A LABORATORY MANUAL FOR

Chemical Engineering Laboratory –II

(Cycle – I)

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Contents

Experiment No. 1
ISOTHERMAL BATCH REACTOR

OBJECTIVE
Study of a non-catalytic homogeneous reaction in a Batch reactor

AIM
To determine the reaction rate constant (k) for the given saponification reaction of ethyl acetate in aqueous sodium hydroxide solution.
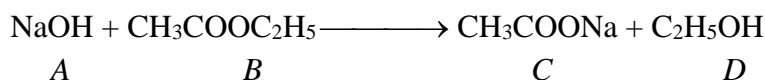
APPARATUS

1. Batch reactor set up	2. Conical flasks (250 ml) - 6 Nos.
3. Stop watch (supplied with set-up)	4. Burette (25 ml) - 1 No
5. Sampling pipette (10 ml) - 1 No.	6. Thermometer (0-110°C) - 1 No.

CHEMICALS REQUIRED

1.   N/10 Sodium hydroxide   2.   N/10 Ethyl acetate

3.   Phenolphthalein indicator and   4.   N/40 Hydrochloric acid

CHEMICAL REACTION

$$NaOH + CH_3COOC_2H_5 \longrightarrow CH_3COONa + C_2H_5OH$$
$$\quad A \qquad\qquad B \qquad\qquad\qquad C \qquad\qquad D$$

THEORY
A batch reactor is a closed system such that no stream enters or leaves the reactor. In homogeneous reactions, all reacting species remain in a single phase. The rate of reaction of any reaction component A is defined as

$$-r_A = \frac{1}{V}\left(\frac{dN_A}{dt}\right) = \frac{moles\ of\ A\ which\ disappear\ by\ reaction}{unit\ time * unit\ volume}$$

Rate of reaction is influenced by variables like temperature, pressure, and concentration. The rate of reaction is a function of concentration at constant temperature, i.e. $-r_A = kC_A$ where n is the order of reaction. The interpretation of the kinetic data is a trial and error procedure. A kinetic model is first selected with a corresponding rate equation, and the concentration-time relationship predicted by the model is matched with experimental data.

For a first order irreversible reaction

$$A \longrightarrow Product$$
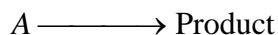
Rate of reaction can also be expressed as moles of A disappearing per unit volume per unit time.

$$-r_A = \frac{1}{V}\left(\frac{dN_A}{dt}\right) = \frac{dC_A}{dt} \tag{1}$$

Where, C_A = concentration of A

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$$-r_A = kC_A \tag{2}$$

Combining equations (1) and (2) integration yields

$$\ln C_A = \ln C_{A0} - kt$$

$$C_A = C_{A0} e^{-kt}$$

$$-\ln \frac{C_A}{C_{A0}} = kt$$

Where,

C_{AO} = Conc. of A at t = 0; $\qquad$ C_A = Conc. of A at t = t

If we plot $-\ln\left(\dfrac{C_A}{C_{A0}}\right)$ vs. t we must get straight line if the reaction is a first order reaction.

For second order irreversible reaction

$$2A \xrightarrow{\hspace{2cm}} \text{Product} \qquad\qquad -\frac{dC_A}{dt} = kC_A^2$$

Integration yields:

$$\frac{1}{C_A} = kt + \frac{1}{C_{A0}}$$

If we plot $\left(\dfrac{1}{C_A}\right)$ vs. t, we must get a straight line for a second order reaction with slope = k and

intercept on y-axis $= \left(\dfrac{1}{C_{A0}}\right)$.

The reaction rate constant is a strong function of reaction temperature. The temperature dependence of k is expressed in terms of Arrhenius equation:

$$k = A\exp\left(\frac{-E}{RT}\right)$$

Where,

A = Arrhenius constant; E = activation energy, J/g mole, cal /g mole
R = ideal gas law constant = 8.314 J/g mole K = 1.98 cal/g mole K

This equation can be written in the form:

$$\ln k = \frac{-E}{R}\left(\frac{1}{T}\right) + \ln k_0$$

Thus, $\left(\dfrac{E}{R}\right)$ may be evaluated from the slope of a plot of $\ln k$ vs. $\dfrac{1}{T}$.

k is known at four temps. : T_1, T_2, T_3, and T_4.

EXPERIMENTAL SET UP DATA

The experimental set up consists of a batch reactor, constant temperature water bath and stirrer etc.

Volume of reactor	-	1000 ml
Volume of water Bath	-	13.5 L (approx)
Reactor stirrer speed range	-	0 - 75 RPM
Water bath stirrer speed range	-	0 - 150 RPM
Temp. Controller range	-	Ambient to 200 ºC with 1 ºC resolution.
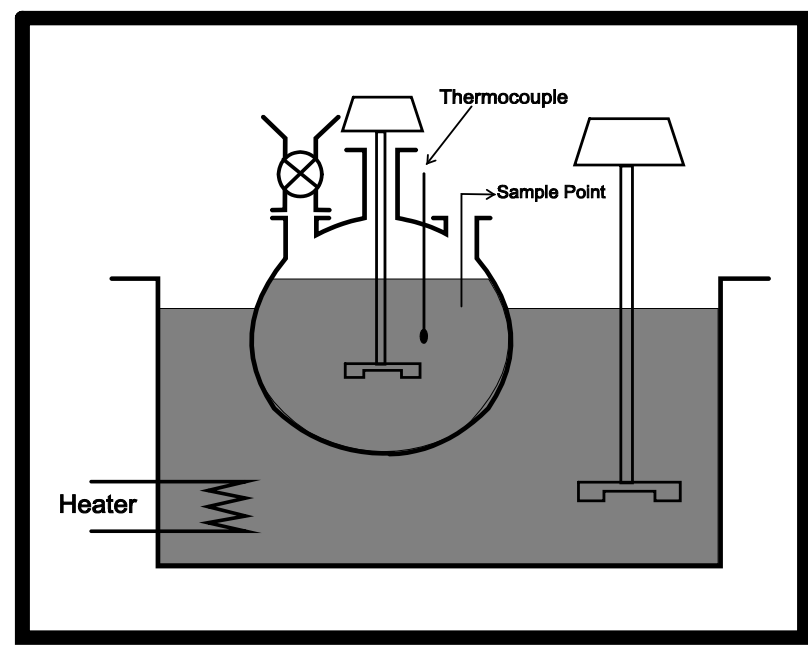
Figure: Batch reactor set up.

PROCEDURE

1. Draw a neat schematic diagram of the experimental set up

2. Prepare 5 L solution of N/100 ethyl acetate and 5 L solution of N/100 NaOH.

3. Prepare a solution of N/40 HCl and N/40 NaOH solutions for the titration.

4. Prepare oxalic acid solution by dissolving 3.15 gm of oxalic acid in 500 ml of water.

5. Standardize NaOH solution with 0.1N oxalic acid.

6. Use phenolphthalein as indicator.

7. Take 6 Nos. of 250 ml conical flasks and put 20 ml of N/40 HCl in each flask.

8. Take N/40 NaOH solution in the burette.

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9. Adjust the temperature of water bath at 35 ºC.

10. Take 400 ml each of N/100 $CH_3COOC_2H_5$ and N/100 NaOH in two separate flasks and keep these in the water bath for about 15 min to preheat the solutions.

11. Transfer these solutions quickly in the batch reactor. Start immediately the mixer and the stop watch.

12. At regular intervals of 3-5 min., withdraw 10 ml of reaction mixture and put it in the conical flask containing N/40 HCl. Take at least 6 samples at regular interval of time.

13. Titrate the excess N/40 HCl in each flask using N/40 NaOH from burette and phenolphthalein as indicator.

14. Record the reaction temperature.

15. In order to study the effect of temp, on k, the above steps may be repeated at temp. = 40 ºC and 45 ºC. All the reactants must be pre-heated in the water bath before mixing in the reactor.

OBSERVATIONS AND CALCULATIONS
REACTION:

$$A + B \longrightarrow C + D \qquad \left(C_{A0} = C_{B0}\right)$$

Reaction temp = ºC
Conc. of NaOH (A) = N/100
Conc. of Ethyl acetate (B) = N/100
Vol. of Ethyl acetate taken = 400 ml
Vol. of Sodium hydroxide = 400 ml
Initial Conc. of NaOH (A) in the mixture, C_{AO} = 0.005 M (g mole/L)
Initial Conc. of Ethyl acetate (B) in the mixture, C_{BO} = 0.005 M (g mole/L)

Table 1

S.No	Sampling time, min.	Vol. of sample taken, ml	Vol. of N/40 HCl added to sample, ml	sample taken for titration, ml	Vol. of N/40 NaOH consumed in titration, ml

Estimation of un-reached NaOH in the reaction mixture (C_A)

Volume of N/40 HCl taken in conical flask = 20 ml
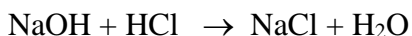Volume of reaction mixture sample added = 10 ml
Volume of N/40 NaOH used in titration for neutralization of excess N/40 HCl, V_{NaOH} =   ml
Hence,

No. of moles of N/40 NaOH used $\qquad$ = V_{NaOH} x (1/40) x 10^{-3}
$\qquad$ = 2.5 x 10^{-5} x V_{NaOH} g moles

No. of moles N/40 HCl present initially in conical flask   = 20 x 1/40 x 10^{-3} g moles
$\qquad$ = 5 x 10^{-4} g moles.

Reaction between NaOH and HCl is:
$\qquad$ NaOH + HCl $\rightarrow$ NaCl + H_2O
$\qquad$ i.e., 1 mole of NaOH reacts with 1 mole of HCl
$\qquad$ Hence, No. of moles of HCl reacted with excess moles of NaOH in the reaction mixture
$\qquad\qquad$ = (5 x 10^{-4} - 2.5 x 10^{-5} x V_{NaOH}) g moles
$\qquad$ Hence, No. of moles of NaOH unreacted in the reaction mixture
$\qquad\qquad$ = (5 x 10^{-4} − 2.5 x 10^{-5} x V_{NaOH}) g moles

Volume of sample collected = 10 ml
$\qquad$ ∴ Concentration of unreacted NaOH is

$$C_A = \frac{(5 \text{ x } 10^{-4} \text{ - } 2.5 \text{ x } 10^{-5} \text{ x } V_{NaOH})}{10 \text{ x } 10^{-3}} \quad \text{gmole / L}$$

Table 2

S. No.	Time, t, m	Conc. of unreacted NaOH in the reaction mixture, C_A (g mole/L)

Plot the curves for 1st order and 2nd order reaction and find out the order of reaction and rate constant. Tabulate *k* values at 3 or 4 different temperatures.

PRECAUTIONS
1.   All solutions should be prepared accurately and must be standardized.
2.   Titrations should be carried out precisely.
3.   The individual reactants must be first pre-heated to the reaction temperature.

4.   While taking sample, care should be taken that tip of the pipette does not touch the agitator blade.

5.   For setting of reaction temperature, initially the temperature should be set at around 6 $^{\circ}$C less than the desired temperature to avoid over shooting. Before starting the flow rate of ethyl acetate, temperature should be set at desired temperature.

6.   All the glass wares to be used should be properly rinsed prior to use.

CONCLUSIONS

Write down the points you have concluded from the experiment.

REFERENCES

Fogler H. Scott (2008). *Elements of Chemical Reaction Engineering*. 4th Ed. Pearson Education, New Delhi, India

Levenspiel,"Chemical Reaction Engineering," 2nd edition, John Wiley & Sons, Singapore, 1995.

Experiment No. 2
ISOTHERMAL SEMIBATCH REACTOR

OBJECTIVE

To study a second order saponification reaction between ethyl acetate and sodium hydroxide in a semi batch reactor under isothermal condition (i.e. at a fixed temperature).

AIM

To determine experimentally the reaction rate constant (k) and variation of concentration in sodium hydroxide with time in the reactor.

APPARATUS

| 1. Semi Batch reactor set up	2. Conical flasks (250 ml) - 6 Nos.
3. Stop watch (supplied with set-up)	4. Burette (25 ml) - 1 No
5. Sampling pipette (10 ml) - 1 No.	6. Thermometer (0-110°C) - 1 No.

INTRODUCTION

One of the best reason to use semi batch reactors is to enhance selectivity in liquid phase reactions. In semi batch reactor a reactant is added to another 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.

THEORY

In this type of reactor, reactant A, in solution of concentration C_{Ao} (mole/L)is charged to the reactor to a volume V_o (L), then the reactant B is pumped in at the rate of v_o (LPH) and the reaction is allowed to take place. There is no overflow and assuming no change in density.

Reaction:

$$NaOH + CH_3COOC_2H_5 \longrightarrow CH_3COONa + C_2H_5OH$$
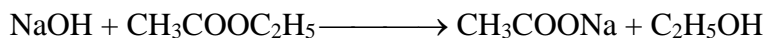
The above reaction is second order reaction (n = 2)

For second order reaction.

$$- r_A = kC_A C_B \qquad (1)$$

Where $- r_A$ is rate of disappearance of A, C_A, C_B is concentration of A and B at any time t, k is rate constant.

$$\frac{- dC_A}{dt} = -kC_A C_B - \frac{v_0}{V} C_A$$

$$V = V_0 + v_0 t$$

$$X_A = \frac{C_{A0}V_0 - C_A V}{C_{A0}V_0}$$

$$k = \frac{\left(\dfrac{v_0 C_A}{V} - \dfrac{dC_A}{dt} \right)}{C_A C_B}$$

Where, $\dfrac{-dC_A}{dt}$ derivative of concentration of A with respect to time t is, v_o is volumetric flow rate, V is volume of feed solution at any time t, V_o is initial volume of feed solution, C_{A0} is initial concentration of A, X_A is degree of conversion.

DESCRIPTION

The set up consists of a reactor fitted in a constant temperature water bath. Before starting the reaction the reactor is charged with measured quantity of one reactant. From a feed tank another reactant is fed to the reactor. Using a sampling pipette samples can be taken out for analysis after fixed interval. Rotameter is provided to measure the flow of chemicals. The compressed air is used for circulation of feed. One stirrer is fitted for mixing the reactants in reactor and other is fitted in water bath to keep the uniform temperature throughout in the bath. Temperature is controlled with the help of digital temperature indicator cum controller. Pressure regulator, pressure gauge and safety valve are fitted in the compressed air line.

UTILITIES REQUIRED

1. Electricity supply: Single phase, 220 V AC, 50 Hz, 5-15 Amp combined socket with earth connection.
2. Compressed air supply continuous @ 0.5 CFM at 1 Bar.
3. Water supply (Initial fill)
4. Floor drain required
5. Laboratory glass ware:-

Beaker (1000 ml)	01 No.
Conical flask (250 ml)	02 Nos.
Measuring cylinder (50 ml)	02 Nos.
Burette (50 ml)	01 Nos.

6. Chemicals:

Distilled water	25 Lit
NaOH Pellets	10 gm
N/10 NaOH	100 ml
N/10 HCl	100 ml
Ethyl acetate	15 ml
Indicator (phenolphthalein)	

EXPERIMENTAL PROCEDURE

1.   Prepare 5L of N/10 sodium hydroxide solution by dissolving 20 gm of sodium hydroxide in 5L of distilled water.

2. Prepare 20L of N/10 ethyl acetate solution by mixing 176 gm (or 196 ml) of ethyl acetate in 20L of distilled water.
3. Prepare oxalic acid solution 0.1 N by dissolving 3.15 gm of oxalic acid in 500 ml of water.
4. Standardize NaOH with oxalic acid 0.1 N.
5. Put 500 ml sodium hydroxide solution in the 1000 ml beaker.
6. Close all the valves V_1-V_7.
7. Open the valve V_3-V_4 fill 20L of the ethyl acetate solution in the feed tank.
8. Close the valve V_3-V_4.
9. Fill the reactor with 500 ml of N/10 sodium hydroxide feed solution.
10. Take 20 ml of N/10 hydrochloric acid in measuring cylinders.
11. Fill the burette with N/10 sodium hydroxide (Standard Solution).
12. Ensure that switches given on the panel are at OFF position.
13. Fill water in the water bath.
14. Set the reaction temperature (ambient to 70°C) by increment, decrement .
15. Connect electric supply to the set-up.
16. Start the heater and stirrer of the bath and wait till constant temperature is attained.
17. Start motor of the reactor.
18. Open valve V_1 and set air pressure 0.5 to 1 kg/cm^2 by pressure regulator and pressure gauge
19. Pass ethyl acetate into the reactor by allowing constant flow rate controlled by valve V_2.
20. At regular intervals of 3-5 min withdraw 10ml of reaction mixture from outlet, in measuring cylinder that already contain 20 ml N/10 hydrochloric acid.
21. Note down the reaction temperature.
22. Transfer the sample solution in conical flask.
23. Titrate the solution, using phenolphthalein as an indicator against N/10 sodium hydroxide (add NaOH from burette).
24. Repeat the experiment for different flow rate of ethyl acetate.

OBSERVATION & CALCULATION
STANDARD DATA

Real gas constant R	= 1.9872 cal/mole K
Initial volume of feed solution V_o	= 20 Lit
Volume of H_{Cl} V_{HCl}	= 20 ml
Volume of sample V_{SAMP}	= 10 ml
Normality of NaOH in feed solution N_{NaOH}	= 0.1 g eq/L
Normality of NaOH used for titration N_1	= 0.1 g eq/L
Normality of HCl N_{HCl}	= 0.1 g eq/L
Normality of ethyl acetate N_{EA}	= 0.1 g eq/L

OBSERVATION TABLE

S. No.	t(min)	V_1(ml)	V_B(LPH)

CALCULATIONS

$$C_{A0} = N_{NaOH}(mole/L)$$

$$HCLO = \frac{V_{HCl}N_{HCl}}{1000}(mole)$$

$$HCLR = HCLO - \frac{V_1 N_1}{1000}(mole)$$

$$C_A = \frac{HCLR}{V_{SAMP}} * 1000(mole)$$

$$V = V_0 + \frac{V_B t}{60}(L)$$

$$X_A = \frac{(C_{A0}V_0 - C_A V)}{C_{A0}V_0}$$

$$C_B = \frac{\frac{V_B t}{60} - N_{NaOH}VX_A}{V}$$

$$\frac{dC_A}{dt} = \underline{\hspace{2cm}}(mole/L\,\min)$$

$$k = \frac{\left(\frac{V_B C_A}{V} - \frac{dC_A}{dt}\right)}{C_A C_B}(L/mole\,\min)$$

Calculation Table

S. No.	t(min)	C_A(mole/L)	X_A	k(L/mole min)

Block Diagram

FRONT VIEW

VALVE	
1.	V1 - AIR SUPPLY VALVE FOR FEED TANK
2.	V2 - FLOW CONTROL VALVE FOR ROTAMETER
3.	V3 - FEED INLET VALVE FOR FEED TANK
4.	V4 - AIR VENT VALVE FOR FEED TANK
5.	V5 - DRAIN VALVE FOR FEED TANK
6.	V6 - DRAIN VALVE FOR REACTOR
7.	V7 - DRAIN VALVE FOR WATER BATH

BACK VIEW

BLOCK DIAGRAM

NAME - ISOTHERMAL SEMI BATCH REACTOR

CODE - CRE-10B
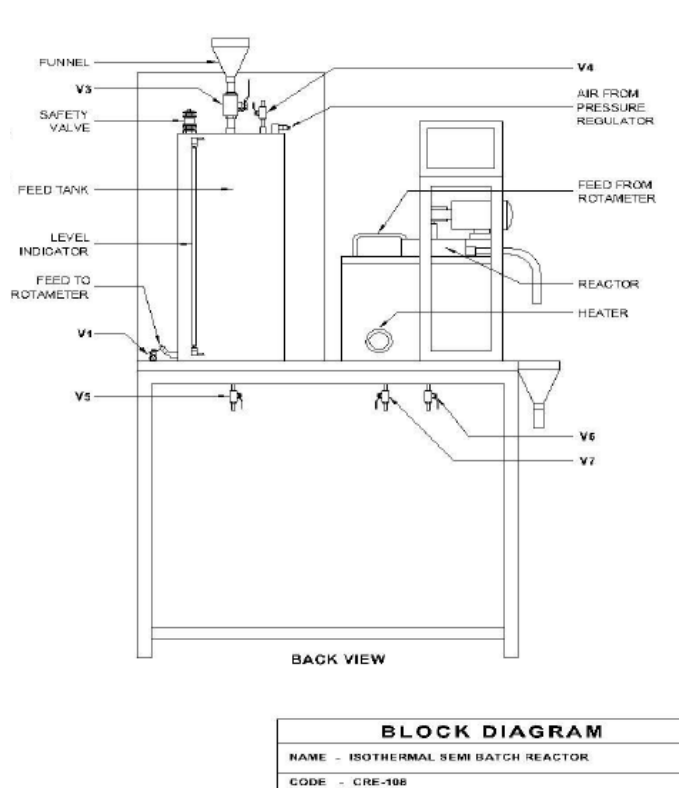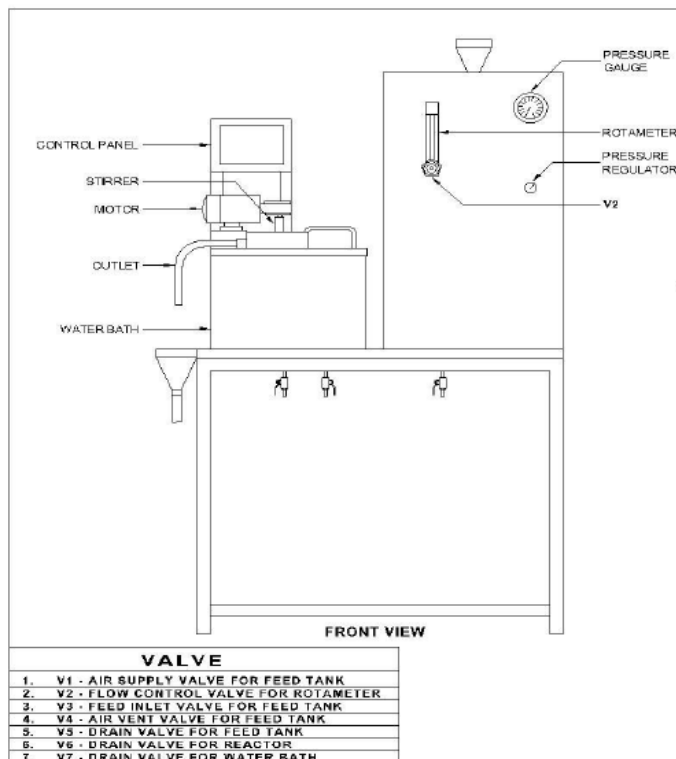
NOMENCLATURE

Nom	Column Heading	Units
C_A	Concentration of unreacted NaOH in the reactor	Mole/L
C_{A0}	Initial concentration of NaOH in the feed mixture	Mole/L
$\dfrac{dC_A}{dt}$	Derivatives of concentration of NaOH with respect to time	Mole/L min
HCLO	Amount of HCl taken for quench	Mole
HCLR	Amount of HCl reacted with NaOH	Mole
K	Rate constant	L/mole min
N_1	Normality of NaOH used for titration	g eq/L
T	Temperature	℃
N_{EA}	Normality of ethyl acetate	g eq/L
N_{HCl}	Normality of HCl	g eq/L
N_{NaOH}	Normality of NaOH in feed solution	g eq/L
R	Real gas constant	cal/mole K
T	Time	Min
V_{SAMP}	Volume of sample	Ml
V	Volume of reactor solution at any time t	Lit
V_1	Volume of NaOH used for titration	Ml
V_B	Volumetric flow rate of ethyl acetate	LPH
V_{HCl}	Volume of N/10 HCl taken for quench	Ml
V_0	Initial volume of feed solution	Lit
X_A	Degree of conversion	

PRECAUTIONS
1. Measure the exact volume of water and weight of chemicals.
2. Always use distilled water, good quality chemicals and standard solution for titration.
3. Keep close all the drain valves V_5-V_7, and vent valve V_4 should be open while filling the reactants in feed tank and reactor.
4. Handle the chemicals carefully.
5. Don't ON the switch of heater before filling water in the water bath

CONCLUSIONS
Write down the points you have concluded from the experiment.

REFERENCES

Fogler H. Scott (2008). *Elements of Chemical Reaction Engineering*. 4th Ed. Pearson Education, New Delhi, India

Levenspiel,"Chemical Reaction Engineering," 2nd edition, John Wiley & Sons, Singapore, 1995.

Experiment No. 3
## ISOTHERMAL CSTR (JACKET HEATING AND COIL HEATING)

OBJECTIVES
- To study a non-catalytic homogeneous second order liquid phase reaction in a CSTR under atmospheric conditions.
- To determine the reaction rate constant for saponification of ethyl-acetate with NaOH at atmospheric conditions.

Reaction: $NaOH + CH_3COOC_2H_5 \rightarrow CH_3COONa + C_2H_5OH$

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.
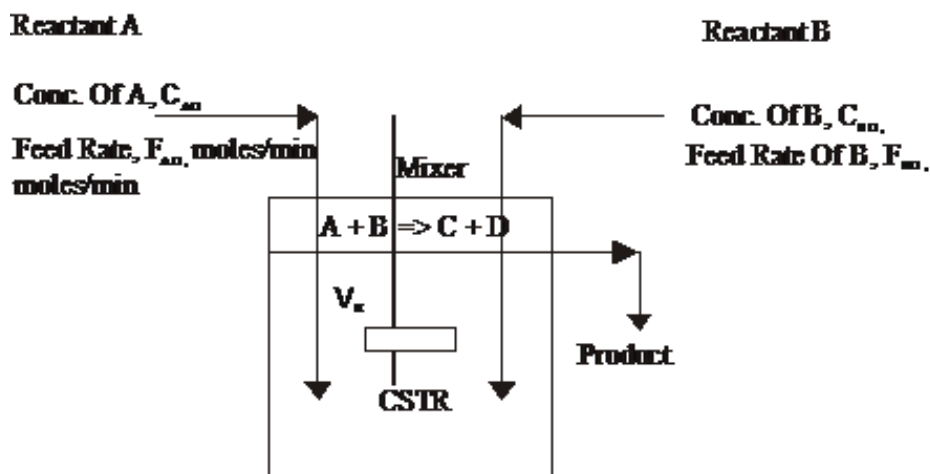
Reactor volume (volume of fluid in the reactor) = V_R, Liters,
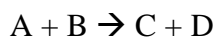Volumetric feed rate (volumetric feed rate of A + volumetric feed rate of B) = V_0 , LPM
Space time = $\tau = V_R / V_o$, min

The performance equation for the CSTR at steady state is:

$$\tau = \frac{V_R}{Vo} = \frac{C_{A0}X_A}{-r_A} = \frac{(C_{A0} - C_A)}{-r_A}$$

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:
$$A + B \rightarrow C + D$$
With $C_{AO} = C_{Bo}$ and negligible change in density of reaction mixture

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The reaction rate (rate of disappearance of A),
$- r_A = k\, C_A^2$
Hence,

$$\tau = \frac{V_R}{Vo} = \frac{C_{A0} X_A}{-r_A} = \frac{(C_{A0} - C_A)}{k C_A^2}$$

Degree of conversion, $\quad X_A = (C_{Ao} - C_A)\,/\,C_{Ao}$

and rate of reaction, $\quad -r_A = C_{Ao}\, X_A\,/\,\tau \qquad\qquad$, gmol/l-min

the rate constant, $\quad k = (C_{AO} - C_A)\,/\,(\tau\, C_A^2)$, $\quad$ l/gmol-min

DESCRIPTION

The reactor is made up of stainless steel. 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, 100 cc titration flasks, 50 cc burette and one stopwatch (supplied with equipment), conductivity cell (optional).

CHEMICALS

1. N/10 NaOH,
2. N/10 HCI,
3. N/10 Ethylacetate (8.8 gms of ethylacetate in 1 L of water),
4. Indicator (Phenolphthalein)
5. N/40 HCl

UTILITIES REQUIRED

1. Compressed Air Supply to be maintained @ 1-2 kg/cm²
2. Water Supply and Drain.
3. Electricity Supply: 1 Phase. 220 V AC. 0.5 kW.
4. Instruments. Laboratory Glassware and Chemicals required for analysis as per the system adopted (to be arranged by institute).

SPECIFICATIONS

Reactor	:	Material Stainless Steel 304 Grade, Capacity 2 Ltrs (Approx).
Stirrer	:	Stainless Steel Impeller and shaft coupled with **FHP Motor**
Feed Tank (2Nos.)	:	Material Stainless Steel 304 Grade, Capacity - 20 Ltrs.
Feed Circulation	:	By compressed air
Flow Measurement	:	Rotameter 2Nos. (one each for Reactants)
Piping	:	Stainless Steel/PVC/GI
Pressure Regulator	:	0-2 Kg/cm²
Pressure Gauge	:	Bourdon type 0-2 Kg/cm²
Stop Watch	:	Electronic
Control Panel	:	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 is painted with auto paints.

PROCEDURE

1. Prepare 10L of N/10 NaOH solution by dissolving 40 gm of NaOH in 20L of water.
2. Prepare 10L of N/10 ethyl acetate solution by mixing 88 gms of ethyl acetate in 10L of water.
3. Prepare 0.5 liter of 0.1 N standard oxalic acid solution by dissolving 3.15 gm of oxalic acid crystals in 500ml of water.
4. Standardize NaOH solution by titrating against 0.1 N oxalic acid and phenolphthalein as indicator.
5. Fill the respective tanks with these solutions.
6. Adjust the flow rate of the two streams so that in the feed mixture $C_{Ao} = C_{Ho}$ i.e have equal flow rates (e.g. 2 LPH each you may use the flow range 2 LPH to 20 LPH for each stream) using calibrated rotameter.
7. Pass equi-molar 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.
8. After about 1.5 times the residence time of the reactor, collect 10 ml sample of the liquid at the out let.
9. Titrate this solution against N/40 HCl (add HCl from burette).
10. Using phenolphthalein as indicator, note the volume of N/40 HCl used (V_{HCl})
11. Note the volumetric flow rate of liquid at the end of the reactor.
12. Note the reaction temperature.
13. 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 HCI using phenolphthalein as indicator. Note the volume of N/10 HCI used.
14. 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 temperature	T	=	^{O}C
Reactor diameter =	id	=	123 mm
Effective height of liquid in the reactor,	L	=	mm
Vol. of the reactor =	V_R	=	2 liters
Volumetric feed rate of feed,	Vo	=	L/min
Initial Conc. of NaOH in the feed mixture = C_{AO}		=	gmol/L
Initial Conc. of Ethyl acetate in the feed mixture C_{BO}		=	gmol/L

Residence Time =

$$\tau = \frac{V_R}{Vo}$$

Conversion

$$X_A = \frac{(C_{A0} - C_A)}{C_{A0}}$$

Concentration of un-reacted NaOH in the reaction mixture, C_A

Estimation of uncreated NaOH in the reaction mixture (C_A):

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 neutralize sample = V_{HCl}
      N/40 x V_{HCl} = V_1 x Normality of reaction mixture
      Normality of reaction mixture=
      C_A= …..gmole/L

Run. No	τ, min residence time	C_A, gmol/L (conc. NaOH)	X_A, degree of conversion	$X_A / (C_{AO} [1-X_A]^2)$

Plot τ vs. $X_A / (C_{AO} [1-X_A]^2)$, the slope is = 1/(k): 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 tilting 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 re-fix that again after wrapping Teflon tape.
3. If Rotameter fluctuating more than average tight control knob of that.
   (Procedure: There are two nuts, lose first nut and tight the second slightly, and then first also. Both nuts are on Rotameter)

REFERENCES

Fogler H. Scott (2008). *Elements of Chemical Reaction Engineering*. 4th Ed. Pearson Education, New Delhi, India

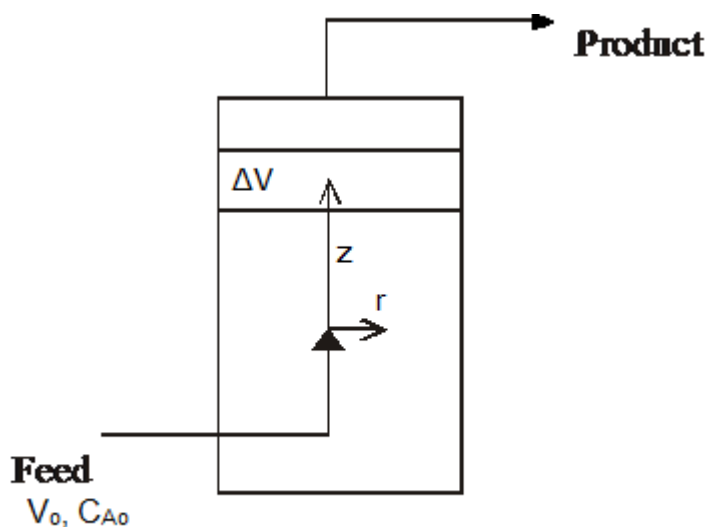Levenspiel,"Chemical Reaction Engineering," 2nd edition, John Wiley & Sons, Singapore, 1995.

Experiment No. 4a
ISOTHERMAL PFR (STRAIGHT TUBE TYPE)

OBJECTIVE
- To study a non-catalytic homogeneous reaction in a plug flow reactor
- To determine the reaction rate constant for saponification of ethyl acetate with NaOH at atmospheric conditions.
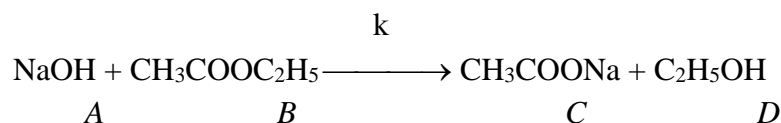
THEORY

In an ideal plug flow reactor (PFR) there is no mixing in the direction of flow and complete mixing perpendicular to the direction of flow. Concentration of the reactant varies along the length of the reactor in the axial direction but not in the radial direction.

For steady state rate operation:

$$\frac{V}{F_{A0}} = \int_0^X \frac{dX}{-r_A}$$

For the reaction

$$NaOH + CH_3COOC_2H_5 \xrightarrow{\quad k \quad} CH_3COONa + C_2H_5OH$$
$$\quad A \qquad\qquad B \qquad\qquad\qquad C \qquad\qquad D$$

Rate of reaction is given by

$$-r_A = kC_A C_B$$

For the Condition:

$C_{A0} = C_{B0}$ and neglecting change in density

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$$-r_A \quad = kC_A{}^2$$
$$C_A \quad = C_{A0}(1-X_A)$$

Therefore

$$\frac{V}{F_{A0}} = -\frac{1}{kC_{A0}{}^2}\int_1^X \frac{dX}{(1-X_A)^2}$$

Where,

F_{AO} = molar flow rate of NaOH

F_{AO} = $V_0 C_{A0}$

V_o = Volumetric flow rate of feed, LPM

V_R = Volume of the PFTR, L

Time constant, $\tau = V_R / V_0$ and thus substituting in above equation,

$$\tau = -\frac{1}{kC_{A0}}\int_0^X \frac{dX}{(1-X_A)^2}$$

when $C_{AO} \neq C_{BO}$, then,

$$\tau = \frac{1}{kC_{A0}[\frac{C_{B0}}{C_{A0}}-1]}\, ln\, \frac{\frac{C_{B0}}{C_{A0}}-X_A}{\frac{C_{B0}}{C_{A0}}(1-X_A)}$$

C_A= Concentration of un-reacted NaOH at steady state.

k = Reaction rate constant

DESCRIPTION
The reactor is a glass tube. The air compressed feed reactants are flowing through Rotameters. The concentration of feed and the product coming out from the reactor is analyzed by titration to check the effect and the performance of the reactor.

GLASSWARE REQUIREMENTS
50 cc graduated cylinders, 2 No. 100 cc titration flasks, 50 cc burette and one stopwatch

CHEMICALS
1. N/10 NaOH,
2. N/10 HCI,
3. N/10 Ethyl acetate (8.8 grams of ethyl acetate 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	:	Material Borosilicate Glass
		Dia 1", length 4 feet, 2" inclined upward at outlet
Feed Tank (2Nos.)	:	Material Stainless Steel 304 Grade. Capacity - 20Ltrs.
Feed Circulation	:	By compressed air
Flow Measurement	:	Rotameter 2Nos. (one each for Reactants)
Piping	:	SS 304 and PVC
Pressure Regulator	:	0-2 Kg/cm^2
Pressure Gauge	:	Bourdon type 0-2 Kg/cm^2
Stop Watch	:	Electronic
Control Panel	:	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 rests are painted with auto paints.

PROCEDURE

1. Prepare 10L of N/10 NaOH solution by dissolving 40 gm of NaOH in 10L of water.
2. Prepare 10L of N/10 ethylacetate solution by mixing 88 gm of ethylacetate in 10L of water.
3. Fill the respective tanks with these solutions.
4. Standardize NaOH solution by titrating against 0.1 N oxalic acid and phenolphthalein as indicator.
5. Adjust the flow rate of the two streams so that in the feed mixture $C_{Ao} = C_{BO}$ i.e have same volumetric flow rates (e.g. 2 LPH each you may use the flow range: 3 LPH to 15 LPH for each stream) using calibrated rotameters.
6. Pass equimolar feed rates of ethyl acetate and NaOH into the PFR by allowing equal volumetric feed rates of reactants in to the reactor
7. After 1.5 time the residence time of the reactor, collect the sample of the liquid at the outlet
8. Titrate this solution against N/40 HCl (add HCl from burette).
9. Using phenolphthalein as indicator, note the volume of N/40 HCl used.
10. Note the volumetric flow rate of liquid at the end of the reactor.
11. Note the reaction temperature.

12. 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/40 HCI using phenolphthalein as indicator. Note the volume of N/40 HCI used.
13. Repeat all the steps for four to six different flow rates of feed.

Note: Record k at the reaction temperature

OBSERVATION & CALCULATIONS
Run No.

Reaction temp. = $\qquad$ T = --------^{O}C

Reactor diameter $\qquad$ d = 28 mm

Reactor Length $\qquad$ L = 1300 mm

Vol. of the reactor = V_R = $(\pi /4) * d^2 * L$, $\qquad$ V_R = ---------L

Volumetric feed rate of feed, L/min $\qquad$ V_O = ----------

Initial Conc. Of NaOH in the feed mixture, gmol/L $\qquad$ C_{AO} = ----------

Initial Conc. of Ethyl acetate in the feed mixture, gmol/L $\qquad$ C_{BO} = ----------

Residence Time= $\tau = V_R/V_O$, min

$X_A = [C_{AO} - C_A] / C_{AO}$

Volume of N/40 HCl used = V_1 ml

Volume of sample taken for titration purpose = V_2 ml

N_1V_1 (HCL SIDE) = N_2V_2 (REACTANT MIXTURE/SAMPLE SIDE)

Implies that N2 i.e strength of NaOH in reaction mixture is = $(N_1 . V_1) / V_2$ gm mole per liter

Concentration of NaOH at outlet:

Concentration of un-reacted NaOH in the reaction mixture, C_A

$C_A = (N_1. V_1) / V_2$

Run. No	τ, min	C_A, gmol/L (conc.NaOH)	X_A	$\dfrac{X_A}{(1 - X_A)C_{A0}}$

Plot of τ vs. $X_A/((1-X_A)C_{A0})$ is a straight line with slope 1/k

PRECAUTION
1. Measure the exact volume of water and weigh the chemicals exactly.

2. Always use distilled water and good quality chemicals and standard solution for titration.
3. Use precision electronic balance for weighing of chemicals. Don't mix the droppers of different chemicals.
4. Keep all the drain valves closed and vent valve should be open while filling the reactant in feed tanks.
5. Flow should not be disturbed during the experiments.
6. Handle the chemicals carefully. Refer Material Safety Data Sheets (MSDS) of chemicals being used.
7. Use primary standards for standardizing/determining chemical strengths.
8. Do not open the vent of pressurized reactant tanks suddenly. Reduce the pressure in feed tanks by adjusting pressure setting knob and open vent valves only after pressure gets released slowly.

TROUBLE SHOOTING

1. If any type of suspended particles are come in the Rotameter. Remove the Rotameter clean the tube and fit that at its place.
2. If there is any leakage; tight that part or remove that and fix that again after wrapping Teflon tape.
3. If Rotameter is fluctuating either feed tank may have got emptied or tank pressure is getting reduced. Determine appropriate cause and rectify that.
   (Procedure: two nuts are there lose first nut and tight the second slightly, and then first also. Both nuts are on Rotameter)

REFERENCES

Fogler H. Scott (2008). *Elements of Chemical Reaction Engineering*. 4th Ed. Pearson Education, New Delhi, India

Levenspiel,"Chemical Reaction Engineering," 2nd edition, John Wiley & Sons, Singapore, 1995.

Experiment No. 4b
ISOTHERMAL PFR (COILED TUBE TYPE)

OBJECTIVE
- To study a non-catalytic homogeneous reaction in PFR (Coil Type)
- To determine the reaction rate constant for saponification of ethyl acetate with NaOH at atmospheric conditions.

Reaction: $NaOH + CH_3COOC_2H_5 \rightarrow CH_3COONa + C_2H_5OH$

APPARATUS REQUIRED
Plug flow reactor set up, conical flask, pipette, burette, etc.

THEORY
Plug flow reactor is characterized by the fact that the flow of the liquid through reactor is orderly with no element of fluid overtaking or mixing with any other element ahead or behind. Actually there may be lateral mixing of fluid in a plug flow reactor; however there must be no mixing or diffusion along the flow path. The necessary and sufficient condition for plug flow is the residence time in the reactor to be the same for all elements of fluid.

The steady state flow reactor is ideal for industrial purpose when large quantity of material is to be processed and when the reaction is fairly high to extremely high. Support equipment needs are great. However extremely good product quality control can be obtained.

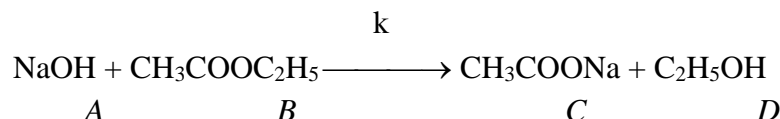By comparing the performance equation for batch and plug flow reactor we find.

   1.For system of constant density (constant volume batch and constant density plug flow) the performance equations are identical. For plug flow τ is equivalent to 't' for the batch reactor and the equation can be used interchangeably.

   2.For systems of changing density. There is no direct correspondence between the batch and the plug flow equation and the correct equation must be used for each particular situation. In this case performance equation cannot be used interchangeably.

For steady state rate operation:

$$\frac{V}{F_{A0}} = \int_0^x \frac{dx}{-r_A}$$

For the reaction

$$NaOH + CH_3COOC_2H_5 \xrightarrow{\quad k \quad} CH_3COONa + C_2H_5OH$$
$$\quad A \qquad\qquad B \qquad\qquad\qquad C \qquad\qquad D$$

The above reaction is second order reaction (n = 2)
For second order reaction:

   $-r_A = kC_A C_B$

For the Condition:

C_{AO} = C_{BO} and neglecting change in density

$-r_A$ = kC_A^2

C_A = $C_{AO}(1-X_A)$

Therefore,

$$\frac{V}{F_{A0}} = -\frac{1}{KC_{A0}^2} \int_0^x \frac{dX_A}{(1-X_A)^2}$$

F_{AO} = molar flow rate of NaOH

F_{AO} = V_oC_{AO}

V_o = Volumetric flow rate of feed, LPM

V_R = Volume of the PFTR, L

Or

$$\frac{V}{Vo} = -\frac{1}{K\,C_{A0}} \int_0^x \frac{dX_A}{(1-X_A)^2}$$

Or

$$\tau = \frac{1}{K\,C_{A0}} \frac{X_A}{(1-X_A)}$$

When $C_{AO} \neq C_{BO}$

$$\tau = \frac{1}{K\,C_{A0}[\left(\frac{C_{B0}}{C_{A0}}\right) - 1]} \, ln\left[\frac{\left(\frac{C_{B0}}{C_{A0}}\right) - X_A}{\left(\frac{C_{B0}}{C_{A0}}\right)(1-X_A)}\right]$$

$$X_A = \frac{C_{A0} - C_A}{C_{A0}}$$

C_A = Concentration of un-reacted NaOH at steady state.

k = Reaction rate constant

PROCEDURE

1. Prepare 0.5 liter of 0.1 N standard oxalic acid solution by dissolving 3.15 gm of oxalic acid crystals in 500ml of water.
2. Prepare 1 liter of 0.1 N HCl by dissolving 9 ml of HCl in 1 liter of water.
3. Prepare 5 liters of 0.1 N NaOH solution by dissolving 20 gm of NaOH flakes in 5 liters of water.
4. Prepare 5 liter ethyl acetate solution by adding 10 ml per liter of water.
5. Standardize NaOH solution by titrating against 0.1 N oxalic acid and phenolphthalein as indicator.
6. Transfer NaOH and ethyl acetate solution to two overhead tanks provided in experimental set up.

7. Adjust the flow rates after steady state has been attained.
8. Perform the experiment with condition $C_{AO} \neq C_{BO}$.
9. Note down the flow rates after steady state has been attained which is 1.5 times of space time.
10. Collect 10 ml of mixtures from the outlet and titrate against N/40 HCl solution.
11. Repeat the experiment for different flow rates.

OBSERVATIONS

Internal Diameter of the Tubular reactor (D) 13mm = 0.013 m
Length of the reactor (L)   20 foot = 6.096 m
(1 foot = 0.3048 meters)

Standardization of NaOH:

Volume of oxalic acid taken in conical flask, V_1 =
Normality of oxalic acid, N_1 =
Volume of NaOH run down from the burette (x), V_2 =
Normality of NaOH ,N_2 = $(N_1 V_1)/ V_2$

Standardization of HCl:

Volume of HCI taken in conical flask = V_3
Volume of NaOH run down from the burette = V_4
Normality of HCI = $(N_2 V_4)/ V_3$

Volume of N/40 HCl used = V_1 ml
Volume of sample taken for titration purpose = V_2 ml

$N_1 V_1$ (HCL SIDE) = $N_2 V_2$ (REACTANT MIXTURE/SAMPLE SIDE)

Implies that N2 i.e strength of NaOH in reaction mixture is = $(N_1. V_1) / V_2$ gm mole per liter
Concentration of NaOH at outlet:
Concentration of un-reacted NaOH in the reaction mixture, C_A
$C_A = (N_1. V_1) / V_2$

OBSERVATIONS

S.No	Flow rate of ethyl acetate cc/sec.	Flow rate of NaOH cc/sec	C_A gmole/ltr

SPECIMEN CALCULATION

Vol flow rate of ethyl acetate $= V_A$

Vol flow rate of NaOH $= V_B$

$$C_{A0} = \frac{C_{AS} V_A}{V_A + V_B} \qquad \& \qquad C_{B0} = \frac{C_{BS} V_B}{V_A + V_B}$$

$$M = \frac{C_{B0}}{C_{A0}}$$

$$X_A = \frac{(C_{A0} - C_A)}{C_{A0}}$$

$$C_B = C_{B0} - C_{A0} X_A$$

$$V = V_A + V_B$$

$V_R = \frac{\pi D^2}{4} * L,$ Volume of Reactor

$\tau = \frac{V_R}{V},$ Space time

$$\tau k = \varphi = \frac{1}{C_{A0}[\left(\frac{C_{B0}}{C_{A0}}\right) - 1]} \, ln\left[\frac{\left(\frac{C_{B0}}{C_{A0}}\right) - X_A}{\left(\frac{C_{B0}}{C_{A0}}\right)(1 - X_A)}\right]$$

$$\tau = \varphi/k$$

Sl.no	τ, min	ϕ

Plot ϕ versus τ and the slope of the line is the rate constant for the reaction, k.

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 tilting the reactant in feed tanks.
5. Flow should not be disturbed during the experiments.
6. Handle the chemicals carefully.

REFERENCES

Fogler H. Scott (2008). *Elements of Chemical Reaction Engineering*. 4th Ed. Pearson Education, New Delhi, India

Levenspiel,"Chemical Reaction Engineering," 2nd edition, John Wiley & Sons, Singapore, 1995.

Experiment No. 5a, 5b & 5c
JAW CRUSHER, ROLL CRUSHER AND BALL MILL

AIM

To study the operation of crushing and grinding equipment, namely jaw crusher (Blake crusher), smooth roll crusher and ball mill.

OBJECTIVE

- Calculate the specific surface (total surface per unit mass of particles), volume-surface mean diameter, mass mean diameter, volume mean diameter, and number of particles.
- Calculate K_k and K_r for only jaw crusher.

THEORY

Size reduction or comminution is usually carried out to make smaller particles from bigger ones, and to produce a product of desired size range or of specific surface are. Different ways are employed for size reduction in different size-reduction machines like compression, impact, attrition, shear and cutting. Different machines are used for different size ranges. A number of size reduction units are used when the particle size is to be reduced considerably because it is not generally economical to effect a large reduction ratio (ratio of feed to product particle size) in a single machine.

Jaw crusher is used for coarse crushing (feed size: 100-40 mm; product size: 50-5 mm). It has two jaws, one fixed vertically (called fixed or anvil jaw), and another swinging jaw, which reciprocates in a horizontal plane. These jaws form a V-open at the top. The feed is introduced between the jaws and is broken down due to compression.

Smooth roll crusher is intermediate crusher (feed size: 50-5 mm; product size: 5-0.1 mm). It has two heavy smooth-faced rolls rotating in opposite directions on parallel horizontal axes. The clearance between the rolls can be adjusted according to the size of the feed and the required size of the product. The feed particles are nipped and pulled downward through the rolls by friction. The size reduction is small (4:1).

Ball mill is fine crusher (feed size: 5-2 mm; product size: $<= 0.1$ mm) and is used for grinding. It has a rotating hollow cylinder, partially filled with iron or steel balls. The axis of rotation is either horizontal or at a small angle to the horizontal. The feed is admitted through a port (for both feeding and discharging) on the surface of the cylinder. The size reduction is accomplished by the impact of the balls as they fall back after being lifted by the rotating chamber.

The cost of power is a major expense in size reduction. It is impossible to estimate accurately the amount of energy required to effect a size reduction of a given material, but a number of empirical laws have been proposed . The two earliest are by Kick and von Rittinger given as

Kick's law : $$\frac{P}{m} = K_k \ln \frac{\overline{D}_{sa}}{\overline{D}_{sb}} \qquad (1)$$

Rittinger's law:
$$\frac{P}{m} = K_r\left(\frac{1}{\overline{D}_{sb}} - \frac{1}{\overline{D}_{sa}}\right) \tag{2}$$

where

P = power consumed for comminution ; M = feed rate to crusher

$\overline{D}_{sa}$ = mean volume-surface diameter of feed particles = $1\left/\sum_{i=1}^{n}\left(x_i\left/\overline{D}_{pia}\right.\right)\right.$

$\overline{D}_{sb}$ = mean volume-surface diameter of product particles = $1\left/\sum_{i=1}^{n}\left(x_i\left/\overline{D}_{pib}\right.\right)\right.$

$\overline{D}_{pi}$ = Average particle diameter, taken as the arithmetic mean of smallest and largest particles diameters in increment of the successive screens.

x_i = mass fraction in a given increment (of screen size)

K_k = Kick's constant

K_r = Rittinger's constant

However, both these equations have been found to be of limited utility. Bond suggested a law intermediate between Kick's and Rittinger's laws, and is given as

$$\frac{P}{m} = \frac{K_b}{\sqrt{D_p}} \tag{3}$$

where D_p is the particle size, and K_b is a constant that depends on the type of the machine and on the material being crushed. A work index W_i is defined as the gross energy requirement in kilowatt-hours per ton of feed needed to reduce a very large feed to such a size that 80% of the product passes through a 100-µm screen. This definition leads to a relation between K_b and W_i. If D_p is in millimeters, P in kilowatts, and m in tons per hour, then

$$K_b = 0.3162W_i \tag{4}$$

$$\frac{P}{m} = 0.3162W_i\left(\frac{1}{\sqrt{D_{pb}}} - \frac{1}{\sqrt{D_{pa}}}\right) \tag{5}$$

Here subscripts a and b signify the feed and the product respectively.

The quality of the comminuted particles is assessed based on the average particle size, surface area or particle population of a mixture. There are different types of averages, some of which are given below.

Volume-surface mean diameter:
$$\overline{D}_s = \frac{1}{\sum_{i=1}^{n}\left(x_i\left/\overline{D}_{pi}\right.\right)} \tag{6}$$

Mass mean diameter:
$$\overline{D}_w = \sum_{i=1}^{n} x_i\overline{D}_{pi} \tag{7}$$

Volume mean diameter:
$$\overline{D}_V = \left[\frac{1}{\sum_{i=1}^{n}\left(x_i \Big/ \overline{D}_{pi}^3 \right)} \right]^{1/3} \qquad (8)$$

The number of particles in a mixture (N_w) is given as

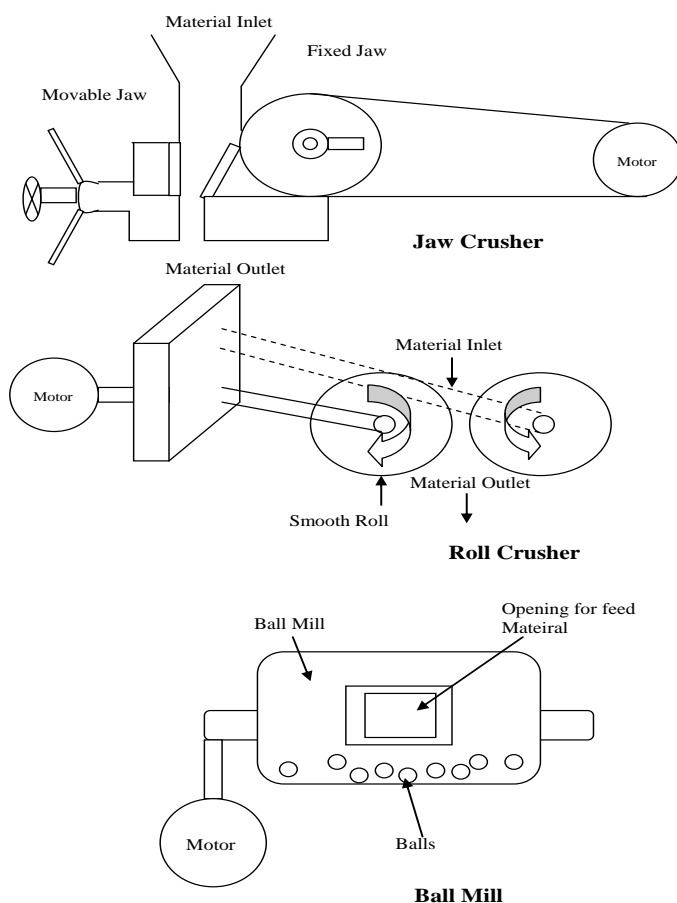$$N_w = \frac{1}{a\rho_p \overline{D}_V^3} \qquad (9)$$

where ρ_p is the density of the particles, and a is the volume shape factor.

EXPERIMENTAL SETUP

REQUIREMENTS
Lime, jaw crusher, roll crusher, ball mill, screens, sieve shaker, weighing machine, physical balance, trays, stop watch.

SCHEMATIC DIAGRAM OF EXPERIMENTAL SETUP

Jaw Crusher

Roll Crusher

Ball Mill

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EXPERIMENTAL PROCEDURE

1. Feed 10 kg of about 6 cm stones continuously into the jaw crusher, and note the reading on the energy meter before and after crushing, and the time of feeding and crushing.
2. Sieve the product using a set of 20mm, 12.5 mm and 6.3 mm screens and a pan. Record the weights of the particles retained on each screen.
3. Take the material retained on 6.3 mm screen and feed it to the roll crusher slowly and steadily. (The reading on the energy meter need not be noted as the reading before and after the crushing does not change significantly.)
4. Sieve (for about 20 minutes) the product using screen of size BSS 6, 8, 10, 12, 14, 16 and pan in the sieve shaker. Weigh the fraction of sample retained on each screen.
5. Take the material retained on BSS 12 screen (pore size 1.4 mm) and feed to the ball mill.
6. Run the ball mill for 15 minutes using the specified number of balls (15-25 balls).
7. Sieve (for about 20 minutes) the product using the screens BSS 100, 150, 200 and pan in the sieve shaker. Weigh the fractions retained on each screen.

OBSERVATIONS

1. Take spherecity of particles $= 0.7$
2. Take specific gravity of material $= 2.66$
3. Take volume shape factor $= 2$
4. Idle power for jaw crusher[$] $= 0.60$ kW
5. For each of the crushers/grinder, note the following

S No	Mesh Size	Screen Opening	Mass Retained

[$] Idle power of a crusher is found by running it without any material for a specified duration of time and recording the energy consumption during this period.

MODEL CALCULATIONS

1. For the three machines, calculate the mass fraction (x_i) and the average particle diameter (D_{pi}) of each fraction as
2. x_i = (mass retained on a screen)/(total mass retained)
3. D_{pi} = (pore size of screen on which mass is retained + pore size of the screen through which the mass passed through)/2
4. Draw plots for differential and cumulative screen analyses in all the cases.

5. Find the specific surface (total surface per unit mass of particles), volume-surface mean diameter, mass mean diameter, volume mean diameter, and number of particles in all the cases.
6. Calculate K_k and K_r for only jaw crusher. To do so, (i) estimate the rate of feed supply to the crusher (m) by dividing the weight of the solid fed by the time taken to feed; and (ii) take $\overline{D}_{sa}$.

RESULTS AND DISCUSSION
(Tables, Graphs, Comparison with those reported in literature)

CONCLUSIONS

PRECAUTIONS

REFERENCES
1. Warren, L McCabe, Smith, J C , and Harriott, P, Unit Operations of Chemical Engineering, 6th edition, McGraw Hill, New Delhi, India, 2000.
2. Richardson, J F, Harker, J H and Backhurst, J R, Coulson and Richardson's Chemical Engineering, 5th edition, Vol-2, Asian Books Private Limited, New Delhi, India, 2002.

NOTE: Pore sizes of the different meshes/sieves/screens are listed below.

SIEVE SIZE

B.S.S.*	A.S.T.M**	Nominal aperture
--	5	4.00 mm
5	6	3.35 mm
6	7	2.80 mm
7	--	2.40 mm
--	8	2.36 mm
8	10	2.00 mm
--	12	1.70 mm
10	--	1.68 mm
12	14	1.40 mm
14	--	1.20 mm
--	16	1.18 mm
16	18	1.00 mm
18	20	850 micron
22	25	710 micron
25	30	600 micron
30	35	500 micron
--	40	425 micron

36	--	420 micron
44	45	355 micron
52	50	300 micron
60	60	250 micron
--	70	212 micron
72	--	210 micron
85	80	180 micron
100	100	150 micron
120	120	125 micron
--	140	106 micron
150	--	105 micron
170	170	90 micron
200	200	75 micron
230	230	63 micron
300	270	53 micron
350	325	45 micron
--	400	38 micron

[*] British standard   ; [**]American standard

Experiment No. 6
PLATE AND FRAME FILTER PRESS

AIM

Study of cake filtration using Discontinuous pressure filter like Plate and frame filter press.

OBJECTIVES

- To study the operation of a plate-and-frame filter press.
- To determine the specific cake resistance (α) and the filter-medium resistance (R_m) for filtering a CaCO$_3$ –slurry for 3 different slurry concentrations.

THEORY

Plate-and-frame filter press consists of alternately placed plates and frames supported on a pair of rails. The plates have ribbed surfaces, and the hollow frames are separated from the plates by filter cloth. The assembly is tightly pressed as a single unit by a hand screw. The feed slurry is admitted from a pump and enters at one end of the filter press. It passes through the conduit formed by the holes in the upper right corner of both the plates and the frames, corresponding holes are also cut in the filter cloths, which themselves act as gaskets. The filtrate runs down the ribbed surface of the plates and is then discharged through a cock into an open launder. The solids accumulate on the cloths on opposite sides of the plates. Filtration is continued until the discharge of liquor becomes zero or very small, or the filtration pressure rises suddenly. These occur because the frames get filled with solid and no more slurry can enter. The press is then disassembled and the cakes are removed. The cloth is washed before being remounted on the frames for subsequent filtration.

The operation can be carried out under either constant-pressure or constant-filtration rate condition. The pressure drop across the filter press during the filtration is held constant in the present experiment. Since the constant pressure filtration starts at time t_s, the filtering time, t, is given by

$$t - t_s = \frac{K_c(V - V_s)^2}{2} + \frac{(V - V_s)}{q_0} \tag{1}$$

where V is the volume of filtrate collected during time t. t_s is the time taken to attain the required pressure and V_s is the filtrate volume collected in this time. K_c and q_0 are given by

$$K_c = \frac{c\mu\alpha}{A^2\Delta p} \tag{2}$$

$$\frac{1}{q_0} = \frac{\mu R_m}{A\Delta p} \tag{3}$$

where A is the total surface area of the plates available for filtration (or cake formation); Δp is the pressure drop applied across the press; c is the mass of the particles deposited in the filter (mass of dry cake) per unit volume of filtrate; α is the specific cake resistance; R_m is the filter-medium resistance, and μ is the viscosity of the filtrate.

Equation 1 shows that $(t-t_s)/(V-V_s)$ varies linearly with $(V-V_s)$. A plot between these two variables will have a slope equal to $K_c/2$ and an intercept of $1/q_0$. From such a plot, the values of α and R_m may be calculated using Eqs. 2 and 3.

EXPERIMENTAL SET-UP

REQUIREMENTS
Plate-and-frame filter press assembly, $CaCO_3$ powder, water, bucket, measuring cylinders, stopwatch, oven, physical balance.

SCHEMATIC DIAGRAM OF EXPERIMENTAL SETUP
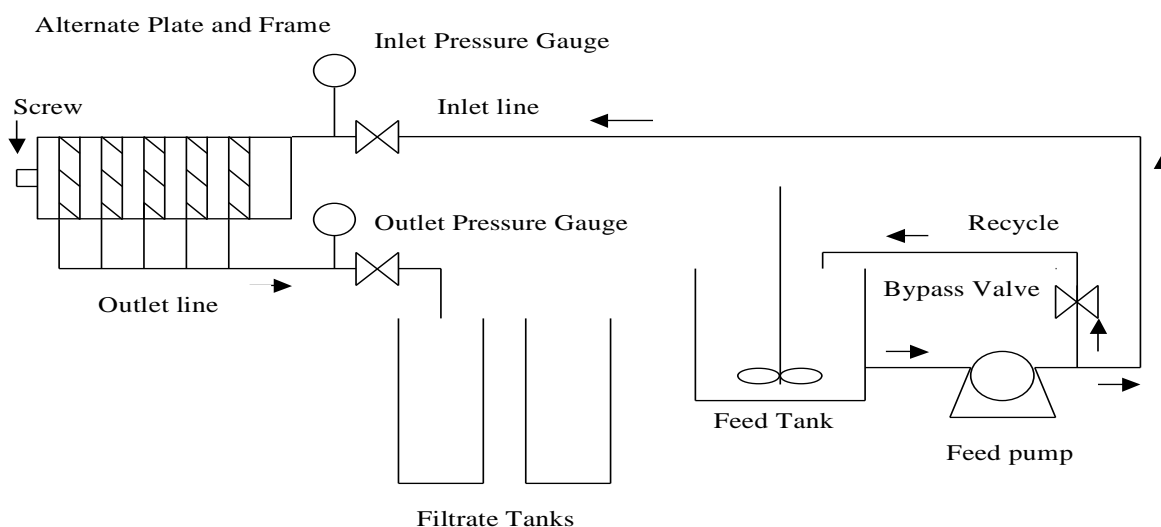
EXPERIMENTAL PROCEDURE
1. Prepare the frames by covering with canvas filter cloth. Place three pairs of plates and frames alternately and secure the arrangement by tightening the screw provided.
2. Fill the filtrate tank with some water to obtain liquid up to the zero level mark (given on scale fixed to tank)
3. Make 20 kg of 5wt% of $CaCO_3$ slurry with water, fill the feed tank and start stirrer.
4. Switch on the feed pump, and note the starting time.

5. Open the inlet valve line. Once the operation starts the pressure will begin to increase. Note the time taken to attain an inlet pressure 0.5 kg/cm^2 (t_s) and the volume of filtrate collected in this time is (V_s).

6. Adjust the Bypass valve to maintain the inlet gage pressure constantly at 0.5 kg/cm^2. From time t_s note the time it takes to move between each succeeding graduations.

7. After collecting 10 liters of filtrate, switch off the feed pump and the stirrer in the feed tank.

8. Disassemble the press by loosening the screw and remove the filter cloth (with the cake deposited on its surface).

9. Remove the cake formed and dry a part of the wet cake in the oven for 15 minutes and estimate the water content of the cake from the difference between the weights of the wet and dried sample. Hence calculate c (corrected value) from Eq (4) of Expt 1 (i.e. Rotary Drum Filter).

10. Rinse the plates and the frames, wash the filter cloths and clean the feed tank with water.

11. Repeat the experiment with 10wt%, 15wt% of slurry.

OBSERVATIONS

1. Area of each plate   = 15.8 x 15.8 cm^2
2. Pressure at the inlet   = 0.5 kg/cm^2 gage
3. Viscosity of filtrate   = 0.886 x 10^{-3} kg/m s
4. Viscosity of water   = 1. 0 x 10^{-3} kg/m s

Set No	t_s (s)	V_s (ml)	Slurry concentration (wt %)	Time, $t^\#$ (s)	Volume of filtrate, $V^\$$ (ml)	Weight (g) Wet Cake	Dry Cake

$^\#$ Includes t_s; $^\$$ Includes V_s

MODEL CALCULATIONS

1. Plot (t-t_s)/(V-V_s) vs. (V-V_s) graph. Fit a straight line through the points using regression analysis. Find the slope and intercept of the straight line.

2. Find the filter surface area, A, as

$A = 2NS$

where,

N = no. of plates

S = surface area of each plate

The factor 2 accounts for the two sides of the plate on which cake is formed.

3. $\Delta p = (0.5 - 0)$ kg/cm^2

RESULTS AND DISCUSSION
1. Determine the specific cake resistance and the filter-medium resistance taking feed-slurry concentration as c in the formulae.
2. Compare these values with those, which were calculated using the corrected c value.
3. Plot α vs. slurry concentration and R_m vs. slurry concentration and discuss the trend.

CONCLUSIONS
Relating to the outcome after accomplishing the aim and the extent to which the objectives were fulfilled.

PRECAUTIONS
1 When setting up the plates and frames see to it that their collection holes are in line with the slurry feed line.
2 Tighten the screw properly in the alternate plate and frame arrangement to avoid leakage

REFERENCES
1. Warren, L McCabe, Smith, J C , and Harriott, P, Unit Operations of Chemical Engineering, 6th edition, McGraw Hill, New Delhi, India, 2000.
2. Richardson, J F, Harker, J H and Backhurst, J R, Coulson and Richardson's Chemical Engineering, 5th edition, Vol-2, Asian Books Private Limited, New Delhi, India, 2002.

Experiment No. 7
ROTARY VACUUM FILTER

AIM

Study of cake filtration using continuous vacuum filter such as Rotary drum filter.

OBJECTIVES

- To study the construction and operation of Rotary drum filter.
- To determine the specific cake resistance (α) and the filter-medium resistance (R_m) for filtering a $CaCO_3$ -slurry for different vacuum conditions.

THEORY

Rotary drum filter (RDF) is one of the most widely used vacuum filters for continuous filtration. It consists of a drum with a slotted face rotating on a horizontal axis at low speed (in the range 0.1 to 3 rpm) in an agitated slurry trough. The surface of the drum is covered with a filter medium like canvas. Under the slotted cylindrical face of the drum is a second drum with a solid surface. Between the two drums are radial partitions dividing the annular space into separate compartments, each connected by an internal pipe to one hole in the rotating plate of a rotary valve. As the drum rotates, each compartment is exposed to vacuum in turn. The vacuum in the segment draws filtrate through the filter medium, depositing the suspended solids on the filter drum as cake. The cake is eventually removed by scrapping it off with a horizontal knife known as a doctor blade.

If the pressure drop across the filter during the cake formation is held constant, the filtering time, t, is given by

$$\frac{t}{V} = \frac{K_c V}{2} + \frac{1}{q_0} \tag{1}$$

where V is the volume of filtrate collected during time t. K_c and q_0 are given by

$$K_c = \frac{c\mu\alpha}{A^2 \Delta p} \tag{2}$$

$$\frac{1}{q_0} = \frac{\mu R_m}{A \Delta p} \tag{3}$$

where A is the surface area of the drum; Δp is the pressure drop applied across the cake; c is the mass of the particles deposited in the filter (mass of dry cake) per unit volume of filtrate; α is the specific cake resistance, R_m is the filter-medium resistance, and μ is the viscosity of the filtrate.

From Eq 1, we find that t/V varies linearly with V, and the plot between these variables will have a slope equal to $K_c/2$ and an intercept of $1/q_0$. From this a plot, the values of α and R_m may be calculated using Eqs 2a and 2b.

Here c (which is the mass of particles deposited in the filter per unit volume of the filtrate) has a value slightly greater that the concentration of solids in the feed slurry. This is because the wet cake includes sufficient water to fill its pores, and V, the actual volume of filtrate, is slightly less that the total liquid in the original slurry. Correction for this retention is made by material balance. If m_F is mass of wet cake, m_c mass of dry cake, ρ density of filtrate and c_s concentration of solids in the slurry the c is given by:

$$c = \frac{c_s}{1 - \left[\left(\dfrac{m_F}{m_c}\right) - 1\right]\dfrac{c_s}{\rho}} \tag{4}$$

This correction may be made after estimating the weight of wet and dry cake experimentally.

EXPERIMENTAL SET-UP

REQUIREMENTS

Rotary drum filter assembly, $CaCO_3$ powder, water, bucket, measuring cylinders, stopwatch, oven, and physical balance.

SCHEMATIC DIAGRAM OF EXPERIMENTAL SETUP
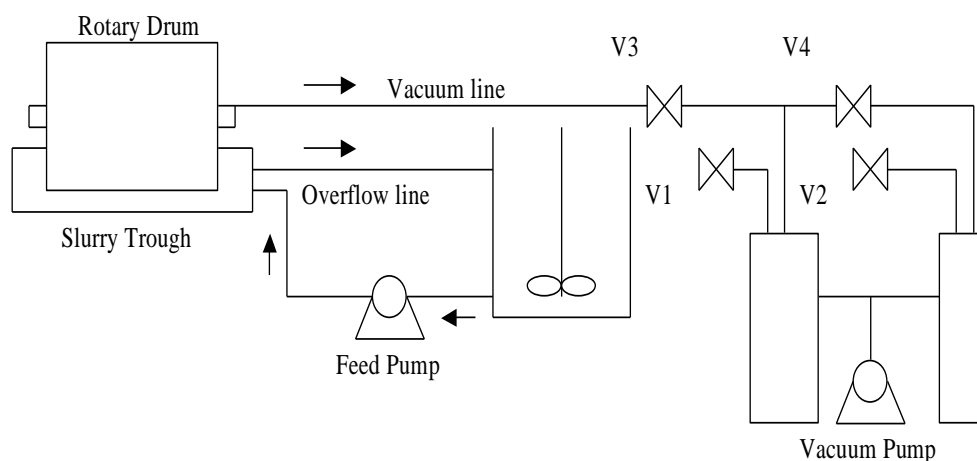
EXPERIMENTAL PROCEDURE

1. Make 50 kg of 10wt% $CaCO_3$ slurry using the following procedure: measuring out the required water and transfer to the slurry tank. Add the calculated amount of $CaCO_3$. Start the feed tank stirrer.
2. Fill the filtrate tank with some water to obtain liquid up to the zero level mark (given on scale fixed to tank).
3. Read the precautions given. Switch on the rotary drum motor, the rotary trough agitator and the feed tank motor. Fill the slurry trough up to the opening of the overflow. You are now ready to start operation.
4. Switch on the vacuum pump and adjust a vacuum pressure of 200 mmHg on the pressure gauge of the first filtrate tank using the corresponding valve (V_1).
5. Open the valve (V_3) of the filtrate tank to allow the filtrate to be sucked into the filter tank from the rotary drum.
6. Note down the height of the filtrate being collected in the filtrate tank at time intervals of say one minute.
7. Once the first filtrate tank is filled use the second tank for collection, operating the appropriate valves.
8. After collecting 10 liters of filtrate, close the valve (V_4) on the filtrate tank. Also switch off the vacuum pump and the stirrer in the feed tank.
9. Remove the cake formed on the drum surface manually since the cake thickness may not be enough to be scraped by the doctor knife.
10. Now switch off the rotary drum motor, trough agitator and feed tank agitator and motor.
11. Dry a part of the wet cake in the oven for 30 minutes and estimate the water content of the cake from the difference between the weights of the wet and dried sample. Hence calculate c (corrected value) from Eq (3) of Exp (1).
12. Drain out the content of the feed tank and clean it with water thoroughly before recharging it with fresh feed slurry. Also clean the surface of the drum and the slurry trough with water before starting a new set of experiments.
13. Repeat the experiment with 400 mmHg of vacuum pressure.

OBSERVATIONS

1. Drum diameter   = 350 mm
2. Drum length   = 450 mm
3. Rotational speed of the filter = 1.5 rpm
4. Viscosity of filtrate   = 0.886 x 10^{-3} kg/m s
5. Viscosity of water   = 1.0 x 10^{-3} kg/m s
6. One liter of liquid (filtrate) results in 2 cm rise in the liquid level in the filtrate tank.

Set No	Slurry conc. (wt %)	Vacuum pressure	Time (s)	Liquid level in the filtrate tank (cm)	Weight (g)	
					Wet Cake	Dry Cake

MODEL CALCULATIONS

1. For each pressure drop: plot t/V vs. V, fit a straight line through the points using regression analysis. Find the slope and intercept of the straight line.
2. Determine the specific cake resistance and the filter-medium resistance (taking c as the feed slurry concentration in Eqs 2 and 3).

RESULTS AND DISCUSSION

1. Report values of specific cake resistance (α) and the filter-medium resistance (R_m) with appropriate units, for different vacuum pressures.
2. Compare your values with those found in literature.
3. Derive Eq (4) using material balance.
4. Discuss significance and origin of Ergun Equation, which is the basis of Eq 1.

CONCLUSIONS

Relating to the outcome after accomplishing the aim and the extent to which the objectives were fulfilled

PRECAUTIONS

1. Ensure that the trough remains filled with the slurry; otherwise the desired vacuum may not be obtained.
2. See that the drain valve of the rotary trough is closed before switching on feed pump.
3. Liquid level in the filtrate tank is not to exceed 22 cm; otherwise liquid will be sucked in to vacuum pump.
4. Switch off feed pump if slurry reaches overflow in rotary trough.

REFERENCES

1. Warren, L McCabe, Smith, J C , and Harriott, P, Unit Operations of Chemical Engineering, 6th edition, McGraw Hill, New Delhi, India, 2000.
2. Richardson, J F, Harker, J H and Backhurst, J R, Coulson and Richardson's Chemical Engineering, 5th edition, Vol-2, Asian Books Private Limited, New Delhi, India, 2002.

Experiment No. 8a

BASKET CENTRIFUGE

1. Aim
To study the characteristics of a basket centrifuge.

2. Objective
To determine the % recovery of water and %water retained by the cake for 5wt% and 10 wt% slurry at different speeds of rotation of basket.

3. Theory
Centrifugal force is widely used for separation when a force greater than the force of gravity is desired for separation of solids and fluids of different densities.

Basket centrifuge is the simplest type of batch centrifugal. It has a basket or bowl mounted on a vertical shaft fixed to a motor. There is another basket inside. The wall of the inner basket is perforated. The feed-slurry is put in a canvas bag, which is placed in the basket. The basket is closed from the top and rotated at a high speed. The liquid passes through the pores of the canvas bag and then the perforations of the inner basket and is collected in the annular space before being drained out of the centrifuge. The solid is retained in the inner surface of the canvas.

4. Experimental Set up

4.1 Requirements
Basket centrifuge (with a canvas bag to hold the slurry), $CaCO_3$ powder, water, oven, physical balance, watch.

4.2. Schematic Diagram of Experimental Setup

Inside cylinder

Filter cloth

Filtrate removal

Shaft for rotating inside cylinder
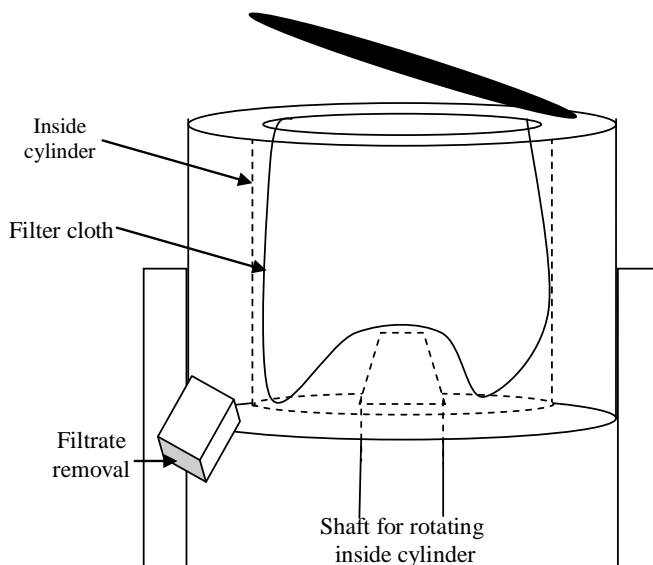
5. Experimental Procedure

1. Prepare 10 kg slurry of 5wt% $CaCO_3$.
2. Place and secure properly the canvas bag inside the basket centrifuge.
3. Transfer the slurry into the canvas bag.
4. Adjust the variac to 50 volts.
5. Switch on the motor and note the time.
6. Collect the filtrate until it stops coming out of the centrifuge, and note the time of filtration.
7. Weigh the amount of water recovered.
8. Collect the cake from the surface of the canvas bag. Take a part of the wet cake, weigh it, put it in the oven [the temperature should be adjusted at about 150^0C]. After 30 minutes weigh the dried cake.
9. Take out the canvas bag from the centrifuge, wash it thoroughly and place it back to carry out further runs.
10. Perform the runs at 60 and 70 volts of variac output.
11. Repeat the experiment (steps 2 to 9) for 10wt% $CaCO_3$ slurry.

6. Observations

Set No	Feed concentration	Variac reading	Weight of the filtrate collected	Time of filtration

7. Model Calculations

Determine the % recovery of water and %water retained by the cake in all the cases.

$$\%\text{water retained} = \frac{(w_2 - w_3)}{(w_2 - w_1)}100$$

Where

w_1 wt of the pan,
w_2 wt of the pan with the wet cake,
w_3 wt of the pan with the dry cake

Assuming the filtrate to be `pure' water, i.e., without any suspended solid, compute %water retained as

$$\left(1 - \frac{\text{wt of filtrate}}{\text{wt of water in feed}}\right)100$$

8. Results & Discussion

Compare the values of %water retained found by two different way as described in model calculations and discuss the discrepancy in the values.

1. Plot %recovery of water vs. variac voltage for 5 wt% and 10 wt% slurry concentration.

2. Plot %water retained by cake vs. variac voltage for 5 wt% and 10 wt% slurry concentration.

9. Conclusions

10. Precautions

References
1. Warren, L McCabe, Smith, J C , and Harriott, P, Unit Operations of Chemical Engineering, 6th edition, McGraw Hill, New Delhi, India, 2000.
2. Richardson, J F, Harker, J H and Backhurst, J R, Coulson and Richardson's Chemical Engineering, 5th edition, Vol-2, Asian Books Private Limited, New Delhi, India, 2002.

Experiment No. 8b

FROTH FLOATATION

1. Aim
Study the separation performance of froth flotation using Denver floatation cell

2. Objective
1. Calculate the percentage recovery of coal using Denver floatation cell.

3. Theory
Flotation includes those operations in which a solid is separated from another by floating one of them at or on the surface of a liquid. In froth floatation, the solid particles are suspended in an aerated liquid and are continuously agitated in the liquid upon which a thick layer of froth is maintained. Because of the difference in the surface properties, one solid more readily adsorbs liquid phase, becomes surrounded by liquid and sinks. The other solid more readily adsorbs gas and becomes at least partially surrounded or covered by the gas. The bulk density of the solid and adsorbed gas bubbles becomes less than that of the liquid, and the whole mass of gas and solid floats to the surface from where the froth is skimmed off. A frothing agent is added to the liquid so that the particles can be held in or at the surface by means of the froth. Surfaces of the specific minerals are treated with chemicals called promoters and collectors, which render those particles air-avid and water repellent. This helps in easier attachment of the air-avid minerals to the air bubbles. Pine oil and kerosene act as collector as well as frothing agent. The reagents are usually specific in their action like kerosene is used as collector for coal. In general, froth floatation processes can be used for particles between about 5 and 250 $\Box$m in size depending on the density. Coarser particles cannot be suitably mixed and suspended in the floatation machine.

Denver floatation machine is an example of a typical froth floatation unit used in the mining industry. It has a floatation cell in which the material is actually separated or floated from the residual tailings. The cell is provided with an overflow for froth removal. An impeller provides the necessary agitation and low pressure air is introduced down the standpipe surrounding the impeller shaft. The liquid is discharged radially from the impeller across the base of the tank, and then rises vertically. There is maximum turbulence at the base of the tank, while the upper part of the cell remains relatively quiescent.
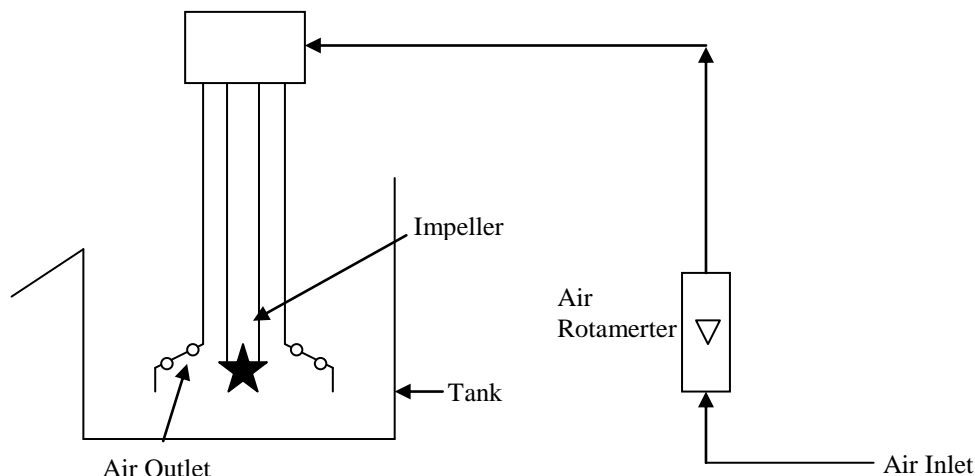
4. Experimental Setup

4.1. Requirements
Denver apparatus, coal, sand, water, compressor, oven, physical balance, pine oil, kerosene, skimmer, filter cloth.

4.2. Schematic Diagram of Experimental Setup
The schematic diagram of the experimental setup is shown below:

5. Experimental Procedure

1. Make a mixture of 100 gm coal and 900 gm sand.
2. Put the mixture of coal and sand in the floatation cell and fill the cell with water (up to the overflow).
3. Add specified amount of pine oil and kerosene.
4. Stir the whole mass manually to get a homogenous mixture.
5. Adjust the air flow from the compressor such that that the rotameters reading is 60, which corresponds to an air velocity of about 0.24 m/s. Froth will now start forming.
6. Start the impeller-agitator and note the time.
7. Place a filter cloth to collect the froth. Keep collecting the froth until the froth formation ceases or diminishes significantly.
8. Note the time taken to complete the separation.
9. Weigh the wet froth along with the filter cloth.
10. Place the filter cloth in the oven for drying for 30 minutes.
11. Take the dry weight of the particles.
12. Carry out the experiments with the following combinations of pine oil and kerosene.

S No	Pine Oil (ml)	Kerosene (ml)
1	20	0
2	12	8
3	8	12
4	0	20

6. Observations

S No	Volume (ml)		Weight of filter cloth (g)	Time of floatation (min)	Weight of filter cloth+Particles (g)	
	Pine Oil	Kerosene			Before drying	After drying

7. Model Calculations
1. Calculate the weight of the initial coal before froth floatation process (w_1) – weight of dry coal collected after froth floatation process (w_2)
2. Calculate the percent recovery of coal = $(w_1 – w_2)/w_1$

8. Results and Discussion
1. Plot % recovery of coal vs. % concentration of pine oil.
1. Discuss the effect of kerosene and pine oil on the effectiveness (% recovery and time of recovery) of froth floatation.

9. Conclusions

10. Precautions

References
1. Perry, R H, and Green, D W, Perry's Chemical Engineers' Handbook, 7th edition, McGraw Hill, New Delhi, India, 1997.
2. Richardson, J F, Harker, J H and Backhurst, J R, Coulson and Richardson's Chemical Engineering, 5th edition, Vol-2, Asian Books Private Limited, New Delhi, India, 2002.

Experiment No. 9a

SINGLE CONE CLASSIFIER

1. Aim
To study the characteristics of a single cone classifier.

2. Objective
To determine the separation efficiency of mixture of a coal and sand particles for different flow rate of water.

3. Theory
Classification is the separation of solid particles into two or more fractions depending on their settling velocities in a fluid. The settling velocities in turn depend on the density and size of the solid particles. Thus it is possible to separate a mixture of two materials into its constituents, or to separate a mixture of particles of the same material into a number of size fractions.

All wet classifiers depend on the difference in settling rate between coarse and fine or heavy- and light-specific gravity particles to be separated. Rates can be controlled to some extent by mild agitation, providing for hindered settling, and centrifugal force versus gravity in centrifuging types of units. Several fundamental laws on classification are:

1. Coarse particles have a relatively faster settling velocity than fine particles of the same specific gravity.
2. Heavy-gravity particles have a relatively faster settling velocity than light-gravity particles of the same size. High solids concentration increases the viscosity and density of the fluid medium.
3. Settling rates of solid particles become progressively slower as the viscosity or density of the fluid medium increases.

Cone classifiers are one of the oldest types but are still used for relatively crude work because of low cost of installation. They are limited in diameter because of high headroom requirements caused by the $\pm 60°$ sloping sides. Units are simple and are often fabricated locally with millwright ingenuity fashioning the apex opening arrangement for adjustment or control of the spigot coarse product. Operating attention is often necessary to a greater degree than for the more positive mechanical types. Cost figures are not available. It is similar to cylindrical tank classifier, except tank is conical to eliminate need for rake. Its low cost (simple enough to be made locally), and simplicity can justify relatively inefficient separation. It is used for desliming and primary dewatering.

Separation efficiency of cone classifier may be defined as:

Separation efficiency $\eta = \dfrac{m_o}{m_i}$ (1)

Where,
η Efficiency of separation

m_o Weight of coal obtained
m_i Initial weight of coal

4. Experimental Procedure
1.   Prepare a mixture of 100gms of coarse sand and 50gm of 600micron coal particles.
2.   Adjust the water flow rate at some constant value.
3.   Connect the water tube to the water inlet of the cone classifier
4.   Pour the mixture slowly and steadily in the cone from top.
5.   Collect the coal particles from the overflow until particles stop coming out.
6.   Weigh the coal particles after drying.
7.   Repeat the experiment for two more ratio keeping the flow rate as constant. Notes:
Note:
(i) Use bigger sand particles.
(ii) Start with low flow rate in the first run and increase the flow rate in the subsequent runs.

5. Observations
1. Density of sand   = 1.4 gm/cc
2. Density of coal   = 0.65 gm/cc

Set No	Water flow rate (ml/min)	Coal Size (mm)	Time of collection (s)	Weight of coal obtained (g)

7. Model calculations
Calculate separation efficiency from eqn. 1.

8. Results & Discussion
1. Determine and compare the efficiency of separation for different water flow rate and different coal sizes of the particles.
2. Plot the separation efficiency vs. the flow rate of water for each size of coal particles, on a single graph paper.
3. Graphically show the effect of the particle size on the time of classification for each of the flow rates.

References
1. Warren, L McCabe, Smith, J C , and Harriott, P, Unit Operations of Chemical Engineering, 6th edition, McGraw Hill, New Delhi, India, 2000.
2. Richardson, J F, Harker, J H and Backhurst, J R, Coulson and Richardson's Chemical Engineering, 5th edition, Vol-2, Asian Books Private Limited, New Delhi, India, 2002.

Experiment No. 9b

THICKENER

Objective

To study the working principle of continuous type thickener.

Aim

1. To determine the concentration of product obtained at the different height of sampling point.

2. Plot the curve for (a) Time vs. Concentration
                              (b) Height of sampling point vs. Concentration

Introduction

The thickener is the industrial plant in which the concentration of a suspension is increased by sedimentation, with the formation of the clear liquid. In most cases, the concentration of the suspension high and hindered settling takes place. Thickeners may operate as batch or continuous units, consists of tanks from which the clear liquid is taken off at the top and the thickened liquor at the bottom.

Industrial sedimentation operations may be carried out batch wise and continuously in equipment called thickener. The equipment is a cylindrical tank with opening for a slurry feed and product draw off. The tank is filled with dilute slurry and slurry is permitted to settle. Continuous thickeners are large diameter, shallow depth tanks with slowly revolving rakes for removing the sludge. Around the top edge of the tank is a clear liquid overflow. The rake serves to scrap the sludge towards the centre of the bottom for discharge. The motion of the rake also stirs only the sludge layer. This gentle stirring aids in water removal from the sludge.

Theory

When large tonnage of slurry of low concentration (3-10%) of solids suspended in a liquid is handled, it is economical to separate solids in the form of thickened slurry sediment by means of gravitational settling. This process of separation of suspension in to a clear supernatant liquid and rather dense slurry containing a higher concentration of solids is termed as SEDIMENTATION or THICKENING of solids. The classification capacity of thickener is determined by the settling rate of suspended solids. This rate may be estimated from the rate of settling of the upper interface in a batch laboratory thickener or measuring cylinder.

Description

The set-up consists of a tank to contain the slurry, feed piping and a feed well to allow the feed stream to enter the tank, a rotating rack mechanism to assist in moving the concentrated solids to

the withdrawal points and underflow solids-withdrawal system and an overflow launder. A feed tank, feed pump, and feed tank agitator is provided.

Utilities Required:

1. Electricity Supply: Single phase, 220 V AC, 50 Hz, 5-15 amp socket with earth connection.
2. Water Supply (Initial fill).
3. Drain required.
4. Floor Area Required: 2m x 1m.
5. $CaCO_3$: 10 kg.

Experimental Procedure:

1. Prepare a calibration chart by taking weights of different concentration.
2. Prepare 5% $CaCo_3$ solution in water.
3. Filter the solution and fed to feed tank.
4. Start the agitator to agitate the slurry.
5. Switch ON the stirrer of thicker.
6. Switch ON pumps to allow feed passing through thickener.
7. Wait till overflow occurs.
8. Take the sample from sampling point at different time intervals.
9. Repeat same procedure for different concentration of feed.

Observation & Calculation:

Observation Table

S. No	t	Density of Sample				
		W_b (at 370 mm)	W_1 (at 230mm)	W_2 (at 155 mm)	W_3 (at 65mm)	W_t (at 0 mm)

Calculations:

C_b = -----------------
C_1 = -----------------
C_2 = -----------------
C_3 = -----------------
C_t = ------------------

Plot the graph for (a) time vs. concentration
(b) Height of sampling point vs. concentration.

Nomenclature:

C_b	=	Concentration of the sample at the base (i.e. 370mm)
C_1	=	Concentration of the sample at a height of 230mm from top
C_2	=	Concentration of the sample at a height of 155mm from top
C_3	=	Concentration of the sample at a height of 65mm from top
C_t	=	Concentration of sample at top (i.e. 0 mm)
W_b	=	Weight of the sample at the base (i.e. 370mm from top)
W_1	=	Weight of the sample at a height of 230mm from top
W_2	=	Weight of the sample at a height of 155mm from top
W_3	=	Weight of the sample at a height of 65 mm from top
W_t	=	Weight of sample at top (i.e. 0mm)

Precaution & Maintenance Instructions:

1. Proper cleaning of valves and pump is required.
2. Feed slurry is to be filtered before feeding it into the tank.
3. Low flow is best for operation.
4. Proper cleaning with running water is necessary after experiment.

Troubleshooting

If the motor is not moving but electric power is showing, Switch off the power and move the motor shaft manually and when it moves freely, then operate it.

References:

1. Coulson & Richardson, " Chemical Engineering Vol-2" 4th ed, Asian Books Pvt. Ltd, ND, 1991, Page 192-194.
2. McCabe, Smith "Unit Operations of Chemical Engineering"7th ed., McGraw-Hill, NY, 2005, Page 1060-1065.

Calibration Chart
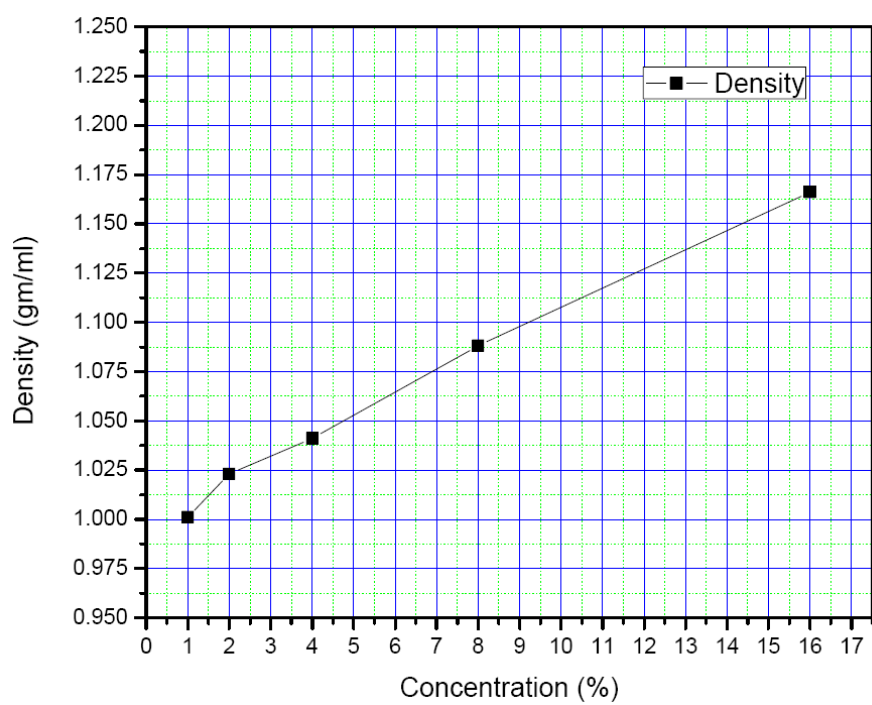
Experiment No. 10

TROMMEL

AIM:
To determine the overall effectiveness for a Trommel.

APPARATUS:
Trommel

APPARATUS DISCRIPTION
A trommel (from the German word for drum, Trommel) is a screened cylinder used to separate materials by size. A trommel screen, also known as rotary screen, is an essential unit which is used mainly in the mineral and solid-waste processing industries. It consists of a perforated cylindrical drum which is normally elevated at an angle at the feed end. Physical size separation is achieved as the feed material spirals down the rotating drum, where the undersized material smaller than the screen apertures passes through the screen, while the oversized material exits at the other end of the drum.
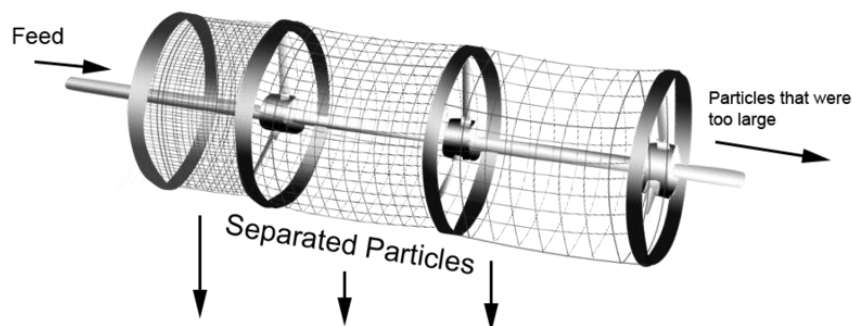
THEORY
Screening is a method of separating particles according to the size alone. In industrial screening the solids are dropped on, or thrown against, a screening surface. The undersized particles, or fines, passed through the screen openings; oversized particles, or tails, do not.

Industrial screens are made from woven wire, silk or plastic cloth, metal bars, perforated, slotted metal plates, or wires that are wedge-shaped in cross section. Various metals are used, with steel and stainless steel the most common. Standard screen range in the size from 4 inch to 400 mesh, and woven metal screens with opening as small as 1μm are commercially available.

An ideal screen would sharply separate the feed mixture in such a way that the smallest particle in the overflow would be just larger than the largest particle in the underflow. Such an ideal separation defines a cut diameter DPC that makes the point of separation between the fractions. Usually DPC is chosen to be equal the mesh opening of the screen. Actual screen do not give a perfect separation about the cut diameter. The closest separation are obtained with spherical particles on standard testing screens but even here there is an overlap between the smallest particles in the overflow and the largest ones in the underflow. The overlap is specially pronounced when the particles are needle like or fibrous or where the particle tends to aggregate into clusters that act as large particles.

MATERIAL BALANCE OVER SCREENS

Let F, D, and B be the mass flow rate of feed, overflow, and underflow respectively and xF, xD , and xB be the mass fraction of oversize material in these three steams. The mass fractions of undersized material in the feed, overflow and underflow are 1-x_F , 1-x_D , and 1-x_B .

Since the total material balance fed to the screen must leave it either as underflow or as overflow,

$$F = D + B$$

The material A in the feed must also leave in these two streams and

$$Fx_F = Dx_D + Bx_B$$

Elimination of B from Esq. –

$$\frac{D}{F} = \frac{x_F - x_B}{x_D - x_B}$$

Elimination of D gives-

$$\frac{B}{F} = \frac{x_D - x_F}{x_D - x_B}$$

SCREEN EFFICIENCY

The efficiency of a screen is a measure of the success of a screen in closely separating material. If the screen functions perfectly, all the oversize material would be in overflow and all the undersize material would in the underflow. A common measure of screen efficiency is the ratio of oversize material that is actually in the overflow to the amount entering with the feed. These quantities are D xF and F xF, respectively. Thus

$$E_A = \frac{Dx_D}{Fx_F}$$

Where, EA is the screen efficiency based on the oversize. Similarly an efficiency EB based on the undersize material is given by

$$E_B = \frac{B(1-x_B)}{F(1-x_F)}$$

The overall effectiveness is given by

$$E = E_A E_B$$

Thus,
$$E = \frac{(x_F - x_B)(x_D - x_F)x_D(1-x_F)}{(x_D - x_B)^2 x_F(1-x_F)}$$

PROCEDURE

- Take 5 kg of sample preferably particle size less than BSS mesh no 5
- Screen it through the trommel.
- Collect the underflow separately from each screen.
- Perform sieve analysis for feed using stand set of sieves (BSS 6, 8, 10, 12, and 14).
- Similarly perform sieve analysis for underflow from each screen.
- For particular screen opening (D_P), find the value of x_D, x_F and x_B.
- Calculate the screen effectiveness.

A LABORATORY MANUAL FOR

Chemical Engineering Laboratory –II

(Cycle – II)

Edited By

Dr Hare Krishna Mohanta &   Dr Pratik N Sheth

Department of Chemical Engineering,
Birla Institute of Technology & Science (BITS), Pilani
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Contents

Experiment No. 11
CASCADE CSTR

OBJECTIVES

1. To study the performance of a cascade of three equal volumes CSTR's in series for the saponification of ethyl acetate with sodium hydroxide.
- To determine the reaction rate constant for saponification of ethyl-acetate with NaOH at atmospheric conditions.

THEORY

The stirred-tank reactor in the form of either a single tank, or more often a series of tanks is particularly suitable for liquid-phase reactions, and is widely used in the organic chemicals industry for medium and large scale production. It can form a unit in a continuous process, giving consistent product quality, easy of automatic control, and low manpower requirements.

Consider a system of N mixed flow reactors that are cascaded (connected in series).Though the concentration is uniform in each reactor, there is a change in the concentration as fluids move from reactor to reactor. This stepwise drop in concentration suggests that the larger the number of units in series.

Reaction: $\quad NaOH + CH_3COOC_2H_5 \rightarrow CH_3COONa + C_2H_5OH$
$$A \qquad\qquad (B) \qquad\qquad (C) \qquad\qquad (D)$$
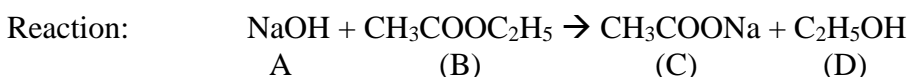
The above reaction is second order reaction (n = 2)
For second order reaction

$$-r_A = \frac{-dC_A}{dt}$$

$$-r_A = kC_A^2 \qquad\qquad (1)$$

Where $-r_A$ is rate of disappearance of A, is concentration of A at any time t, K is rate constant.
The performance equation for the mixed flow reactor at steady state is:

$$\tau = \frac{V_R}{V_O} \qquad\qquad (2)$$

In terms of concentration we can write

$$\tau = \frac{(C_{A0} - C_A)}{-r_A} \qquad\qquad (3)$$

Reactor volume (volume of fluid in the reactor) $\qquad = V_R$, Liters,
Volumetric feed rate (volumetric feed rate of A + volumetric feed rate of B) $\qquad = V_0$, LPM
Space time $\qquad = \tau = V_R / V_o$, min
C_{AO} is initial concentration of A
Substitute the value of $-r_A$ in eqn (3) from eqn (1)
We get,

$$\tau = \frac{(C_{A0} - C_A)}{KC_A^2}$$

$$X_A = \frac{(C_{A0} - C_A)}{C_{A0}}$$

Also, a plot of τ vs. $X_A/(1-X_A)^2$ shall yield a straight line for an assumed second order reaction with slope = $1/(KC_{A0})$. From this slope rate constant, K can be calculated.

DESCRIPTION
The set up consists of three identical stirred tanks made of stainless steel arranged in a cascaded manner, two feed tanks through which chemicals are fed to the reactors. The flow rates can be adjusted by operating the needle valve and measured by rotameter. The compressed air is used for circulation of feed. Pressure regulator & pressure gauge are fitted at the compressed air line. Products coming out from the reactor are analyzed by chemical titration.

CHEMICALS
1. N/10 NaOH,
2. N/10 HCI,
3. N/10 Ethylacetate (8.8 gms of ethylacetate in 1 L of water),
4. Indicator (Phenolphthalein)
5. N/40 HCl

UTILITIES REQUIRED
1. Compressed Air Supply to be maintained @ 1-2 kg/cm²
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 (to be arranged by institute).

SPECIFICATIONS

Reactor	:	Material Stainless Steel 304 Grade, Capacity 2 Ltrs (Approx).
Stirrer	:	Stainless Steel Impeller and shaft coupled with FHP Motor
Feed Tank (2Nos.)	:	Material Stainless Steel 304 Grade, Capacity - 20 Ltrs.
Feed Circulation	:	By compressed air
Flow Measurement	:	Rotameter 2Nos. (one each for Reactants)
Piping	:	Stainless Steel/PVC/GI
Pressure Regulator	:	0-2 Kg/cm^2
Pressure Gauge	:	Bourdon type 0-2 Kg/cm^2
Stop Watch	:	Electronic
Control Panel	:	On / off switch, Mains Indicator etc.

PROCEDURE

1. Prepare 20L of N/10 sodium hydroxide solution by dissolving 80 gm of sodium hydroxide in 20L of distilled water. Standardize using 0.1N Oxalic Acid).
2. Prepare 20L of N/10 ethyl acetate solution by mixing 176 gm (or 196 ml) of ethyl acetate in 20L of distilled water.
3. Take 20 ml of N/10 hydrochloric acid in three measuring cylinders.
4. Fill the burette by N/10 sodium hydroxide (Standard Solution).
5. Close all the valves V1-V12.
6. Open valve V4 and V6 , fill sodium hydroxide solution in feed tank A.
7. Close valve V4 and V6.
8. Open valve V5 and V7 , fill ethyl acetate solution in feed tank B.
9. Close valve V5 and V7.
10. Connect compressed air supply to the set-up at valve V1.
11. Connect electric supply to the set-up.
12. Open valve V1 and set air pressure 0.5 to 1 kg/cm^2 by pressure regulator and pressure gauge.
13. Pass sodium hydroxide and ethyl acetate into the reactor by allowing equal flow rate controlled by valve V2-V3.
14. Start the stirrer of the reactor.
15. After about 10 min or time equal to 1.5times the residence time of the reactor (which ever is greater) collect exact 10 ml of the sample from the outlets of reactor 1, 2 & 3 in measuring cylinders.
16. Titrate the sample solution, using phenolphthalein as an indicator against N/40 HCl (add HCl from burette).
17. Repeat experiment for different flow rates of feed.

OBSERVATION & CALCULATION

Data	
Working volume of reactor-1 VR1	= _____ Lit
Working volume of reactor-2 VR2	= _____ Lit
Working volume of reactor-3 VR3	= _____ Lit

Standardization of NaOH:

    Volume of oxalic acid taken in conical flask, V_1 =

    Normality of oxalic acid, N_1 =

    Volume of NaOH run down from the burette (x), V_2 =

    Normality of NaOH ,N_2 = $(N_1 V_1)/ V_2$

Standardization of HCl:

    Volume of HCI taken in conical flask   = V_3

    Volume of NaOH run down from the burette   = V_4

    Normality of HCI   = $(N_2 V_4)/ V_3$

Volume of N/40 HCl used = V_1 ml

Volume of sample taken for titration purpose = V_2 ml

N_1V_1 (HCL SIDE) = N_2V_2 (REACTANT MIXTURE/SAMPLE SIDE)

Implies that N2 i.e strength of NaOH in reaction mixture is = $(N_1 . V_1) / V_2$ gm mole per liter

Concentration of NaOH at outlet:

Concentration of un-reacted NaOH in the reaction mixture, C_A

$C_A = (N_1 . V_1) / V_2$

Observation Table

S No	V_A (LPH)	V_B (LPH)	C_{A1} Conc of NaOH at reactor 1 outlet	C_{A2} Conc of NaOH at reactor 2 outlet	C_{A3} Conc of NaOH at reactor 3 outlet

CALCULATIONS

$C_{AO} = N_{NaOH}/2$ (mole/L)

For Reactor 1

$X_{A1} = (C_{AO} - C_{A1})/C_{AO}$

$\tau_1 = V_{R1} * 60/(V_A + V_B)$ (min)

Calculation Table 1

S.No	τ_1 (min)	X_{A1}	$X_{A1} / [1-X_{A1}]^2$

Plot τ_1 vs. $X_{A1} / [1-X_{A1}]^2$ and find slope S_1.

$k_{\tau_1} = 1/(C_{AO}S_1)$ (L/mole min)

Reactor 2

$X_{A2} = (C_{AO} - C_{A2})/C_{AO}$

$\tau_2 = V_{R2} * 60/(V_A + V_B)$ (min)

Calculation Table 2

S.No	τ_2 (min)	X_{A2}	$X_{A2}/[1-X_{A2}]^2$

Plot τ_2 vs. $X_{A2}/[1-X_{A2}]^2$ and find slope S_2.

$k_{\tau2} = 1/(C_{AO}S_2)$ (L/mole min)

Reactor 3

$X_{A3} = (C_{AO}-C_{A3})/C_{AO}$

$\tau_3 = V_{R3}*60/(V_A+V_B)$ (min)

Calculation Table 3

S.No	τ_3 (min)	X_{A3}	$X_{A3}/[1-X_{A3}]^2$

Plot τ_3 vs. $X_{A3}/[1-X_{A3}]^2$ and find slope S_3.

$k_{\tau3} = 1/(C_{AO}S_3)$ (L/mole min)

Calculation Table 4
Calculate
$$-r_A = kC_{Ao}^2(1-X_A)^2$$

S.No	F_{A0}	$F_{A0}/-r_A$	X_A

Plot the $F_{A0}/-r_A$ vs X_A for each F_{A0} value and shade the area in the graph by showing the volume of the reactor.

NOMENCLATURE

Nom	Column Heading	Units	Type
C_{A1}	Concentration of unreacted NaOH in reactor-1	Mole/L	Calculated
C_{A2}	Concentration of unreacted NaOH in reactor-2	Mole/L	Calculated
C_{A3}	Concentration of unreacted NaOH in reactor-3	Mole/L	Calculated
C_{A0}	Initial Concentration of unreacted NaOH in feed	Mole/L	Calculated
k_1	Rate constant for individual run of reactor-1	L/mole min	Calculated

k_2	Rate constant for individual run of reactor-2	L/mole min	Calculated
k_3	Rate constant for individual run of reactor-3	L/mole min	Calculated
N_1	Normality of NaOH used for titration	g eq /L	Given
N_{HCl}	Normality of HCl	g eq /L	Given
N_{NaOH}	Normality of NaOH in feed solution	g eq /L	Given
S_1	Slope of graph τ_1 vs. $X_{A1}/[1-X_{A1}]^2$	Min	Calculated
S_2	Slope of graph τ_2 vs. $X_{A2}/[1-X_{A2}]^2$	Min	Calculated
S_3	Slope of graph τ_3 vs. $X_{A3}/[1-X_{A3}]^2$	min	Calculated
V_1	Volume of NaOH used for titrate the sample solution taken from reactor-1	ml	Measured
V_2	Volume of NaOH used for titrate the sample solution taken from reactor-2	ml	Measured
V_{Samp}	Vol of sample	ml	Given
V_3	Volume of NaOH used for titrate the sample solution taken from reactor-3	ml	Measured
V_A	Volumetric flow rate of NaOH	LPH	Measured
V_B	Volumetric flow rate of $CH_3COOC_2H_5$	LPH	Measured
V_{HCl}	Volume of N/10 HCl taken for quench	ml	Given
V_{R1}	Working volume of reactor-1	L	Given
V_{R2}	Working volume of reactor-2	L	Given
V_{R3}	Working volume of reactor-3	L	Given
X_{A1}	Degree of conversion from reactor-1		Calculated
X_{A2}	Degree of conversion from reactor-2		Calculated
X_{A3}	Degree of conversion from reactor-3		Calculated
τ_1	Residence time for reactor-1	min	Calculated
τ_2	Residence time for reactor-2	Min	Calculated
τ_3	Residence time for reactor-3	Min	Calculated

Block Diagram

VALVES

1.	V1 - AIR SUPPLY VALVE FOR FEED TANK
2.	V2 - FLOW CONTROL VALVE FOR ROTAMETER A
3.	V3 - FLOW CONTROL VALVE FOR ROTAMETER B
4.	V4 - FEED INLET VALVE FOR FEED TANK A
5.	V5 - FEED INLET VALVE FOR FEED TANK B
6.	V6 - AIR VENT VALVE FOR FEED TANK A
7.	V7 - AIR VENT VALVE FOR FEED TANK B
8.	V8 - DRAIN VALVE FOR FEED TANK A
9.	V9 - DRAIN VALVE FOR FEED TANK B
10.	V10 - DRAIN VALVE FOR REACTOR 1
11.	V11 - DRAIN VALVE FOR REACTOR 2
12.	V12 - DRAIN VALVE FOR REACTOR 3

BLOCK DIAGRAM

NAME - CASCADE C S T R

CODE - CRE-103

FRONT VIEW

VALVES

1.	V1 - AIR SUPPLY VALVE FOR FEED TANK
2.	V2 - FLOW CONTROL VALVE FOR ROTAMETER A
3.	V3 - FLOW CONTROL VALVE FOR ROTAMETER B
4.	V4 - FEED INLET VALVE FOR FEED TANK A
5.	V5 - FEED INLET VALVE FOR FEED TANK B
6.	V6 - AIR VENT VALVE FOR FEED TANK A
7.	V7 - AIR VENT VALVE FOR FEED TANK B
8.	V8 - DRAIN VALVE FOR FEED TANK A
9.	V9 - DRAIN VALVE FOR FEED TANK B
10.	V10 - DRAIN VALVE FOR REACTOR 1
11.	V11 - DRAIN VALVE FOR REACTOR 2
12.	V12 - DRAIN VALVE FOR REACTOR 3

BLOCK DIAGRAM

NAME - CASCADE C S T R

CODE - CRE-103

BACK VIEW

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 tilting the reactant in feed tanks.
5. Flow should not be disturbed during the experiments.
6. Handle the chemicals carefully.

REFERENCE

1. Chemical Reaction Engineering by Octave Levenspeil. Chapter 2 to 5
1. Fogler H. Scoot (2008). Elements of Chemical Reaction Engineering. 4th Ed. ND: Prentice-Hall of India Pvt. Ltd. pp 55-57.

Experiment No. 12
COMBINED FLOW REACTOR

OBJECTIVE

To perform kinetic studies to establish rate constant using a series of reactors i.e. PFR followed by CSTR.

APPARATUS

Experimental set up, conical flasks, pipettes, burette, stopwatch etc.

THEORY

The concentration of the reactant decreases progressively through the system in a plug flow reactor whereas in mixed flow reactor the concentration drops immediately to a low value. Because of this a plug flow reactor is more efficient than a mixed reactor for reactions whose rates increase with reactant concentration, such as nth order irreversible reactions. When N mixed reactors are connected in series, though the concentration in each reactor is uniform in each reactor, there is change in concentration when the fluid moves from reactor to reactor. This stepwise drop in concentration suggests that the large number of mixed flow reactors in series perform like a plug flow reactor.

CHEMICALS
1. N/10 NaOH,
2. N/10HCl,
3. N/10Ethylacetate (8.8 gms of ethylacetate in 1 L of water),
4. Indicator (Phenolphthalein)
5. N/10 oxalic acid

UTILITIES REQUIRED
1. Compressed Air Supply to be maintained @ 1-2 kg/cm²
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 (to be arranged by institute).

PROCEDURE
1. Prepare 1 litre of 0.1 N HCL by dissolving 9ml HCL in 1 litre of water.
2. Prepare 5 litre of 0.1 N NaOH solution by dissolving 20 gm of NaOH flakes in 5 litre of water.

3. Prepare oxalic acid solution 0.1 N by dissolving 3.15 gm of oxalic acid in 500 ml of water.
4. Standardise NaOH with 0.1 N oxalic acid.
5. Prepare 5 litre of ethyl acetate solution by adding 10 ml per litre of water.
6. Transfer the NaOH and ethyl acetate solutions to the overhead tanks provided.
7. Adjusted the flow rates of ethyl acetate and NaOH solution into the first CSTR of the given series of reactors such that the flow rate of NaOH is slightly higher than that of ethyl acetate.
8. Note down the flow rates after steady state has attained.
9. Collect 10 ml of sample from the outlet of the both reacotrs and titrate against N/40 HCl solution.
10. Repeat the experiment for different flow rates and tabulate the readings.

OBSERVATIONS:

Standardization of NaOH:

Volume of oxalic acid taken in the conical flask	$= V_1 =$	
Normality of oxalic acid	$= N_1 =$	
Volume of NaOH run down from the burette (x)	$= V_2 =$	
Normality of NaOH	$= N_2 = (N_1 V_1) / V_2$	

Standardization of HCl:

Volume of HCl taken in the conical flask	$= V_3 =$	
Volume of NaOH run down from the burette	$= V_4 =$	
Normality of HCl	$= (V_2 V_4) / V_3 =$	

Volume of N/40 HCl used $= V_1$ ml

Volume of sample taken for titration purpose $= V_2$ ml

$N_1 V_1$ (HCL SIDE) $= N_2 V_2$ (REACTANT MIXTURE/SAMPLE SIDE)

Implies that N2 i.e strength of NaOH in reaction mixture is $= (N_1 . V_1) / V_2$ gm mole per liter

Concentration of NaOH at outlet:

Concentration of un-reacted NaOH in the reaction mixture, C_A

$C_A = (N_1 . V_1) / V_2$

OBSERVATION

Diameter of the reactor	$= D =$
Length of the reactor	$= L =$

Sr. No.	Flow rate of ethyl acetate cc/sec.	Flow rate of NaOH cc/sec.	C_{A1} Conc of NaOH at reactor 1 (PFR) outlet	C_{A2} Conc of NaOH at reactor 2 (CSTR) outlet

SPECIMEN CALCULATION

Vol flow rate of ethyl acetate $=V_A$

Vol flow rate of NaOH $=V_B$

$$C_{A0} = \frac{C_{AS}V_A}{V_A+V_B} \qquad \& \qquad C_{B0} = \frac{C_{BS}V_B}{V_A+V_B}$$

$$M = \frac{C_{B0}}{C_{A0}}$$

$$X_A = \frac{(C_{A0} - C_A)}{C_{A0}}$$

$$C_B = C_{B0} - C_{A0}X_A$$

$$V = V_A + V_B$$

$$V_R = \frac{\pi D^2}{4} * L, \qquad \text{Volume of Reactor}$$

$$\tau = \frac{V_R}{V}, \qquad \text{Space time}$$

$$\tau k = \varphi = \frac{1}{C_{A0}[\left(\frac{C_{B0}}{C_{A0}}\right) - 1]} \ln\left[\frac{\left(\frac{C_{B0}}{C_{A0}}\right) - X_A}{\left(\frac{C_{B0}}{C_{A0}}\right)(1 - X_A)}\right]$$

$$\tau = \varphi/k$$

Sr.no	τ, min	ϕ

Plot ϕ versus τ and the slope of the line is the rate constant for the reaction, k.

Calculation Table 4

Calculate

$$-r_A = kC_{Ao}^2(1 - X_A)(M - X_A)$$

S.No	F_{A0}	$F_{A0}/-r_A$	X_A

Plot the $F_{A0}/-r_A$ vs X_A for each F_{A0} value and shade the area in the graph by showing the volume of the reactor.

PRECAUTIONS

1. Measure the exact volume of water and weigh the chemicals exactly.

2. Always use distilled water and good quality chemicals and standard solution for titration.

3. Use precision electronic balance for weighing of chemicals. Don't mix the droppers of different chemicals.

4. Keep all the drain valves closed and vent valve should be open while filling the reactant in feed tanks.

5. Flow should not be disturbed during the experiments.

6. Handle the chemicals carefully. Refer Material Safety Data Sheets (MSDS) of chemicals being used.

7. Use primary standards for standardizing/determining chemical strengths.

8. Do not open the vent of pressurized reactant tanks suddenly. Reduce the pressure in feed tanks by adjusting pressure setting knob and open vent valves only after pressure gets released slowly.

TROUBLE SHOOTING

1. If any type of suspended particles are come in the Rotameter. Remove the Rotameter clean the tube and fit that at its place.

2. If there is any leakage; tight that part or remove that and fix that again after wrapping Teflon tape.

3. If Rotameter is fluctuating either Feed tank may have got emptied or tank pressure is getting reduced. Determine appropriate cause and rectify that.
(Procedure: two nuts are there lose first nut and tight the second slightly, and then first also. Both nuts are on Rotameter)

REFERENCES

Fogler H. Scott (2008). *Elements of Chemical Reaction Engineering*. 4th Ed. Pearson Education, New Delhi, India

Levenspiel,"Chemical Reaction Engineering," 2nd edition, John Wiley & Sons, Singapore, 1995.

Experiment No. 13
PACKED BED REACTOR

OBJECTIVE

To determine the reaction rate constant for saponification of ethyl acetate with NaOH at atmospheric conditions.

INTRODUCTION

In an ideal plug flow reactor (PFR) there is no mixing in the direction of flow and complete mixing perpendicular to the direction of flow. Concentration of the reactant varies along the length of the reactor but not in the radial direction. In case of a non- catalytic packed bed reactor, an additional contact surface is provided for the reaction by the packed bed. It is expected that the yield of the reaction should be high in the case of a packed bed reactor as compared to an equal volume plug flow reactor.
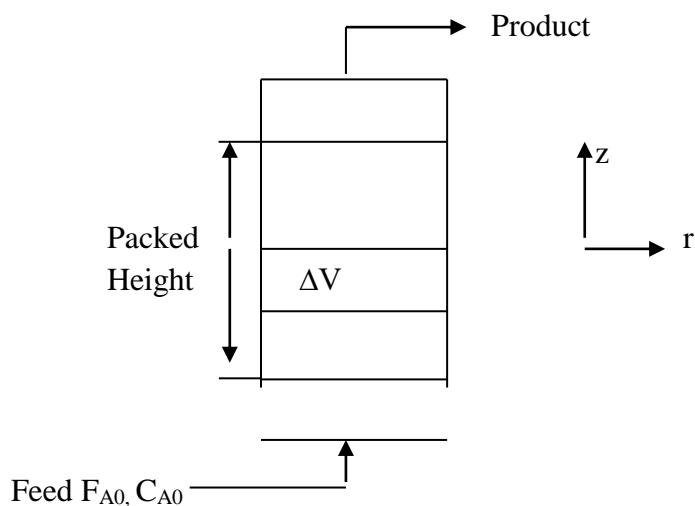
Assuming plug flow condition to exist in the reactor, then under steady state operation:

$$\frac{V}{F_{A0}} = \int_0^X \frac{dX}{-r_A}$$

Where, V is the active volume of the packed bed reactor (total volume of the packed section-solid volume =void volume)

For the reaction

$$NaOH + CH_3COOC_2H_5 \xrightarrow{\ k\ } CH_3COONa + C_2H_5OH$$
$$A \qquad\quad B \qquad\qquad\qquad C \qquad\qquad D$$

70
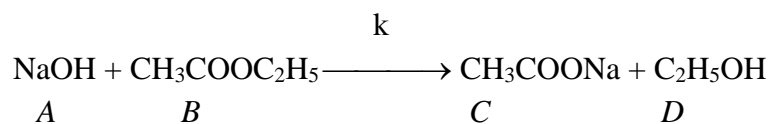
$-r_A$ = $kC_A C_B$

For the condition:

C_{A0} = C_{B0} and neglecting change in density.

$-r_A$ = kC_A^2

C_A = $C_{A0}(1-X_A)$

$$\frac{V}{F_{A0}} = \frac{1}{kC_{A0}^2}\int_0^X \frac{dX_A}{(1-X_A)^2}$$

Where,

F_{A0} = molar flow rate of NaOH

F_{A0} = vC_{A0}

v = Volumetric flow rate of feed

V = Active volume of the packed bed reactor

$$\frac{V}{v} = \frac{1}{kC_{A0}}\int_0^X \frac{dX_A}{(1-X_A)^2}$$

$$\tau = \frac{1}{kC_{A0}}\frac{X_A}{(1-X_A)}$$

When $C_{AO} \neq C_{BO}$, then,

$$\tau = \frac{1}{kC_{A0}\left(\frac{C_{B0}}{C_{A0}}-1\right)}\ln\frac{\left(\frac{C_{B0}}{C_{A0}}\right)-X_A}{\left(\frac{C_{B0}}{C_{A0}}\right)(1-X_A)}$$

$$X_A = \frac{C_{A0}-C_A}{C_{A0}}$$

C_A = Concentration of unreacted NaOH at steady rate.

k = Reaction rate constant

APPARATUS

A packed bed reactor with the following dimensions:

Column I.D. = 43 mm

Column height = 1 m

Packed height = 1 m

Packing material: Glass reaching rings (1=d= 9 mm)

Effective volume of the reactor = V_R = 1.1 L

- 6 No. 100 cc graduated cylinder

- 6 No. 25 cc titration flasks
- 50 cc burette
- Stopwatch

CHEMICALS
N/10 NaOH, N/10 HCL, N/10 Ethyl acetate, Phenolphthalein Indicator

PROCEDURE
1. Prepare 10L of M/10 NaOH solution by dissolving 40 gm of NaOH in 10L of water.
2. Prepare 10L of M/10 ethyl acetate solution by mixing 88 gm of ethyl acetate in 10L of water.
3. Fill the respective tanks with there solutions.
4. Standardize NaOH solution by titrating against 0.1 N oxalic acid and phenolphthalein as indicator.
5. Adjust the flow rate of the two streams so that in the feed mixture $C_{A0} = C_{B0}$ (i.e. have equal flow rates (eg. 2 LPH each).
6. Pass equimolar feed rates of ethyl acetate and NaOH into the PBR.
7. After about 1.5 times the residence time collect exact 10 ml of the sample of the liquid at the out let and titrate against N/40 HCl.
8. Note the volumetric flow rate of liquid at the end of the reactor.
9. Collect the feed mixture at the bottom of the packed bed reactor and measure the initial cone. Of NaOH in the feed. Also, determine the cone. Of feed NaOH solution in the feed tank.
10. Note the reaction temperature.
11. For calculating the conversion at equilibrium condition, collect the sample 25 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/40 HCI using phenolphthalein as indicator. Note the volume of N/40 HCI used.
12. Repeat all the steps for four (4) different flow rates of feed (individual feed rate in the range of 21ph to 10 1ph)

CALCULATIONS & OBSERVATIONS
Run No

Reaction temp.	$T =$ _____ ^{0}C
Reactor diameter	$d =$ _____ m
Reactor packed height	$Z =$ _____ m
Effective volume of the reactor	$V_R=$ _____ L
Volumetric feed rate of feed	$v =$ _____ m^3 /min
Initial Cone. Of NaOH in the feed mixture	$C_{AO} =$ _____ gmole/L
Initial Conc. Of Ethyl acetate in the feed mixture	$C_{BO} =$ _____ gmole/L

Standardization of NaOH:

Volume of oxalic acid taken in the conical flask	$= V_1 =$
Normality of oxalic acid	$= N_1 =$
Volume of NaOH run down from the burette (x)	$= V_2 =$
Normality of NaOH	$= N_2 = (N_1 V_1) / V_2$

Standardization of HCl:

Volume of HCl taken in the conical flask	$= V_3 =$
Volume of NaOH run down from the burette	$= V_4 =$
Normality of HCl	$= (V_2 V_4) / V_3 =$

Volume of N/40 HCl used $= V_1$ ml
Volume of sample taken for titration purpose $= V_2$ ml

$N_1 V_1$ (HCL SIDE) $= N_2 V_2$ (REACTANT MIXTURE/SAMPLE SIDE)

Implies that N2 i.e strength of NaOH in reaction mixture is $= (N_1 . V_1) / V_2$ gm mole per liter

Concentration of NaOH at outlet:

Concentration of un-reacted NaOH in the reaction mixture, C_A

$C_A = (N_1 . V_1) / V_2$

Residence Time $= \tau$, min $= V_R/v$

$$X_A = [C_{AO} - C_A] / C_{AO}$$

Run No.	τ, min	C_A, gmol/L (conc NaOH)	X_A	$\dfrac{1}{C_{A0}} \dfrac{X_A}{(1 - X_A)}$

Plot τ vs. $\dfrac{1}{C_{A0}} \dfrac{X_A}{(1-X_A)}$ on a simple graph, the slope is $= 1/k$.

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Levenspiel,"Chemical Reaction Engineering," 2nd edition, John Wiley & Sons, Singapore, 1995.

Experiment No. 14a
RTD STUDIES CSTR

OBJECTIVE

Residence Time Distribution (RTD) Studies in CSTR

AIM

- To plot the RTD curve for a CSTR using a pulse tracer.
- To determine the dispersion number (D/ul) and Number of tanks for a CSTR using a pulse tracer.

INTRODUCTION

Real reactors do not satisfy the idealized flow patterns, back mix flow or plug flow deviation from ideality can be due to channeling of fluid through the vessel, recycling of fluid within the vessel or due to the presence of stagnant region or pockets of fluid in the vessel.

To predict the exact behavior of a vessel as a chemical reactor, RTD or stimulus response technique is used. The exit age distribution function of fluid leaving a vessel or RTD of a fluid in a vessel is called the E-CURVE.

$$\int_0^\infty Edt = 1 \tag{1}$$

i.e. area under E curve is unity.

Where,

E - Residence time distribution function and, $E = \dfrac{C(t)}{\int_0^\infty C(t)dt}$

$C(t)$ = concentration of tracer.

In stimulus-response experimentation the system is perturbed and then sees how the system reacts or responds to this stimulus. The analysis of the response gives the desired information. A pulse tracer input signal could be used as a stimulus.

The analysis of the response gives the desired information. A pulse tracer input signal could be used as the stimulus The concentration time curve for pulse signal at the vessel outlet is called the C-CURVE. Considering steady-state flow of fluid through a closed vessel:

$$C \quad = \quad E \tag{2}$$

Fig 6.1 CSTR

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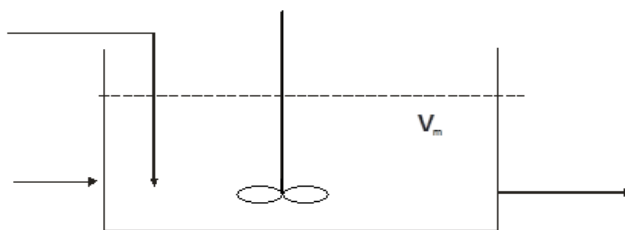
The mean age of the exit stream or mean residence-time is:

$$\tau = \tau_E = \tau_C = \int_0^\infty tEdt = \sum tE\,\Delta t \qquad (3)$$

And the variance of the E or C distribution is

$$\sigma_t{}^2 = \int_0^\infty t^2 Edt - \tau^2 = \sum t^2 E\Delta t - \tau^2$$

Defining the reduced time as:

$$\theta = t/\tau$$
$$E_\theta = \tau E$$
$$C_\theta = \tau C$$
$$\sigma_\theta^2 = \sigma_t^2/\tau^2$$

For an ideal flow in a CSTR the E-Curve should be as below.
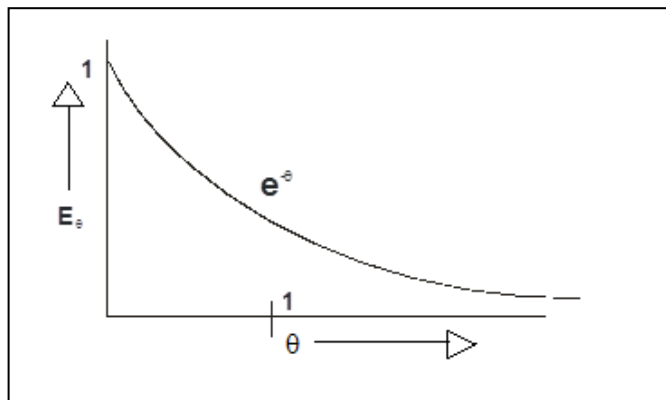
APPARATUS

The experimental set-up shown in fig. 1 consists of a stirred tank made of steel. The characteristics and dimensions of the vessel are:

STIRRED TANK

Height of the tank	= 200mm
Inside diameter	= 140mm
Volume of the tank (V_m)	=
Height of the liquid in the tank	=
Agitation	=
FLUID	=
FLUID flow rate	=

Fluid flows by gravity and a rotameter indicated

TRACER = H_2SO_4

EXPERIMENTAL PROCEDURE:
1. Prepare 5L of N/10 NaOH solution by dissolving 20 gm of NaOH in 5L of water.
2. Prepare 0.5 litre of 0.1 N standard oxalic acid solution by dissolving 3.15 gm of oxalic acid crystals in 500ml of water and standardize above preapared NaOH.
3. Select a volumetric feed rate of water. (Preferably < 1LPM)
4. Allow water to flow through the CSTR and attain the steady state (i.e. when liquid height inside the tank remains constant)
5. Input into the system and H_2SO_4 injection (10ml) (i.e. tracer: H_2SO_4, input signal: pulse)
6. At regular intervals of time (say1 min for high water rate e.g. 20-50 LPH, 2-5 min for low water rate e.g. 10-20 LPH). Collect the samples at the outlet in beakers/measuring jar (about 20ml) until all tracer leaves the vessel.
7. Analyze these samples with N/10 NaOH using phenolphthalein as indicator.
8. Thus obtain the concentration vs time data. (i.e. conc. of H_2SO_4 (mol/L) in the outlet stream).
9. Convent C vs t data to $E(\theta)$ data and E_θ vs θ. Plot E_θ vs θ and compare it with the ideal black mix curve ($E_\theta = e^{-\theta}$) for a CSTR.
10. Obtain the value of variance of the distribution:

$$\sigma_\theta^2 \quad = \quad \sum \theta^2 E_\theta \, \Delta\theta - 1$$

Dispersion number (D/uL) from:

$$\sigma_\theta^2 \quad = \quad 2(D/uL) - 2(D/uL)^2 \left(1 - e^{-uL/D}\right) \text{ ----} \rightarrow \text{ Dispersion model}$$

(Using Newton-Raphson method)
And the number of tanks in series (N) from

$$\sigma_\theta^2 \quad = \quad 1/N$$

11. Discuss the significance of D/uL & N.
12. Theoretical mean residence time in the tank

$$\tau_t = \frac{V_m}{Q} \quad = \quad \text{Volume / volumetric flow rate}$$

$$= \quad \text{L/LPM} = \quad \text{min.}$$

13. Calculate the experimental means residence time

$$\tau \quad = \quad \sum t \, E \, \Delta t$$

14. Compare τ_t and τ and discuss the significance of the deviation between the two.

SAMPLE CALCULATIONS-
Tracer	=	H_2SO_4		
Water flow rate	=	48.6 LPH	=	0.81 LPM

Volume of the tank   = V_m =   3.078L

$\therefore \tau_t$ =   (3.078/0.81)   =   3.8 min.

TRACE CONCENTRATION DATE:

For calculating the concentration of H_2SO_4 in the reactor, take 10 ml of solution in a titration flask. Add 2 – 3 drops of phenolphthalein indicator against NaOH (taken in a burette).

Let the volume of N/10 NaOH used   = V_1(mL)

Therefore

$$[V_1 (N/10)]_{NaOH} \quad = \quad (10 \, x \, N_2) H_2 SO_4$$

Normality of H_2SO_4 in solution = N_2 = V_1/100 = gm eq/L

Concentration of H_2SO_4 =   [(V_1/100) x (49.038/98.076)] moles/L

Concentration of H_2SO_4 at exit   =   5 x 10-3 V_1 (mole/L)

Time (min) T	Volume of N/10 NaOH used per 10ml of Soln., V_1, mL	Conc. of H_2SO_4 mole/L, C_i	$E_i = C_i / \sum C_i \, \Delta t$	$\theta = t/\tau$	$E_\theta = \tau E$
0	0	0	0	0	0
1	9.6	0.048	0.3038		0.815
2	8.8	0.044	0.2785		0.747
3	4.8	0.024	0.1519		0.4076
4	3.2	0.016	0.1013	1.490	0.2718
5	2.4	0.012	0.076	1.863	0.204
6	1.6	8 x 10^{-3}	0.0506	2.236	0.1358
7	0.8	4 x 10^{-3}	0.0253	2.608	0.0679
8	0.4	2 x 10^{-3}	0.01266	2.918	0.0339

Here $\Delta t = 1$ min.

Plot Concentration Ci Vs t:

$$\sum C_i \, \Delta t = \sum C_i = 0.158 \text{mol/L min.}$$

$$\tau \quad = \quad \sum t \, E \, \Delta t = \quad \sum t \, E \; = 1 \text{ x } 2.684 = 2.684 \text{ min}$$

$$\sigma_\theta^2 \quad = \quad \sum \theta^2 \, E_\theta \, \Delta \theta - 1 = 1.4123 - 1 = 0.4123$$

$$\sigma_\theta^2 \quad = \quad 2(D/uL) - 2(D/uL)^2 \left(1 - e^{-uL/D}\right)$$

for

σ_θ^2 =   0.4123, the dispersion number, D/uL =   0.286,

using Newton's Raphson Method and for tanks in series model:

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$$\sigma_{\theta}^{2} \quad = \quad 1/N$$

$$N \quad = 1/\sigma_{\theta}^{2} \quad = 1/0.4123 = 2.42$$

The high value of D/uL & N indicates that there is a large amount of dispersion from plug flow. (As is expected in the case of CSTR)

Theoretical mean residence time in tank,

$$\tau_{t} = \frac{V_m}{Q} = 3.078/0.81 = \qquad 3.8 \text{ min.}$$

Experimental mean residence time $\quad \tau = 2.684$ min.

Theoretical mean residence time is above the experimental mean residence time, indicating the existence of channeling or short-circuiting. (The fluid leaves the system before the time predicted theoretically.

Conc. of H₂SO₄ Vs Time
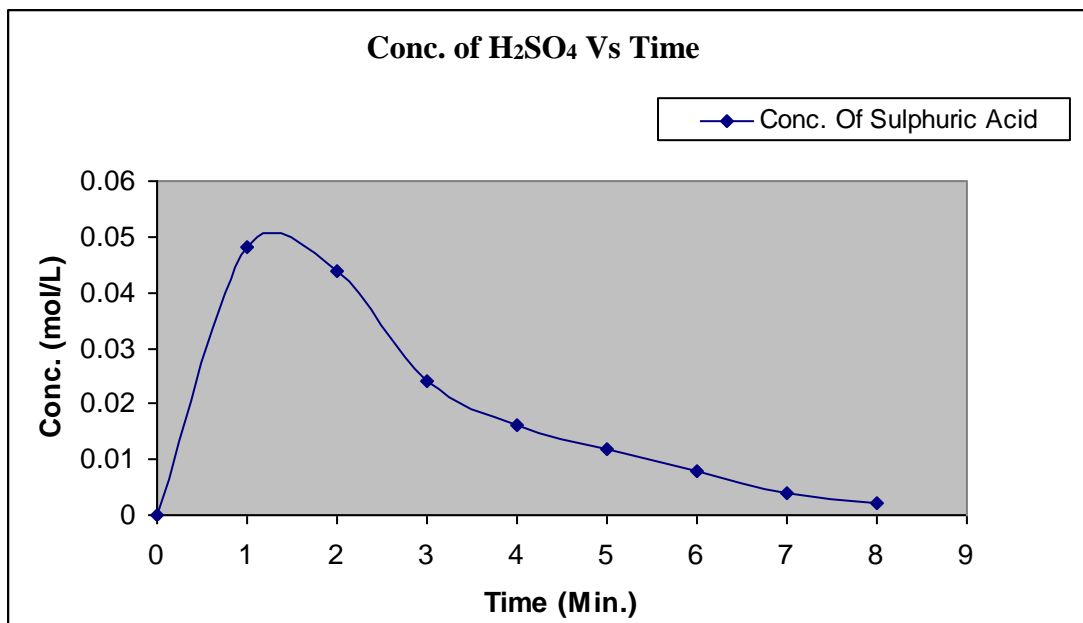
Experiment No. 14b
RTD STUDIES PFR

OBJECTIVE
RTD Studies in PFTR.

AIM
- To plot the RTD curve for a PFTR using a pulse tracer.
- To determine the dispersion number (D/uL)

INTRODUCTION
Real reactors do not satisfy the idealized flow patterns, back mix flow or plug flow deviation from ideality can be due to channeling of fluid through the vessel, recycling of fluid within the vessel or due to the presence of stagnant region or pockets of fluid in the vessel.
To predict the exact behavior of a vessel as a chemical reactor, RTD or stimulus response technique is used.

THEORY
The exit age distribution function of fluid leaving a vessel or RTD of a fluid in a vessel is called the E-CURVE.
The normalized curve is such that

$$\int_0^\infty E\,dt = 1 \tag{1}$$

In stimulus-response experimentation the system is perturbed and then sees how the system reacts or responds to this stimulus. The analysis of the response gives the desired information. A pulse tracer input signal could be used as a stimulus.
The concentration time curve for pulse signal at the vessel outlet is called the C-CURVE.
Considering steady-state flow of fluid through a closed vessel:

$$C = E \tag{2}$$

Where,

$$E(t) = \frac{C_i}{\sum C_i \Delta t}$$

The mean age of the exit stream or mean residence-time is:

$$\tau \;=\; \tau_E \;=\; \tau_C \;=\; \int_0^\infty tE\,dt \;=\; \sum tE\,\Delta t \qquad\qquad (3)$$

also,

$$\tau = \left[\frac{\sum t_i C_i}{\sum C_i}\right]$$

and the variance of the E or C distribution is

$$\sigma_t^2 = \int_0^\infty t^2 E\,dt - \tau^2 = \sum t^2 E\Delta t - \tau^2$$

Or $\quad \sigma_t^2 = \left[\frac{\sum t_i^2 C_i}{\sum C_i}\right] - \left[\frac{\sum t_i C_i}{\sum C_i}\right]^2$

DEFINITIONS: (Dimensions quantities)

$$\theta \;=\; t/\tau$$
$$E_\theta \;=\; \tau E$$
$$C_\theta \;=\; \tau C$$
$$\sigma_\theta^2 \;=\; \sigma_t^2/\tau^2$$

Thus σ_θ^2 can be evaluated from the experimental data on C Vs t and for known values of σ_θ^2 the dispersion number $(1/P_e)$ can be obtained from:

$$\frac{D}{uL} = \frac{1}{Pe}$$

For a closed vessel:
(Dispersion model)

$$\sigma_\theta^2 = 2\left[\frac{D}{uL}\right] - 2\left[\frac{D}{uL}\right]^2 [1 - e^{-\frac{uL}{D}}]$$

Dispersion no. = D / uL

If D/uL (the dispersion number) $\to 0$, it corresponds to negligible dispersion, hence Plug Flow.

If D/uL (the dispersion number) $\to\infty$, it corresponds to large, hence mixed flow.

D is the axial or longitudinal dispersion coefficient.

For open vessel condition:

$$C_\theta = \frac{1}{2\sqrt{\pi\theta\left(\dfrac{D}{uL}\right)}}\exp\left[-\frac{(1-\theta)^2}{4\theta\left(\dfrac{D}{uL}\right)}\right]$$

From the experimental data using a pulse of tracer, we obtain the concentration of the tracer (C_i) vs time (t) data.

From this data we obtain the following:

$$E(t) = \frac{C_i}{\sum C_i \Delta t}$$

Find $\sum t_i C_i =$ and $\sum t_i^2 C_i =$

Then calculate:

$$\tau = \left[\frac{\sum t_i C_i}{\sum C_i}\right]$$

And obtain the corresponding values of Eθ

Plot: C_i vs t_i ; E_i vs t_i ; and E_θ vs θ

$$\sigma_t^2 = \sum t^2 E \Delta t - \tau^2 = \left[\frac{\sum t_i^2 C_i}{\sum C_i}\right] - \left[\frac{\sum t_i C_i}{\sum C_i}\right]^2$$

$$\sigma_\theta = \frac{\sigma^2}{\tau^2}$$

Using this values of σ_θ^2 , calculate the dispersion Co-efficient, D/uL from Equation 5 by hit and trial method. First neglect the second term on RHS of the equation and obtain the approximate value of D/uL, then improve upon this value till you get LHS of equation 5 equal to RHS.

DESCRIPTION

The setup consists of one feed tank through which water is fed to the reactor. The flow rate can be adjusted by opening the needle valve and measured by rotameter. The compressed air is used for circulation of feed. Helical coil tube type plug flow reactor made of stainless steel pipe is provided. Reactants enter at lower end and exit at the top of coil from where samples are collected for analysis.

For understanding the RTD characteristics, a special arrangement to inject tracer into the lower end of reactor, using a syringe is provided. Pressure Regulator & Pressure Gauge are fitted at the compressed air line.

UTILITIES REQUIRED
1. Compressed air supply at 2 Bar, 0.25 CFM
2. Water Supply
3. Drain
4. Laboratory Glassware

CHEMICAL REQUIRED:
1. 1N NaOH
2. N/20 HCl
3. Phenolphthalein indicator

EXPERIMENTAL PROCEDURE:
1. Fill the feed tank with water and connect compressed air line to the apparatus.
2. Place the syringe containing known amount of 1N NaOH at the Tracer inlet.
3. Start the supply of water to the reactor at particular flow rate with the help of Rotameter.

4. Allow water to flow through the PFTR and attain the steady state.
5. inject 1N NaOH into the system as a pulse signal from the tracer inlet.
6. At regular time intervals (say 1 minute), collect the samples at the outlet in pre-marked beakers/measuring cylinders (about 20 ml) until all tracer leaves the vessel.
7. Analyze these samples with N/20 HCL using phenolphthalein as indicator.
8. Repeat the experiment for different flow rates.

SPECIFICATION

Reactor	:	Material stainless steel, capacity 0.7 Ltrs. (Approx) (helical coiled tube type)
Feed tank	:	Material stainless steel, capacity – 20 Ltrs.
Feed Circulation	:	By compressed air
Flow measurement	:	Rotameter
Piping	:	Material Stainless Steel and PVC
Pressure Regulator	:	0-2 Kg/cm^2
Pressure gauge	:	Bourdon type 0-2 Kg/cm^2
Stop watch	:	Electronic

The whole unit is assembled rigidly on a base plate and mounted on a stand.
Most of the parts are powder coated and rests are painted with auto paints.

FORMULAE

1. Concentration of NaOH in sample,

$$C_i = \frac{V_1 N_1}{V_2} \times \frac{40}{1000} \, gm/ml$$

2. Experimental mean residence time,

$$\tau = \frac{\sum t_i C_i}{\sum C_i} \, , s$$

3. Theoretical mean residence time,

$$\tau_t = \frac{V_R}{V_O} \, , s$$

4. $\sigma_\theta^2 = 2\left[\frac{D}{uL}\right] - 2\left[\frac{D}{uL}\right]^2 [1 - e^{-\frac{uL}{D}}]$

OBSERVATION & CALCULATION:

For calculating the concentration of NaOH in the exit stream, take 10 ml of solution in a titration flask. Add 2-3 drops of phenolphthalein and titrate against N/20 HCL (taken in a burette). The end point is light pink.

Let the volume of N/20 HCl used   = V_1

Normality of N/20 HCl used   = N_1

Volume of sample taken   = V_2

            V_1N_1 = V_2N_2

            $[V_1 (N/20)]_{HCl}$ = $(10 \times N_2)_{NaOH}$

$\therefore$ Normality of NaOH in the solution, N2 = $V_1/200$ gram eq/L

$\therefore$ Concentration of NaOH   = $[(V_1/200) \times 40.0]$ gram eq/L

Concentration of NaOH at exit   = $0.2\ V_1$ gm eq/L

Time Min	Volume of N/20 HCl used per 10 ml of Soln.	Conc. Of NaOH gm/L, C_i	$t_i \times C_i$	$t_i^2 \times C_i$
		$\sum C_i =$	$\sum t_i \times C_i =$	$\sum t_i^2 \times C_i =$

$$\sum C_i \ =$$
$$\sum t_i C_i \ =$$
$$\sum t_i^2 C_i \ =$$
$$\tau \ = \ [\sum t_i^2 C_i \times \sum C_i]$$
$$\sigma_t^2 \ = \ [\sum t_i^2 C_i \times \sum C_i] - \tau^2$$
$$\sigma_\theta^2 \ = \ \sigma_t^2 / \tau^2$$

using the dispersion model:

$$\sigma_\theta^2 = \ 2\left[\frac{D}{uL}\right] - 2\left[\frac{D}{uL}\right]^2 [1 - e^{-\frac{uL}{D}}]$$

For an initial trial method, neglect the second term on rhs

Therefore, $2(D/uL)$ = σ_θ^2

By hit and and trial method (improve upon the previous value exact value of D/uL) is:

The dispersion number D/uL =

Theoretical mean residence time is

      $\tau_t = V_R / V_O$, (L/LPM) $\rightarrow$ min

Calculate the experimental means residence time is

      $\tau = \sum t\ E\ \Delta t = \tau = [[\sum t_i C_i \times \sum C_i]$

Compare τ_t and τ and discuss your results

To plot RTD curve (E curve)

Time Min	C_i , gm/L	$E_i = \sum C_i \Delta t$	$\theta = t/\tau$	$E_\theta = \tau E_i$

Plot E curve, θ vs E_θ

NOMENCLATURE

θ	=	reduced time
E	=	RTD function
τ	=	Experimental mean residence time
τ_t	=	theoretical mean residence time
σ_θ	=	variance at time, θ
t	=	time
V_1	=	volume of N/10 NaOH
V_R	=	Volume of reactor
V_O	=	Volumetric flow rate
D/uL	=	dispersion number

PRECAUTIONS & MAINTENANCE INSTRUCTIONS

1. Always use clean water, good quality chemicals and standard solution for titration.
2. Keep close all the drain valves. Vent valve should open while filling the water in feed tank.
3. Air pressure should not be more than 1 kg/cm^2.
4. Flow should not be disturbed during the experiments.
5. Handle the chemicals carefully.
6. If any types of suspended particles are in the rotameter, stop the flow, drain the water tank and reactor. Fill the tank with clean water after proper cleaning of feed tank and the reactor.

REFERENCE:

1. Levenspil, Octave: "Chemical Reaction Engineering", John Wiley.

2. Fogler H. Scoot (2008). *Elements of Chemical Reaction Engineering*. 4th Ed. ND: Prentice-Hall of India Pvt. Ltd.

Experiment No. 14c
RTD STUDIES PACKED BED REACTOR

OBJECTIVE

Residence Time Distribution (RTD) Studies in Packed Bed Reactor (PBR).

AIM

- To plot the RTD curve for a PBR using a pulse tracer.
- To determine the dispersion number (D/uL).

INTRODUCTION

Axial diffusion and dispersion of fluid in packed beds are important for design and operation of separation equipment and chemical reactors. The tracer technique, the most widely used method for the study of axial dispersion, is usually used in the form of:

1.   Pulse input.
2.   Step input.
3.   Cyclic input.
4.   Random input.

In stimulus-response experimentation, we perturb the system using either a pulse input of tracer or a step input and then see how the system reacts or responds to this stimulus. The analysis of the response gives the desired information about the system. The concentration time curve at the vessel outlet is called the F-Curve when the input signal is a step signal and is called C-curve when the input signal is a pulse signal.

THEORY

The exit age distribution function of fluid leaving a vessel or RTD of fluid in a vessel is called the E-CURVE. The normalized curve is such that

$$\int_0^\infty E \, dt = 1 \qquad \text{------- (1)}$$

i.e. area under E curve is unity. Where, E - Residence time distribution function and,

$$E = \frac{C}{\int_0^\infty C \, dT}$$

where, C(t)= concentration of tracer.

In stimulus-response experimentation the system is perturbed and then observes how the system reacts or responds to this stimulus. The analysis of the response gives the desired information. A pulse tracer input signal could be used as a stimulus. The concentration - time curve for pulse signal at the vessel outlet is called the C-CURVE. Considering steady-state flow of fluid through a vessel:

$C = E$ (2)

The mean age of the exit stream or mean residence time is:

$$\tau = \tau_E = \tau_C = \int_0^\infty tE\,dt = \sum tE\,\Delta t \qquad (3)$$

$$\tau = \frac{\sum t_i C_i}{\sum C_i} \qquad (4)$$

When a tracer is injected into a packed bed at a location more than two or three particle diameters downstream from the entrance and measured some distance upstream from the exit, the system is analogous to an open-open system. For such a system where there is no discontinuity in type of flow at the point of tracer injection or at the point of tracer measurement, the variance for open system is

$$\sigma_t{}^2 = \int_0^\infty t^2 E\,dt - \tau^2 = \sum t^2 E\Delta t - \tau^2 \qquad (5)$$

$$\sigma_t{}^2 = \frac{\sum(t_i^2 C_i)}{\sum C_i} - \left[\frac{\sum(t_i C_i)}{\sum C_i}\right]^2 \qquad (6)$$

Variance or a measure of the spread of the curve at time Θ.

$$\sigma_\theta{}^2 = \frac{\sigma_t{}^2}{\tau^2} \qquad (7)$$

Models are useful for representing flow in real vessels, for scale up, and for diagnosing poor flow. We have different kind of models depending on whether flow is close to plug, mixed, or somewhere in between. For small deviations from plug flow dispersion model is used. Suppose an ideal pulse of tracer is introduced into the fluid entering a vessel. The pulse spreads as it passes through the vessel, and to characterize the spreading this model, we assume diffusion like process superimposed on plug flow. we call this dispersion, the dispersion coefficient D represents the spreading process. $\left(\frac{D}{uL}\right)$ is the dimensionless group characterizing the spread in the whole vessel.

For open vessel:

$$\sigma_\theta^2 = 2(D/uL) - 8(D/uL)^2 \qquad (8)$$

Solving the above equation we get.

$$\frac{D}{uL} = \frac{-1+\sqrt{1+8\sigma_\theta{}^2}}{8} \qquad (9)$$

Defining the reduced time as:

$$\theta = \frac{t}{\tau} \qquad (10)$$

Exit age distribution at time i

$$E = \frac{C_i}{\sum C_i \Delta t} \qquad (11)$$

Exit age distribution at time θ

$$E_\Theta = \tau \times E_i \qquad (12)$$

Plot a graph between Θ vsE_{Θ}.

DESCRIPTION

The setup consists of a glass column packed with rasching rings and one feed tank. Water is fed to the reactor through liquid distributor, fitted at the bottom of the column. Rotameter is provided to measure the flow of water. The flow rate can be adjusted by operating the needle valve provided on rotameter. A special arrangement is provided to inject tracer at the lower end of reactor, using a syringe. Samples can be taken periodically from the top outlet of reactor. Pressure regulator & pressure gauge are fitted in the compressed air line.

UTILITIES REQUIRED

1. Electricity supply: Single phase, 220 V AC, 50 Hz, 5-15 Amp combined socket with earth connection.
2. Compressed air supply @ 0.25 CFM at 1 Bar.
3. Water supply (Initial fill)
4. Floor drain required.
5. Laboratory glassware required:-

Burette (50 ml)	: 01 No.
Conical flask (250 ml)	: 01 No.
Syringe (20 ml)	: 01 No.
Measuring cylinder (250 ml)	: 01 No.

6   Chemicals:-

N/10 NaOH	: 200 ml
Concentrated H2SO4	: 100 ml
Phenolphthalein indicator	: Few drops

EXPERIMENTAL PROCEDURE

STARTING PROCEDURE

1. Close all the valves V1-V6.
2. Open the valve V3-V4 and fill the feed tank with water.
3. Connect compressed air supply to the set up at valve V1.
4. Connect electric supply to the set up.
5. Open valve V1 and set air pressure 0.5 to 1 kg/cm^2 by pressure regulator and pressure gauge.
6. Start the supply of water to the reactor at particular flow rate with the help of valve V2.
7. Fill N/10 sodium hydroxide in burette.
8. Fill the concentrated H_2SO_4 (10 ml or 20 ml) in the syringe.
9. Place the syringe at the tracer inlet.
10. Wait till the water comes out from the outlet.
11. Push the syringe into the system.

12. At regular time intervals (say 30 sec for high water rate, 1 min for low water rate), collect 20 ml sample from the outlet, in measuring cylinder.
13. Transfer the sample solution in conical flask.
14. Titrate the sample solution, using phenolphthalein as an indicator against N/10 sodium hydroxide (add NaOH from burette).
15. Repeat the experiment for different flow rates (before changing the flow rate, drain the reactor first).

OBSERVATION & CALCULATION:

DATA:	
Working volume of reactor V_R	1.3 Lit
Volume of sample V_2	20 ml
Normality of NaOH used for titration N1	0.1 g eq/L

OBSERVATION TABLE:			
S.No.	**t_i (min)**	**V_1 (ml)**	**Vo (LPH)**

CALCULATIONS

$$N_2 = \frac{V_1 N_1}{V_2} (g\ eq/L)$$

$$C_1 = \frac{N_2}{2} (mole/L) \qquad \& \qquad \tau_t = \frac{V_R * 60}{V_0} (\text{min})$$

$$\Delta t = t_i - t_{i-1} (\text{min})$$

S. No.	t_i(min)	C_i(mol/L)	$t_i C_i$	$t_i^2 C_i$	**Δt(min)**	$C_i \Delta t$
		Σ C_i	**Σ$t_i C_i$**	**Σ $t_i^2 C_i$**		

$$\tau = \frac{\Sigma t_i C_i}{\Sigma C_i} \, (\text{min})$$

$$\sigma_t^2 = \left(\frac{\Sigma t_i^2 C_i}{\Sigma C_i} \right) - \tau^2$$

$$\sigma_\theta^2 = \frac{\sigma_t^2}{\tau^2}$$

$$\frac{D}{uL} = \frac{-1 + \sqrt{1 + 8\sigma_\theta^2}}{8}$$

$$E_i = \frac{C_i}{\Sigma C_i \Delta t}$$

$$\theta = \frac{t}{\tau} \qquad \& \qquad E_\theta = \tau * E_i$$

θ	E_i	E_θ

Plot a graph between E_θ vs. θ

Block Diagram

BLOCK DIAGRAM

NAME _ RTD OF PACKED BED REACTOR

CODE CRE-116
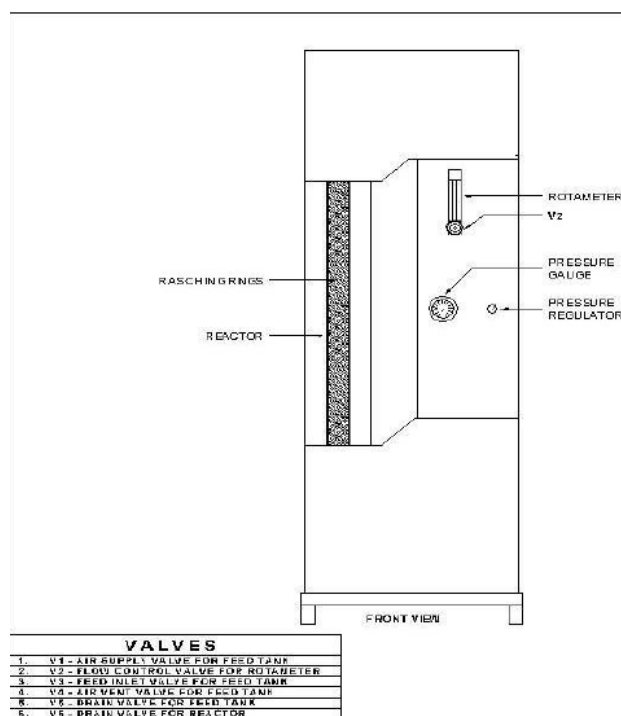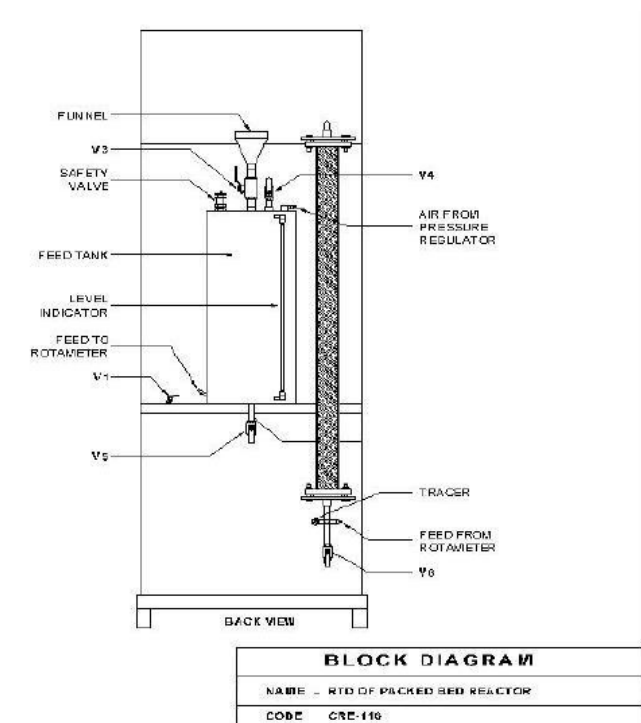
NOMENCLATURE

Nom	Column Heading	Units	Type
C_i	Concentration of H_2SO_4 insample	mole/L	Calculated
D/uL	Dispersion number	*	Calculated
E_i	Exit age distribution at time i	*	Calculated
E_Θ	Exit age distribution at time θ	*	Calculated
N_1	Normality of NaOH used for titration	g eq/L	Given
N_2	Normality of H_2SO_4 in sample solution	g eq/L	Calculated
t	Time	min	Measured

90

V_1	Volume of NaOH used for tritation	ml	Measured
V_2	Volume of sample	ml	Given
Vo	Volumetric flow rate	LPH	Measured
V_R	Working volume of reactor	Lit	Given
τ	Experimental mean residence time	min	Calculated
τ_t	Theoretical mean residence time	min	Calculated
Θ	Reduced time	*	Calculated
σ_Θ	Variance at time Θ	*	Calculated
σ_t	Variance at time t	min^2	Calculated
Δt	Average time difference	min	Calculated

* Symbols are unitless

PRECAUTION & MAINTENANCE INSTRUCTIONS

1. Always use distilled water, good quality chemicals and standard solution for titration.
2. Keep close all the drain valves V5-V6 and vent valve V4 should be open while filling water in the feed tanks.
3. Air pressure must be set below 1 kg/ cm^2.
4. Flow should not be disturbed during the experiments.
5. Handle the chemicals carefully.

TROUBLE SHOOTING

1. If any type of suspended particles are come in the rotameter, remove the rotameter clean the tube and fit it at its place.
2. If there is any leakage tight that part or fix that again after wrapping teflon tape.
3. If rotameter fluctuating more than average tight the control knob properly.

REFERENCES

1. Levenspiel, Octave (2001). *Chemical Reaction Engineering*. 3rd Ed. NY: John Wiley & Sons. pp 293-294, 299-301, 305.

2. Fogler H. Scoot (2008). *Elements of Chemical Reaction Engineering*. 4th Ed. ND: Prentice-Hall of India Pvt. Ltd. p 869.

Experiment No. 15

LEVEL CONTROL TRAINER

1. Aim
Study of Level Control Trainer

2. Objective(s)
   i.   To study the effect of controller parameters in a single loop feedback control system with a liquid level setup.
   ii. To control the level in the process tank using high/low level switch.
   iii. To control the level in the process tank using time proportioned relay output to a solenoid valve controlling outflow from the tank.

3. Theory
The output of a PID controller is given by

$$c(t) = K_c \varepsilon(t) + \frac{K_c}{\tau_I} \int_0^t \varepsilon(t)dt + K_c \tau_D \frac{d\varepsilon}{dt} + c_s \tag{1}$$

Where ε = error (i.e., set point - measured value of the output), c_s = controller's bias signal (i.e., its actuating signal when error, $\varepsilon = 0$), K_c = proportional gain of the controller (Proportional band, PB = $100/K_c$), τ_I = integral time constant (minutes) and τ_D = derivative time constant (seconds).

4. Experimental Set-up
The experimental setup consists of Electrical Console (PCT 10), Process Module (PCT 9), Level Control Accessory (PCT 11) and Connectors.

4.1. Electrical Console (PCT 10)
Any control accessory (Temperature, Level, Pressure, etc.) must be operated in conjunction with Electrical Console PCT10. The appropriate electrical supplies are provided by PCT10. The Electrical Console is shown in Figure 1.The main parts of Electrical Console are
1. Process Controller
2. Signal Conditioning Circuit
3. Motor Positioner
4. Voltmeter and Ammeter

4.2. Process Controller

The controlling parameters and set point value can be set by process-controller. The controller is a typical industrial model, which utilizes a microprocessor and allows re-configuration from the front panel controls. This means that all normal operating or commissioning settings can be adjusted without removing the controller from the console.

4.3. Signal Conditioning Circuit
The conditioning circuitry is contained in a plastic case, which is electrically connected to the PCT10 via 8 pin connector at the rear. The signal to be conditioned is sent to the module via the 2 pin connector on the front of the module using the connecting lead supplied.

Figure 1: Side and front panels of the Electrical Console PCT 10.

4.4. Motor Positioner
In Motor Positioner the signal (4-20mA from controller) is converted in corresponding analog signal and it is then sent to final control element to adjust the manipulated variable.

4.5. Voltmeter and Ammeter
These are used to measure the voltage and current reading manually at any point in the controlling procedure. The voltmeter reading when multiplied by 100 gives temperature in degree centigrade. Ammeter directly gives the current in mA.

4.6. Process Modules (PCT 9 and PCT 11)

Process module PCT 9 and level control accessory PCT 11 are shown in Figure 1. Process module PCT 9 consists of a sump tank at the backside and a pump, which pumps the water in the bottom tank to the top tank PCT 11 through a manual control valve, a rotameter, a solenoid valve and finally a motorized valve. The motorized valve is used as the final control element. There are three drain valves (one manual and two solenoid valves) and one overflow line. There is a linear

millimeter scale attached to the top tank to directly read the liquid level. The level in the tank is measured using a float time sensor, which measures the liquid level from 40 mm to 140 mm.

Figure 1: Process Module PCT 9 and Level Control Accessory PCT 11.

The schematic diagram of the experimental setup is shown in the Figure below:
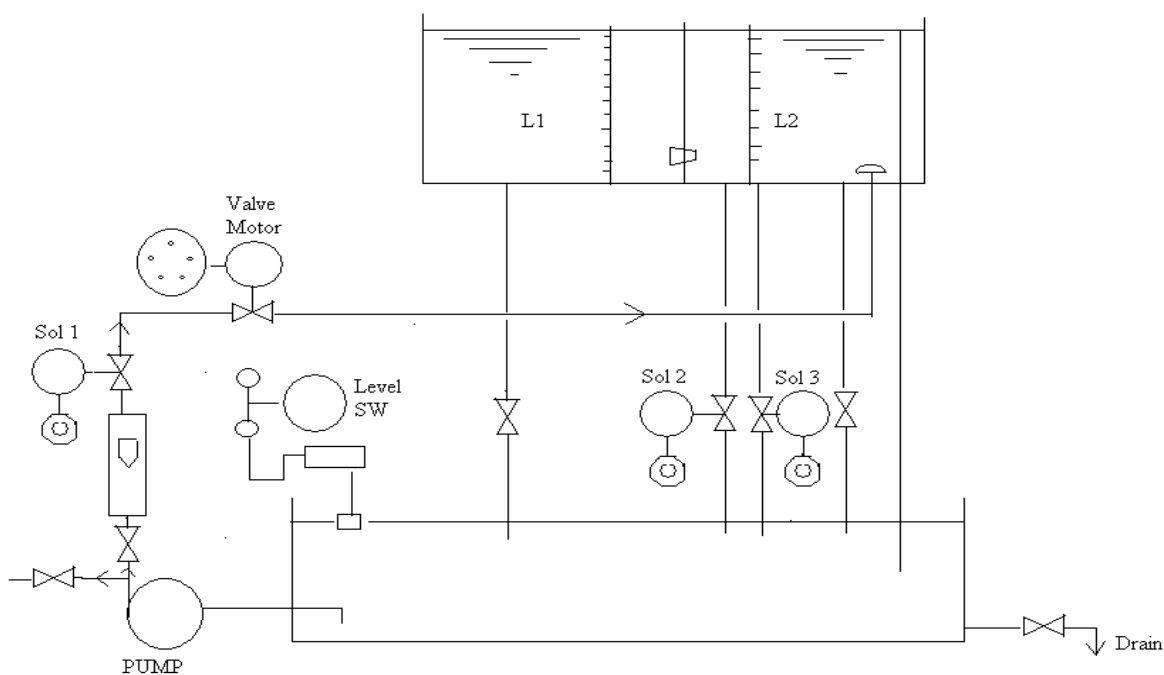
5. Experimental Procedure

1. Fill the sump tank of the process module with distilled water.
2. Switch on the electrical console and connect the heater plug of the process module to 240 V AC supply in the Switched Output of the electrical console.
3. Insert the signal-conditioning block marked as "LEVEL" to either of the signal conditioning channels.
4. Insert the input from the potentiometric level sensor to three ports of the input in the signal conditioning channel. Here 0% in the process controller corresponds to 40 mm and 100% to 140 mm in the tank level.
5. Connect 0-1V output from the signal-conditioning block to the voltmeter. The voltmeter reading multiplied by 100 plus 0% reading (40 mm) gives the level of the tank.
6. Start the water flow through the tank by connecting the power plug of the pump to 240 V AC power supply in the side panel of the electrical console.
7. Open the motorized valve to its maximum opening position. This can be done by connecting a 4-20mA manual output from the electrical console to input of the motor positioner and the output from the motor positioner to the input of the motorized valve using a 5-pin plug and rotating the manual knob to its maximum position. Alternatively the level in the tank can be controlled by motorized valve at the inlet and at the outlet of the tank flow.
8. Wait till the level in the tank reaches to a steady state.
9. Connect 4-20mA output of the signal conditioner to 4-20mA input of the process controller in electrical console (PCT 10).
10. Change the connectors from 4-20mA manual output to 4-20mA output of the process controller. The other ends of the connectors will remain connected to 4-20mA input of the motor positioner. Also the output from the motor positioner will remain connected to the motorized valve.
11. Keep the initial setpoint at 50% and increase to 70% while giving the step change to study the effect of the PID parameters.
12. In the microprocessor based controller set Prop = 4%, Int = 0.1 min and dEr = 5 sec. Please refer to Reference 3 for detailed operation of the process controller.
13. Wait till steady state is reached in the liquid level.
14. Change the set point to 80% and note down the liquid level at each second interval for about 5 minutes.
15. See the effect of proportional band by changing Prop to 2% and keeping other parameter constant and giving the same step change in the setpoint.
16. See the effect of integral time by changing Int to 0.05 min and keeping other parameters constant.
17. See the effect of derivative time by changing dEr to 0.5 second and keeping other parameters same as before.

6. Observations

Steady state reading

Set point (%)   =
PB   =
Int   =
dEr   =

S No	Water level (mm)	Inlet flow rate

After step change
New set point (%)   =
Step change   = New set point – set point in (I)

S No	Time (s)	Water level (mm)
1	5	
2	10	
.	.	
.	.	
.	.	
Steady state		

7. Model Calculations

Plot the response curves using graphics and study the effects of different parameters.

8. Results and Discussion

Set what is obtained and what you understood from this study.

9. Conclusions

State the conclusions relating to the outcome after accomplishing the aim and the extent to which the objectives were fulfilled.

10. Precautions

1. Always follow the colour codes while using the connectors.

2. Don't connect mA output to voltage input or vice versa.

References

1. Stephanopoulos, G., Chemical Process Control – An Introduction to Theory and Practice, 6th edition, Prentice-Hall of India, New Delhi, India, 1993.
2. Instruction Manual for Electrical Console PCT 10, Feedback Instruments Ltd., Park Road, Crowborough, UK.
3. Instruction Manual for Process Module PCT 9, Feedback Instruments Ltd., Park Road, Crowborough, UK.
4. Instruction Manual for Level Control Accessory PCT 11, Feedback Instruments Ltd., Park Road, Crowborough, UK.

(ii) Level control using High/Low Level Switch

Objective:

To control the level in the process tank using a high/low level switch with a normally closed solenoid valve (SOL3) controlling outflow from the process tank.

Equipment Required:

PTC9	Process Module
PTC10	Electrical Console
PTC11	Level Control Accessory: High/Low level switch

Initial Settings (Refer to equipment set-up diagram):

Manual control valves:

V1, V3, V4, V5	Closed
V2	Open

Adjust V2 TO GIVE 1200 cm^3/min on F1 with the motorized valve open.

The process controller is not required for this experiment.

Experimental Details:

To maintain a steady level in the process tank, the flow from the tank must be interrupted when the water falls below the required level. The object of the demonstrations is to maintain the level at the mid-range in the process tank (80 mm on L1).

Procedure:

- Connect the equipment as shown in the set-up diagram with the high/low level switch mounted on the front face of the process tank. Adjust the height of the left- hand electrode (connected to 'C' on the switched output of PTC10) so that the tip is approximately at 90 mm height in the tank. Adjust the right- hand electrode (connection 'B') so that the tip is approximately 20 mm below the left- hand electrode using the scale on the switch assembly.
- If correctly adjusted, the level should rise to the upper electrode before the solenoid valve (SOL3) opens, then fall to the lower electrode before the valve closes again.
- Observe that the level oscillates between the upper and lower limit with the solenoid/ relay opening and closing as required.
- Effect of change in rate of inflow to the process tank.
- Adjust valve V2 to give a reading of 800 cm^3/min on F1. Observe the change in characteristics. Namely, the level slower than before then falls at the same rate as before.
- Adjust valve V2 to give a reading of 400 cm^3/min on F1.
- Observe the change in characteristics. Namely, the level rises even more slowly than before then falls at the same rate as before.
- Adjust valve V2 to give a reading of 3000 cm3/min on F1.
- Observe the change in characteristics. Namely, that the level cannot be maintained in the process tank (the tank fills faster than it can drain).

PCT 10

Banana plugs

Height clamp

Mounting clamp

Scale
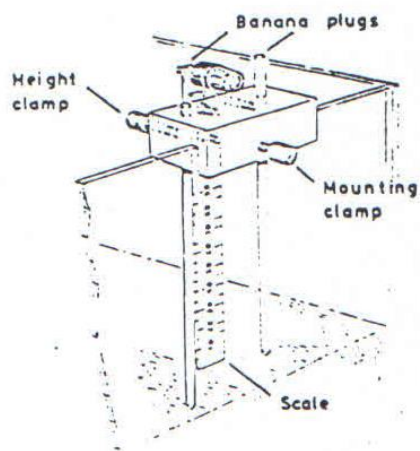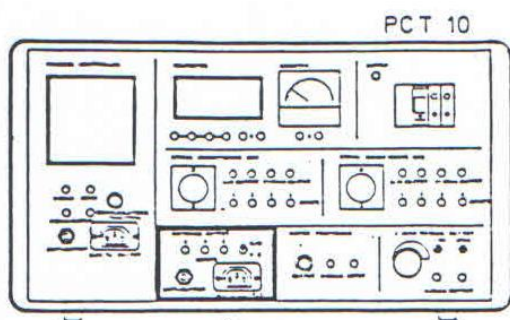
DETAILS OF HIGH LOW LEVEL SWITCH
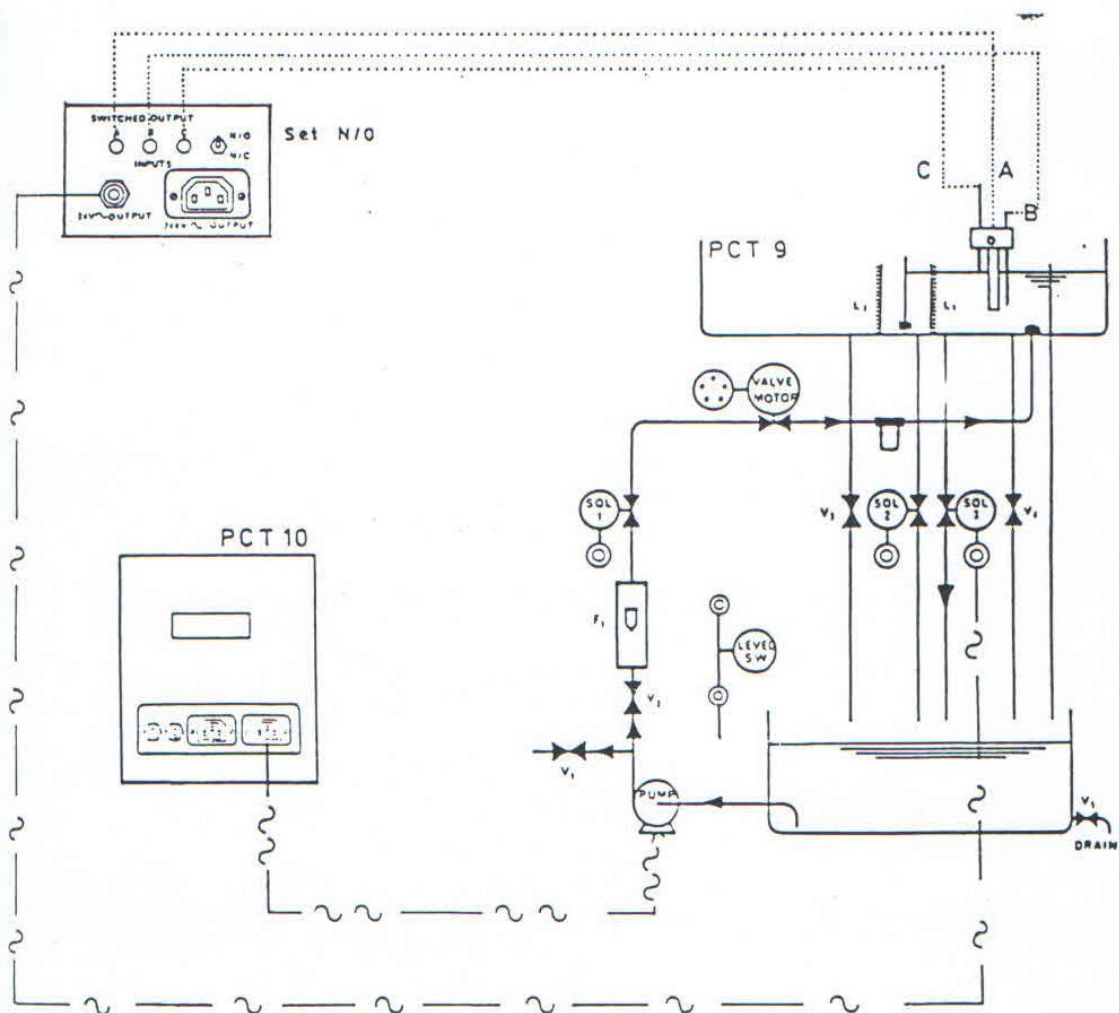
Further experiment:

Effect of hysteresis (deadband) in the switch:

- Repeat the above procedure for different settings of the electrodes on the switch i.e. different heights between upper and lower electrodes. Observe the effect on the level in the tank and the operation of the solenoid valve.
- Effect of an additional outlet on the process tank:
- Open solenoid valve SOL2 by connecting it to the 24V A.C supply. Observe the effect of the additional outlet on the level in the process tank. Open drain valve V4 fully. Observe that the level cannot be maintained.

Conclusions:

- Level in the process tank can be controlled using a switch in the tank to open and close a solenoid valve at the outlet from the tank.
- Control of outflow from the tank ensures that the tank cannot empty.
- Control of outflow from the tank cannot prevent the tank from overfilling. When inflow exceeds outflow, the tank will overfill.
- The time taken for the level to rise between the lower and upper electrodes is dependent on the inflow.
- The time taken for the level to fall between the upper and lower electrodes is dependent on the difference between the outflow and the inflow.
- The cycling of the relay/ solenoid valve is dependent on the hysteresis of the level switch, the inflow to the process tank and the outflow from the tank.
- Addition of outlets in the tank, other than the controlled outlet, will change the time for the level to rise and fall between the set limits. The level cannot be maintained if the outflow through the additional outlet exceeds the available inflow to the tank.

(iii) Level control using Solenoid valve in the outlet of the tank

Objective:

To control the level in the process tank using a level sensor and process controller with time proportioned relay output to a normally closed solenoid valve (SOL3) controlling outflow from the process tank.

Equipment Required:

PTC9	Process Module
PTC10	Electrical Console
PTC11	Level Control Accessory: Level sensor, Level conditioning module

Optional Equipment (if available):

PCT 10/10	Two Channel Laboratory recorder or
PCT 10/11	Two channel process recorder

Initial Settings (Refer to equipment set-up diagram)

Manual control valves:

V1, V3, V4, V5	Closed
V2	Open

Adjust V2 to give 1000cm3/min on F1 with the motorized valve open.

Process Controller:

Full details on how to set and operate the controller are given in the Operating instructions.

For this set of demonstrations, the controller should be configured for three- term control with the following initial settings:-

Controller Settings	Code	Setting	Units
Set point		50	%
Power output	Pr		
Proportional band	Prop	5	%
Integral time	Int	0.2	mins
Derivative time	Der	0	secs.
Cycle time	CY-t	5	secs.
Not used	CL-G		
Not used	HC-O		
Not used	UP-t		
Hysteresis	HYSt		%
Power limit	Pr-L	100	%
Set point limit	Sp-L	100	%
Range	CS-1	-058	
Direct action	CS-2	-d--	
Not used	CS-3-7	----	
Calibration:			
Span	SPAn	100% at 20 m A input	
Zero	ZERo	0% at 4 m A input	

Sensor calibration/ setting:

30 mm level on L1 = 4Ma and 0.000 V from level conditioner = 0% of range

130 mm level on L1 = 20Ma and 1.000 V from level conditioner = 100% of range

Experimental Details:

To maintain a steady level in the process tank, the flow from the tank must match the outflow. In time proportioning, the output relay in the controller is either on or off but the ratio of the on and off periods is continuously variable. Over a period of time, the effect is a proportioning of the flow through the solenoid valve.

The object of the demonstrations is to maintain the level at the mid-range in the process tank (50% set point on the process controller corresponds to 80 mm on L1 in the tank), by opening and closing the outlet solenoid valve (SOL3) to allow the required amount of water to leave the tank.

As SOL3 is normally closed and low level in the process tank (process variable below set point) requires the valves to be closed, the controller is configured for DIRECT action.

Procedure:
- Connect the equipment as shown in the set-up diagram with the level sensor mounted on the front of the process tank. A recorder may be connected to the level conditioner output/ controller input 4-20 m A loop to provide a record of the response.
- If the setup is correct, water should be delivered to the process tank and drain back to the sump tank via solenoid SOL3 (when required). As the inflow is less than the outflow, the process tank will fill and SOL3 will open and close repeatedly on a 5 second cycle, the open and closed periods varying to achieve the outflow required.
- Observe that the level is controlled at the set point.
- Adjust V2 to give an inflow of 600 cm^3/min on F1. Observe that the level is maintained at the set point, that the on and off cycle is still 5 seconds but the open and closed periods have changed to suit the conditions.

Effect of changing the Cycle Time:
- Set the cycle time 'CY-t' on the controller to 30 seconds. The outflow from the tank will be proportioned as before but the level will oscillate about the set point because of the long cycle time relative to the response of the process.
- Set 'CY-t' to 1 second. The level will be controlled accurately at the set point but the relay and solenoid valve will chatter because of the short cycle time.
- Adjust the set point to 70% on the process controller (100 mm corresponding level on L1). Observe how the level changes to the required setting and the open and closed periods of the solenoid valve vary to suit the conditions.
- Adjust the set point to 40% on the process controller (70 mm on L1). Once again observe the changes in the level and the valve operation.
- Controlling inflow to the process tank:
- Disconnect SOL3 from the relay output on the controller and connect SOL1 in its place.
- To maintain a steady level in the process tank, the flow into the tank must match the flow out of the tank. As SOL1 is normally open and low level requires the valve to be open, the controller will remain configured for DIRECT action.
- Repeat the above experiment.
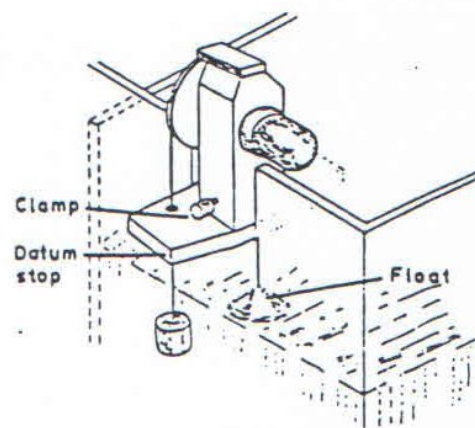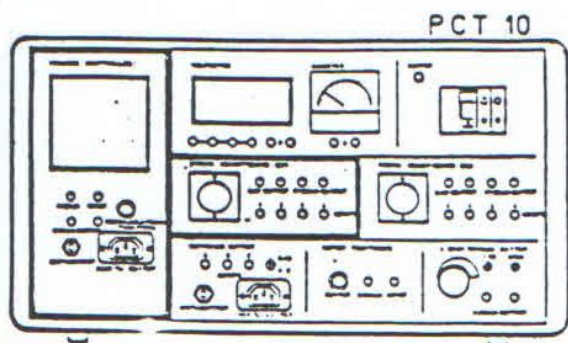
DETAILS OF LEVEL SENSOR
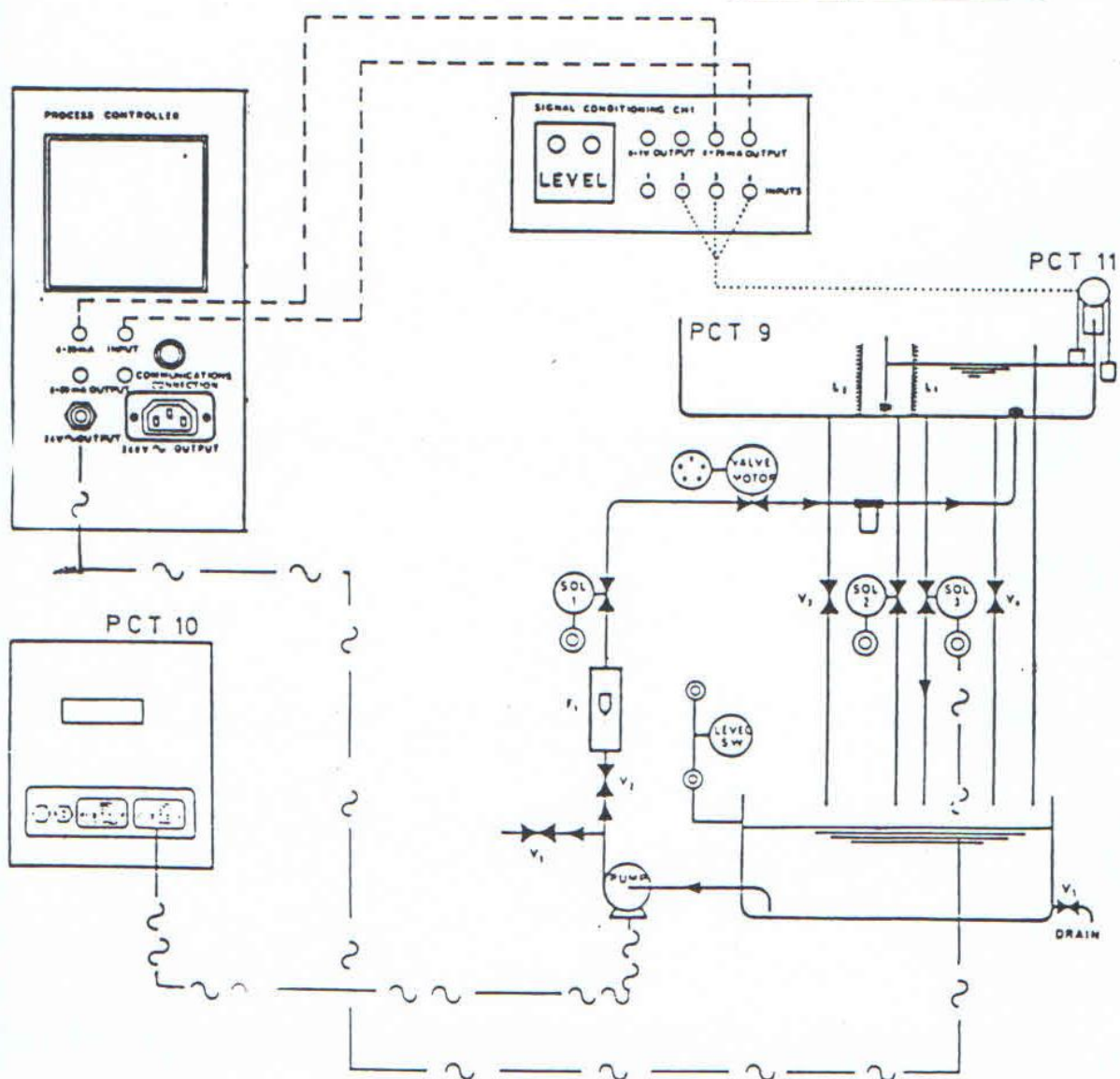
Conclusions:

- Level in the process tank can be controlled using a level sensor and process controller configured for time proportioned relay output, to open and close a solenoid valve at the outlet from the tank. Alternately, a solenoid valve at the outlet can be controlled.

- The level in the tank may be changed by setting an alternative set point on the controller. No adjustment to the process or sensor is required provided the inflow and outflow rates are compatible with the new setting.

- Cycle time is set on the process controller and must be adjusted to suit the process. The maximum variation in level which is tolerable should be used as the basis for this setting (minimum operation of relay and solenoid valve).

- Note: This technique is mainly used where the process has a slow response (e.g. heating processes) and cyclic changes in the input will not affect the process variable.

- Time proportioning using a simple on/off control element allows three-term control of a process without the need for expensive proportioning valves or other proportioning control elements.

Experiment No. 16

PRESSURE CONTROL TRAINER

1. Aim
Study of Pressure Control Trainer

2. Objective(s)
  i. To study the effect of Proportional Band and Derivative time for controlling pressure in a process vessel.
  ii. Tuning of controller by closed-loop continuous cycling (ultimate gain) method.
  iii. To study the stability of the system (Bode Plot)

3. Theory

Process Controllers
In automatic controlled process the parameter to be controlled is measured and compared with the set point by process controller. The difference between the measured signal and the set point is error. The controller performs on-line calculations based on error and other setting parameters and generates an output signal. The output signal drives the final control elements like control valve or a damper to control the process to the set point.
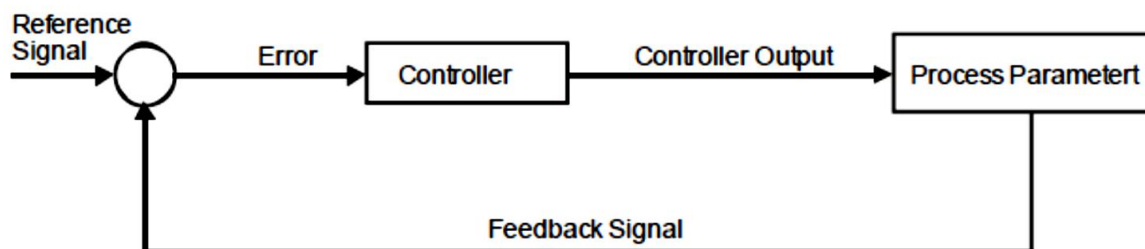
Block Digaram of Typical Controller

Proportional (P) controllers

In proportional controller the control algorithm generates a linear control output proportional to deviation. In proportional action the amount of change in the measured value (or deviation) is expressed in percent of span that is required to cause the control output to change from 0 to 100 % is called the proportional band.
The controller output is given by:

$$OP = \frac{100}{PB} . e$$

where, OP is the output, PB is proportional band in %, b is the bias value, and e is the error signal. If there is no biasing, output OP will become zero when error is zero. Hence bias value decides the value of output when error is zero. The proportional controllers usually show some difference between the set point and process variable called offset. The offset can be reduced by decreasing proportional band or by readjusting the bias. With decrease in proportional band the process becomes oscillatory.

There are two types of controller actions:

1) Increase-increase in which output increases as measurement increases. (error e = measurement – set point)
2) Increase-decrease in which output decreases as measurement increases. (error e = set point – measurement)

Proportional- Integral (PI) controllers

Propertional - Integral Controller
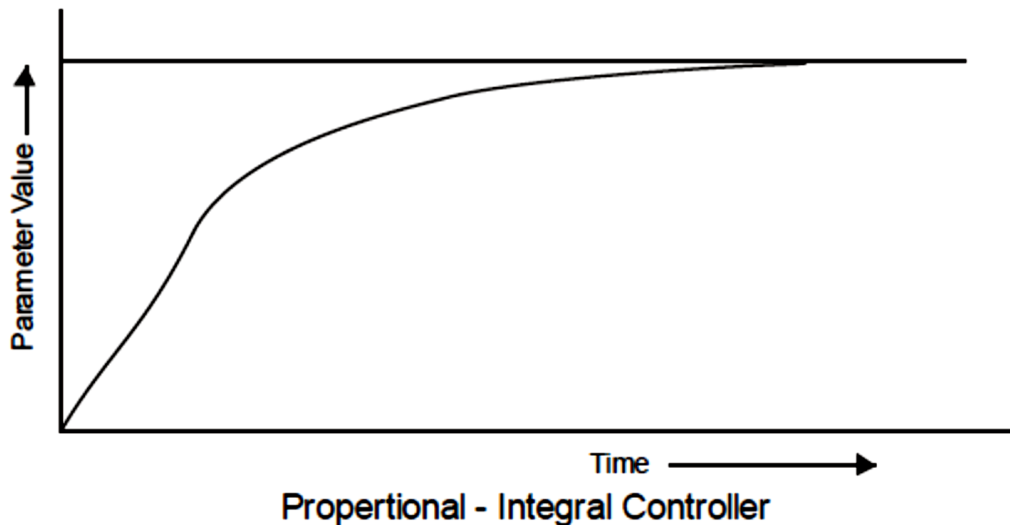
The offset in proportional controller can be overcome by adding integral action. The control algorithm that applies changes in output as long as deviation exits, so as to bring the deviation to zero, is called integral action. Output of proportional-Integral controllers is given by:

$$OP = b + \frac{100}{PB}\left(e + \frac{1}{Ti}\int e.\,dt\right)$$

Where OP is the output, b is the bias, PB is the proportional band in %, e is the error signal, Ti is integral time; this is the time required to repeat proportional action.

Proportional-Derivative (PD) controller

This mode of control is described by the relationship

$$OP = b + \frac{100}{PB}\left(e + Td.\frac{de}{dt}\right)$$

Where OP is the output, b is the bias, PB is the proportional band in %, e is the error signal, Td is derivative time.

Larger the derivative time larger is the action. Smaller is the proportional band the larger is the derivative action.
In order to achieve faster response and more stable operation in slow processes derivative action is added to apply an output component proportional to the rate of change of input (error). Derivative action is used with P action or PI action.

Proportional–Integral–Derivative (PID) controllers

PID controllers are used for controlling almost all process variables like temperature, flow, level, pressure etc. in a continuous or batch process.
The output of a PID controller is given by:

$$OP = b + \frac{100}{PB}\left(e + Td.\frac{de}{dt} + \frac{1}{Ti}\int e.dt\right)$$

Where OP is the output, b is the bias, PB is the proportional band in %, e is the error signal, Ti is the integral time and Td is derivative time.
Selection of proportional band, integral time and derivative time to achieve desired process response to load changes is called tuning of controller.

Controller tuning

The three-mode controller (PID) is the most common feedback controller used in industrial control. The method of determination of the optimum mode gains, depending on the nature and complexity of the process is known as loop tuning. The three parameters should be selected to meet a set of defined goals. These goals typically require a plant response with minimum steady state error, insensitivity to load disturbances and an acceptable transient response to set point changes and disturbances. In practice the choice of proportional band, integral time and derivative time is a compromise between the set point tracking and disturbances. If a mathematical model of the process is known, selecting the controller parameters is relatively simple. But in many industrial applications, a reliable mathematical model is not available or it is difficult to determine. So empirical rules are developed for PID tuning which do not require

mathematical model. A widely used set of rules is proposed by Ziegler- Nichols by open loop method and close loop method in 1942.

Closed loop method (Ultimate gain method)

This method is also called as ultimate gain method. The term ultimate was attached to this method because its use requires the determination of the ultimate proportional band and ultimate period. The ultimate proportional band, PBu is the minimum allowable value of proportional band (for a controller with only proportional mode) for which the system continuously oscillates at constant amplitude. The ultimate period, Tu is the period of response with the proportional band set to its ultimate value. To determine the ultimate proportional band and ultimate period the proportional band of the controller (with all integral and derivative action turned off) is gradually reduced until the process cycles continuously.
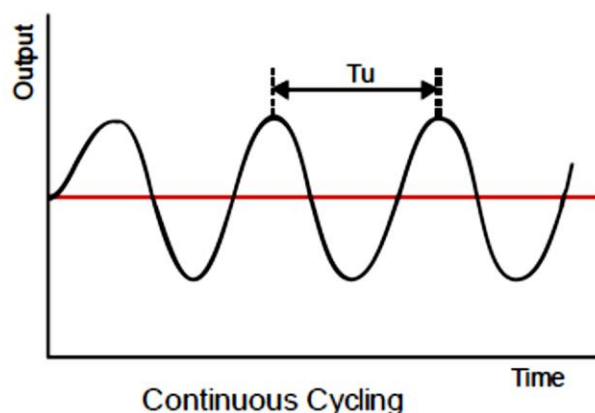
Response curve for ultimate gain and period

The process is placed in the closed loop with a proportional controller. The Proportional band is decreased until the process goes to continuous oscillations. The corresponding value of proportional band is called as ultimate proportional band PBu and the period of oscillation is called the ultimate period Tu. The PID controller parameters are selected from the following table.

Mode	Proportional	Integral	Derivative
P	2 PBu		
P+I	2.2 PBu	Tu/1.2	
P+I+D	1.65 PBu	0.5 Tu	Tu/8.0

Laplace transform techniques are used to express mathematical models in terms of transfer functions in order to calculate transient response. An alternative way to interpret the transfer function model, referred to as the complex transfer function or the frequency response. It shows

how the output response characteristics depend upon the frequency of the input signal. This is the origin of the term, frequency response.

Frequency response analysis

Stability analysis by using Bode plots

Frequency response may be plotted in three ways:
i. The rectangular plot with amplitude ratio verses frequency and phase lag verses frequency.
ii. A polar plot with magnitude and phase shown in vector form with frequency as a parameter.
iii. A phase margin plot with magnitude shown verses a function of phase with frequency as a parameter.
If the system is linear, the output signal for sine wave input must be a sine wave having the same frequency as the input.
Assume that the input to the first order system is suddenly switched from a constant input to a sine wave input. The initial slope of the sine wave input signal may be approximated by a ramp input or even exponential input. If the input frequency is very low, period of the sine wave will be very long. Stated in another way, period is very much larger than the system time constant. Under this condition the output will have sufficient time to approach the condition, which is nearly in equilibrium with the input signal. Thus, at low frequencies both magnitude and phase angle of the output signal will be close to magnitude and phase angle of the input signal. However as the frequency increases period decreases and the system does not have sufficient time to achieve the equilibrium status between input and output. Under these conditions output magnitude will decrease, and output phase angle will fall behind or lag the input phase angle. Thus it is evident that any solution must show a decrease in ratio of output to input magnitude as frequency increases.

Magnitude and phase angle calculation:

Let,
M   Magnitude ratio
A1   Input amplitude (%)
A2   Output amplitude (%)
ϕ Phase angle (degrees)
x   lag (seconds)
T   Period (seconds)
f   frequency (cycles per seconds) = 1/T.
Then,

$M = A2/A1$

$\phi = (x/T) \times 360$

Frequency Response
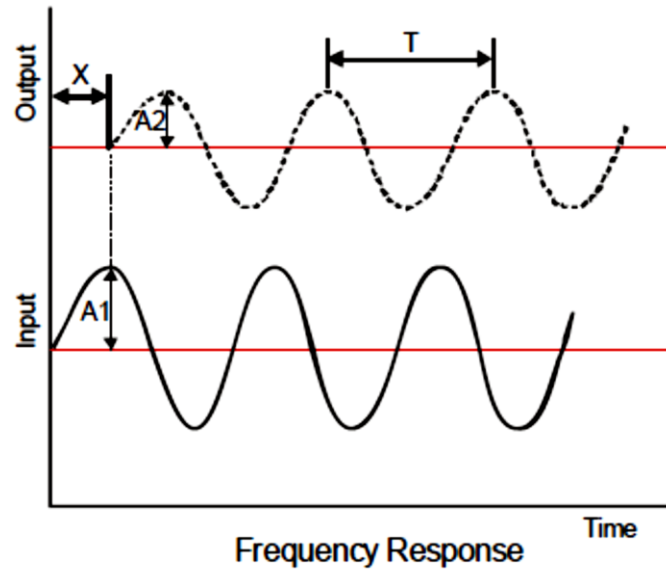
BODE diagram

In Bode diagram the magnitude ratio verses frequency is plotted on log-log co ordinates and the phase angle verses frequency on semi-log coordinates.


Bode Diagram
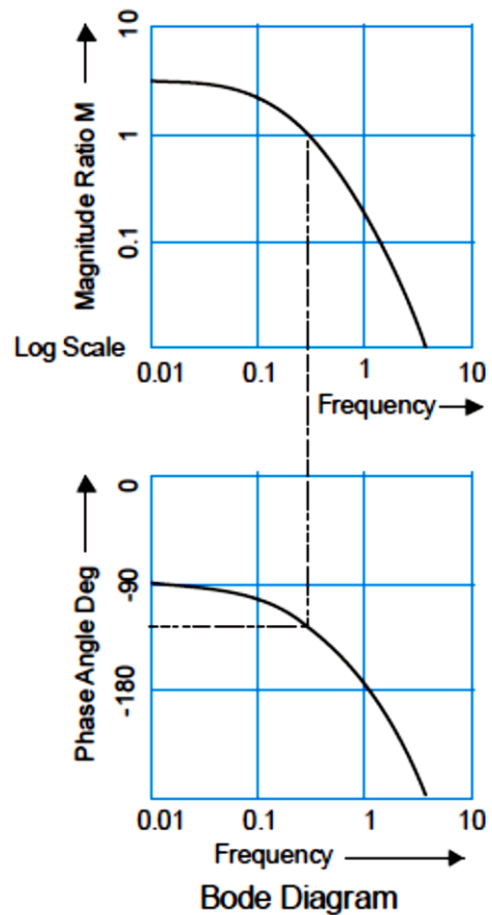
Stability criteria

- A system is stable if the phase lag is less than 180 Deg. at the frequency for which the gain is unity (one).
- A system is stable if the gain is less than one (unity) at the frequency for which the phase lag is 180 Deg.

4. Experimental Set-up

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.
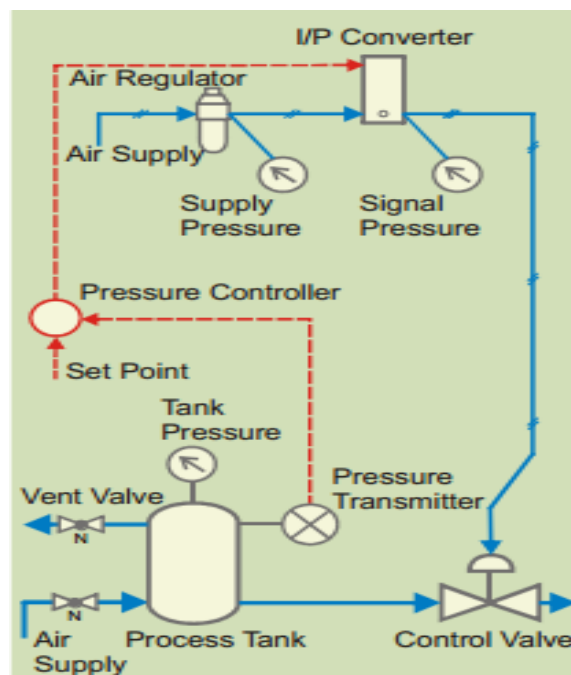
5. Experimental Procedure

Startup Setup:
- Switch on Mains.
- Switch on the compressed air source and adjust the *Air regulator* to set supply air pressure at @ 2 kg/cm^2.

Startup software:
- Click on Start|All programs|Wonderware|InTouch|Yes to open InTouch Appilcation manager.
- Open "Pressure Control Trainer" under InTouch Appplication Manager
- Click "Ignore" under "WindowMaker" for License not available
- Click "Select All" and "OK" under "Windows to Open"
- Click on "Runtime" at Top Right corner under "Intouch- WindowMaker"
- Click "Ignore" under "WindowViewer" for License not available

- Click "OK" under " WindowViewer" for Demo mode

Open loop response (Manual control):
- **Startup Setup** and **Startup software** as explained before.
- Select **open loop** Experiment (click on "Change Expt." Button, click on "Change", Click on "Open Loop" button.)
- Close the control valve by increasing the controller output to 100%. Note down steady state process value.
- Apply the step change by 10% to controller output and wait for the process value to reach the steady state value. Note down the process value.
- Repeat the above step until the controller output reaches to minimum 0%.
- Set the output of the controller to the noted value and at steady state apply the load change to the process. Load change can be given by slightly varying the vent valve. Observe new steady state process value.

Effect of Proportional Band:
- **Startup Setup** and **Startup software** as explained before.
- Select **P Mode** option for control from software. (click on "Change Expt." Button, click on "Change", Click on "P Mode" button.)
- Keep the set point to 60%. Change output mode to Manual. Adjust output value so as to match the process value with set point and apply this output value as bias value to the controller. Adjust the proportional band to 50%.
- Switch the controller to Auto mode.
- Apply step change of 10% to set point and observe the response.
- Switch the controller to Manual mode. Decrease proportional band to half of the previous value & then shift controller to Auto mode. With each decrease, obtain a new response of the step change. Ensure that the set point changes are around the same operating point (@ 5-10% only).
- Using trial and error approach, find a value of proportional band so that the response to a step change has at most one overshoot and one undershoot.

- Set the controller to the settings obtained in the above step and wait for the system to reach at steady state.

Effect of Derivative time:
- **Startup Setup** and **Startup software** as explained before.
- Initialize Modbus communication on the computer i.e.Start| All programs |
- Wonderware FactorySite | IO Servers | Modicon MODBUS
- Execute Programs | Wonderware | InTouch for Pressure control. Select All in
- Windows to open and Click "Runtime".
- Select **PD Mode** option for control from software. (Click on "Change Expt."
- Button, click on "Change", Click on "PD Mode" button.)
- Select PD controller. Set the proportional band estimated from Proportional control (P only). Start with derivative time=0 and integral time=6000 sec., which will cut off the derivative action and widen the effect of integral action.
- Set the set point to desired pressure (@60%). Allow the process to reach at steady state. Note the response of the system.
- Switch on the controller to manual mode. Increase the derivative time by 1 sec.
- Switch to Auto mode and apply step change to the set point by 2 to 3%. Note the response of the system.
- Increase the derivative time gradually and observe the process response for step change.

Tuning of controller (Closed loop method):
- **Startup Setup** and **Startup software** as explained before.
- Select **Close Loop** option for control from software. (Click on "Change Expt." Button, click on "Change", Click on "Close Loop" button.)
- Set the proportional band value to maximum (Say 100). Set the controller to manual mode and adjust the output so that the process is nearly at set point (60%).
- Set controller to auto mode and impose step on the process by moving the set point for a few seconds & then return to its original value (or apply the step change to the set point of 5%). Wait for some time & observe the response.
- Decrease the proportional band to the half of previous and impose step on the process as mentioned above. Wait for some time & observe the response.
- Repeat the above procedure and find out correct value of proportional band for which the system just goes unstable i.e. continuous oscillations are observed in the output of controller.
- Record the ultimate proportional band and ultimate period from the response.
- Calculate the PID values from the table. Select the PID controller and apply the parameter values obtained from the above steps. Observe the response of the process to a step change with these settings.

Stability of the system (Bode plot):
- **Startup Setup** and **Startup software** as explained before.
- Select **Stability analysis** option for control from software. (Click on "Change Expt." Button, click on "Change", Click on "Stability analysis" button.)
- Select function generator to apply the sinusoidal input to the output of the controller.
- Enter Reference point, Amplitude and Period.

- Observe the sinusoidal output of the controller and sinusoidal response of the process.
- Log the data for records.
- Change the period and repeat the observation for 3-4 different values of the period.
- Form the data file stored note down input amplitude (A1,%).
- Observe the output response of the process and note down the output amplitude (A2,%).
- Measure output wave period and note down as T sec. Measure the phase lag X and note down in sec.

6. Observations

Manual Control:

Controller output in %	Process Value in %
100	
90	
80	

0	

From the above data, note the output required for maintaining the process at desired set points. (for particular vent valve opening).

Effect of Proportional Band:

Proportional Band	PV %	SP%	Offset

- Observe steady state error decreases as proportional band decreases.
- Observe the effect of very low proportional band values (system works in oscillatory mode).
- Observe the response of the system at load change. Load change can be given by slightly manipulating the vent valve of the tank.

Effect of Derivative time:

Derivative time	PV %	SP%	Offset	Oscillation or No oscillations

- Observe the effect of increasing derivative time. Also note that the process may show offset as effect of integral action is cut off.

Tuning:

Ultimate Proportional Band, PBu   =

Ultimate period   Tu   =

Settings for PI Controller:

    Proportional band, PB   =

    Integral time, T_I =

Settings for PID Controller:

    Proportional band, PB   =

    Integral time, T_I =

    Derivative time, T_D =

Stability of the System (Bode Plot):

Observation number	Input Amplitude	Output Amplitude	Output period	Lag x in Sec	Frequency
	A1%	A2%	T in sec		

7. Model Calculations

***

***

Stability of the System (Bode Plot)

- Calculate for each observation
    - Magnitude ratio as M = A2/A1
    - Phase angle = (x/T) x 360
    - Frequency = 1/T cycles / sec.
- Draw the graphs of:
    - Magnitude Vs frequency on log - log scale
    - Phase angle Vs frequency on semi-log coordinates.
    - Study the graph for stable conditions mentioned in theory

8. Results and Discussion

9. Conclusions

10. Precautions

References

1. Instruction Manual for Pressure Control Trainer 314A, Apex Innovations, Sangli, Maharastra, India.
2. Theory Process Control, Apex Innovations, Sangli, Maharastra, India.
3. Seborg, D. E., Edgar, T. F. and Mellichamp, D.A., "Process Dynamics and Control", 2nd Ed., John Wiley and Sons, 2004.
4. Stephanopoulos, G., Chemical Process Control – An Introduction to Theory and Practice, 6th edition, Prentice-Hall of India, New Delhi, India, 1993.
5. Coughanowr, D.R., Process Systems Analysis and Control, 2nd Ed., McGraw-Hill, 1991.

Experiment No. 17

FLOW CONTROL TRAINER

1. Aim
      Study of flow control trainer

2. Objective(s)
    i.   To study the effect of hysteresis in on/off control of flow rate.
    ii. To study the effect of Proportional Band and Integral time for controlling flow rate.
    iii. Tuning of controller by open-loop (process reaction curve) method.

3. Theory
Basic control theory is same as that is given in Expt. 14.

On/Off controllers
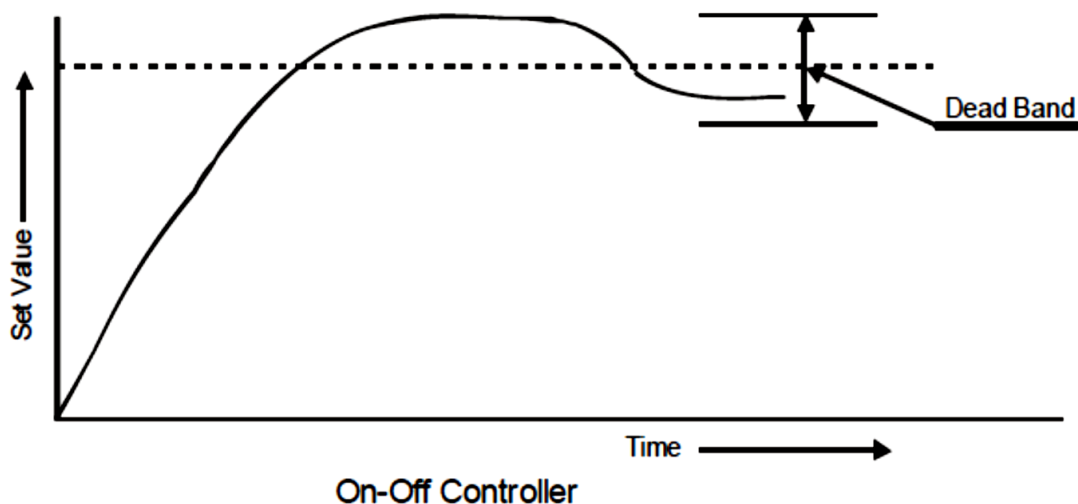
On-Off Controller

A special case of proportional control is On-Off control. If the proportional band of the controller is made very low (=0) the controller output will move from one extreme position to other for slight deviation of process value from the set point. This very sensitive action is called On-Off control because final control element is either open (On) or close (Off) i.e. operates like a switch. These are the simplest controllers.

These controllers incorporate a dead band to keep the output from cycling rapidly between on and off. The controller will not turn on or off until the error signal moves out of the dead band. The process variable controlled by an on/off controller always cycles back and forth about the set point as shown in the fig. dark line and dotted line shows process parameter and reference values respectively. Hysteresis is a value set in the vicinity of on-off operating point. Upper hysteresis is value or band in which process value is allowed to operate above the set point and lower hysteresis is value or band in which process value is allowed to operate below the set point.

Controller tuning

The three-mode controller (PID) is the most common feedback controller used in industrial control. The method of determination of the optimum mode gains, depending on the nature and complexity of the process is known as loop tuning. The three parameters should be selected to meet a set of defined goals. These goals typically require a plant response with minimum steady state error, insensitivity to load disturbances and an acceptable transient response to set point changes and disturbances. In practice the choice of proportional band, integral time and derivative time is a compromise between the set point tracking and disturbances. If a mathematical model of the process is known, selecting the controller parameters is relatively simple. But in many industrial applications, a reliable mathematical model is not available or it is difficult to determine. So empirical rules are developed for PID tuning which do not require mathematical model. A widely used set of rules is proposed by Ziegler- Nichols by open loop method and close loop method in 1942.

Open loop method (Process reaction curve method)

In open loop method the process is assumed to be model of first order. The step response i.e. process reaction curve, allows to obtain the approximate values of P, I and D parameters. With the feedback loop open, a step response is applied to manipulated variable and the values of P, I and D are estimated.
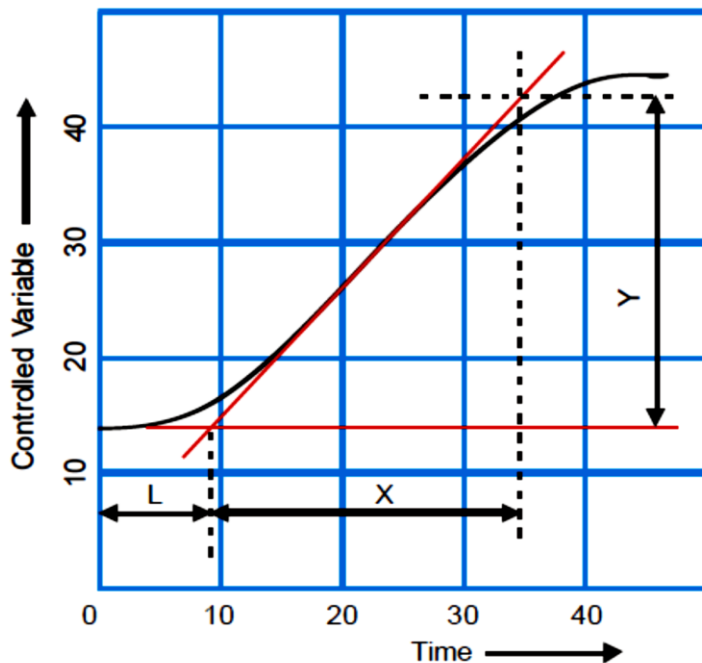
Fig: Open loop response for input step change (Process reaction curve)

Where, Slope R: Slope of line drawn tangent to the point of inflection.

$$R = \frac{\% \ change \ in \ variable}{time \ (\text{min})} = \frac{x}{y}$$

Dead time L: Time between the step change and the point where tangent line crosses the initial value of the controlled variable (in min.).

ΔP = Step change applied in %

Using these parameters, the empirical equations are used to predict the controller settings for a decay ratio of 1/4. For P, PI and PID controller the parameters are calculated as follows.

Mode	Proportional band (in %)	Integral time (in Min)	Derivative time (in Min)
P	100RL/ΔP		
P+I	110RL/ ΔP	L/0.3	
P+I+D	83RL/ ΔP	L/0.5	0.5L

4. Experimental Set-up

Flow control trainer is designed for understanding the basic principles of flow control. The process setup consists of supply water tank fitted with pump for water circulation. A DP transmitter is used for flow sensing which measures differential pressure across orifice meter. The process parameter (flow) is controlled by microprocessor based digital indicating controller which manipulates pneumatic control valve through I/P converter. The control valve is fitted in water flow line. These units along with necessary piping are fitted on support housing designed for tabletop mounting.

The controller can be connected to computer through Ethernet port for monitoring the process in SCADA mode.
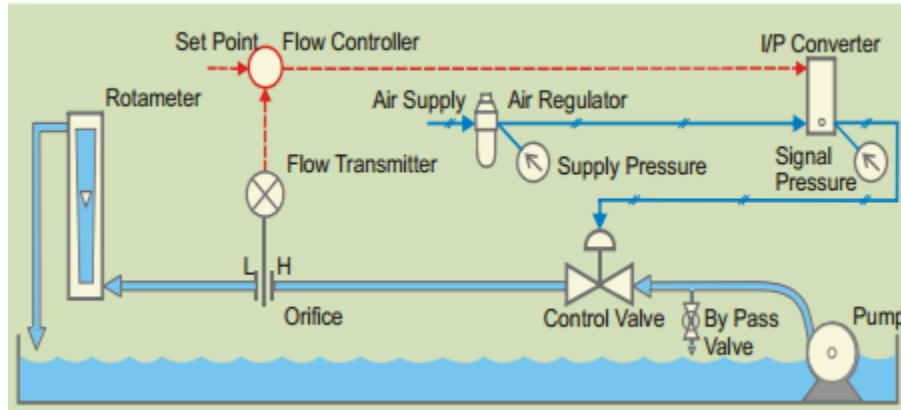
Experiments: The experiments from 1 to 6 are to get feel of the process and PID settings.

5. Experimental Procedure

Startup Setup:

- Switch on electric supply. Switch on Mains.
- Switch on the pump and ensure that flow through rotameter is above 100 LPH.
- Switch on the compressed air source and adjust the air regulator to set supply air pressure at @ 2 kg/cm^2

Startup software:

- Click on Start|All programs|Wonderware|InTouch|Yes to open InTouch Appilcation manager.
- Open "Flow Control Trainer" under InTouch Appplication Manager
- Click "Ignore" under "WindowMaker" for License not available
- Click "Select All" and "OK" under "Windows to Open"
- Click on "Runtime" at Top Right corner under "Intouch- WindowMaker"
- Click "Ignore" under "WindowViewer" for License not available
- Click "OK" under " WindowViewer" for Demo mode

Study of open loop response (Manual control):

- Startup Setup and Startup software as explained before.
- Select open loop Experiment (click on "Change Expt." Button, click on "Change", click on "Open Loop" button.)
- Close the control valve by increasing the controller output to 100%.
- Apply the step change by 10% to controller output and wait for process value to reach the steady state value.
- Repeat the above step until the controller output reaches to minimum 0%.

Effect of Integral time:
- **Startup Setup** and **Startup software** as explained before.
- Select **PI Mode** option for control from software. (click on "Change Expt." Button, click on "Change", Click on "PI Mode" button.)
- Adjust the process value by changing the output of controller in manual mode to a particular pressure (set point =60%).
- Set the proportional band estimated from Proportional control (from previous experiment). Start with derivative time=0 and integral time=1000 sec., which will cut off the derivative action and widen the effect of integral action.
- Set the set point to desired pressure (@60%). Allow the process to reach at steady state. Record the steady state error.
- Switch on the controller to manual mode. Reduce the integral time to half of the previous value. Switch to Auto mode and apply step change to the set point by 2 to 3%. Note the response of the system.

- Using trial and error, find out an integral time, which gives satisfactory response to the step change in set point.

Tuning of controller (Open loop method)

- Startup Setup and Startup software as explained before.
- Select Process Reaction option for control from software. (Click on "Change Expt." Button, click on "Change", Click on "Process Reaction" button.)
- Adjust controller output, so that the process value is maintained at 70%.
- Start data logging.
- Apply a 20 - 30 % change to controller output. (Open the control valve) Record the step response. Wait for the steady state.
- Stop data logging.
- Plot the step response (Process reaction curve) from stored data. Find out the value of slope at the point of inflection and time lag.
- Calculate P I D settings for different modes.
- Select close loop, switch auto manual key to auto mode and then select controller to study. Set the PID values obtained from the calculations. Apply the step change & observe the response of the system. Allow the system to reach steady state.

Tuning of controller (Using Auto Tuning method)

1. Startup Setup and Startup software as explained before.
2. Select Autotune option for control from software. (Click on "Change Expt." Button, click on "Change", Click on "Autotune" button.)
3. Wait Till Autotune is complete. (Blinking of green LED stops).
4. Controller automatically finds the PB, IT & DT values.
5. Find out PID values at different set points /flow rates

6. Observations

Study of open loop response (Manual control):

Controller Output OP%	Process Variable PV%

From the above data, note the output required for maintaining the flow at desired set points.

Effect of Integral time:

Integral time	PV %	SP%	Offset	Oscillation or No oscillations

Observe the effect of reducing integral time on offset and on the response of the process.

Tuning of controller (Open loop method)

1. Step change to the system ΔP = Initial output - Final output of the controller.
2. Plot the graph of process value Vs Time on a graph paper.
3. From process reaction curve:
4. Slope of the process reaction curve R =
5. Time lag L=
6. Calculate P, PI, PID setting from above values.
7. Observe response of the system for different PID settings.

Tuning of controller (Using Auto Tuning method)

Compare the PID settings obtained from Open loop method with Auto tuning method. Observe the performance of both for a step change in set point.

7. Model Calculations

8. Results and Discussion

9. Conclusions

10. Precautions

References

1. Instruction Manual for Flow Control Trainer 312A, Apex Innovations, Sangli, Maharastra, India.
2. Theory Process Control, Apex Innovations, Sangli, Maharastra, India.
3. Seborg, D. E., Edgar, T. F. and Mellichamp, D.A., "Process Dynamics and Control", 2nd Ed., John Wiley and Sons, 2004.
4. Stephanopoulos, G., Chemical Process Control – An Introduction to Theory and Practice, 6th edition, Prentice-Hall of India, New Delhi, India, 1993.
5. Coughanowr, D.R., Process Systems Analysis and Control, 2nd Ed., McGraw-Hill, 1991.

Experiment No. 18

CONTROL VALVE CHARACTERISTICS

1. Aim
Study of control valve characteristics

2. Objective
i.   To study inherent characteristics of different control valves
ii. To study installed characteristics of different control valves
iii. To study hysteresis of different control valves

3. Theory

Types of Control valves
Valve is essentially a variable orifice. Control valve is a valve with a pneumatic, hydraulic, electric (excluding solenoids) or other externally powered actuator that automatically, fully or partially opens or closes the valve to a position dictated by signals transmitted from controlling instruments. Control valves are used primarily to throttle energy in a fluid system and not for shutoff purpose. The figure shows basic elements and internal parts of typical pneumatic control valve. Depending upon the valve plug design the control valves can be classified as quick opening, linear and equal percent type.

Linear: Flow is directly proportional to valve lift.

$$Q = ky$$

Where
Q = flow at constant pressure drop
y = valve opening
k = constant

Equal%: Flow changes by a constant percentage of its instantaneous value for each unit of valve lift.

$$Q = b\, e^{ay}$$

Where
Q = flow at constant pressure drop
y = valve opening
e = base of natural logarithms
a and b = constants
Constants a and b can be evaluated to give more convenient form

$$Q = Q_0 e^{\{(\log R / y_{\max})y\}}$$

Where

Q_o = Flow at constant drop at zero stroke

R = Flow range of valve, maximum to minimum at constant drop.
$y_{\max}$ = maximum rated valve opening

Quick opening: Flow increases rapidly with initial travel reaching near its maximum at a low lift. It is generally not defined mathematically.

Valve actions and actuator mechanism

Different types of actuators are used to control the stem travel of the valve, like electrical actuators, pneumatic actuator, Hydraulic actuators etc.

In this product pneumatic actuators are used for control valves. Spring opposed diaphragm actuator positions the valve plug in response to the controller signals. Mostly the controller signals are in the range of 3 - 15 psig.

Direct acting actuator (air to close):

Direct acting actuators basically consist of a pressure tight housing sealed by a flexible fabric reinforced elastomer diaphragm. A diaphragm plate is held against the diaphragm by a heavy compression spring. Signal air pressure is applied to upper diaphragm case that exerts force on the diaphragm and the actuator assembly. By selecting proper spring rate or stiffness, load carrying capacity, and initial compression, desired stem displacement can be obtained for any given input signal.

Reverse acting actuator (air to open):

In case of reverse acting actuators the stem gets retracted with increase in pressure.

Control valve flow coefficient

A control valve regulates the flow rate in a fluid delivery system. In general a close relation exists between the pressure along the pipe and the flow rate so that if pressure is changed, then the flow rate is also changed. A control valve changes the flow rate by changing the pressure in the flow system because it introduces the constriction in the delivery system so we can say that the flow rate through the constriction is given by

$$Q = K\sqrt{\Delta P} \qquad\qquad (1)$$

The correction factor K in above equation allows selection of proper size of valve to accommodate the rate of flow that the system must support. This correction factor is called as valve coefficient and is used in valve sizing.

Valve coefficient:

$$C_v = 1.16\, Q \sqrt{\frac{G}{\Delta P}} \qquad \text{(In SI Units)} \qquad\qquad (2)$$

Where G is specific gravity of liquid, Q flow in m^3/h, ΔP pressure drop in bar.

Valve Characteristics

The amount of fluid passing through a valve at any time depends upon the opening between the plug and seat. Hence there is relationship between stem position, plug position and the rate of flow, which is described in terms of flow characteristics of a valve. Inherent and Installed are two types of valve characteristics.

Inherent characteristics:
The inherent flow characteristic of control valve is the relation between the flow and the valve travel at constant pressure drop across the valve. Following are the inherent characteristics for different types of valves
.

Installed characteristics:
The Inherent characteristics of the valves described are subject to distortion due to variations in pressure drop with flow. Line resistance distorts linear characteristics towards that of quick opening valve and equal% to that of linear.
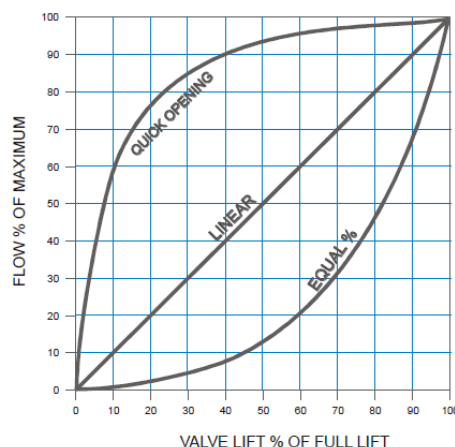
Hysteresis of control valve
Hysteresis is a predictable error resulting from the differences in the transfer functions when a reading is taken from above and below the value to be measured. In case of control valves for same actuator signal different stem travel (hence valve coefficients) are obtained depending upon the direction of change in the signal. The maximum error in stem travel (or valve coefficient) expressed in % for same actuator pressure while opening and closing the valve is indicated as hysteresis.

Rangeability of equal % valve
Equal% valve has characteristics such that given percent change in stem position produces an equivalent change in flow. Generally this type of valve does not shut off the flow completely in its limit of stem travel. The Rangeability (R) is defined as the ratio of maximum to minimum controllable flow.

$$R = \frac{F_{max}}{F_{min}}$$
(3)

Where F_{max} is the flow when the valve stem is at extreme open position F_{min} is the flow when valve stem is at extreme closed position.
(F_{max}, F_{min} represents flow rates measured at constant pressure drop across control valve. Hence rangeability R also can be defined as ratio of Cv_{max} to Cv_{min}.) For equal percent valve flow has exponential characteristics of rangeability,

$$F = R^{m-1}$$
(4)
Where R is the rangeability of the valve and m is its fractional stem position.

Valve positioner

Valve positioner is a device used with actuator. The actuator stem motion is accurately compared with the signal from controller. Any deviation from the desired position results in an error signal which activates pneumatic relay having an independent air supply. Some of the advantages of positioner are as follows:

- Helps in overcoming valve stem friction
- Matches input signal with valve stroke
- Increases speed of response of control valve

Possibility of split ranging, alteration in valve characteristics and action reversal

4. Experimental Set-up

The setup is designed to understand the control valve operation and its flow characteristics. It consists of pneumatic control valves of linear, equal% (& quick opening for product 318B) type, stainless steel water tank with pump for continuous water circulation and rotameter for flow measurement. An arrangement is made to measure pressure at the valve inlet in terms of mm of water. An air regulator and pressure gauge is provided for the control valve actuation. In case of additional optional requirement a valve positioner is fitted on linear valve. The set up is stand-alone type.
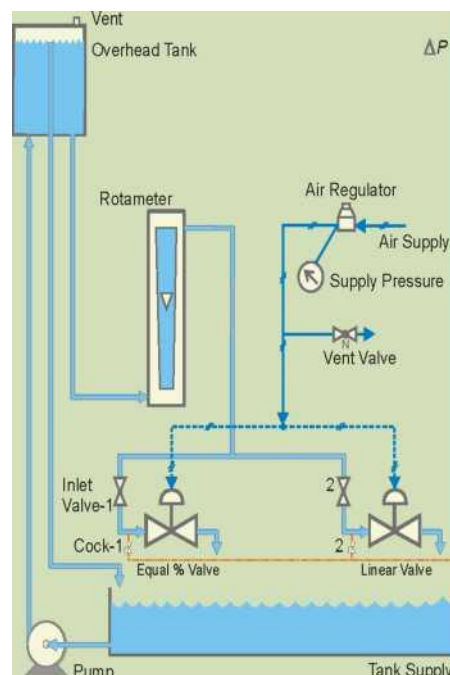
5. Experimental Procedure

Study of inherent characteristics

- Start up the set up. Open the flow regulating valve of the control valve to be studied (Linear/ Equal%/quick opening). Open the respective hose cock for pressure indication. (Close the flow regulating valves and hose cocks of other control valves.)
- Ensure that pressure regulator outlet is connected to the valve actuator of the control valve under study. Keep the control valve fully open by adjusting air regulator.
- Adjust the regulating valve and set the flow rate. (Set 400 LPH flow for linear/equal% valve   or
- 600 LPH for quick opening valve). Note for measuring flow rates below rotameter minimum range use measuring jar.
- Note the pressure drop at control valve at full open condition.
- Slowly increase/decrease air pressure by regulator and close the control valve to travel the stem by 4mm. Note the pressure drop at control valve and corresponding flow rate.

- Repeat above step and take the readings at each 2mm-stem travel till the valve is fully closed.

Study of installed characteristics

- Start up the set up. Open the flow regulating valve of the control valve to be studied (Linear/ Equal%/quick opening). Open the respective hose cock for pressure indication. (Close the flow regulating valves and hose cocks of other control valves.)
- Ensure that pressure regulator outlet is connected to the valve actuator of the control valve under study. Keep the control valve fully open by adjusting air regulator.
- Adjust the regulating valve and set the flow rate. (Set 400 LPH flow for linear/equal% valve or 600 LPH for quick opening valve). Note for measuring flow rates below rotameter minimum range use measuring jar.
- Note the flow rate at full open condition.
- Slowly increase/decrease air pressure by regulator and close the control valve to travel the stem by 4mm. Note the flow rate.
- Repeat above step and take the readings at each 4mm stem travel till the valve is fully closed.

Study of hysteresis of control valve

- Start up the set up. Open the flow regulating valve of the control valve to be studied (Linear/ Equal%/quick opening). Open the respective hose cock for pressure indication. (Close the flow regulating valves and hose cocks of other control valves.)
- Ensure that pressure regulator outlet is connected to the valve actuator of the control valve under study. Keep the control valve fully open by adjusting air regulator.
- Adjust the regulating valve and set the flow rate. (Set 400 LPH flow for linear/equal% valve or 600 LPH for quick opening valve). Note for measuring flow rates below rotameter minimum range use measuring jar.
- Note the pressure drop at control valve at full open condition.
- Slowly increase/decrease air pressure in the step of 3 psi by regulator to close the control valve fully. Note the actuator pressure, pressure drop across the control valve and the flow rate. (Do not try to correct the actuator pressure by reversing the regulator direction if it inadvertently exceeds desired value.)
- Slowly decrease/increase air pressure in the step of 3 psi by regulator to open the control valve fully. Note actuator pressure, pressure drop across control valve and the flow rate.

6. Observations

Study of inherent characteristics:

Type of control valve: Linear/ Equal%/ Quick opening

(Fill up the column "valve coefficient" after calculations)

Sr. No	Lift (mm)	Flow (LPH)	Pressure drop AP (mm of H_2O)	Valve coefficient Cv
1				
2				
3				
4				
5				
6				

Plot the graph of valve coefficient versus lift to obtain inherent characteristic
of the control valve.

- Inherent characteristics of control valve can also be studied by keeping constant pressure drop across the control valve.
- Keep the valve fully open and adjust the flow rate. (400 LPH for linear/equal% valve or 600 LPH for quick opening valve). Note the pressure drop.
- Gradually close the control valve in steps of 4mm of stem travel. The pressure drop across the valve increases. Manipulate flow rates to maintain pressure drop constant. Note the flow rates.

Plot the graph of flow versus lift.
- Note that the nature of the graph is same as inherent valve characteristics.

Study of installed characteristics
Type of control valve: Linear/ Equal%/ Quick opening

Sr. No	Lift (mm)	Flow (LPH)
1		
2		

Study of hysteresis of control valve
Type of control valve: Linear/ Equal%/ Quick opening
(Fill up columns "valve coefficient" and " Hysteresis" after calculations).

Sr No	Actuator pressure (psig)	Flow (LPH)	Pressure drop (mm of H2O)	Valve coefficient Cv	Flow (LPH)	Pressure drop (mm of H$_2$O)	Valve coefficient Cv	Hysteresis (%)
		Increasing actuator pressure			Decreasing actuator pressure			
1	0							
2	3							
3	6							
4	9							
5	12							
6	15							
7	18							

Plot the graph of actuator pressure versus valve coefficient. Ratio of maximum difference between flow coefficients at same actuator pressure, to that of maximum flow coefficient is termed as hysteresis.

7. Model Calculations

Study of inherent characteristics / Study of hysteresis of control valve

$$C_v = 1.16\, Q \sqrt{\frac{G}{\Delta P}} \qquad \text{(In SI Units)}$$

where,
Q = Flow (m^3/h)= Q in LPH/1000
ΔP = Pressure drop across valve (bar) = (ΔP in mm of H$_2$0) $\times$ 1.013 / (10.33 $\times$ 10^3).
G = Specific gravity = 1 for water

8. Results and Discussion

9. Conclusions
- The inherent valve characteristics plotted for each valve fairly tallies with theoretical valve characteristics.
- Installed characteristics of linear valve slightly approaches to the characteristic of quick opening valve because of the pipe friction and other resistance to the flow.
- Installed characteristics of equal % valve approaches to the characteristics of linear valve because of the pipe friction and other resistance to the flow.
- The experiment gives idea about hysteresis of control valve.

10. Precautions

References

1. Instruction Manual for Control Valve Characteristics 318B, Apex Innovations, Sangli, Maharastra, India.
2. Theory Process Control, Apex Innovations, Sangli, Maharastra, India.
3. Seborg, D. E., Edgar, T. F. and Mellichamp, D.A., "Process Dynamics and Control", 2nd Ed., John Wiley and Sons, 2004.
4. Stephanopoulos, G., Chemical Process Control – An Introduction to Theory and Practice, 6th edition, Prentice-Hall of India, New Delhi, India, 1993.
5. Coughanowr, D.R., Process Systems Analysis and Control, 2nd Ed., McGraw-Hill, 1991.

Experiment No. 19

MULTIPROCESS TRAINER

1. Aim
Study of advanced control strategies with multi-process trainer
2. Objective(s)
    i.   To study cascade control system (LC loop in cascade with FC loop)
    ii. To study feed-forward plus feedback control system.
    iii. To study the ratio control system

3. Theory
Advanced control strategies

Cascade control

The cascade controller consists of two controllers

1) Primary Controller
2) Secondary Controller

In cascade control arrangement the output of primary controller is used as set point of secondary controller. Each controller has its own measurement input, but only the primary controller has independent set point and the secondary controller has an output to the process. The manipulated variable and, the secondary controller, and its measurement constitute a closed loop within the primary loop.

Tuning of cascade controllers
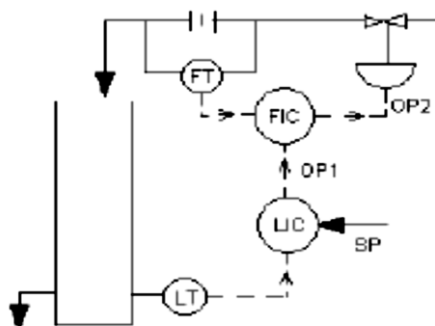
Because the secondary loop exists as an element in the primary loop, the secondary controller should be properly tuned before the primary controller, with the primary controller in manual. Whether the secondary should be tuned for optimum set point or load response depends on whether severe load changes are expected in the secondary loop. If so, observing response to load changes simulated by stepping the secondary controller output while in manual should do tuning, followed by transfer to automatic. If not, set point steps should be introduced.

In a cascade system that is cycling, it is often difficult to determine which controller needs detuning. If the period of cycles is too short to belong to the primary loop, the safest action is to widen the proportional band of

secondary controller- a few percent changes is enough. In this case, both periods may appear in either or both of the controlled variables. But only one period is evident, widening the proportional band of either controller could dampen the cycle, although it is probably more effective to de-tune the primary controller.

Feed forward control
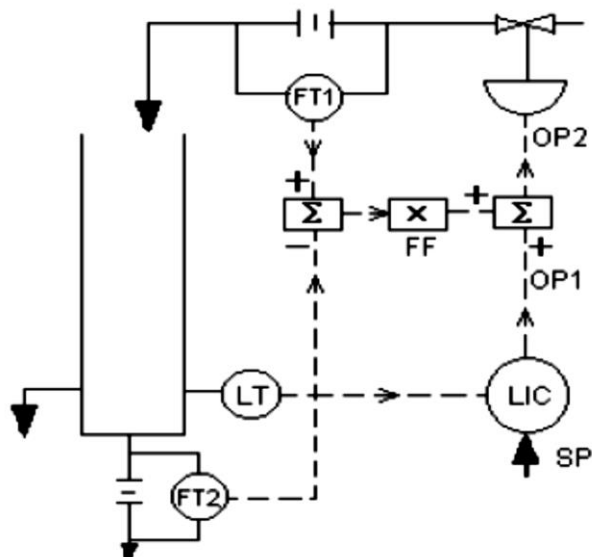
In feed forward control the correction is based upon the changes in load variable(s), hence the change in this variable does not affect the controlled variable. This overcomes the serious limitation of economic loss associated with the feed back controller. It applies faster correction. The feed forward control system alone cannot maintain the controlled variable at the set point because of unknown disturbance or any un-measurable process variables. Hence combination with feed back loop helps in maintaining the process variable at the set point.

The inlet flow signal **FT1** and the outlet flow signal **FT2** are measured and compared. A multiplying factor is added for the calculation. The difference between the two-flow signals is multiplied by the **feed forward** factor and added to the output of the level controller. If the out flow is less than the inlet flow the positive output of the calculation is added to the LIC output i.e. the output of the controller is increased. This reduces inlet flow to match with outlet. Any finer correction is applied by output of LIC loop.

OP2 = OP1+ FF(FT1-FT2)

There is no feed forward action when FT1=FT2 or when FF=0.

Ratio control

Ratio control systems are feed forward systems wherein one variable is controlled in ratio to another to satisfy some higher-level objective. In ratio control systems, the true controlled variable is controlled in the ratio of controllable flow F1 to wild flow F2

R = F1 /F2

In the above system the ratio set is multiplied by the wild flow and the value is given as a set point to flow controller FIC. The controllable flow F1 and the wild flow F2 is measured with orifice meters and DP transmitters.

Rc = FT1 / FT2
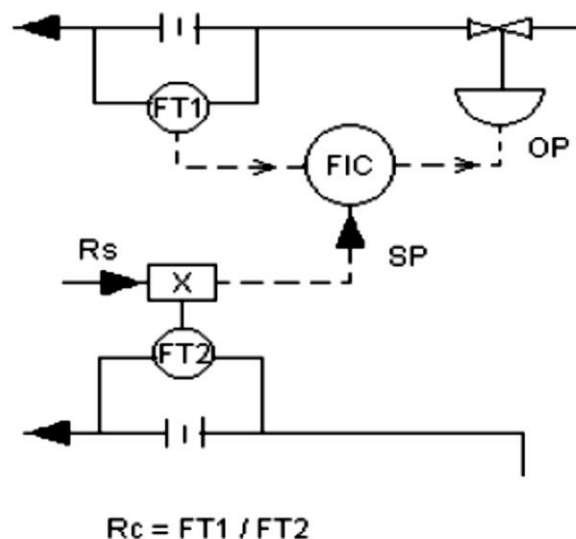
Ratio Station: The ratio station is the calculation box where the set point is generated for FIC. Calculations to be carried out in the ratio station block:

SP= F2 X Rs

Where
SP: Set point for controllable flow loop F1
F1: Controllable flow displayed in %.
F2: Wild flow displayed in %. (Flow values displayed are square rooted values obtained from respective DP transmitter)
Rs: Ratio set. (Range 0.5 – 2)
Rc: Current ratio = Ratio of current flow values **(F1 / F2)**.

Ratio control is utilised in processes where two process variables have to be fed to a process in a definite ratio. For example, in a gas-fired furnace, the flow of gas has to match in a fixed ratio with combustion air, blending of reactants entering a chemical reactor etc.

4. Experimental Set-up

The setup is designed to understand the advanced control methods used for complex processes in the industries. Different experiments like Flow, level, cascade, feed forward and ratio control can be configured and studied with the setup. It consists of water supply tank, pump, level transmitter, transparent level tank, orifice meters with differential pressure transmitters, rotameters, pneumatic control valve, I/P converter and serial based dual loop controller. These units along with necessary piping are mounted on stand-alone type structure. The set up is connected to computer through USB port for monitoring and control by using PID logics. Product is supplied with 32 tag demo version of SCADA software package.
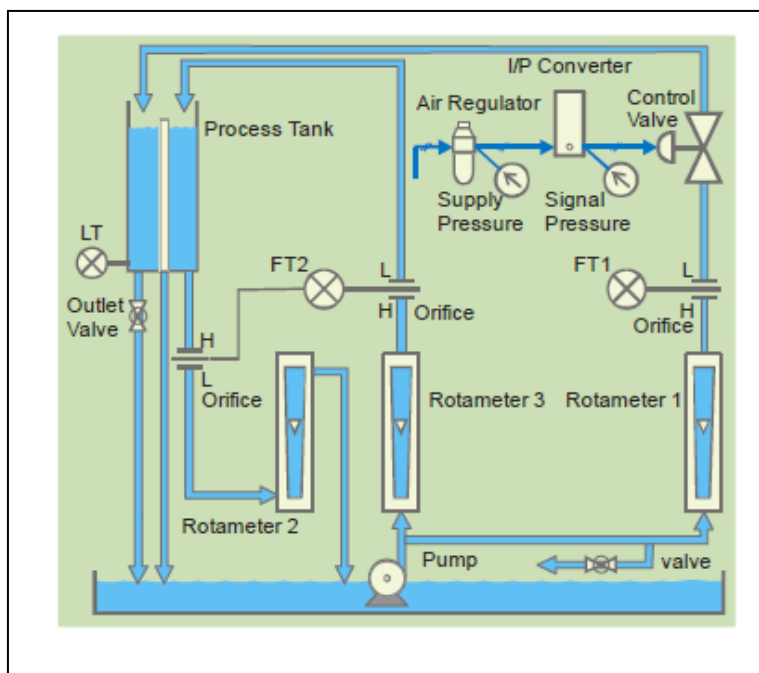
5. Experimental Procedure
For cascade loop:
- Start up the set up and adjust the Rotameter-1 (extreme right) to 100 LPH.
- Provide air supply to the regulator and adjust it to 2 kg/cm^2
- Click on Start|All programs|Wonderware|InTouch|Yes to open InTouch Appilcation manager.
- Open *"Multi Process trainer "ADAM-4022T"*
- Click "Ignore" under "WindowMaker" for License not available
- Click "Select All" and "OK" under "Windows to Open"
- Click on "Runtime" at Top Right corner under "Intouch- WindowMaker"
- Click "Ignore" under "WindowViewer" for License not available Click "OK" under " WindowViewer" for Demo mode
- For selecting Cascade experiment: Click "Experiment On"; Click "Experiment off". Then Click "Cascade"
- Close the Rotameter-2 (Extreme Left) and close the drain valve provided at the inlet of Rotameter-2.

- Open the control valve by changing secondary loop to "Manual" mode and decreasing the controller output to 0%.
- Adjust the tank drain valve such that the tank level shall remain between 90 and 100% . Take Secondary loop to "Auto" mode.
- Default values of Primary loop are SP=50, PB=20, IT=30, DT=2, Act=Revese, and that of secondary loop are PB= 75, IT=8, DT=2, Act=Direct. Note that primary loop is "Level control" loop and secondary loop is "Flow Control" loop. The output of primary loop is connected to secondary loop as a set point.
- Wait till the Process is at steady state. Change the set point of primary loop and observe the response.
- For tuning cascade controller first tune **Secondary Controller.** Tune the controller independently by keeping the Primary controller in manual mode. The set point of secondary loop can be changed by changing output of primary loop.
- Tune the **Primary Controller** using trial and error approach, select the proportional band and integral time, which gives a satisfactory response to step change in set point.
- Conduct the experiment without cascading the primary loop but with the same settings and same step change.

For feed forward-feedback loop:
- If the tappings of orifice placed in the process tank outlet line are short circuited by a PU tube loop, remove the loop. Connect pressure signal tappings of this orifice to FT2 with proper polarity.
- Short circuit the pressure tappings of orifice placed in the line, at the outlet of the middle rotameter.
- Start up the set up and adjust the Rotameter-1 (extreme right) to 100LPH.
- Provide air supply to the regulator and adjust it to 2 kg/cm^2
- Remove entrapped air, if any from the FT2. For removing air open the vent valves on the DP transmitter.
- Click on Start|All programs|Wonderware|InTouch|Yes to open InTouch Appilcation manager.
- Open *"Multi Process trainer ADAM-4022T"*
- Click "Ignore" under "WindowMaker" for License not available
- Click "Select All" and "OK" under "Windows to Open"
- Click on "Runtime" at Top Right corner under "Intouch- WindowMaker"
- Click "Ignore" under "WindowViewer" for License not available
- Click "OK" under " WindowViewer" for Demo mode
- For selecting Feed Forward experiment: Click "Experiment On"; Click "Experiment off". Then Click " Feed Forward +feedback"
- Close the drain valve of the tank and close the Rotameter drain valve provided at the inlet of Rotameter-2
- Open Rotameter-2 and adjust flow to 75 LPH.
- From the default values of SP=50, PB=20, IT=30, DT=2, FF=1 observe the response of the system. Note for FF=0 the loop is purely feedback loop.
- With Feed forward factor =1 apply load disturbance by changing the output flow rotameter. Observe the effect of feed forward on LIC output and on process variable.

- Conduct the experiment without feed forward loop but with the same settings in the feedback loop and for same step change.

For Ratio control loop:
- If the tappings of orifice placed in the second inlet flow line to the process tank are short circuited by a PU tube loop, remove the loop. Connect pressure signal tappings of this orifice to FT2 with proper polarity.
- Short circuit the pressure tappings of orifice placed in the outlet line of the process tank. (i.e. at the inlet of the left rotameter).
- Start up the set up and adjust the Rotameter no. 3 (middle) and Rotameter-1 (extreme right) to 100LPH. Remove air entrapped, if any, from the FT2. For removing air open the vent valves on the DP transmitter.
- Provide air supply to the regulator and adjust it to 2 kg/cm^2
- Click on Start|All programs|Wonderware|InTouch|Yes to open InTouch Appilcation manager.
- Open *"Multi Process trainer ADAM-4022T"*
- Click "Ignore" under "WindowMaker" for License not available
- Click "Select All" and "OK" under "Windows to Open"
- Click on "Runtime" at Top Right corner under "Intouch- WindowMaker"
- Click "Ignore" under "WindowViewer" for License not available
- Click "OK" under " WindowViewer" for Demo mode
- For selecting Ratio Control: Click "Experiment On"; Click "Experiment off". Then Click " Ratio Control"
- Adjust Rotameter no.3 to @ 75 LPH.
- From the default values of PB=75, IT=8, DI=2, Ratio Set (RS)=1, observe that the flow in rotameter no.1 is automatically adjusted and become equal to that of rotameter no.3.
- Manipulate the flow in Rotameter no. 3 and observe the effect on Rotameter no.1.
- Change the ratio (range 0.5-2) and observe the effect.

6. Observations

Cascade control loop:
- Plot the responses with and without cascade on the same graph paper.
- It is observed that the speed of response of the level control system is improved. As the flow loop is fast responsive than the level control loop, for a small change in primary controller output, the secondary controller tries to achieve the set point.
- Time required to achieve the primary controller set point is less than that of single loop control.

Feed forward-Feedback Control Loop:
- Plot the responses with and without feed forward loop on the same graph paper.
- Because of the feed forward loop the controller responds immediately to the disturbance before the process value is affected. The final correction is done by PID control loop.
- Any error observed in the flow measurement (due to manufacturing inaccuracies or air in signal lines or DP calibration) has no impact on controlling.

Ratio Control:
- The ratio of controlled variable (Flow1) to wild variable (Flow2) can be set and Controlled.
- For a given RS, change wild variable and observe the value of controlled variable.

RS	CV	WV	RC

7. Model Calculations

8. Results and Discussion

9. Conclusions

10. Precautions

References

1. Instruction Manual for Multi Process Trainer 326A, Apex Innovations, Sangli, Maharastra, India.
2. Theory Process Control, Apex Innovations, Sangli, Maharastra, India.
3. Seborg, D. E., Edgar, T. F. and Mellichamp, D.A., "Process Dynamics and Control", 2nd Ed., John Wiley and Sons, 2004.
4. Stephanopoulos, G., Chemical Process Control – An Introduction to Theory and Practice, 6th edition, Prentice-Hall of India, New Delhi, India, 1993.
5. Coughanowr, D.R., Process Systems Analysis and Control, 2nd Ed., McGraw-Hill, 1991.

Experiment No. 20

FIRST ORDER AND SECOND ORDER SYSTEMS

1. Aim

Study of First-order and Second-order systems.

2. Objective

    i.   To study the step response of thermometer and thermo-well.

    ii.   To study the sinusoidal response of the thermo-well.

    iii. To study the step response of mercury- and water manometers.

3. Theory

First-order system: Thermometer and Thermo-well

A thermometer bulb is a first-order system, whose response can be described by a first order linear differential equation. The dynamic response of first-order type instruments to a step change can be represented by

$$T\frac{d\theta}{dt} + \theta = \theta_F$$

Where θ = Temperature indicated by thermometer

    θ_F = Final steady state temperature

    t = time

    T = time constant

The linear first order differential has the particular solution for given initial conditions,

$$\frac{\theta}{\theta_F} = 1 - e^{\frac{-t}{T}}$$

Which, represent a single exponential response as shown below:

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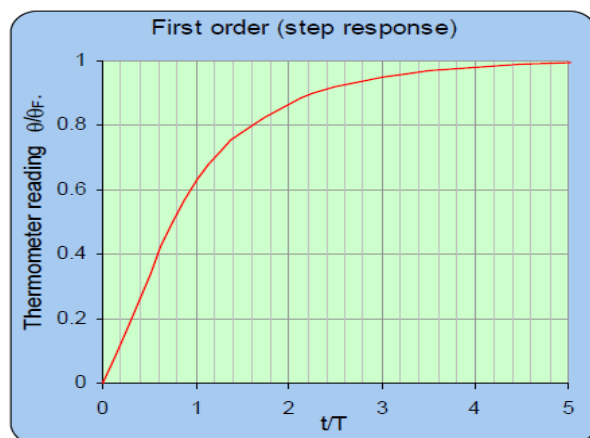
The time constant T is the time required to indicate 63.2% of the complete change. The time constant T is numerically equal to the product of resistance and capacitance.

In case of a <u>thermo-well</u> a thermometer as described above is added with additional resistance (thermo-well) at its bulb to increase its time constant. The system can generally be considered as first order system and response can be described as a first-order linear differential equation.

The dynamic response of first-order type instruments to a <u>sinusoidal change</u> can be represented by:

$$T\frac{d\theta}{dt} + \theta = A\sin\omega t$$

Where θ = Indicated Temperature

t = time

T = time constant

A = amplitude of cycle of measured variable

ω = circular frequency of cycle

A solution with given initial conditions and with the transient terms omitted is,

$$\left[\frac{\theta}{A}\right]_{SS} = \frac{1}{\sqrt{1+\omega^2 T^2}}\sin(\omega t - \phi)$$

Where ϕ = arctan ωT = lag angle and the subscript ss denotes steady state.

This equation shows that:

1) The output is a sine wave with a frequency ω equal to that of the input signal.
2) The instrument lags the measured variable by a geometric angle ϕ.

   Where $\phi = \tan^{-1}(\omega T)$

3) The amplitude is reduced or attenuated. The ratio of output amplitude to input amplitude is $\dfrac{1}{\sqrt{1+\omega^2 T^2}}$

To investigate the response of a first-order system to a sinusoidal forcing function, a response of thermo well will be considered in a sinusoidal temperature bath. The general response shall be as shown below:


139
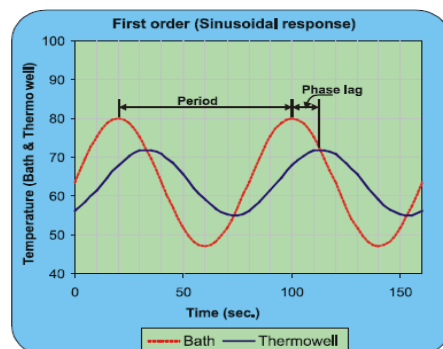
Second-order system: Manometer

The dynamic response of a second order system to a step change can be described by a second-order differential equation. The solutions to above equation involve three cases: an under damped condition [$\zeta < 1$], critical damped condition [$\zeta = 1$] and over damped condition [$\zeta > 1$]. The response for under damped system [i.e. $\zeta < 1$] can be written as:

…….1

Following figure shows response of second order system for different damping coefficient.
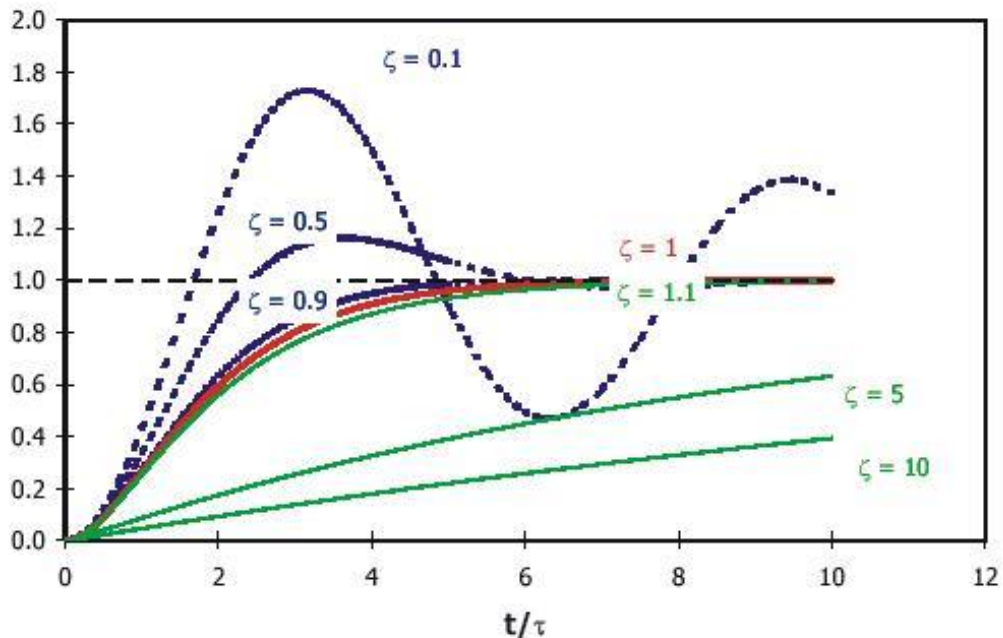
In case of manometer:

y(t) = response at any time t after step change (deviation value).

K= Gain factor =1

M= magnitude of step change

Damping coefficient …….2

Where,

L = Column length in meter,

μ = Dynamic viscosity in Kg/m.s.

ρ = Mass density of the manometer fluid in kg/m3,

D = tube diameter in m,

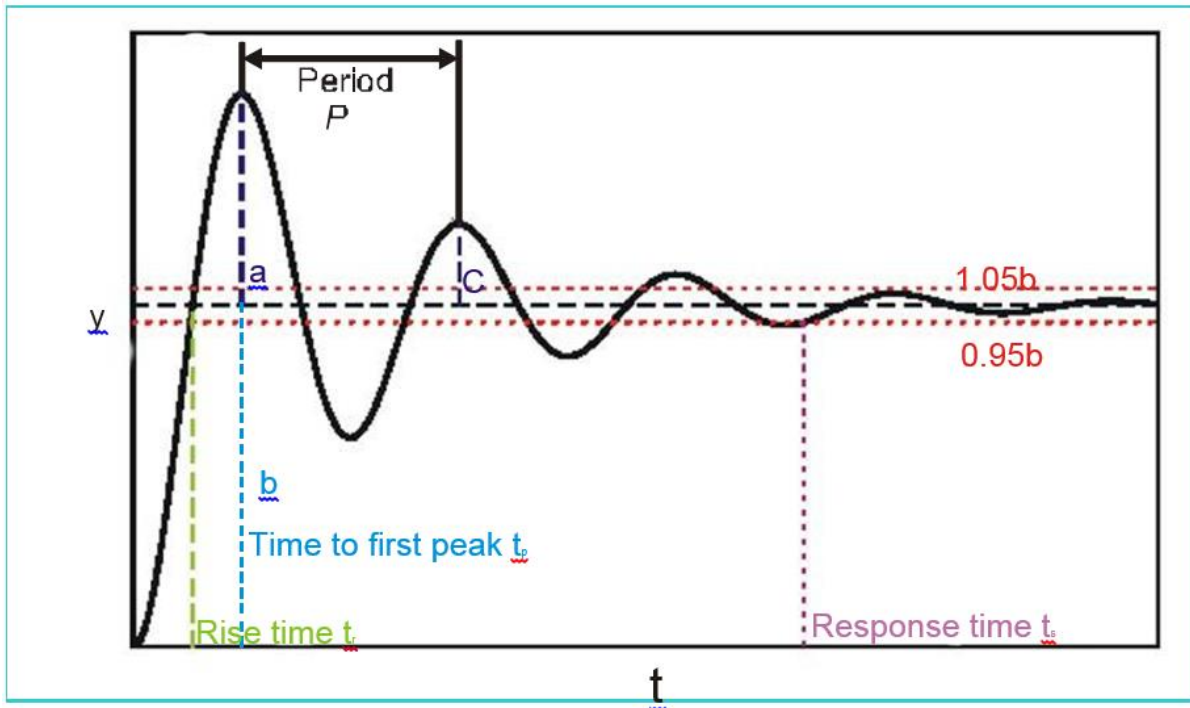g = Gravitational acceleration in m/sec^2

Characteristics time in secs   ……..3

Frequency of damped oscillation   in cps   ..…….4

Where Natural frequency in   rad/sec   ……….5

Performance characteristics for the step response of an under damped system is shown below

1. Rise time = t_r is the time the indicated value takes to first reach the new steady-state value.
2. Time to first peak = t_p is the time required for the indicated value to reach its first maximum value.
3. Response/settling time = t_s is defined as the time required for the indicated value to reach and remain inside a band whose width is equal to +/-5% of the total change in θ. The term 95% response time sometimes is used to refer to this case. Also, values of +/-1% sometimes are used.
4. Decay ratio (DR) = c/a (Where c is the height of the second peak).

..……………6

5. Overshoot (OS) = a/b = $\sqrt{decay\ ratio}$ ……………..7

6. Period of oscillation = P is the time between two successive peaks or two successive valleys of the response.

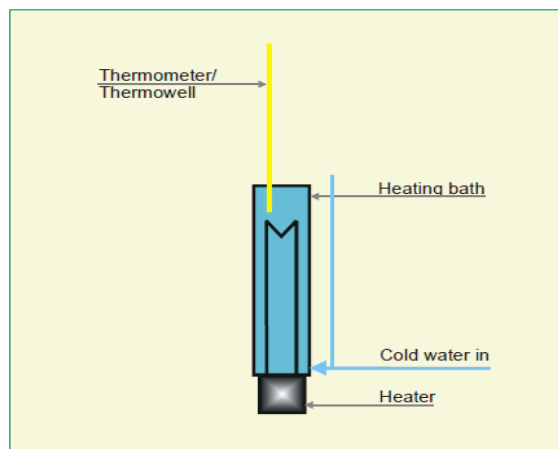..……………..8

4. Experimental Set-up

Setup consists of two 'U' tube manometers, heating bath, thermometer, thermo-well, beeper for recording observations and timer for heater on-off operation. The components are mounted on base plate. The set up is tabletop mountable.

5. Experimental Procedure

Step response of Thermometer/Thermo-well:
Prepare the set up as shown below.

- Fill the heating bath with clean water by opening the inlet valve of heating bath.
- Switch on beeper and set beep interval to 3 seconds.
- Ensue that cyclic timer is set to 30 seconds on time and 30 seconds off time. Switch on Mains to heat the water in heating bath to its boiling point. Switch off the mains.
- The water in heating bath is now near its boiling point. Insert the thermometer in heating bath suddenly after noting its initial temperature.
- Note the thermometer reading at each beep interval till the temperature reaches at steady state.
- Switch off beeper and fill up the readings observed in "Observations" below.

Sinusoidal response of Thermo-well:
Prepare the set up as shown below.
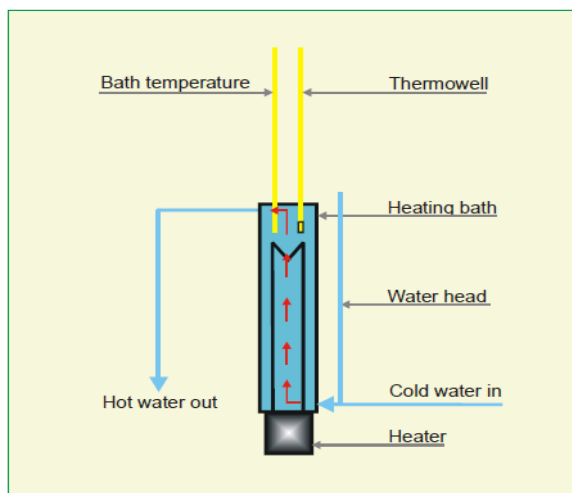
- Start the clean water supply by opening the inlet valve of heating bath and maintain constant water flow through the heating bath. Keeping constant level in "Water head" indication tube can ensure this.
- Insert the thermometer and thermo well in heating bath.

- Ensue that cyclic timer is set to @30 seconds on time and @30 seconds off time. Switch on Mains to heat the water in heating bath.
- After some time observe sinusoidal response of the heating bath temperature on thermometer. The amplitude (temperature range) can be changed by adjusting water flow rate and period can be changed by adjusting on time, off time of the cyclic timer. (Period = on time + off time)
- At steady state note amplitude ratio and phase lag (Refer observations)

**Step response of mercury manometer:**
Prepare the set up as shown below.
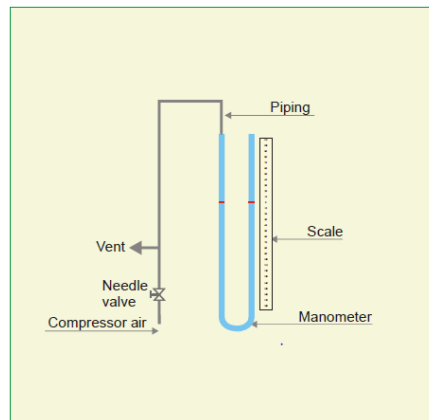
- Ensure that mercury level in manometer is set at '0' on the scale.
- Close vent connection by putting finger on it.
- Adjust the needle valve and vent to raise the mercury level to @200mm from '0' level.
- Note the mercury level reading and quickly open the vent to apply step change.
- Note the top peak and bottom peak readings. Also simultaneously note the period of oscillation. (This can be noted by measuring time required for 4-5 oscillations and then calculating for each oscillation)
- Repeat process 2-3 times for different step changes.

Step response of water manometer:
 Prepare the set up as shown below

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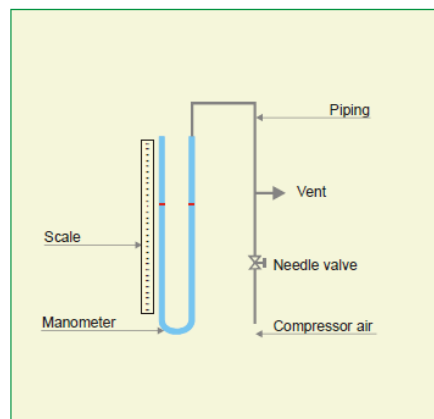
- Ensure that water level in manometer is set at '0' on the scale.
- Close the vent of water manometer by putting hand on it.
- Adjust the needle valve and vent to deflect the water column to @ 450mm from '0' level.
- Note the water level reading and quickly open the vent to apply step change.
- Note the top peak and bottom peak readings. Also simultaneously note the period of oscillation. (This can be noted by measuring time required for 4-5 oscillationsand then calculating for each oscillation)
- Repeat process 2-3 times for different step changes.

6. Observations

Step response of Thermometer/Thermowell:

1) Initial temperature (^{o}C)
2) Final temperature (^{o}C)

Sr. No.	Time (sec)	Actual Temperature(°C)
1.		
2.		
3.		
4.		

Sinusoidal response of Thermo-well:

1) Maximum bath temperature (^{o}C)   =
2) Minimum bath temperature (^{o}C)   =
3) Period of oscillation (sec)   =
4) Maximum thermo well temperature (^{o}C)   =
5) Minimum thermo well temperature (^{o}C)   =
6) Observed phase lag (sec)   =

Step response of Mercury Manometer:

Manometer fluid   = Mercury
Dynamic viscosity (μ)   = 0.0016 Kg/m.s.
Mass density (ρ)   = 13550 Kg/m3.
Column length (L)   = 0.760 m
Tube diameter (d)   = 0.005 meter
Step change (mm):   =
Period of oscillation (sec):   =

S.No.	Actual response*(mm)	Period of oscillation**(sec)
1		
2		
3		
4		

*: Note peak values observed during oscillations.
**: Measure the period of 4-5 oscillations and note average time required for each oscillation.

Step response of water manometer:

Manometer fluid   = water
Dynamic viscosity (μ)   = 0.001Kg/m.s.
Mass density (ρ)   = 998Kg/m^3

Column length (*L*)   = 1.050m

Tube diameter (*d*)   = 0.022 meter
Step change (mm):   =
Period of oscillation (sec): =

S.No.	Actual response*(mm)	Period of oscillation**(sec)
1		
2		
3		
4		

*: Note peak values observed during oscillations.
**: Measure the period of 4-5 oscillations and note average time required for each oscillation.

7. Model Calculations

Step response of Thermometer/Thermo-well:
1) *Step change = (Final temp. – Initial temp.)*
2) *Value of 63.2% of step = 0.632 x (Final temp. –Initial temp.) + Initial temp.*
3) Plot the graph of Actual temperature V_s time and note the value of time at 63.2% of step change. This value is observed time constant of the thermometer.
4) Calculate theoretically predicated temperature by following equation:
   Theoretical Temp. =

$$\text{Initial Temp.} + \left(\text{Step change} * \left(1 - \text{EXP}\left(\frac{-1*\text{Time}}{Time\, \text{constant}(\text{From Graph})} \right) \right) \right)$$

5) Plot the graph of Theoretical temperature V_s time on the same graph plotted above.

Sinusoidal response of Thermowell:

1) Input amplitude $= \dfrac{\text{Maximum bath temp.} - \text{Minimum bath temp.}}{2}$ °C

2) Frequency of oscillation $= \dfrac{2*\Pi}{\text{Peroid of oscillation}}$ Rad/sec

3) $\text{Output amplitude} = \dfrac{\text{Maximum thermowell temp.} - \text{Minimum thermowell temp.}}{2}$ °C

4) $\text{Amplitude ratio} = \dfrac{\text{Output amplitude}}{Input \text{ amplitude}}$

5) $Time \text{ constant from amplitude ratio} = \sqrt{\dfrac{1 - \text{Amplitude ratio}^{2}}{\text{Amplitude ratio}^{2} * \text{Frequency of oscillation}^{2}}}$ sec

6) $\text{Phase lag} = \dfrac{\text{Observed phase lag} * 360}{Period \text{ of oscillation}}$ Deg

7) $\text{Time constant from phase lag} = \dfrac{\text{Tan (Phase lag)}}{\text{Frequency of oscillation}}$ sec

8) Plot the graph of bath & thermo well temperature V_s time to note phase lag and amplitude ratio.

Step response of Mercury/Water Manometer:

- Calculate natural frequency of oscillations using equation no. 5
- Calculate damping coefficient using equation no. 2
- Calculate period of oscillations using equation no. 8
- Calculate decay ratio using equation no. 6
- Calculate overshoot using equation no. 7
- Calculate frequency of damped oscillations using equation no. 4
- Calculate characteristics time using equation no. 3
- Calculate theoretical response for different time values using equation no.1

Plot the graphs of Actual & Theoretical response V_s Time.

8. Results and Discussion

9. Conclusions

10. Precautions

References

1. Instruction Manual for First Order and Second Order Systems 320B, Apex Innovations, Sangli, Maharastra, India.
2. Theory Process Control, Apex Innovations, Sangli, Maharastra, India.
3. Seborg, D. E., Edgar, T. F. and Mellichamp, D.A., "Process Dynamics and Control", 2nd Ed., John Wiley and Sons, 2004.
4. Stephanopoulos, G., Chemical Process Control – An Introduction to Theory and Practice, 6th edition, Prentice-Hall of India, New Delhi, India, 1993.
5. Coughanowr, D.R., Process Systems Analysis and Control, 2nd Ed., McGraw-Hill, 1991.