER
- LEVEL PROCESS CONTROLLER
- TEMPERATURE PROCESS CONTROLLER
- STUDY OF COMPLEX CONTROL SYSTEM USING MATLAB

Experiment 09: To study the performance of ON-OFF/P/PI/PD/PID controllers on Pressure process.

Experiment 10: To study the performance of ON –OFF/P/PI/PD/PID controllers on level process
Experiment 13: To study the performance of ON –OFF/P/PI/PD/PID controllers on flow process

Experiment 11: To study the performance of ON-OFF/P/PI/PD/PID controllers on temperature process.

Experiment 12: To study the complex control system using matlab and to compare the response of simple and cascade loop.

HEAT TRANSFER: DRYING OPERATION: TRAY DRIER

EXPERIMENT 1: Determination drying characteristics of a solid material under batch drying condition.

AIM:

- ❖ To study the drying characteristics of a solid material under batch drying condition.

OBJECTIVE:

- ❖ The rate of drying curve for a tray dryer.
- ❖ The effect of velocity of air on drying rate of the sample by performing drying process.
- ❖ To learn how to operate the tray dryer.
- ❖ To compare tray dryers with other types of batch dryers

INTRODUCTION:

Dryers are used to remove liquids or moisture from bulk solids, powders, parts, continuous sheets or other liquids by evaporation or sublimation. Dryers can be broken up into two main types: direct and indirect. Direct dryers convectively heat a product through direct contact with heated air, gas or a combusted gas product. Indirect dryers conductively heat a product through contact with a heated wall. Tray Dryer is used for the best drying results in conventional process. It is a double walled cabinet with Single or Two doors. The gap between two walls is filled with high density fiber glass wool insulation material to avoid heat transfer. Doors are provided with gaskets. Stainless steel trays are placed on the movable trolleys. Tray Dryer is provided with control panel board, process timer, Digital temperature controller cum indicator etc.

THEORY:

Drying of solids is considered to occur in two stages, a constant rate period followed by a falling rate period. In the constant rate period, the rate of drying corresponds to the removal of water from the surface of the solid. The falling rate period corresponds to the removal of water from the interior of the solid. The rate in either case is dependent on:

- ❖ Air wet and dry bulb temp.
- ❖ Flow rate of air
- ❖ The solid characteristics
- ❖ Tray material

Drying can be described in terms of gas mass transfer and heat transfer co-efficient. The rate of drying is given by:

$$q = h_c (T_g - T_s) + h_r (T_r - T_s) + h_k (T_g - T_s) \quad \text{---(2)}$$

Where h_c , h_r , h_k are the heat transfer coefficients for convection, radiation and conduction respectively. These coefficients are given by:

$$h_c = 14.3 G^{0.8} \quad \text{----- (3)}$$

$$h_r = \epsilon \sigma (T_r^4 - T_s^4) / (T_r - T_s) \quad \text{.....(4)}$$

$$h_k = \left[\frac{l}{h_c} \frac{A}{A_U} + \frac{Z_m}{k_m} \frac{A}{A_U} + Z_s \frac{k}{k_s} \frac{A}{A_m} \right]^{-1} \dots\dots\dots(5)$$

From equation 1 and 2 we get

$$\frac{(Y_s - Y_s)\lambda_s}{(h_c/(k_y))} = (1 + h_k/h_c)(T_g - T_s) + h_r/h_c(T_r - T_s) \dots\dots\dots(6)$$

Ratio of h_c/k_y = Lewis number (Le) and is given by:

$$\frac{h_c}{k_y C_s} = (S_c/P_r)^{0.567} = Le^{0.567} \dots\dots\dots(7)$$

C_s = specific heat of saturated gas at T_s .

For an air-water system $Le=1$. Eq. (3) & (7) yields h_c & k_y respectively.

Simultaneously iterative solution of Eq. (6) with saturation humidity curve provides the solid surface temp. (T_s) and the corresponding value of humidity, Y_s .

Knowing Y_s , eq. (1) is used to predict the theoretical drying rate, N_c . The absolute air humidity Y is measured experimentally from the dry and wet bulb temperatures.

DESCRIPTION:

The set up consists of a Stainless-Steel Chamber which is insulated by puff insulation to prevent it from heat loss. One temperature controller is fitted to maintain the temperature in the chamber. A plate and weight balance is provided for measurement of weight.

EXPERIMENTAL PROCEDURE:

1. Load the pre-weighed tray with solid (e.g., saw dust or sand).
2. Place it over the weighing balance.
3. Record the weight of sand & tray.
4. Set the temperature of chamber with the help of temp. controller.
5. Keep the tray gentle in the drying chamber and start the stopwatch.
6. Record the balance reading with time at about 3-5 min interval.
7. Similarly note down the readings in every 5-minute interval.
8. Drying is assumed to be complete when at least 3 consecutive readings are same.
9. Switch off the main power supply.

OBSERVATION & CALCULATIONS:

Solid dry wt. = S, kg

Initial moisture content = x

Table 1: Raw data of Sample

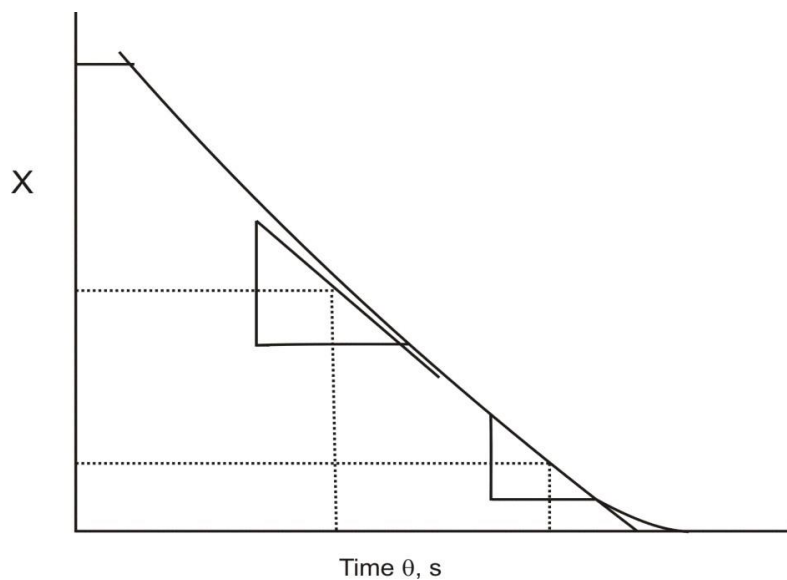
Time (mins)	Mass of tray + solid (g)	Mass of solid (g)

TABLE 2

Time (minutes)	Mass of tray + solid (g)	Mass of solid (g)	% Moisture content	Drying rate
0				
5				
10				
15				
20				
25				
30				
35				
40				
45				

Plot X Vs θ and draw a straight line through all the points. Fit a second-degree polynomial to the X vs θ data obtain the slope $dX/d\theta$ corresponding to various of θ .

Diagram



Plot drying rate N ($\text{kg}/\text{m}^2\text{-s}$) Vs moisture content X (kg of water/ kg of dry solid (Drying curve) From this plot critical moisture (X_c) content is obtained.

Calculations:

Mass of dry solid = mass of final product – mass of equilibrium-moisture*

*assume that the equilibrium-moisture content is 0.05 g water/g dry solid

% Moisture content at time t = $[(\text{mass of solid at time } t - \text{mass of dry solid})/\text{mass of dry solid}] \times 100$

solid] X 100

Rate of drying = - (moisture content at time t2 – moisture content at time t1)/(t2 – t1) min

NOMENCLATURE:

A = drying surface area, m²

N_c = constant drying rate, kg/ m²- s

Θ = time, s

kg/m³

X =moisture constant of a solid (kg of water/kg of dry solid)

N = drying rate, kg/m²-s

S = mass of dry solid (kg)

M = viscosity kg/m-s ρ = density

PRECAUTIONS & MAINTENANCE INSTRUCTIONS:

- ❖ Measure the exact volume of water and weigh the Mixture.
- ❖ Always use clean water and good quality saw dust for mixing.
- ❖ Use electronic balance for weighing of mixture.
- ❖ Keep close the front door if chamber while working.
- ❖ Don't ON heater switch before putting the mixture in chamber.

COOLING TOWER

EXPERIMENT 2: Study of the heat & mass transfer in Water Cooling Tower for different flow & thermodynamic conditions.

Objectives:

To find out tower characteristics:

- ❖ Range
- ❖ Approach
- ❖ Water to air mass flow ratio (L/G)
- ❖ Effectiveness of the tower
- ❖ Evaporation losses
- ❖ Percentage evaporation losses
- ❖ Blow down losses
- ❖ Drift losses
- ❖ Make up water requirements

INTRODUCTION:

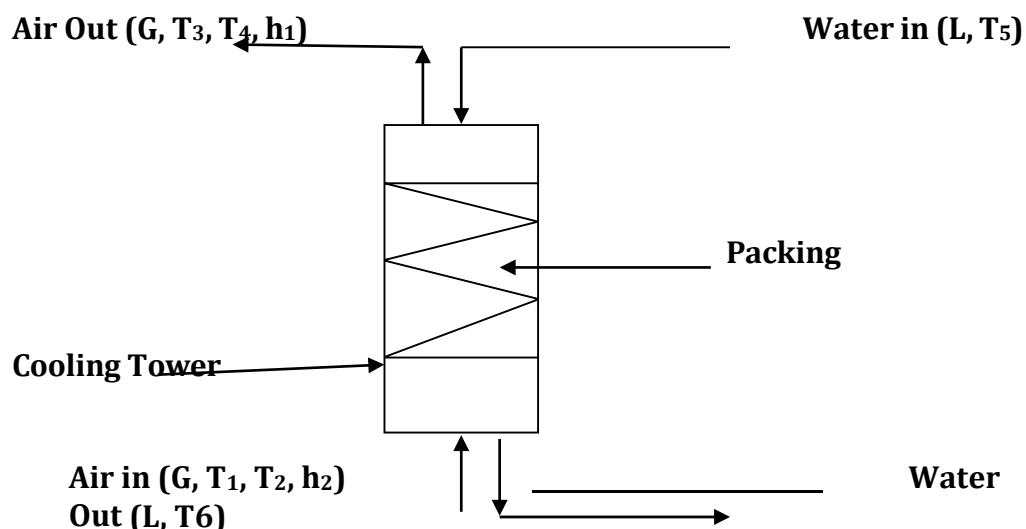
Water from condensers and heat exchangers is usually cooled by an air stream in spray ponds or in Cooling Towers using natural draft or forced flow of the air. Mechanical draft towers are of the forced draft type, where the air is blown into the tower by a fan at the bottom. The forced draft materially reduces the effectiveness of the cooling.

THEORY:

Water may be cooled by the air as long as its temperature is above the wet bulb temperature of the entering air. In a counter flow design, the air flow is directly opposite to the water flow. Air flow first enters an open area beneath the fill media, and is then drawn up vertically. The water is sprayed through pressurized nozzles near the top of the tower, and then flows downward through the fill, opposite to the airflow.

Advantages of the counter flow design:

- ❖ Spray water distribution makes the tower more freeze-resistant.
- ❖ Breakup of water in spray makes heat transfer more efficient



The carrying of liquid with the gas stream is termed as **Liquid Entrainment**. This may be due to a high rate of air flow. This should be avoided to get better performance. This can be avoided by following the Operational limits of the equipment.

DESCRIPTION:

The apparatus is provided for the process of Forced draft countercurrent cooling of hot water using air. The water to be cooled is heated in a heating tank using a heater. It is then circulated; through a rotameter; to the top of the cooling tower mounted over the heating tank. Cooled water is then re-circulated to the heating tank. A blower is provided for the cooling air. Speed of blower can be regulated with the help of variable speed drive(VSD). A valve is provided in airline to regulate the flow rate of air. There is an orifice meter mounted with its taps connected to a manometer to find the flow rate of air. A set of two temperature sensors is provided at both inlet and outlet of air stream. These sensors give Dry bulb & Wet bulb air temperatures. The cooling tower is packed with Aluminum expanded wire mesh.

Description of Temperature Sensors Are As Follows:

T₁ = inlet dry bulb temp air.

T₂ = inlet wet bulb temp air.

T₃ = outlet dry bulb temp air.

T₄ = outlet wet bulb temp air.

T₅ = inlet water temp.

T₆ = outlet water temp.

EXPERIMENTAL PROCEDURE:

- Fill the heating tank with water, set the temperature with the help of D.T.C. and switch on heater.
- Switch on pump & blower after desired temperature achieved.
- Set the flow rate of water and air.
- Record the flow rate of water and manometer reading after steady state achieved.
- Record the temperatures.
- Steps 3 to 5 may be repeated for different water & air flow rates within operational range.

OBSERVATION & CALCULATION DATA

Orifice dia., (d₁) = 0.026 m

area of Orifice, (a₁) = 5.30 × 10⁻⁴ m²

Dia. of Pipe) (d₂) = 0.052 m

area of Orifice (a₂) = 2.12 × 10⁻³ m²

Coefficient of Discharge, (Cd) = 0.64

Density of water (ρ_{water}) = 1000 kg/m³

Density of water(ρ_{air}) = 1.21 kg/m³

Acceleration due to gravity(g) = 9.81 m/s².

Observation Table:

S.N	h (in cm)		Flow rate of water Fw (LPH)	Water temp. (°C)		Air Temperature (°C)			
	h1	h2		T5 Inlet	T6 Outlet	Inlet		Outlet	
						T1 Dry bulb	T2 Wet bulb	T3 Dry bulb	T4 Wet bulb
1									
2									
3									

CALCULATION:

$$\text{Range (r)} = T_5 - T_6,$$

$$\text{Approach (A)} = T_6 - T_2,$$

$$\text{Effectiveness } (\varepsilon) = \frac{\text{Range}}{\text{Range} + \text{Approach}} \times 100 \%$$

$$\text{Head in terms of air, } (\Delta H) = \frac{h_1 - h_2}{100} \left(\frac{\rho_{\text{water}}}{\rho_{\text{air}}} - 1 \right), \text{ m of water}$$

$$\text{flowrate of air } (Q_a) = C_d \frac{a_1 a_2}{\sqrt{a_1^2 - a_2^2}} \sqrt{2g\Delta H}, \text{ m}^3/\text{sec}$$

$$\text{Flowrate of air, } G = Q_a \times 3600, \text{ m}^3/\text{hr}$$

$$\text{Flowrate of water, } L = F_w / 1000, \text{ m}^3/\text{hr}$$

$$L/G =$$

$$\text{Evaporation losses } (E_i) = 0.00085 \times 1.8 \times \text{mass flowrate of water} \times (T_5 - T_6), \text{ m}^3/\text{hr}$$

$$\text{Percentage Evaporation loss } (E_l) = \frac{\text{water loss due to evaporation}}{\text{mass flowrate of water}} \times 100 = \%$$

NOMENCLATURE:

Nom	Column Heading	Units	Type
d ₁	Diameter of Orifice	m	Given
d ₂	Diameter of the pipe in which Orifice meter is installed	m	Given
a ₁	Cross Section area of orifice	m ²	Given
a ₂	Cross Section area of pipe	m ²	Given
C _o	Coefficient of discharge of orifice	---	Given
g	Acceleration due to gravity	m/s ²	Given
G	flow rate of air	m ³ /hr	Calculated
L	Flow rate of water	m ³ /hr	Calculated
h ₁	Manometric higher scale reading	cm	Observed
h ₂	Manometric lower scale reading	cm	Observed
ΔR	Manometric difference	m	calculated
ΔH	Head in term of air	m of air	calculated
ρ _{water}	Density of manometer fluid (water)	Kg/m ³	Given
ρ _{air}	Density of the fluid whose flow rate is to be measured (air)	Kg/m ³	Given
Q _a	Flow rate of air	m ³ /sec	Calculated
L/G	Water to air flow ratio	-----	Calculated
T ₁	Air inlet dry-bulb temperature	oC	Observed
T ₂	Air inlet wet-bulb temperature	oC	Observed
T ₃	Air outlet dry-bulb temperature	oC	Observed
T ₄	Air outlet wet-bulb temperature	oC	Observed
T ₅	Water inlet temperature	oC	Observed
T ₆	Water outlet temperature	oC	Observed
E _L	Evaporation losses	m ³ /hr	Calculated

D_L	Drift losses	m^3/hr	Calculated
B_D	Blow down losses	m^3/hr	Calculated
M	Makeup Water Required	m^3/hr	Calculated
r	range	$^{\circ}C$	Calculated
A	Approach	$^{\circ}C$	Calculated
ε	Effectiveness of tower	%	Calculated

PRECAUTIONS & MAINTENANCE INSTRUCTIONS:

- ❖ Heater should not be switched on before filling the water in heating tank.
- ❖ Pump should not be switched on at low voltage.
- ❖ Water in heating tank should be properly drained after experiment is over.
- ❖ Cotton jacket over the wet bulb arrangement should be in its place properly.
- ❖ Wet bulb bottle should be filled with water before starting the experiment.

TROUBLESHOOTING:

In case of any problem regarding operation of the apparatus; the apparatus should be quickly switched off and electric supply should be cut off.

Electrical:

- ❖ Electric Shock: It means that either earth wire inside the panel is loose or there is no earth provided in the socket to which the equipment is plugged. So, make it sure that the equipment is earthed properly.
- ❖ Display of '1' on D.T.C/D.T.I.: It means sensors connections are loose. Locate the point of loose connection and solder it properly.
- ❖ No rise in temperature despite heater is ON: It means there is some loose connection. Check out for the same and then either solder it or tight it, whatever is applicable.

Leakage: The point of leakage should be detected & the concerned part is tightened properly. If the problem still persists then the part is removed & Teflon tape is wrapped on the threads properly & the part is then refitted carefully.

CHEMICAL REACTION ENGINEERING II

ISOTHERMAL OPERATION

EXPERIMENT 3

Determination of the effect of reactant concentration on the reaction rate

ADIABATIC OPERATION

EXPERIMENT 4

To determine the rate equation for the hydrolysis of acetic anhydride to acetic acid in an adiabatic reactor.

BATCH REACTOR

The reactor vessel (1) which is in the form of a vacuum insulated flask is set on a baseplate (2) which is designed to be located on four studs on the service unit and secured by the thumbnuts (3). The positioning of the reactor on the service unit is illustrated on page 3. Springs (4) are used to secure the reactor to the baseplate.

The reactor is equipped with a stainless-steel heat transfer coil (5) to which the hot water circulator of the CEX service unit can be attached. Alternatively, for reaction demonstrations below ambient temperature, the accessory CW- 16, chilled water circulator can be connected. The coil is supported by glands (6) and (7) in the lid of the reactor.

To assist the heat transfer and also to provide good mixing of the reactants, a propeller agitator (8) driven by an electric motor (9) is also mounted in the lid. The motor speed can be varied from the service unit. **FOR**

ISOTHERMAL BATCH OPERATION

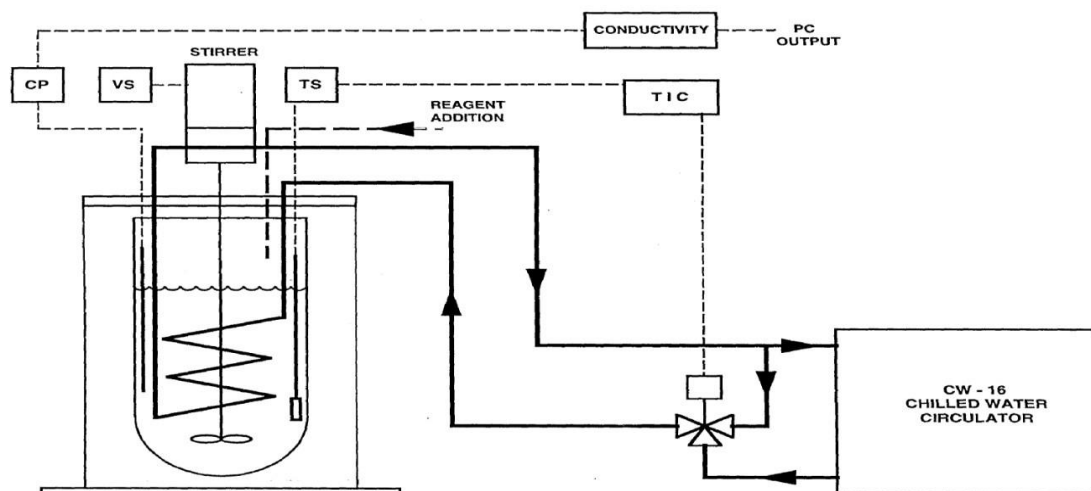
In order to maintain a constant operating temperature within the reactor, the coil contains chilled water, the flow of which is automatically adjusted by the temperature controller in the console. The actual temperature is relayed to the controller by a sensing probe (10) (part of the CEX service unit) which is held in gland (11) in the lid. The controller then opens or closes the solenoid valve in the chilled water circulator CW - 16 (optional accessory) to maintain automatically the desired set point. See OPERATIONAL PROCEDURES section in the CEX manual for more information on the temperature controller.

During the reaction the conductivity of the reactants changes as they are converted and this conversion is monitored using a conductivity probe (12) (part of the CEX service unit) which is secured in a gland (13) in the lid.

FOR ADIABATIC BATCH OPERATION

An efficient vacuum insulation surrounding the reactor allows negligible heat to be removed or added to the system during the course of a reaction. This allows the progress of an exothermic reaction to be monitored simply by observing the rise in temperature over a period of time. A temperature probe and transmitter are supplied; the probe being

inserted in the gland (11) in the place of the sensor supplied with the CEX service unit. The intention is that the probe then be connected to the Armfield interface unit (CEX-303



BATCH REACTOR

EXPERIMENTAL PROCEDURES

The Armfield batch reactor is designed to demonstrate the mechanism of a chemical reaction in such a reactor as well as the effects of varying the process conditions such as reaction temperature, reagent concentrations etc.

The reaction chosen for the isothermal demonstration is the saponification of ethyl acetate by sodium hydroxide as it can be carried out under safe conditions of temperature and pressure and is well documented.

The reaction chosen for the adiabatic demonstration is the hydrolysis of acetic anhydride. This involves the use of glacial acetic acid and sulphuric acid as well as acetic anhydride. Although it may be possible to carry out demonstrations using other chemicals it is not advisable as the materials of construction of the reactor may not be compatible.

Before carrying out reactions involving any other reagents, please refer to Armfield Ltd. for advice.

DILUTION OF ETHYL ACETATE FOR USE WITH CEM LIQUID PHASE CHEMICAL REACTOR

Armfield recommends the use of a 0.1M solution of Ethyl Acetate in the CEB Mid reactor. This should be made by diluting concentrated Ethyl Acetate as follows:

$$\text{Volume of concentrate} = \frac{\text{Mol Wt}}{10} \times \frac{1}{\text{Density}} = \frac{88.11}{10 \times 0.90} = 9.79 \text{ ml per litre of solution}$$

Therefore, add 9.79 ml of concentrated Ethyl Acetate to 900 ml of deionized or distilled water. Shake the mixture vigorously until the two liquids have mixed. Add further water to make up the final volume to 1000 ml.

Note: The practice of making a strong solution (eg. 1M) then further diluting this to the required concentration (eg. 0.1M) cannot be applied when using Ethyl Acetate, the required dilution should be made directly as stated above.

EXPERIMENT 4

Objective: To determine the effect of reactant concentration on the reaction rate

THEORY

In homogenous reaction systems, reaction rates depend on the concentration of the reactants. Collision theory indicates a rate increase if the concentration of one or both of the reactants is increased. However, the specific effect of concentration changes in a reaction system has to be determined by experimental methods. Increasing the concentration of substance, A in reaction with substance B could increase the reaction rate, decrease it, or have no effect on it - depending on the particular reaction. It is important to recognize that the balanced equation for the net reaction does not indicate how the reaction rate is affected by a change in concentration of reactants.

The general form of the rate law is

$$R = k [A]^n [B]^m$$

where: R = reaction rate
 k = rate constant

[A]ⁿ, [B]^m = molar concentration of reactants
n, m = appropriate powers (based on experimental data)

METHOD

- The same procedure as for Experiment A. Run the experiment using different initial concentrations of the reagents. For example, 0.1M, 0.08M and 0.02M (0.5 litre of each solution will be required for each mixture tested).
- Using the Armfield Data Logger in each experiment, graphs can be plotted relating reaction rate to concentration level for each of the reactants tested.
- A logarithmic plot of reaction rate to concentration level will give a straight line of slope equal to the power of the relationship.
- Hence write the rate law for sodium hydroxide and ethyl acetate in the form

$$R = k[A]^n [B]^m$$

ADIABATIC OPERATION

EXPERIMENT 5

WARNING: Extreme care should be taken whilst handling either acetic acid or acetic anhydride. Both chemicals are highly corrosive and care should be taken to avoid contact or inhalation of vapour.

DO NOT add acetic anhydride directly to sulphuric acid, as the reaction is violent and will cause the chemicals to be sprayed into the air.

The reaction described in this experiment should be carried out in a fume cupboard. On completion, the reaction products should be discarded into a copious flow of cold water, taking care to avoid inhalation of vapour.

OBJECTIVE:

To determine the rate equation for the hydrolysis of acetic anhydride to acetic acid in an adiabatic reactor.

THEORY:

The hydrolysis of acetic anhydride in water, in the presence of a catalyst of sulphuric acid $(\text{CH}_3\text{CO})_2\text{O} + \text{H}_2\text{O} \rightarrow 2\text{CH}_3\text{COOH}$

This reaction can be assumed to have simple power law kinetics of the form $R = kC$ if the reaction is zero order in water. The concentration of the protonic acid remains constant for the duration of the run and thus k is a catalytic rate constant that includes this concentration, also in power law form. For any system of this particular type the instantaneous value of the acetic anhydride concentration can be eliminated between the material and thermal balance equations to give a linear equation (which can therefore be subjected to a statistical test). Use of this equation requires that values of the gradient of temperature with respect to time be found.

If the concentration of acetic anhydride at any time is C , then the rate of the reaction is given by:

$$R = -\frac{dC}{dt}$$

with $C = C_0$ at $t = 0$. The heat generation rate is:

$$(-\Delta H)R = (-\Delta H)R = \overline{\rho C_p} \frac{dT}{dt} \quad (2)$$

with $T = T_0$ at $t=0$. Substituting equation (1) in equation (2), integrating with respect to time and using the boundary conditions, we obtain:

$$(C_0 - C)(-\Delta H) = (C_0 - C)(-\Delta H) = \overline{\rho C_p}(T - T_0) \quad (3)$$

For the n th order case, the rate may be expressed in Arrhenius form:

$$R = kC^n = A \cdot e^{-E/RT} \cdot C^n \quad (4)$$

Using $\beta = \frac{\overline{\rho C_p}}{-\Delta H}$ and rearranging, equation (3) becomes:

$$C = C_0 - \beta(T - T_0)$$

Using equations (4) and (2), we obtain:

$$\beta \frac{dT}{dt} = A \cdot e^{(-E/RT)} \cdot [C_0 - \beta(T - T_0)]^n \quad (5)$$

Using $T=T_\infty$ when the reaction is complete gives:

$$(C_0 - 0) = \beta \cdot (T_\infty - T_0) \text{ and thus } \beta = C_0 / (T_\infty - T_0)$$

Substituting for β in equation (5) and rearranging, we have:

$$\frac{\frac{dT}{dt}}{\frac{T_\infty - T_0}{C_0} \left[C_0 - C_0 \cdot \frac{T - T_0}{T_\infty - T_0} \right]^n} = A \cdot e^{(-E/RT)}$$

Taking logarithms of both sides of this equation gives:

$$\ln|\text{LHS}| = \ln|A| - (E/RT) \quad (6)$$

and so a plot of $\ln(\text{LHS})$ vs. $1/T$ should be linear with an intercept of $\ln(A)$ and a gradient of $-E/R$.

METHOD

- i. Ensure the reactor has been set up with the special temperature sensor and computer interface for the adiabatic reaction as described previously in the CONNECTION TO SERVICES section.
- ii. If possible, the reactants should be at approximately 30°C before mixing. If they are cooler than this, the reaction will take much longer to complete.
- iii. Charge the reactor with 100ml of acetic anhydride and 250ml of demineralised water.
- iv. Start the reactor agitator and adjust the variable speed drive to position '5.0'.
- v. Add 150ml of glacial acetic acid (since the water and acetic anhydride are mutually insoluble, but are both soluble in acetic acid, this ensures that the reaction mixture is homogeneous - the acetic acid does not form part of the reaction).
- vi. Start the data logger program choosing 15 seconds as the interval between samples and 1500 seconds as the total running time of the experiment (a longer duration will be required if the temperature of the reactants is less than 30°C). It is essential to log data until the temperature in the reactor begins to fall again.
- vii. Immediately add 25ml of 0.5M sulphuric acid as the catalyst. The reaction will commence.
- viii. Switch off the agitator.
- ix. When the reaction has completed, the temperature data can be viewed within the software, either in tabular or graphical format. The data should be saved as an excel file. It is then possible to carry out the second part of the analysis described above, by calculating the terms of equation (6), and plotting the graph.
- x. In order to find dT/dt , it will be necessary to extract the linear region of data from a graph of temperature vs time for the experiment. This will entail discarding the first few points, and also any points after the temperature reaches the peak value.
- xi. It will then be necessary to find the best value for n in equation (6) by trial and error, refining the value until the best straight-line fit is obtained.
- xii. Several runs similar to that described above should be performed, using differing quantities of sulphuric acid catalyst (eg. 15ml to 40ml in 5ml increments).

NOMENCLATURE

a_{μ}	sodium hydroxide conc. in feed vessel	(mol/dm ³)
a_0	sodium hydroxide conc. in mixed feeds	(mol/dm ³)
a_1	sodium hydroxide conc. in reactor at time t	(mol/dm ³)
a_{∞}	sodium hydroxide conc. in reactor after ∞ time	(mol/dm ³)
b	ethyl acetate conc.	(mol/dm ³)
(same subscripts as above for a)		
c	sodium acetate conc.	(mol/dm ³)
(same subscripts as above for a)		
k	specific rate constant	
r	reaction rate	
t	elapsed time	(s)
T	reactor temperature	(K)
V	volume of reactor	(dm ³)
X_a	conversion of sodium hydroxide = $\frac{a_0 - a_1}{a_0}$	
X_c	conversion of sodium acetate = $\frac{c_1 - c_0}{c_{\infty}}$	
Λ	conductivity (Siemens)	
Λ_0	initial	
Λ_1	at time t	
Λ_{∞}	at ∞ time.	

CONTINUOUS STIRRED TANK REACTOR (CSTR)

EXPERIMENT 6: Determination of reaction rate constant for saponification of ethyl-acetate with NaOH at ambient conditions.

The purposes of this Continuous Stirred Tank Reactor experiment are:

- To determine the order of saponification reaction
- To determine the reaction rate constant, k by plotting the graph

INTRODUCTION

Every industrial chemical process is designed to produce economically a desired product from a variety of starting materials through a succession of treatment steps. The raw materials undergo a number of physical treatment steps to put them in the form in which they can be reacted chemically. They then pass through the reactor. The products of the reaction must then undergo further physical treatment like separations, purification, etc for the final desired product to be obtained in chemical engineering, chemical reactors are vessels designed to contain chemical reactions. The design of a chemical reactor deals with multiple aspects of chemical engineering. Chemical engineers design reactors to maximize the net present value for the given reaction. Designers ensure that the reaction proceeds with the highest efficiency towards the desired output product, producing the highest yield of product while requiring the least amount of money to purchase and operate.

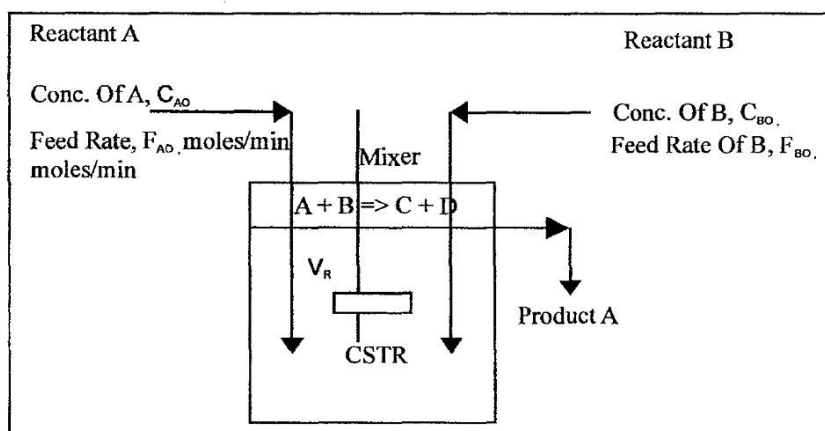
Reactor design uses information, knowledge and experience from a variety of areas such as thermodynamics, chemical kinetics, fluid mechanics, heat transfer, mass transfer and economics. Chemical reaction engineering is the synthesis of all these factors with the aim of properly designing a chemical reactor.

The reactor is the equipment in which empirical information is obtained can be divided into two types, the batch and flow reactors. The batch reactor is simply a container to hold the contents while they react. The flow reactor is used primarily in the study of the kinetics of heterogeneous reactions.

Reaction: $\text{NaOH} + \text{CH}_3\text{COOC}_2\text{H}_5 \rightarrow \text{CH}_3\text{COONa} + \text{C}_2\text{H}_5\text{OH}$

THEORY:

In an ideal CSTR (that is an ideal steady state flow reactor) the contents in the reactor are well mixed and have uniform composition throughout. Thus, the exit stream has the same composition as the fluid within the reactor. This type of reactor is also known as MIXED FLOW



REACTOR.

Reactor volume (volume of fluid in the reactor) = V_R , Liters, Volumetric feed rate (volumetric feed rate of A + volumetric feed rate of B) = V_0 , LPM
 Space time = V_R / V_0 , mm

$$\tau = \frac{V_R}{V_0} = \frac{C_{A0} X_A}{-r_A} = \frac{C_{A0} - C_A}{-r_A}$$

The performance equation for the mixed flow reactor at steady state is:

X_A & r_A are evaluated at exit stream conditions, which are the same as the conditions within the reactor.

For a second order reaction:



With $C_{A0} = C_{B0}$ and negligible change in density of reaction mixture.

DESCRIPTION:

The reactor is made up of SS -304. The air compressed feed reactants are given through Rotameter. The concentration of feed and the products coming out from the reactors are analyzed by chemical titration and check the effect and the performance of the reactor.

A Continuous stirred tank flow reactor (CSTR) required. 6 No. 50 cc graduated cylinder. No. 100 cc titration flasks, 50 cc burette and one stopwatch, conductivity cell (optional).

CHEMICALS:

1. N/10 NaOH,
2. N/10 HCL,
3. N/10 Ethylacetate (8.8 gms of ethylacetate in 1 L of water),
4. Indicator (Phenolphthalein)

UTILITIES REQUIRED:

1. Compressed Air Supply at 2 Bar, 0.5 CFM.

2. Water Supply.
3. Drain.
4. Electricity Supply: 1 Phase. 220 V AC. 0.5 kW.
5. Instruments. Laboratory Glassware and Chemicals required for analysis as per the system adopted.

SPECIFICATIONS:

Reactor (Approx).	: Material Stainless Steel 304 Grade, Capacity 2 Ltrs
Stirrer coupled with FHP Motor	: Stainless Steel 304 Grade Impeller and shaft
Feed Tank (2Nos.)	: Material Stainless Steel 304 Grade. Capacity - 20 Ltrs.
Feed Circulation	: By compressed air
Flow Measurement for Reactants) Piping and PVC	: Rotameter 2Nos. (one each : Stainless Steel 304 Grade
Pressure Regulator	0-2 Kg/cm ²
Pressure Gauge	: Bourdon type 0- 2 Kg/cm ² Stop Watch
Control Panel	: Electronic : On / off switch, Mains Indicator etc.
The whole unit is assembled rigidly on a base plate and mounted on a stand. Most of the parts are powder coated and rest are painted with auto paints.	

PROCEDURE:

1. Prepare 20L of N/10 NaOH solution by dissolving 80 gm of NaOH in 20L of water.
1. 2. Prepare 20L of N/10 ethylacetate solution by mixing 176 gms of ethylacetate in 20L of water.
2. Fill the respective tanks with these solutions.
3. Adjust the flow rate of the two streams so that in the feed mixture $CA_0 = CH_0$ i.e have equal flow rates (e.g. 2 LPH each you may use the flow range :2 LPH to 16 LPH for each stream) using calibrated rotameters.
4. Pass equimolar feed rates of ethyl acetate and NaOH into the CSTR by allowing equal volumetric feed rates of reactants in to the reactor and simultaneously start the mixer of the CSTR. Ensure constant stirring throughout the experiment.
5. After about 10 mm or time equal to the residence time of the reactor (whichever is greater) i.e when steady state is achieved, collect sample of the liquid at the out let
6. Titrate this solution against N/40 HCL (add HCL from burette).
7. Using phenolphthalein as indicator, note the volume of N/40 HCL used (VHCL)

8. Note the volumetric flow rate of liquid at the end of the reactor.
9. Note the reaction temperature.
10. For calculating the conversion at equilibrium condition, collect the sample 10 ml, in an empty conical flask and allow the reaction to proceed for completion for two hours. After two hours titrate the solution with N/10 HCl using phenolphthalein as indicator. Note the volume of N/10 HCl used.
11. Repeat all the steps for four to six different flow rates of feed.

Note: report k at the reaction temperature STANDARD DATA Run No.

Reaction temp. °C	=	T °C
Reactor diameter	=	d =
Effective height of liquid in the reactor,		cm L
		= cm
Vol. of the reactor	=	$VR = (11/4) * d^2 * L$, VR
		= L
Volumetric feed rate of feed	$V_0 =$	L/m in
Initial Conc. Of NaOH in the feed mixture	=	CA_0 gmol/L

Initial Conc. of Ethyl acetate in the feed mixture		$C_{BO} =$
gmol/L Residence Time	=	$t = V_r / V_0$
mm		
X_A	=	$[CA_0 - CA] / CA_0$

Concentration of unreacted NaOH in the reaction mixture, CA Estimation of unreacted NaOH in the reaction mixture (CA):

Titrate the sample collected immediately with N/40 HCl using phenolphthalein as indicator.

Volume of reaction mixture taken as sample = V_1

Volume of acid used to neutralise sample = V_{HCl}

$N/40 \times V_{HCl} = V_1 \times \text{Normality of reaction mixture}$

Normality of reaction mixture = CA = gmole/L

Run. No	τ , min residence time	C_A (conc. NaOH) gmol/L	X_A , degree of conversion	$k = [C_{A0} - C_A] / \tau C_A^2$, l/gmol-min

Or plot τ vs $X_A/[1-X_A]^2$ on a simple graph, the slope is $= 1/(k C_{AO})$: from the slope find k and report k at the reaction temp.

PRECAUTION:

1. Measure the exact volume of water and weigh the chemicals.
2. Always use clean water and good quality chemicals and standard solution for titration.
3. Use electronic balance for weighing of chemicals. Don't mix the droppers of different chemicals.
4. Keep close all the drain valves and vent valve should open while tilling the reactant in feed tanks.
5. Flow should not be disturbed during the experiments.
6. Handle the chemicals carefully.

TROUBLE SHOOTING:

1. If any type of suspended particles come in the Rotameter. Remove the Rotameter clean the tube and fit again at its place.
2. If there is any leakage; tight that part or remove that and refix that again after wrapping Teflon tape.
3. If Rotameter fluctuating more than average tight control knob of that. (Procedure: two nuts are there lose first nut and tight the second slightly, and then first also. Both nuts are on Rotameter)

REFERENCE:

1. Chemical Reaction Engineering by Octave Levenspil. Chapter 2 to 5

SAMPLE CALCULATIONS

STANDARD DATA:

Effective Volume of the Reactor	$V_R = 2.649\text{L}$
Initial Conc. Of NaOH, CAO	$= 0.0455\text{gmol}$
	$/\text{L}$
Reaction Temp., T	$= 30.0^\circ\text{C}$
Sample volume taken = ml	
Strength of HCL	$= \text{N}/40$

OBSERVATIONS TABLE:

RU N NO	Total Volume of Feed Rate to the reactor, Vof LPM	Vol. Of Std.N/40 HCL used for titration ml
1	0.52 LPM	
2	0.425 LPM	
3	0.333 LPM	
4	0.167 LPM	

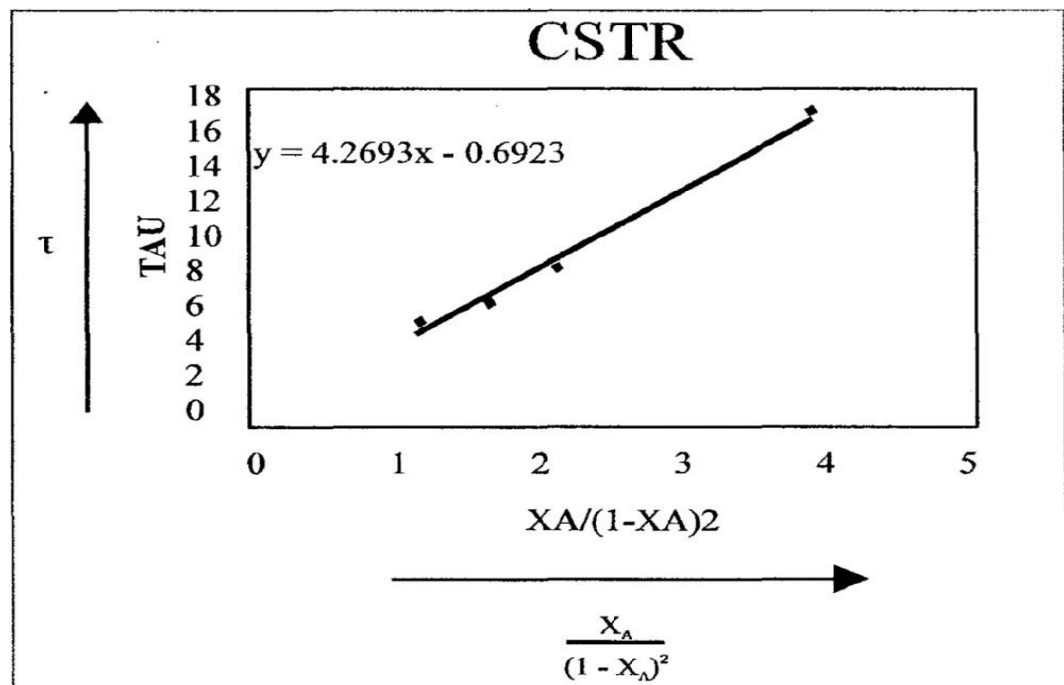
CALCULATION FOR EACH RUN:

RUN NO. 1

$$\begin{aligned}\text{Conversion, } X &= (C_{AO} - C_A) / C_{AO} \\ &= (0.0455 - 0.027) / 0.0455 \\ &= 0.4066 \\ \text{Residence time, } \tau &= V_R / V_0, \text{ min} \\ &= 2.649 / 0.52 \\ &= 5.0942, \text{ min} \\ \text{Rate of reaction } -r_A &= (V_0 * C_{AO} * X) / V_R, \text{ gmol/l-min} \\ &= (0.52 * 0.0455 * 0.4066) / 2.649 \\ &= 0.00363 \\ \text{Rate constant, } k &= (C_{AO} - C_A) / (\tau * C_A^2), \text{ l/gmol-min} \\ &= (0.0455 - 0.027) / 5.0942 * (0.027)^2 \\ &= 4.98156\end{aligned}$$

RESULTS:

$\tau, \text{ min}$	$C_A, \text{ gmol/L}$	X	$k, \text{ l/gmol-min}$	$-r_A, \text{ gmol/l-min}$
5.0942	0.027	0.4134	4.98156	0.00363
6.2329	0.0240	0.4783	6.128	0.00353
7.79	0.0220	0.5258	6.3641	0.0030
16.2577	0.0180	0.6087	5.2178	0.00172



MULTI-FUNCTIONAL PLATE DISTILLATION COLUMN

EXPERIMENT 7: To investigate the basic principles and calculation techniques of SIEVE PLATE Distillation and to determine the number of theoretical plates

AIM:

The objective of this experiment is to put into practical use of the concepts that you learned about distillation in mass transfer course.

OBJECTIVES:

- ❖ To investigate the basic principles and calculation techniques of SIEVE PLATE Distillation.
- ❖ To determine the number of theoretical plates.

INTRODUCTION:

The unit operation distillation is used to separate the components of a liquid solution, which depends upon the distribution of these various components between a vapor and a liquid phase. All components are present in both phases. The vapor phase is created from the liquid phase by vaporization at the boiling point. If a homogeneous liquid solution is boiled, the vapor is richer in the more volatile components than is the liquid, whereas the remaining liquid is richer in the less volatile components. The separation of crude petroleum into gasoline, kerosene, fuel oil and lubricating stock and the separation of a mixture of alcohol and water into its components are examples of distillation.

THEORY:

Distillation may be carried out by either of two principal methods.

- Based on the production of a vapour by boiling the liquid mixture to be separated and condensing the vapors without allowing any liquid to return to the still in contact with the vapors.
- Based on the return of part of the condensate to the still under such conditions that this returning liquid is brought into intimate contact with the vapors on their way to the condenser. Either of these methods may be conducted as a continuous process or as a batch process.
- Batch distillation, which is the process of separating a specific quantity of a liquid mixture into products, is used extensively in the laboratory and in small production units that may have to serve for many mixtures. In batch distillation, a batch of liquid is charged to the reboiler and the system is first brought to uniform operation under total reflux. Then apportion of the overhead product is continuously withdrawn in accordance with the established reflux policy. The column operates as an enriching section. The progress of batch distillation can be controlled in several ways.
- Constant reflux, varying overhead composition. The reflux is set at a predetermined value where it is maintained for the run. Since the composition of the pot is changing

the composition of the distillate also changes.

- Constant overhead composition, varying reflux. If it is desired to maintain a constant overhead composition, the amount of reflux returned to the column must be increased. As time proceeds, the reboiler is gradually depleted of the lighter component. Finally, a point is reached where the reflux ratio attains a very high value. The receivers are then changed, the reflux is reduced, and an intermediate cut is taken.

A SIEVE PLATE DISTILLATION COLUMN consists of a number of perforated plates with a downspout as well. Ideally, the liquid coming down from the top flows across the surface of each plate. The vapour flows vertically upwards through the perforations of each plate. Thereby the contact between liquid and vapour occurs. The streams leaving each plate are considered to be in equilibrium for a perfect plate. Through this experiment the number of practical stages are obtained using McCabe Thiele method and compared with the actual number plates.

DESCRIPTION:

The column is made of Stainless Steel with seven plates for mounting the sieve plates. An electrically heated re-boiler is installed at the bottom of the column. The bottom product is collected in the tank. The vapours from the top of column are condensed in the shell and tube type condenser by cooling water supplied by laboratory overhead tank. The condensate is divided into reflux and distillate by automatic reflux divider and R/D Ratio can be varied. Reflux is fed back to the column and distillate is received in a receiving tank. The complete column is insulated for minimizing the heat loss. Instrumentation provided for pressure & temp, measurement wherever necessary.

EXPERIMENTAL PROCEDURE:

- ❖ Make a feed solution at least 20 liters, by adding 15 wt % methanol and 85% water, then mix the liquid well before fed in to the reboiler.
- ❖ The column takes a long time (about 1 hour) to heat up and come to steady state under total reflux conditions. Familiarize yourself with the operation of the column and the Refractometer. A total run time of 4+ hours is not uncommon.
- ❖ Charge the feed to the Reboiler.
- ❖ Start heaters and adjust the temperature of the boiler using DTC. Using the highest allowable setting will speed the experiment considerably. It is important to monitor the column carefully during the startup period and reduce the setting of DTC on which the column floods + 5°C. Flooding is when the space between two of the trays in the column becomes filled with liquid. The top tray tends to flood first. If this occurs, the column will take some time to return to normal operation following a reduction of heat.
- ❖ Open the vent valve time to time to release air from column.
- ❖ As the column heats up, adjust the cooling water flow rate to the condenser so that the entire vapor is condensed.
- ❖ Allow the mixture to boil in the re-boiler and close the vent valve of the condenser shell as the vapors coming out from the shell.

- ❖ Run the apparatus at total reflux until the column reaches steady state. This will be indicated by steady temperatures and full, flooded in trays.
- ❖ When steady state is reached, set the reflux ratio at (in the range of 1.5 to 3), and begin collecting product.
- ❖ Continue distilling under these conditions until 10% of the charge has been collected as distillate. While the distillation is progressing, periodically record the following information:
 - The tray compositions and temperatures,
 - The instantaneous distillate composition,
 - The condenser flow rate and temperature rise,

When the distillation is finished, take a final set of readings and switch off the heaters. Shut off all column inputs.

Prepare a calibration curve for CH₃OH-Water by plotting R-I as a function of mole fraction CH₃OH at the ambient temperature or at 25°C. Prepare different mixtures of CH₃OH + Water by volume and measure the R-I of each mixture. Measure the density of CH₃OH ($\rho_A=803 \text{ kg/m}^3$) and water ($\rho_B= 1000\text{kg/m}^3$) at the ambient temperature or 25°C and knowing the molecular weight of CH₃OH ($M_A= 32$) and Molecular weight of Water ($H_2O=18$). Convert your volume fractions to mole fractions and plot mole fraction CH₃OH vs R-I on a simple graph.

If V_A ml of CH₃OH mixed with V_B mL of water.

Then Mole Fraction of CH₃OH

$$x = \frac{\frac{V_A \rho_A}{M_A}}{\frac{V_A \rho_A}{M_A} + \frac{V_B \rho_B}{M_B}}$$

% of methanol	% of water	Mole fraction of methanol (x)	R.I at 25°C
100	0	1	1.3268
90	10	0.802	1.3300
80	20	0.643	1.3330
70	30	0.512	1.3356
60	40	0.403	1.3381
50	50	0.310	1.3398
40	60	0.231	1.341
30	70	0.162	1.3411
20	80	0.101	1.3394
10	90	0.047	1.3363
0	100	0	1.3321

% of methanol in water	Density(kg/m ³)
0	998.2
10	981.5
20	966.6
30	951.5
40	934.5
50	915.6
60	894.6
70	871.5
80	846.9
90	820.2
100	791.7

Plot R.I vs x using excel or plot x vs R.I and fit two polynomials one for CH₃OH rich section (to be used for finding the distillate composition) and the other for water rich section (to be used for finding the feed and bottoms composition).

At 25°C

e.g. for CH₃OH rich section: $x = 57.3625 - 42.5319 R.I$ --
 -- 1 and for water rich section :
 $x = 1865.11 - 2827.05 R.I + 1071.23 R.I^2$ 2

OBSERVATION& CALCULATION:

DATA:

Density of Feed : kg/m³ at ambient temp.
 Density of Distillate : kg/m³ at ambient temp.
 Density of Bottoms : kg/mm³ at ambient temp.
 R-I of Feed : -----
 R-I of Distillate : -----
 R-I of Bottoms : -----

From the calibration curve, corresponding mole fractions of CH₃OH are:

Xf = -----
 XD = -----
 XB = -----
 L/D R = -----
 Cooling Water Rate = LPH
 Cooling Water inlet temp = °C
 Cooling water outlet temp = °C

Feed Temperature = 0°C

Steady state tray temperature and composition:

OBSERVATION TABLE:

Sieve Plate No, with sensor no (From Bottom)	Temperature 0°C	R.I	Mole Fraction (x) from calibration curve
1			
2			
3			
4			
5			
6			
7			

x-y data for CH₃OH-H₂O System

Temperature (°C)	x	Y
100	0	0
96.4	0.020	0.134
93.5	0.040	0.230
91.2	0.060	0.304
89.3	0.080	0.365
87.7	0.100	0.418
84.4	0.150	0.517
81.7	0.200	0.579
78	0.300	0.665
75.3	0.400	0.729
73.1	0.500	0.779
71.2	0.600	0.825
69.3	0.700	0.870
67.5	0.800	0.915
66	0.900	0.958
65	0.950	0.979
64.5	1	1

Using the above x-y data for CH₃OH-H₂O, step off the number of ideal stages required From McCabe-Thiel diagram, we obtain eight ideal stages including re-boiler.

Total number of ideal stages (including re-boiler) = NT
 No. of ideal stages required = NT^{-1}
 No. of actual stages provided = N

$$\text{Overall Efficiency of column} = \frac{\text{No of ideal Trays (Plates)}}{\text{No of actual trays}} \times 100$$

Reflux Ratio = $R = L/D$

$$\text{Intercept} = \frac{X_D}{R+1}$$

NOMENCLATURE:

Nom	Description	Unit	Value
T1	Vapour temperature at sieve plate No. 1	°C	Measured
T2	Vapour temperature at sieve plate No. 2	°C	Measured
T3	Vapour temperature at sieve plate No. 3	°C	Measured
T4	Vapour temperature at sieve plate No. 4	°C	Measured
T5	Vapour temperature at sieve plate No. 5	°C	Measured
T6	Vapour temperature at sieve plate No. 6	°C	Measured
T7	Vapour temperature at sieve plate No. 7	°C	Measured
T8	Vapour temperature inlet to calorimeter	°C	Measured
T9	Condensate temperature outlet from calorimeter	°C	Measured
T10	Water temperature inlet to calorimeter	°C	Measured
T11	Water temperature outlet from calorimeter	°C	Measured
x _F	Mole fraction of feed		Calculated
x _D	Mole fraction of distillate		Calculated
x _B	Mole fraction of Bottom product		Calculated
L/D=R	Reflux ratio	-	Observed
x	Mole fraction of CH ₃ OH in solution	-	Calculated
y	Mole fraction of H ₂ O in solution	-	Calculated

PRECAUTIONS & MAINTENANCE INSTRUCTIONS:

- ❖ Use the stabilize A.C. Single Phase supply only.
- ❖ Never switch on mains power supply before ensuring that all the ON/OFF switches given on the panel are at OFF position.
- ❖ Voltage to heater starts and increases slowly.
- ❖ Keep all the assembly undisturbed.
- ❖ Never run the apparatus if power supply is less than 180 volts and above than 240 volts.
- ❖ Operate selector switch for temperature indicator gently.

- ❖ Do not start heater supply unless water is filled in the test unit.
- ❖ Always keep the apparatus free from dust

TROUBLESHOOTING:

- ❖ In case of any problem regarding operation of the apparatus ; the apparatus should be quickly witch OFF and electric supply should be cut OFF.
- ❖ Electric shock: It means that either earth wire has loose connection or the reiso no earth provided in the socket to which the equipment is plugged in. So, make it sure that the equipment is earthed properly.
- ❖ Leakage : The point of leakage should be detected & the concerned part is tightened properly . If the problem persists then that part should be removed & Teflon tape is wrapped on the threads properly and refitted carefully.

pH MEASUREMENT AND ITS APPLICATION

DETERMINATION OF pH OF A GIVEN SOLUTION

AIM:

To determine the pH of the given water sample with the conditions.

INTRODUCTION:

The term pH refers to the measure of hydrogen ion concentration in a solution and defined as the negative log of H^+ ions concentration in water and wastewater. The values of pH 0 to a little less than 7 are termed as acidic and the values of pH a little above 7 to 14 are termed as basic or alkaline. When the concentration of H^+ and OH^- ions are equal then it is termed as neutral pH.

$$pH = -\log [H^+], \text{ high } [H^+] \gg pH < 7, \text{ low } [H^+] \gg pH > 7$$

ENVIRONMENTAL SIGNIFICANCE:

Determination of pH is one of the important objectives in biological treatment of the wastewater. In anaerobic treatment, if the pH goes below 5 due to excess accumulation of acids, the process is severely affected. Shifting of pH beyond 5 to 10 upsets the aerobic treatment of the wastewater. In these circumstances, the pH is generally adjusted by addition of suitable acid or alkali to optimize the treatment of the wastewater. pH value or range is of great importance for any chemical reaction. A chemical shall be highly effective at a particular pH. Chemical coagulation, disinfection, water softening and corrosion control are governed by pH adjustment. Lower value of pH below 4 will produce sour taste and higher value above 8.5 a bitter taste. High pH causing cancer in human beings.

Apparatus

pH meter with temperature device, accurate and uniform to 0.1 pH unit with a range of 0 to 14. Follow manufacturer's instructions on use and care of device.

SAMPLE HANDLING AND PRESERVATION:

- Preservation of sample is not practical. Because biological activity will continue after a sample has been taken. Changes may occur during handling and storage.
- The characteristics of the water sample may change. To reduce the change in samples taken for the determination of pH, keep samples at 4°C. Do not allow the samples to freeze.
- Analysis should begin as soon as possible.

PRECAUTIONS:

The following precautions should be observed while performing the experiment:

1. Temperature affects the measurement of pH at two points. The first is caused

by the change in electrode output at different temperatures. This interference can be controlled by the instrument having temperature compensation or by calibrating the electrode-instrument system at the temperature of the samples. The second is the change of pH inherent in the sample at different temperatures. This type of error is sample dependent and cannot be controlled; hence both the PH and temperature at the time analysis should be noted.

2. In general, the glass electrode, is not subject to solution interferences like color, high salinity, colloidal matter, oxidants, turbidity or reductants.
3. oil and grease, if present in the electrode layer, should be removed by gentle wiping or detergent washing, followed by rinsing with distilled water, because it could damage the electrode response.
4. Before using, allow the electrode to stand in dilute hydrochloric acid solution for at least 2 hours.
5. Electrodes used in the pH meter are highly fragile. Hence handle it carefully.

PROCEDURE:

Three major steps are involved in the experiment. They are:

1. Preparation of Reagents.
2. Calibrating the Instrument.
3. Testing of Sample.

TESTING OF SAMPLE:

- In a clean dry 50 or 100 mL beaker take the water sample and place it for test.
- Now place the electrode in the beaker containing the water sample and check for the reading in the pH meter. Stir the sample gently while measuring pH to insure homogeneity. Wait until you get a stable reading.
- The pH of the given water sample is --.
- Take the electrode from the water sample. Wash it with distilled water and then wipe gently with soft tissue.

to determine the value of pH of the given water sample the readings obtained are required to be tabulated.

SAMPLE NO.	TEMP. OF SAMPLE (C°)	pH
1		
2		
3		
4		

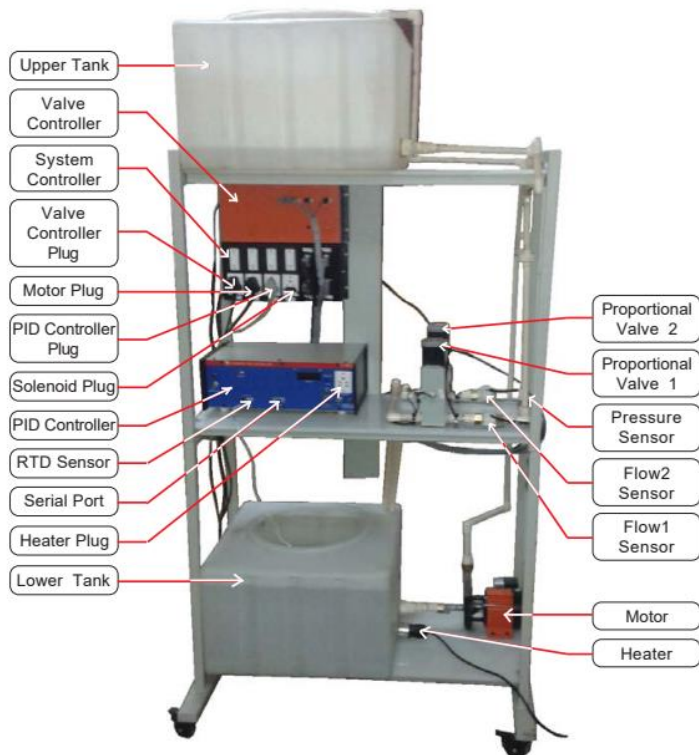
- *What factors affect the electrode life?*

- *How does the temperature affect PH measurements?*
- *Can pH be negative or higher than 14?*

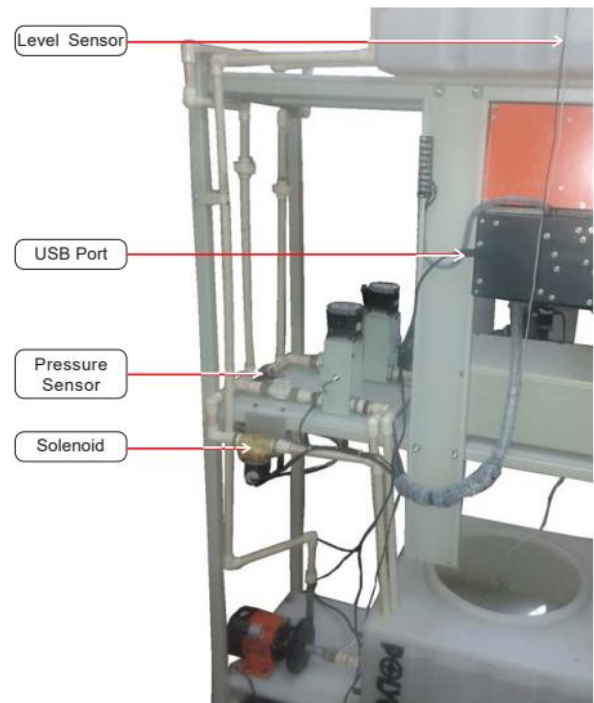
PROCESS CONTROL AND INSTRUMENTATION

VPL-PLC-MPCT MULTIFUNCTION PROCESS CONTROL SYSTEM

System Parts Details



System Front View



System Rear View

SYSTEM DESCRIPTION

The process control trainer helps us to study the dynamics and control of multi process system.

This system consists of two tanks. The lower tanks have a heater unit and RTD which helps us to control the temperature. There is a PID control box with RTD as input and heater control to maintain the temperature according to the set point.

The upper tank has a level indicator to measure the level. The upper tank and lower tank are connected by pump and solenoid valve.

The motor pump is used to pump water from lower tank to upper tank. There is a safety switch in the lower tank which prevents the motor to run dry. The solenoid valve when switched ON descends the water from the upper tank to the lower tank.

There are two proportional valves which also delivers water from upper tank to the lower tank. The positions of these valves are controlled by valve controller.

An impeller type Flowmeter is included in the flow circuit of each proportional valve to measure the flow and provide a calibrated output signal for display or control purposes. Water flowing in the flow circuit causes the impeller to rotate. Optical sensing of the impeller produces a series of pulses. These pulses are proportional to the flow rate. A frequency-to- voltage converter produces a D.C. signal proportional to the flow rate that

can be used for display or control purposes.

There is a pressure sensor in the flow circuit of Proportional valve 1. It measures the pressure in the flow line connecting the upper tank to lower tank via Proportional valve 1.

There is a main controller unit called System controller. It has all the inputs of sensor and a communication block which connects to PC via USB. This controller block is also linked to valve controller and provides the appropriate signal to position the Valves at desired location.

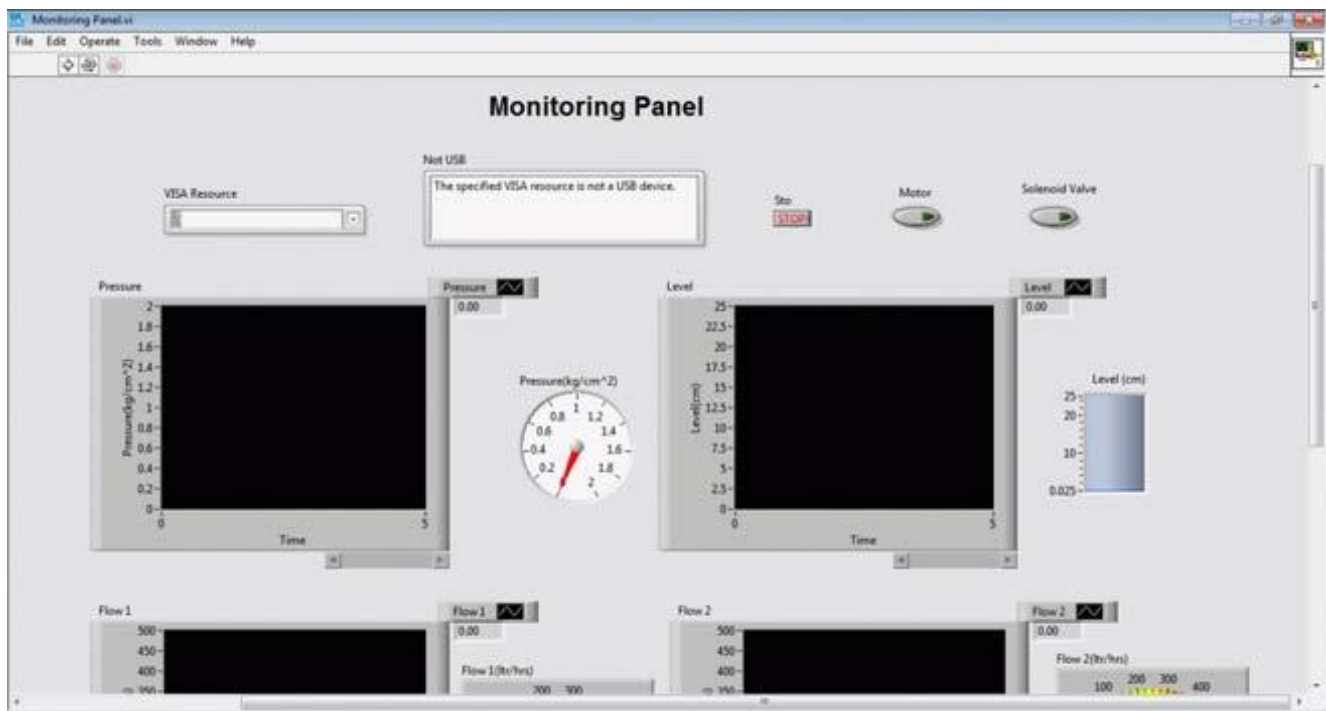
The Monitoring panel of the whole processes:

Level Control System

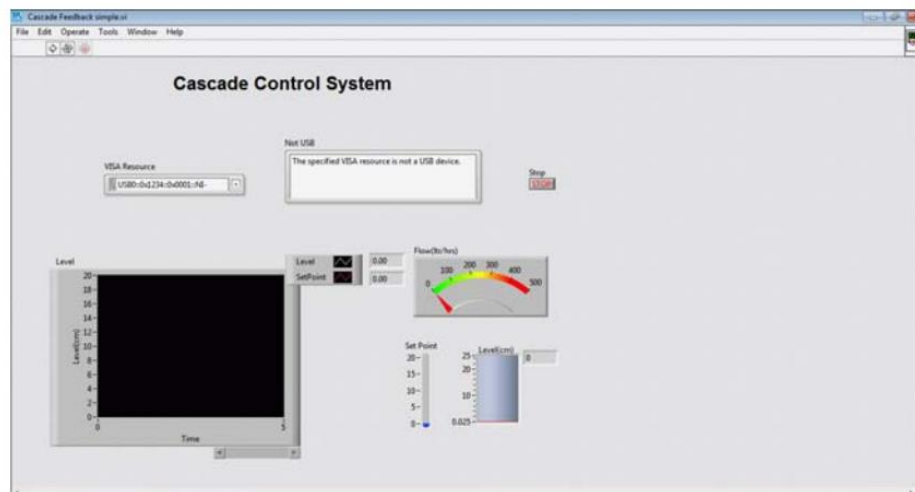
Flow 1 Control System Flow 2 Control System

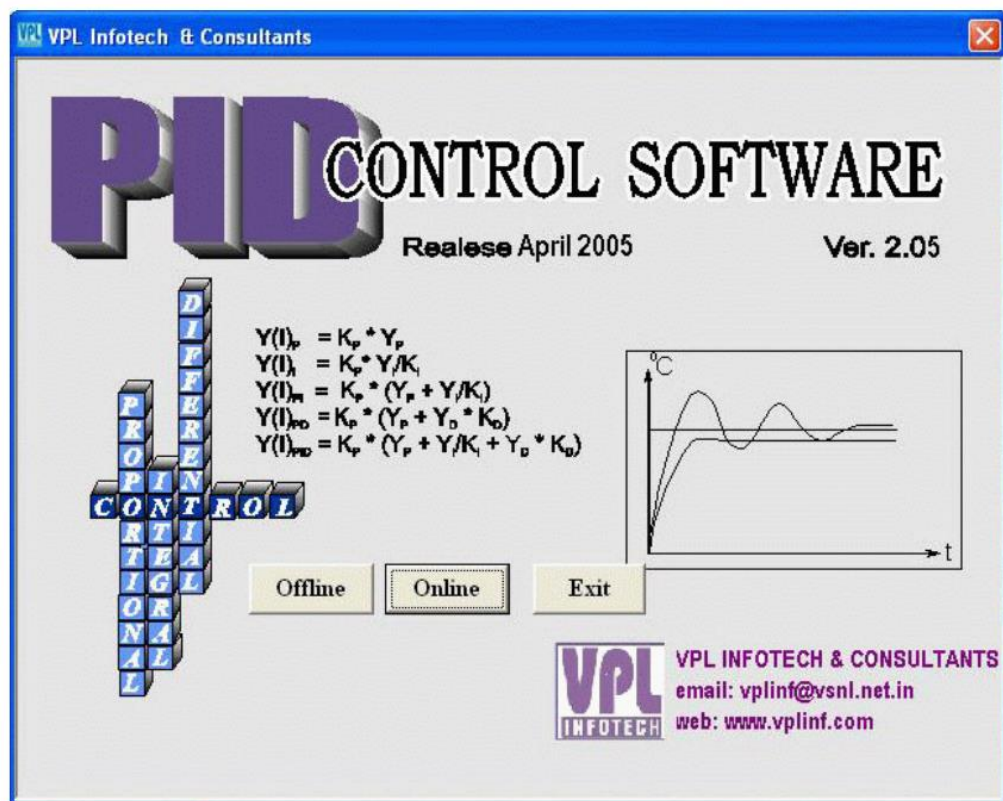
Pressure level Controller

Temperature level controller



Multi-Process Control System Trainer





PID controller interface

EXPERIMENTS

- PRESSURE PROCESS CONTROLLER
- LEVEL PROCESS CONTROLLER
- TEMPERATURE PROCESS CONTROLLER
- STUDY OF COMPLEX CONTROL SYSTEM USING MATLAB

Experiment 09: To study the performance of ON-OFF/P/PI/PD/PID controllers on Pressure process.

Experiment 10: To study the performance of ON –OFF/P/PI/PD/PID controllers on level process Experiment 13: To study the performance of ON –OFF/P/PI/PD/PID controllers on flow process

Experiment 11: To study the performance of ON-OFF/P/PI/PD/PID controllers on temperature process.

Experiment 12: To study the complex control system using matlab and to compare the response of simple and cascade loop.

THEORIES

PRESSURE PROCESS CONTROLLER

Theory: - Pressure control trainer is designed for understanding the basic principles of pressure control. The process set up consists of pressure vessel fitted with pneumatic control valve. Pressure transmitter is used for pressure sensing. The process parameter (Pressure) is controlled by microprocessor based digital indicating controller which manipulates pneumatic control valve fitted at Outlet of pressure tank outlet through I/P converter. These units along with necessary piping are fitted on support housing designed for tabletop mounting.

LEVEL PROCESS CONTROLLER

The level process controller is used to perform the control action of level process and study the characteristics of I/P converter. The RF capacitance level transmitter is used to measure the level of the process tank. In level control action, a pump sucks the air from reservoir and gives it to control valve. Every internal transaction is in voltage. Here, IBM-PC acts as error detector and controller. According to error signal, corresponding control signal is given to the I/P converter. It controls the flow of the liquid in pipeline by varying stem position of the control valve. For maintaining the level of the process tank, flow is manipulated level signal is given to the data acquisition card. By pass line is provided to avoid the pump overloading.

From this controller also study the characteristics of the level transmitter, I/P converter, control valve and justify the various control actions.

FLOW PROCESS CONTROLLER

Flow process controller is used to perform the control action on Flow process. In this unit flow is the process variable and is sensed and given to controller. A Differential Pressure Transmitter is used to measure the Flow of the fluid through orifice plate.

In this unit, pump sucks the water from the reservoir tank and gives it to the control valve. Every internal transaction are in voltage. Here, PC acts as error detector and controller. According to the error signal, computer develops a control signal.

This control signal is given to I/P Converter which operates the control valve

Control valves act here as final control element which controls the flow of the fluid in pipe line by varying stem position of the control valve.

Flow is the manipulated level signal here and is given to the Data acquisition card. By pass line is provided to avoid the pump over loading.

TEMPERATURE PROCESS CONTROLLER

Temperature process controller is used to perform the control action on temperature process. In this unit, temperature is the process variable. An RTD sensor is being used to measure the temperature of the process and is transmitted by RTD transmitter.

In this unit, water is stored up in the overhead tank by pumping water from reservoir tank through pump. Water from overhead tank is allowed to heater through a rota meter.

Rota meter is being provided for giving disturbance to the process and for operational safety of the heater. For conducting experiment flow rate in minimum value (say 30 Lph). In the outlet of heater, an RTD is provided for sensing the temperature and is transmitted by RTD transmitter.

Every internal transaction are ion voltage. Here PC acts as error detector and controller. According to the error signal, computer develops a control signal. This control signal is given to the SCR power driver circuit by which firing angle varies. Accordingly, the supply delivered to heater varies and thus process gets controlled.

Data acquisition card has the ADC and DAC so that it acts as an effective link between the process and controller.

TYPES OF CONTROL

ON/OFF Control

One of the most widely used type of control is the ON/OFF control. ON/OFF control is also referred as “TWO POSITION” control or “OPEN AND CLOSE” control. Two position controls is a position type of controller action in which the manipulated variable is quickly changed to either a maximum or minimum value depending upon the controlled variable is greater or less than the set point.

If the controlled variable is below the set point, the controller output is 100% (i.e. control valve is fully closed). If the controlled variable is above the set point, the controller output is 0% (i.e. control valve is fully opened), when the differential gap is zero. The tuning parameter for ON/OFF control are differential gap and time delay.

Differential Gap

Differential gap is the region in which the control causes the manipulated variable to maintain its previous until the controlled variable has moved slightly beyond the set point. Small differential gap is not preferred. Because it introduces oscillations and reduces the life of final control element.

Proportional Control

Two position control applied to a process results in a continuous oscillation in the quantity to be controlled. This drawback was overcome by a continuous control action which could maintain the continuous balance of input and output mode of control which will accomplish this is known as "PROPORTIONAL CONTROL"

Proportional control is defined as follows.

"It is a controller action in which there is a continuous linear relationship between value of the controlled variable and position of the final control element within the proportional band".

The tuning parameters for proportional control are,

Proportional Gain $[K_p]$,

Time Delay $[T_d]$

Proportional Band $[P_b]$

Proportional band is defined as the percent deviation in measurement of its full scale required to give 100% valve deviation. Narrow band proportional control gives a comparatively large corrective action to the valve for a small change in the measurement. For wide band proportional the corrective action to the valve is small band therefore the offset will be large. Usually, narrow proportional band is preferred. If proportional band is zero, the controller behaves as two position control.

i. Time Delay

Time required to take the successive samples of process variable.

Proportional + Integral (P+I)

The proportional control mode provides a stabilizing influence while the integral mode will help to overcome OFFSET. Integral controller will provide corrective action as long as there is deviation in the controlled variable from the set point value.

Integral control has a phase lag of 90 degrees over proportional control. This lagging feature of reset will result in a slow response and oscillation will come into picture.

This is suitable for flow control and pressure control where the process has little lag. But a wide proportional band is required for stability. The small process lag permits the use of a large amount of integral action.

Proportional derivative (P+D)

Derivative control action combined with proportional gives a controller which is good on process containing appreciable lag. Because the process lag can be compensated by the anticipatory nature of derivative action (i.e.) derivative action provides the boost necessary to counter at the time delay associated with such control by 90 degree.

Since this controller combination is most effective where the system lags are high, it could be used on most multi capacity process applications. Where the process lag is

short, this combination could not be used. This controller combination does not eliminate OFFSET after a sustained load disturbance. It does reduce the magnitude of OFFSET because of narrow proportional band.

A Proportional plus derivative controller properly fitted and adjusted to the process acts to prevent the controlled variable from deviating excessively and reduces time required to stabilize.

Proportional + Integral derivative (P+I+D)

This controller offers the benefit of each control action and moreover the effect duplicates the action of good human operator of the process. A three mode controller contains the “stability “of ability to provide an immediate correction for the magnitude of a disturbance because of rate control.

Pre-Lab Questions:

Pressure controller

- 1.Name some pressure transducer.
- 2.Define PB.
- 3.What is two position control/
- 4.Define offset.
- 5.What is integral windup?

Post Lab Questions:

- i. Why derivative mode is nor used alone?
- ii. What are the advantages of pi controller?
- iii. What are the drawbacks of p controller?
- iv. Comment on the responded of P, PI, PID controllers.
- v. What is differential gap?

Level Controller

Pre-Lab Questions:

1. Name some level transducer.
2. Define PB.
3. What is two position control/
4. Define offset.
5. What is integral windup?

Post Lab Questions:

1. Why derivative mode is nor used alone?
2. What are the advantages of pi controller?
3. What are the drawbacks of p controller?
4. Comment on the responded of P, PI, PID controllers.
5. What is differential gap?

FLOW PROCESS CONTROLLER

1. Name some flow transducer.
2. Define PB.
3. What is two position control/
4. Define offset.
5. What is integral windup?

Post Lab Questions:

1. Why derivative mode is not used alone?
2. What are the advantages of pi controller?
3. What are the drawbacks of p controller?
4. Comment on the response of P, PI, PID controllers.
5. What is differential gap?

Temperature Controller

Pre-Lab Questions:

1. List out some temperature transducers.
2. What is the principle of RTD?
3. Write the equation of RTD.
4. What are the different types of RTD?
5. Mention some of the safety factors to be followed before starting the experiment?

Post Lab question:

1. What is the purpose of using rotameter?
2. Which component is used as FCE in this process?
3. What is controller tuning?
4. What are the different tuning methods of controller?
5. What is the principle of SCR?

EXPERIMENT 11: SIMULATION OF PID CONTROLLER USING MATLAB/ SIMULINK

Aim: To analyze the response of a second order system using PID controller

Apparatus: Matlab software

Theory:

Proportional-Integral-Derivative (PID) controller is widely employed tool to reduce the transient and steady state behavior of a control system. The PID controller is very understandable it is quite effective. One attraction of the PID controller is that all engineers understand conceptually differentiation and integration, so they can implement the control system even without a deep understanding of control theory. Further, even though the compensator is simple, it is quite sophisticated in that it captures the history of the system (through integration) and anticipates the future behavior of the system (through differentiation). We will discuss the effect of each of the PID parameters on the dynamics of a closed-loop system and will demonstrate how to use a PID controller to improve a system's performance.

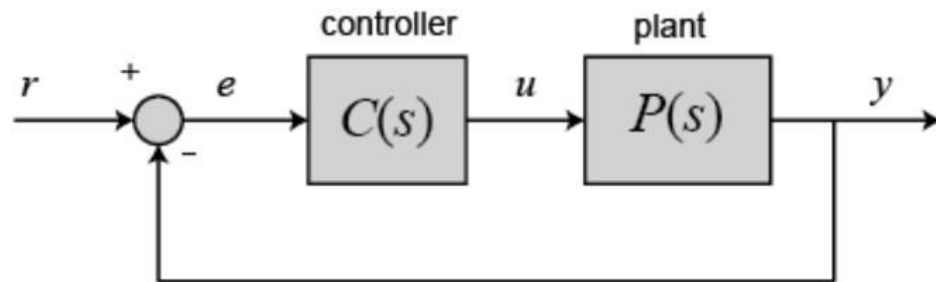


Figure: Controller based feedback system

The output of a PID controller, which is equal to the control input to the plant, is calculated in the time domain from the feedback error as follows:

$$u(t) = K_p e(t) + K_i \int e(t) dt + K_d \frac{de}{dt} \quad (1)$$

First, let's take a look at how the PID controller works in a closed-loop system using the schematic shown above. The variable (e) represents the tracking error, the difference between the desired output (r) and the actual output (y). This error signal (e) is fed to the PID controller, and the controller computes both the derivative and the integral of this error signal with respect to time. The control signal (u) to the plant is equal to the proportional gain (K_p) times the

magnitude of the error plus the integral gain (K_i) times the integral of the error plus the derivative gain (K_d) times the derivative of the error.

This control signal (u) is fed to the plant and the new output (y) is obtained. The

new output (y) is then fed back and compared to the reference to find the new error signal (e). The controller takes this new error signal and computes an update of the control input. This process continues while the controller is in effect.

The transfer function of a PID controller is found by taking the Laplace transform of Equation (1),

$$K_p + \frac{K_i}{s} + K_d s = \frac{K_d s^2 + K_p s + K_i}{s}$$

In equations (1) & (2) K_p = proportional gain, K_i = integral gain, and K_d = differential gain

Characteristics of PID controller:

Increasing the proportional gain (K_p) has the effect of proportionally increasing the control signal for the same level of error. The fact that the controller will "push" harder for a given level of error tends to cause the closed-loop system to react more quickly, but also to overshoot more. Another effect of increasing K_p is that it tends to reduce, but not eliminate, the **steady-state error**.

The addition of a derivative term to the controller (K_d) adds the ability of the controller to "anticipate" error. With simple proportional control, if K_p is fixed, the only way that the control will increase is if the error increases. With derivative control, the control signal can become large if the error begins sloping upward, even while the magnitude of the error is still relatively small. This anticipation tends to add damping to the system, thereby decreasing overshoot. The addition of a derivative term, however, has no effect on the steady-state error.

The addition of an integral term to the controller (K_i) tends to help reduce steady-state error. If there is a persistent, steady error, the integrator builds and builds, thereby increasing the control signal and driving the error down. A drawback of the integral term, however, is that it can make the system more sluggish (and oscillatory) since when the error signal changes sign, it may take a while for the integrator to "unwind."

The general effects of each controller parameter (K_p , K_d , K_i) on a closed-loop system are summarized in the table below. Note, these guidelines hold in many cases, but not all. If you truly want to know the effect of tuning the individual gains, you will have to do more analysis, or will have to perform testing on the actual system.

CL RESPONSE	RISE TIME	OVERSHOOT	SETTLING TIME	S-S ERROR
K_p	Decrease	Increase	Small Change	Decrease
K_i	Decrease	Increase	Increase	Decrease

Kd	Small Change	Decrease	Decrease	No Change
-----------	-----------------	----------	----------	-----------

Objective: Analyze the response of the PID controller based feedback system with transfer function given by,

$$G(s) = \frac{1}{s^2 + 10s + 20}$$

And obtain fast rise time, minimal overshoot and zero steady state error.

Procedure:

1. Open a new .m file in MATLAB.
2. Write the code to analyze the open loop step response of $G(s)$ and note down the values of rise time, overshoot and settling time.
3. Now, enter $K_p = 300$ and include in $G(s)$ as **plant (s) = $K_p * G(s)$** and analyze the positive feedback response of the plant and note down the values of rise time, overshoot and settling time.
4. Now, include $K_d = 10$ and $K_p = 300$, PD controller, include in $G(s)$ as **plant (s) = $(K_d s + K_p) * G(s)$** and analyze the positive feedback response of the plant and note down the values of rise time, overshoot and settling time.
5. Before PID controller, we analyze the PI controller effect also. To do that, include $K_i = 70$ and $K_p = 300$, PI controller, include in $G(s)$ as **plant (s) = $(K_i/s + K_p) * G(s)$** and analyze the positive feedback response of the plant and note down the values of rise time, overshoot and settling time.
6. For PID controller case, take $K_p = 300$, $K_d = 10$, and $K_i = 70$ and formulate **plant (s) = $(K_i/s + K_p + K_d s) * G(s)$** , and obtain the step response of the plant. Also note down the values of rise time, overshoot and settling time.
7. Record the observations seen from step 3. To step 6. And tabulate the observations.

MATLAB CODE:

```

%%Analysis of PID controller clc;clear;
%% Transfer function s = tf('s');
G = 1/(s^2 + 10*s + 20);
figure(1) step(G)
%% Proportional control Kp = 300;
C = pid(Kp) plant = C*G;
T = feedback(plant,1)
t = 0:0.01:2;
figure(2) step(T,t)

```

%% Proportional-derivative control Kp = 300;

Kd = 10;

C = pid(Kp,0,Kd) plant = C*G;

T = feedback(plant,1)

t = 0:0.01:2;

figure(3) step(T,t)

%% Proportional-integral control Kp = 30;

Ki = 70;

C = pid(Kp,Ki) plant =

C*G;

T = feedback(plant,1) t = 0:0.01:2;

figure(4) step(T,t)

%% Proportional-integral-derivative control Kp = 350;

Ki = 300;

Kd = 50;

C = pid(Kp,Ki,Kd) plant = C*G;

T = feedback(plant,1) t = 0:0.01:2;

figure(5) step(T,t)

Observations:

Method	Rise time (tr)	Peak over shoot (Mp %)	Settling time (ts)
P			
PD			
PI			
PID			

Field controlled method:

S.No.	Field resistance in ohms	Field current in amperes	Speed in rad/s	Speed in rpm
1	1			
2	50			
3	100			
4	150			
5	200			

Result:

Questionnaire:

1. Define a proportional controller.
2. Define a PD controller.

3. Define a PI controller.
4. Define a PID controller.
5. What is the effect on rise time if the close loop response of the system is incorporated with PD control?
6. How can you differentiate between a PI response and a PD response?
7. Which control best suits to minimize the steady state error of a second order system and why?
8. What is the variation in natural and damped frequencies of the second order system's step response applied with a proportional control?
9. If an integral control is used in second order system response, its stability will increase or decrease? Please explain.

EXPERIMENT 12: STUDY OF COMPLEX CONTROL SYSTEM USING MATLAB

Aim: To study the complex control system using matlab and to compare the response of simple and cascade loop.

Apparatus Required: Personal computer

MATLAB Software

Theory:

Cascade control configuration we have one manipulated variable and more than one measurement. It is clear that with a single manipulation we can control only one output. Consider a process consisting of two parts as shown in process 1&2 processes; 1 has an output the variable we want to control process 2 has an output that we are not interested in controlling but which affect the output which we want to control. The two controllers of a cascade control system are standard feedback controllers. Generally, a proportional controller is used for a secondary loop, although a pi controller with small integral action is not unusual any offset caused by a p control in the secondary loop is not important since we are not interested in controlling the output of the secondary loop are much faster than those of the primary loop, consequently the phase lag of the closed secondary loop will be less than that of the primary loop. This feature leads to the following important result which constitutes the rationales behind the use of cascade control.

Procedure:

Cascade loop.

1. For cascade control open loop transfer function $G_c(S)$ by Block diagram reduction technique
2. Find value of W and K_c for Cascade Loop Transfer Function $G_c(S)$
3. Determine block diagram of Cascade Control Loop in by substituting Gain K_u for Proportional Controller
4. View Response Using Scope

Simple Loop

- I. Find Simple Loop Transfer Function $G_c(S)$ By Block Diagram Reduction
- II. Repeat Steps from 2 to 4 of Cascade Control Loop
- III. To Find W :
- IV. Write the Phase Angle $\phi\omega$ for Above $G_c(S)$
- V. Equate It To -180°
- VI. Find Ω Value

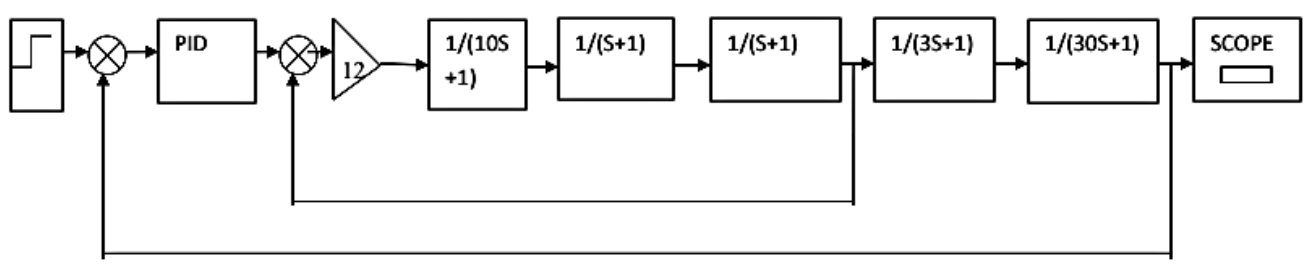
To Find K_u

- I. Write Amplitude Ratio Expression for $G_c(S)$

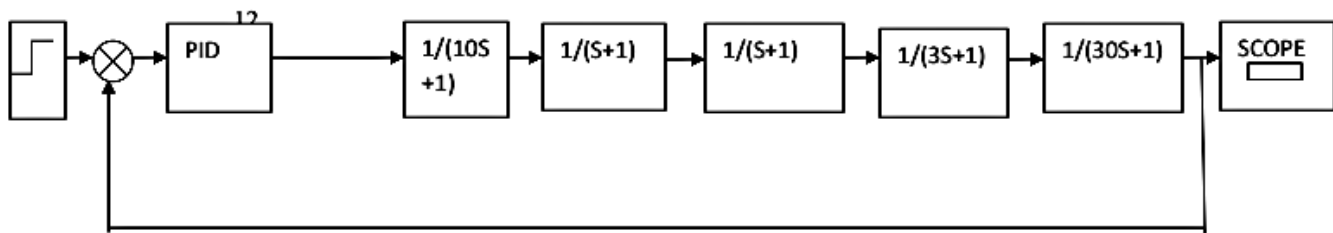
- II. Equate It to "1"
- III. Find Kc Value
- IV. Find Ku Value, $K_u = K_c / 2$

BLOCK DIAGRAM:

CASCADE CONTROL LOOP



SIMPLE CONTROL LOOP



CALCULATIONS:

CASCADE CONTROL LOOP:

$$\frac{G(s)}{1+G(s)H(s)} = \frac{\left[\frac{12}{(s+1)(s+1)(10s+1)} \right]}{\left[1 + \frac{12}{(s+1)(s+1)(10s+1)} \right]}$$

$$= (s+1)^2 \frac{10s+1}{(s+1)^2(10s+1)+12} * \frac{12}{(s+1)^2(10s+1)}$$

$$\frac{12}{10s^3+21s^2+12s+13}$$

$$G(s) \frac{12}{[10s^3+21s^2+12s+13]} \left[\frac{1}{(3s+1)(30s+1)} \right]$$

$$\phi = \tan^{-1} \left[\frac{-10w^3+12w}{-21w^3+13} \right] - \tan^{-1} 3w - \tan^{-1} 30w$$

$$w = 0.54; \phi = -180.02$$

$$Ar = \left[\frac{12Ku}{\sqrt{((-21w^3+13)+(-10w^3+12w^2)(9w^2+1)(900w^2+1))}} \right]$$

$$= \frac{12Ku}{8.446 \cdot 1.9 \cdot 16.23} \approx 1$$

$$Ku = 21.7$$

$$Pu = \frac{2\pi}{w} = 11.6$$

SIMPLE CONTROL LOOP:

$$G(s)/(1 + G(s)H(s))$$

$$\left[\frac{1}{(s+1)(s+1)(10s+1)(3s+1)(30s+1)} \right] \left[\frac{(s+1)(s+1)(10s+1)(3s+1)(30s+1)}{1 + (s+1)(s+1)(10s+1)(3s+1)(30s+1)} \right]$$

$$\emptyset = 1/(jw + 1)(jw + 1)(10jw + 1)(3jw + 1)(30jw + 1)$$

$$\emptyset = -2 \tan^{-1} w - \tan^{-1} 10w - \tan^{-1} 30w - \tan^{-1} 3w$$

$$w = 0.16; \emptyset = -180.04$$

$$Ar = \left[\frac{Ku}{\sqrt{(9w^2+1)(900w^2+1)(100w^2+1)(w^4+2w^2+1)}} \right]$$

$$Ku = 10.47$$

$$Pu = \frac{2\pi}{w} = 39.26$$

SECOND SEMESTER

COURSE TITLE: CHEMICAL ENGINEERING ENTREPRENEURSHIP

COURSE CODE: CHE 410

General Objective 1.0: Entrepreneurial theory

General Objective 2.0 Understand the importance of the environment on entrepreneurship

General Objective 3.0: Understand business ownership types in Nigeria

General Objective 4.0: Understand the need for manufacturing & raw materials availability

General Objective 5.0: Understand small scale business

General Objective 6.0: Understand federal and state governments regulation on chemical industries.

General Objective 7.0 Understand the elements of marketing

General Objective 8.0 Understand market research methods and preparation of feasibility studies.

General Objective 9.0: Understand the personal factors in starting a business.

PART ONE PRODUCTION

1. SOAP PRODUCTION
2. DETERGENT
3. SHAMPOO
4. YOUGHUT PRODUCTION

ENTERPRISE: SOAP MAKING

Products:

1. **Liquid multipurpose wash**
2. **Liquid Anti Septic Soap production**
3. **Air/room freshener**
4. **Body perfume**

INTRODUCTION

Liquid soap are convenience products, compared with powdered detergent dissolve more rapidly, particularly in cold water, they generate less dust. It is not surprising, therefore, those liquid forms of household cleaning product developed by manufacturers. With the exception of fabric softeners and shampoos, the solid form of cleaning preceded the liquid form. This is used as manual and automatic dishwashing, laundry general personal washing products.

As a result, the technical history of liquid detergent is to a large extent one of emulating the performance features of the powder models. Generally, there are two types of liquid detergents:

1. Light duty liquid detergent.
2. Heavy duty liquid detergent.

Production procedure for manufacturing liquid detergent, both the batch as well as continuous blending process is used. Both batch and continuous blending process are used to manufacture liquid and gel cleaning products. Stabilizers may be added during manufacturing to ensure the uniformity and stability of the finished product. In a typical continuous process, dry and liquid ingredients are added and blended to a uniform mixture using in-line or static mixers. Recently, more concentrated liquid products have been introduced of new high-energy mixing process in combination with stabilizing agents. To make liquid detergent, the dry powder is simply mixed back in with a solution consisting of chemicals and water, called as solubilizers.

Uses of Liquid Soap

Liquid soap is formulated with the intension of the following end uses:

1. House hold washing mainly for dish, utensils washing etc.
2. Light duty laundering for synthetic and woolen fabrics.
3. For scouring minerals oiled goods of woolen or worsted piece of fabrics.
4. Car or Rugs cleaning shampoos.
5. For bathing
- . For washing of Hair

Type of Liquid Soap

Liquid Multipurpose Soap

Liquid car wash

Liquid hand wash

Liquid Antiseptic Soap

Shampoo

The equipment's needed for the production are:

- Mixer: This equipment can either be manual or electrical it is made up of a (reactor) bowl and a stirrer. The best material for this is plastic. The size of the mixer depends on the quantity of soap to be produced, as the name implies it is used for mixing the chemicals to form a homogenous product.
- Weighing balance: This is used for the measurement of weight of the chemicals.
- Hydrometer: This is the equipment used for determine the water the water density of the Sodium Hydroxide (caustic soda).
- pH Paper: the approximate pH of a solution can be determined by placing a drop of a solution on a piece of indicator paper. Two types of pH paper are commonly used: litmus paper and universal (Alkacid) paper. The type of pH paper used is dependent on the type of measurement and degree of accuracy required.
- Face Mask: This is an instrument used for nose and mouth protection from direct inhalation of chemicals.
- Lab Coat: it protect the skin from direct contact with the chemicals or from penetration of chemicals.
- Hand Glove: It protect the hand from chemicals.
- Plastic Bowl: Used for storing water
- Packaging containers
- Table spoon: for measuring the desire quantity of some solid chemical
- Storage Tank: This is used for storing water that will be used for diluting the solid chemicals and also for storing liquid products.

Classification of Chemicals used in Liquid Soap Production according to their functions

COLOURATION	Water soluble colour
DEODOURATION	Fragrance of different type
PRESERVATIVES	Methylparaben, Formalin, Sodium Benzoic, Citric acid
SKIN AND COLOUR PROTECTION (ACIDITY REGULATOR)	Industrial salt, STPP
STAIN REMOVAL	Soda Ash, Caustic soda,
THICKENER	Nitrosol, Antisol, Cassava
FOAMING AGENT	Texapon, Sulphurnic, SLS, Cocoamine (foaming Booster)

1.0 LIQUID MULTIPURPOSE SOAP PRODUCTION

Liquid Multipurpose Soap is a type of soap use for domestic use only, it is mainly use in washing our dish, cloth and hand, and it does not contain glycerin which makes the hand mild and soft.

Measurement Table for Liquid Multipurpose Soap Production

Production for 20 litres

Ingredient	Quantity
Water	20litres
Nitrosol/Antisol	kg
Sodium Hydroxide (Caustic soda)	1/8kg or 3 tablespoon
Sodium Carbonate (Soda ash)	1/2 kg
Sulphurnic acid	1/2 litre
Sodium Tripolium phosphate (STPP)	1/2 kg
Sodium Lauryl Sulphate (SLS)	1/2 kg or
Texapon	1/4 kg
Booster (Optional)	1/2 litre
Industrial salt	20gm
Formalin, Methylparaben or Sodium Benzoic	100ml or 1/8kg
Aromatic Agent	as desired
Colour	as desired

Note: Some other chemicals can also be added to the above mentioned chemicals from your own discovery during research.

Procedure for Liquid Multipurpose Soap Production

To produce a standard liquid multipurpose soap, the steps below will guard you during production.

Step 1: Determine the quantity of soap to be produce.

Step 2: Soak the Soda Ash with 1litre of water and label it A

Step 3: Soak the Sodium Tripolium Phosphate (STPP) with 1lire of water and label it B

Step 4: If Methylparaben or Sodium Benzoic is to be use, it should be soaked with 1/zlitre of water and label it C, if otherwise skip this step.

Step 5: Soak also SLS with 1litre of water in a separate container and label D

Step 6: The required quantity of Caustic soda should also be soaked with little: water in a separate bowl and label in E

Step 7: Soak the required quantity of Nitrosol or Antisol (CMC) with about 80% (70litres) of litres of the total volume of water in a production tank or bowl. For Antisol, it should be soaked for 24hours or more,for adequate dissolve and label it F.

NOTE: All the chemicals listed in step 2-7 should be allow soaking for 2-5minutes. Except for Antisol which will soak for 24hours or more.

Step 8: To sample F add sample A, C,D and E and stir well until homogeneity is obtain.Then label it G

Step 9: Add texapon to step G and further stir well

Step 10: Add surlphurnic acid to step 9 above and further stir in one direction until the foam goes down.

Step 11: Add industrial salt to step 10 above

Step 12: If formalin or methylparaben (sample B) is to be used as preservative, it should be added to step 11 and lastly add the required quantity of colour and Fragrant and allow the product to stay for 1-5hours.

Step 13: The last and the important thing to do is packaging and labelling of your product in attractive containers.

The Market of Liquid Multipurpose Soap

Liquid dish soap is now a thing of admiration especially in the restaurants, Hotels,Eatery's, youth and students.

2 LIQUID BODY WASH/BATH GEL PRODUCTION

Liquid body wash/bath gel soap is the type of soap use for bathing. It can either be in Antiseptic body wash, Medical body wash, Liquid lightening/bleaching body wash or ordinary form. The differences in

the categories are the addition of some chemicals.

3. Liquid Antiseptic Soap Production

Liquid Antiseptic soap is types of soap used for bathing. It contains more ingredients than any other liquid soap. Liquid Antiseptic Soap is a double action body soap Antiseptic chemicals which include Pine oil, Phenol, chloroxylenol, Chlorophenol or Salicyclic acid.

Measurement Table for Liquid Antiseptic Soap production

Production

Ingredient	Quantity
Nitrosol/Antisol	1/2kg
Sodium Carbonate (Soda ash)	1kg
Sodium Tripolium phosphate (STPP)	1kg
Sodium Lauryl Sulphate (SLS)	1/2kg
Sodium Hydroxide (Caustic soda)	1/8kg 3 tablespoon
Texapon	1kg
Sulphurnic acid	4-5litres
Booster (Optional)	1litre
Industrial salt	100gm
Methylparaben or Sodium Benzoic	1/8kg
Colour	To taste
Glycerine	1/2litre
Pine oil, Chloroxylenol or Phenol	1/2litre or 1/8litre
Menthol crystal	1spoon
Aromatic Agent	200ml (optional)

Note: Chemical, such as Pine oil, Chloroxylenol and Phenol can be substitute for each other. And some other additions chemical can also be used if discovered during research.

Procedure for Liquid Antiseptic Soap Production

To produce a standard Liquid Antiseptic Soap that will meet the market demand, the steps below will guide you during the production.

Step 1: Determine the quantity of soap to be produce.

Step 2: Soak the Soda Ash with 1litre of water and label it A

Step 3: Soak the Sodium Tripolium Phosphate (STPP) with 1litre of water and label it B

Step 4: If Methylparaben or Sodium Benzoic is to be use, it should be soaked with 1/2litre of water and label it C, if otherwise skip this step.

Step 5: Soak also SLS with 1litre of water in a separate container and label D

Step 6: Soak the required quantity of Nitrosol or Antisol (CMC) with about 80% (70litres) of the total volume of water in a 24hours or more, for adequate dissolve and label it E

Note: All the chemicals listed in step 2-6 should be allow soaking for 2-5minutes. Except for Antisol which will soak for 24hours or more.

Step 7: To sample E add sample A, B, C and D and stir well until homogeneity is obtain. Then label it F.

Step 8: Add Texapon to sample F and further stir well

Step 9: Add surlphurnic acid to step 8 above and further stir in one direction until the foam goes down.

Step 10: Add industrial salt to step 9 above

Step 11: Add sample B (Methylparaben or Sodium Benzoic) to step 10.

Step 12: Add glycerin, pine oil, phenol or Chloroxylenol to step 11 allow the product to stay for about 1-2hours.

Step 13: Lastly add the required quantity of colour and allow the product to settle

Step 13: The last and the most important thing to do is the packaging and labelling of your product in an attractive containers.

4. AIR FRESHNER PRODUCTION

Air freshener are consumer product used in home or commercial product used in restrooms that typically emit fragrance

Types of room freshener: There are two types of room freshener

1. Water type (Non-Transparent type)
2. Alcoholic type (transparent type)

Water Types of Room Freshener

Air freshener is produce from simple combination of Scents, Catalyst, Texapon, Menthol, Methanol, Thickener and water in a standard formula.

Chemicals and ingredients use in water types in water types of Air freshener production

Ingredient	Quantity
Scent (fragrance of different type)	1 litre
Catalyst or Texapon	1/4 litre or 1/4 kg
Menthol a pinch (optional)	
Formalin, methylparaben or sodium benzoic	20ml or 1/2 spoon
Methanol or ethanol (optional)	1 litre
Thickener (white binder, Antisol)	1 litre (optional)
Water	10-15 litres
Colour	as desired

Procedure for Air Freshener's Production

Step 1: Determine the quantity of the product that you wish to produce.

Step 2: Mix 1litre of scent of your choice (Banana, Mango, Vanilla, 212, Strawberry, Andepo, Lemon, Orange etc.) in a separate bowl and label A

Step 3: Add require amount of Texapon to step 2 above stire vigorously and label as B

Step 4: Dissolve the require amount of thickener with little water and add it to step 2 or 3above

Step 4: Add 10 – 15 litres of water to step 4 above and stir well.

Step 5: Add methanol or Ethanol (optional) and menthol to step 4 above and allow for few minutes before packaging.

Step 6: Filter the product with white cloth after the completion of the reaction and package

6. PERFUME PRODUCTION

Perfume is a fragrant liquid made from extract that has been distilled in alcohol and water. A perfume is composed of three notes. The base note is what a fragrance will smell like after it has dried. The smell that develops after the perfume has mixed with unique body chemistry is referred to as middle note. And the top note is the first smell experienced in an aroma. Each perfumery has a preferred perfume manufacturing process, but there are some basic steps.

The Alcoholic type

Ingredient	Quantity
Scent (Fragrant of any type)	1Litre
dCatalyst/Glycerin	100ml (optional)
Methanol or Ethanol	1-5 litres

Procedure for perfume production

The following step should be followed to produce standard perfume

Determine the quantity of perfume to be produce.

Step 1: Measure the required quantity of the scent of your choice in a separate bowl and label it A

Note: Two or more scent (fragrant) can be used together but care must be taking when carrying out scent combination because some scent generate an offensive odor when mix together.

Step 2: Add the required quantity of Glycerin to step 1 above and stir well and label it B

Step 3: Further add B into A and mix well until it blend together then finally add the require.

7. SHAMPOO

Shampoos like detergents also foam in water and are easily washed off from the hair and scalp. They

are easily washed off from the hair and scalp.

The properties of a good shampoo are:

1. It must have a pH of 5.5.
2. It must be easy to rinse off.
3. It must give a rich foaming lather in water.
4. It must have a long shelf life.
5. It must cleanse the hair.

Forms of Shampoo Products in Nigeria

There are various forms of shampoo products in Nigeria. This is because of value being added to shampoo to make different shampoo products. The various forms of shampoo products are:

1. Liquid clear shampoos
2. Liquid cream shampoos
3. Cream paste shampoos
4. Beer shampoos
5. Dry shampoos
6. Liquid dry shampoos
7. Baby shampoos

Tool/Materials/Ingredients Needed Tools

- a. Mixing machine
- b. Cooling machine
- c. Bottle filling machine
- d. Bottle sealing machine
- e. Weighing machine
- f. Laboratory equipment
- g. Open tank with speed stirred

Ingredients

- a. Nitrosol: half cup
- b. Sulphuric acid: one-quarter litre
- c. Caustic soda: one and a half cup
- d. Soda ash: one and half cup
- e. Fragrance: As desired
- f. Industrial camphor: 1 tablespoon
- g. Preservative (Formalin): 1 tablespoon
- h. Colouring: As desired
- i. Distilled water: 10 litres
- j. Vitamin E
- k. Glycerine

Step-by-Step Process of Production

- a. Pour distilled water into a bowl.
- b. Pour Nitrosol into water and mix thoroughly.
- c. Leave the mixture for 24 hours.
- d. Pour caustic soda into water.
- e. Stir the solution.
- f. Add soda ash to water in another bowl.
- g. Stir the solution.
- h. Add the soda ash solution to the caustic soda solution.
- i. Mix the two solutions together thoroughly.
- j. Add sulphuric acid to the soda solution.
- k. Stir well to mix thoroughly.
- l. Add preservative to the mixture and stir thoroughly to mix well.
- m. Add colour to the mixture and stir well.

- n. Add perfume to the mixture and stir well.
- o. Add vitamin E to the mixture and stir well.
- p. Add glycerine to the mixture and stir well.
- q. Stir all the ingredients together to obtain a homogeneous solution.
- r. Leave the mixture and allow it to stay from some hours before packaging into squirt bottle, pump bottle for sale.

Black Shampoo Production

Ingredients:

- a. Black soap- 1 bar
- b. Hot water-1 cup
- c. Honey-1 tablespoon
- d. Essential oil

Steps

- a. Break the bar soap into crumbles.
- b. Drop the crumbs of bar soap into the shampoo bottle.
- c. Add hot water to the soap crumbs.
- d. Add honey to the soap crumbs.
- e. Add some drops of essential oil to the mixture.
- f. Shake the mixture properly.
- g. Allow the mixture to cool down for some time.
- h. Your black soap shampoo is ready for use.
- i. Apple Cider Vinegar Shampoo

Ingredients

- 1. Olive oil-5 tablespoons
- 2. 1 egg
- 3. Lemon juice-5 tablespoons
- 4. Apple Cider Vinegar-5 tablespoons
- 5. Blender
- 6. Plastic bottle

Steps

- a. Pour the olive oil, egg, lemon juice and Apple Cider Vinegar into a blender.
- b. Blend the ingredients until a smooth mixture is obtained.
- c. After blending, allow to settle for some time.
- d. Pour into a plastic bottle.
- e. Take some quantity and use it to massage your scalp.
- f. Rinse off using lukewarm water.
- g. Aloe Vera Hair Shampoo

Materials

- 1. Lemon juice- 2 tablespoons
- 2. Castle soap-1 tin
- 3. Aloe Vera gel-2 tablespoons
- 4. Plastic bottle

Steps

- a. Pour squeezed lemon juice, castle soap and Aloe Vera into a plastic bottle.
- b. Shake to mix the ingredients thoroughly.
- c. Allow the mixture to settle for some time.
- d. Take some quantity and use it to massage your hair.
- e. Rinse your hair with clean water.

Packaging Strategies

- a. Specify the ingredients used for producing the shampoo.

- b. State the name of the product.
- c. State the address of the manufacturer.
- d. Explain how to use the shampoo
- e. List the batch number of the shampoo.
- f. List the manufacturing date if the shampoo.
- g. List the expiry date of the shampoo.
- h. State the NAFDAC number of the product.
- i. Package into plastic bottles, squirt bottles and pump bottles for sale.

Marketing Strategies

- a. Can be marketed directly to wholesalers and retailers.
- b. Can be marketed directly to hairdressing saloons.
- c. Can be marketed online using the producer's website.
- d. Can be marketed on social media.

8. PAINT

Paint is typically made up of four main components: resin, additives, solvent and pigments. Each has an important purpose which we will explore in more detail below.

1. **Pigments** give paint its colour. The basic white pigment typically used is titanium dioxide, and other pigments that add colour include iron oxide and metallic salts for example [\[1\]](#).
2. **Resin** is used in paint to hold all the pigments together and help the paint dry. You can get both natural (e.g. linseed oil, soybean oil) and synthetic (e.g. acrylics, epoxies) resins [\[1\]](#).
3. **Additives** act as fillers (e.g. calcium carbonate [\[1\]](#)) and can sometimes act as anti-fungal agents.
4. **Solvents** [\[2\]](#) or **water-based** are either chemical-based liquids (e.g. alcohols, acetone [\[1\]](#)) or water-based that make paint easier to apply to surfaces.



TABLE 3.0: PERCENTAGE OF COMPONENTS FOR THE FORMULATION OF PAINTS

COMPONENTS	PERCENTAGE BY MASS	
	GLOSS	EMULSION
Alkyd resin binder	54	0
Pigment	25	0
Solvent	17	25 to 50

Additives	4	2 to 5
Co-polymer binder	0	15 to 23
Pigment (white)	0	20
Pigment (color)	0	0 to 5
Extenders	0	15 to 25

2.1

PAINT MANUFACTURING PROCESS

In the process of getting a finished paint product, two stages of production are involved, i. the production process and ii. The post-production process,

- i. **The production process** : this is a step by step procedure taken in charging all the various components of paints into the mixing vessel, one after the other to achieve an homogeneous texture and blend of paint, the steps below may require up to 50minutes for a complete blend of an emulsion paint, while it may require 2hr to 3hours for a gloss paint.

Note : That in paints production the taste or texture of the color demanded, determines the production time or residence time of the paints.

TABLE 3.1: STEP BY STEP CHARGING OF COMPONENTS INTO THE MIXING VESSEL

STEPS	COMPONENTS
1	Solvent
2	Extenders
3	Additives
4	Pigments
5	Binder

- ii. **Post-production process of paints** : After completion of the mixing process, giving an homogeneous product, the post production process listed below are carried out to give the paint a finishing touches,
 - Laboratory Certification
 - Writing of batch number
 - Labeling
 - Packaging and corking
 - Cleaning/washing the equipments.

The student was fully involved in the production process and the post production process of paints in powerseal paints factory.

Our paint is generally manufactured in batch lots from 200 litres for special products and colours to 10,000 litres for mainline white products.

The manufacturing process involves five critical parts:

Part 1 – Accurate measurement of ingredients

Ingredients are typically measured by weight on scales, and in some cases by volume in calibrated vats and graduated measuring containers. For batches larger than about 4000 litres the vats are loaded onto load cells connected to accurate electronic scales. The scales allow addition of ingredients with a measurement accuracy of +/- 5 kg in 20,000 kg. Where greater accuracy of small additions is required highly accurate floor scales are used.

Part 2 – Mill-base preparation and pigment dispersion

Pigments are powders of typically small size that tend to stick together to form clumps or agglomerates. These must be broken down into separate particles that must then be wetted by resin and additives to stop them sticking together again. This is the process of dispersion.

High speed mixers are used for combining materials and dispersing most pigments. These machines rotate stainless steel serrated discs of up to 60cm diameter, at up to about 1000 rpm. The largest machine also has rotating scraper blades to ensure material doesn't stick to the sides of the mill-base vat.

Pigments are added slowly, from containers of 10 kg up to 1000 kg, to a portion of the liquid paint components, with the mixer running, to form the mill-base.

Certain pigments are more difficult to disperse and require ball milling, bead milling or bar milling.

Ball mills are used for small batches of difficult to disperse mill-bases. Ball mills are large porcelain lined rotating drums containing golf ball sized porcelain balls. Rolling and tumbling of the balls provides sufficient force to break up agglomerates.

Bead mills are used for large batches and can maintain semi-continuous output. Operation is similar to a ball mill but the vessel is smaller and balls are 3mm diameter zirconium dioxide beads, and mixing is at a higher speed producing more rapid output.

Bar mills are especially useful for highly viscous (thick) mill-bases. The mill-base is forced through a small clearance between a rotating water cooled roller and a bar pushed against the roller.

Part 3 – Let-down

In a separate, larger, vat the rest of the paint (resin, solvent and additives) is combined and mixed. This is called the let-down. When the let-down and the mill-base are completed, the mill-base is added with stirring to the let-down. At this stage, if required by the formulation, any final additions are made and added in.

Part 4 – Finished product and in process laboratory testing

Product quality is monitored throughout the manufacturing process by the Product Verification Laboratory. Critical ingredients are tested before manufacturing starts. The mill-base may be tested

for dispersion; if necessary further processing may be required. The let-down may be tested to ensure it is sufficiently mixed.

The completed batch (mill-base plus let-down and any final additions) is thoroughly tested by the Product Verification Laboratory. These final tests evaluate properties such as degree of dispersion, viscosity (consistency), density, hiding, tint strength and colour, application, dry time, gloss and dry film appearance.

Part 5 – Canning

When testing is completed the batch is passed for canning. During canning two samples are taken; a retain sample, which is stored in case it is needed for future reference, and a Final Inspection sample. The Final Inspection sample is tested in the Resene Final Inspection Laboratory to guarantee conformance to relevant standards and specifications, such as APAS, Environmental Choice, etc. After this final stage of testing is complete the batch is passed for use in the warehouse and dispatch to branches as required.

How does paint work?

The simplest types of paint are lacquers that form a film by evaporation of solvent.

Waterborne paints, such as Resene Hi-Glo, are usually based on emulsion resins that consist of trillions of tiny resin particles, about 1/100th of the diameter of a human hair. As the water in these paints evaporates the resin and pigment particles get closer and closer together until they begin to touch each other. When the resin particles touch each other and the pigments, they stick together and fuse into a tough elastic solid, which we recognise as the paint film.

Solventborne enamel paints, such as Resene Super Gloss, are based on alkyd resin dissolved in solvent (mineral turps). When the solvent evaporates the first stage is the formation of a tacky lacquer. The alkyd resin progressively reacts with oxygen from the atmosphere and polymerises to form a hard, tough coating.

Two component protective coating paints are unreactive on their own, but when mixed together undergo a chemical reaction. The chemical reaction takes a few hours (depending upon temperature) and results in an extremely tough, hard coating with great adhesion. These paints generally require specialist surface preparation and application, and are often used in extreme environments.

The boundaries between waterborne paints and solventborne enamels, and other reactive coatings, are becoming blurred as new technologies develop. For example, Resene Enamacryl and Resene Lustacryl consist of waterborne emulsion resins and chemistry that produces polymerisation of the dried film similar to solventborne enamels.

Example of the manufacturing process for a semi-gloss waterborne white

Mill-base: High speed mixer

1. Water, propylene glycol, surfactants, dispersants, defoamer and biocide are measured out and added to a dispersion vat. This is the start of the mill-base.
2. The liquid measure is premixed for about five minutes.
White pigment (titanium dioxide) and extender pigment (calcite, talc, barytes) are added in a
3. predetermined order with the disperser running. The disperser blade speed is gradually increased as pigment is loaded. Water is also added in stages to maintain optimum viscosity for

mill-base dispersion. The finished mill-base is then tested.

4. Remaining additives and some thickener and water are added and mixed.

Let-down

Meanwhile in a larger let-down vat, the emulsion resin component is measured out. This has

5. defoamer, thickener and coalescent solvent added with stirring and is mixed for about 30 minutes.
6. When both mill-base and let-down are completed, the mill-base is pumped into the let-down, while the hydraulic mixers attached to the vat maintain good agitation.

Finished product

Once all the mill-base is added, and the mill-base vat washed out, the nearly completed paint is

7. mixed for about 30 minutes. Then additional thickeners and remaining additives are measured out and mixed into the batch.
8. A sample of the batch is taken to the laboratory for analysis.
9. Depending upon the results of testing, the batch may be fine tuned for viscosity and mixed for a further period of time.

A predetermined series of tests are carried out on a sample of the batch. Where tint strength

10. and colour are specified, the batch will also be tested for properties such as tint acceptance and compatibility with tinters.

Canning

11. When the batch passes through the primary stage of testing it is approved for canning and then packed into a specified series of containers. Retain and Final Inspection samples are collected.
12. If the product is APAS and EC approved it is held in quarantine while a sample is subjected to further tests in our Final Inspection Laboratory.
13. When all tests are completed, and results recorded and checked, the Final Inspection Laboratory issues a release from quarantine to the warehouse.
14. When the Final Inspection Laboratory request is received the batch is moved into warehouse general stock for dispatch as required to meet orders.

The normal production time for steps 1-14 is about two days.

Example of the manufacturing process for a gloss solventborne bright red:

Mill-base: High speed mixer and bead mill

1. Check organic bright red pigment for quantity and grade.
2. Solvents, alkyd resin, dispersing resin, dispersants and anti-settling aids are added to the mill-base vat.
3. The liquid mixture is premixed for about five minutes.
4. The organic bright red pigment is added very carefully and mixed in to form the mill-base.
5. The mill-base is dispersed by the high speed mixer for about 20 minutes to predisperse the pigment.

After premixing, the mill-base is pumped through a horizontal bead mill. A sample is taken to

6. test dispersion. Depending on the result the mill-base may be further bead milled. Typically several passes are required to ensure all the agglomerates are broken up.

Let-down

7. Meanwhile in a larger let-down vat, alkyd resin, solvent, driers (metal soaps) and anti-skinning agents are measured out and mixed together to form the let-down.
8. When both mill-base and let-down are completed, the mill-base is pumped into the let-down

while the mixture is stirred. Stirring continues for about 30 minute to ensure a uniform mixture.

Finished product

9. Final additions of solvents and additives are made and mixed into the batch.
10. A sample is taken to the laboratory for analysis.
11. The batch is fine tuned to viscosity as necessary based on the results of the tests.
12. Further samples are taken and the batch is tested for properties including gloss, colour, tint acceptance and drying.

Canning

When the primary stage of testing is completed the batch is passed for straining and canning.

13. While being strained the batch is periodically sampled to ensure satisfactory straining is maintained. Retain and Final Inspection samples are collected.
14. If the product is APAS approved it is held in quarantine while a sample is subjected to further testing in our Final Inspection Laboratory.
15. When all tests are completed, and results recorded and checked, the Final Inspection Laboratory issues a release from quarantine to the warehouse.

The normal production time for steps 1-15 is about four days.

Glossary

Agglomerate:	A clump of many small particles that are stuck together.
Alkyd resin:	A synthetic resin used in oil based paints. An alkyd resin is made by reacting a natural drying oil with a hard, synthetic material.
Biocide:	A chemical used in very small amounts to control the growth of bacteria and fungi in paint. Biocides are used to prevent spoilage of paint in the can and to prevent fungal attack of the dried paint film.
Coalescent:	A solvent used to soften emulsion resin particles so that they stick together properly when the water evaporates and the film forms.
Defoamer:	A specially formulated additive to break down foam bubbles and prevent the formation of bubbles during mixing and application.
Dispersant:	Most often a salt (e.g. sodium) of a poly-acrylic acid. Used to help break up agglomerates.
Organic pigment:	Pigments based on carbon chemistry are described as 'organic'. In contrast pigments based on minerals (such as titanium dioxide and calcium carbonate in calcite) are described as inorganic. Examples of organic pigments are: Quinacridone (magenta), Diketo Pyrrole (red), Copper Phthalocyanine (blue and green), Arylamide (yellow), etc.
Polymerisation:	A chemical reaction where molecules grow larger and longer. The result is to produce a polymer that is tougher and harder than the starting point.
Surfactant:	A detergent or soap used to stabilise emulsions and dispersions of pigment. Surfactants help prevent particles sticking together.

PART TWO

- EQUIPMENT DESIGN CONCEPT
- MODELLING PROTOTYPE
- FABRICATION.

Chemical engineering involves the design and fabrication of equipment used in various chemical

processes. This manual serves as a guide for aspiring chemical engineering entrepreneurs to understand the basics of equipment design, prototyping, and fabrication.

Importance in Chemical Engineering:

- Provides practical knowledge on designing and fabricating equipment.
- Essential for scaling chemical processes for industrial applications.
- Equips entrepreneurs with the skills to create innovative solutions.

Understanding Equipment Design Requirements:

- Define the purpose and functionality of the equipment.
- Determine the design specifications based on process needs.

Types of Equipment:

- Reactors: Used for carrying out chemical reactions.
- Heat Exchangers: Transfer heat between two fluids.
- Pumps: Move liquids through a system.

Material Selection and Safety Considerations:

- Choose materials resistant to corrosion and high temperatures.
- Ensure compliance with safety and regulatory standards.

Basics of Equipment Modeling:

- Use of software like AutoCAD and SolidWorks for 3D designs.
- Simulating equipment performance before fabrication.

Building Prototypes:

- Steps: Conceptual design, creating models, testing prototypes.
- Best practices: Use accurate measurements and high-quality materials.

Fabrication Techniques:

- Welding: Joining metal parts together.
- Cutting: Shaping materials to desired dimensions.
- Bending: Forming materials into specific shapes.

Equipment Assembly and Testing:

- Assemble components based on the design model.
- Perform quality checks and safety tests before deployment.

Market Research and Identifying Opportunities:

- Analyze industry trends and market needs.
- Focus on designing cost-effective and efficient equipment.

Cost Estimation and Budgeting:

- Prepare a budget for materials, labor, and overhead costs.
- Plan for unexpected expenses.

Business Plan Development:

- Define your target market and competitive advantage.
- Outline a clear roadmap for scaling your equipment manufacturing business.

Step-by-Step Guide:

1. Define the reactor's purpose and specifications.
2. Use modeling software to design the reactor.
3. Select materials based on the chemical process.
4. Fabricate components and assemble the reactor.
5. Test for leaks, pressure resistance, and efficiency.

Challenges and Solutions:

- Challenge: Material corrosion ? Solution: Use stainless steel or corrosion-resistant alloys.
- Challenge: High manufacturing cost ? Solution: Optimize design to reduce material usage.

Key Takeaways:

- Equipment design and fabrication are crucial for chemical engineering processes.
- Understanding design principles and fabrication techniques can open doors for entrepreneurship.
- Continuous learning and adaptation are essential for success.

Resources for Further Learning:

- Books on equipment design, online courses, and workshops.
- Industry standards and safety guidelines.