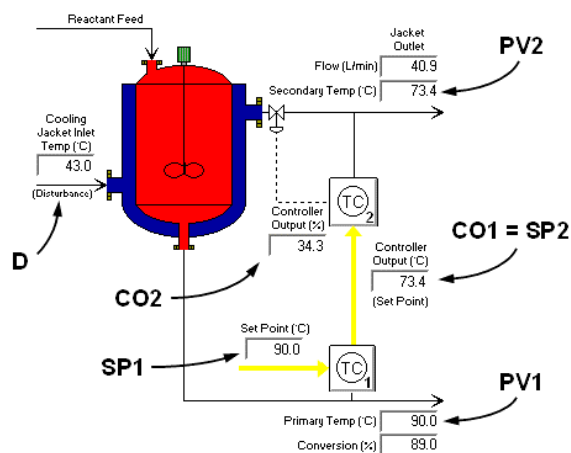


Laboratory Manual

For

Advanced Chemical Engineering Lab (CHC510)

AY: 2021-22



Department of Chemical Engineering
Indian Institute of Technology (ISM), Dhanbad

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Lab Safety Rules

Things to Do

- Be on time. Follow all written and verbal instructions carefully
- Conduct yourself in a responsible manner at all times in the laboratory
- Store your backpacks in the place mentioned by the Instructor and /or TA
- Familiarize yourself with all labs procedure before doing lab experiments
- Bring your lab notebook and an open mind to every lab
- Be aware of others in the lab. Areas of the room may be crowded at times and you should take care not to disturb the experiments of others in the lab
- Be aware of all the safety devices
- Dress properly during a laboratory activity. Personal protective equipment (PPE), such as gloves, safety glasses and lab coats, is an important factor in your safety when working in the laboratory. Proper PPE should be worn during laboratory experiments
- Keep clutter to a minimum. Work areas should be kept clean and tidy at all times
- Inform the instructor and/or TA if there is a problem. You will have their immediate attention if you have cut yourself (even if you consider it minor), if something broke and needs cleaning up, or if you are on fire
- Use extreme care when handling sharp objects
- Heated glassware remain very hot for a long time, picked up with caution. Use tongs or heat protective gloves if necessary
- Read the chemical safety information (MSDS).
- Keep all materials away from open flames
- Dispose of all chemicals, broken glass and other lab materials as directed
- Always be sure that electrical equipment is turned in the "OFF" position before plugging it into an electrical socket
- Always Turn OFF all electrical switches of the equipment before leaving laboratory
- Report ALL incidents, accidents, injuries, hazards or chemical spills to the instructor (no matter how trivial it seems)
- Wash your hands before you leave the lab for the day

Things Not to Do

- DO NOT eat, drink beverages, chew gum in the lab
- DO NOT touch any equipment, chemicals, or other materials in the laboratory area until you are instructed to do so.
- NEVER do any experiment on your own and NEVER work in the lab alone
- NEVER use electrical instrument around water
- DO NOT use the mobile
- NEVER smell, taste or touch chemicals
- DO NOT work with chemicals until you are sure of their MSDS that includes some awareness of their flammability, reactivity, toxicity, and disposal.
- NEVER return unused chemicals to the original container/bottle
- DO NOT casually dispose of chemicals down the drain
- NEVER use chipped, cracked, or dirty glassware.
- DO NOT panic
- NEVER leave the lab without washing your hands

EXPERIMENT NO. 01: FLOW BEHAVIOR OF COMPLEX FLUIDS

AIM: Study the physical properties of complex fluids flow behavior of surfactant /polymer solutions for industrial application.

OBJECTIVES:

- ❖ *To prepare surfactant/polymer systems and determine the physical properties of these polymeric/surfactant systems.*
- ❖ *To evaluate rheological flow models for polymeric/surfactant systems.*

Chemical/apparatus required- Rheometer, sample (surfactant/polymer solutions), distilled water

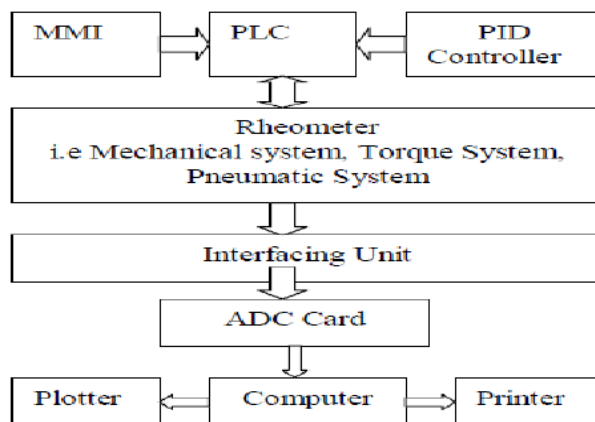
THEORY:

Science of flow and deformation of matter (liquid or solid) under the effect of an applied force. A rheometer is a lab device used to measure how liquid suspension or slurry flows in response to applied forces. It is used for those fluids which cannot be defined by a single value of viscosity and therefore require more parameters to be set and measured than is the case for a viscometer. It measures the rheology of the fluid. There are two distinctively different types of rheometers. Rheometers that control the applied shear stress or shear strain are called rotational or shear rheometers, whereas rheometers that apply extensional stress or extensional strain are extensional rheometers. Rotational or shear type rheometers are usually designed as either a native strain-controlled instrument (control and apply a user-defined shear strain which can then measure the resulting shear stress) or a native stress-controlled instrument (control and apply user-defined shear stress and measure the resulting shear strain). A rheometer is a device used to measure the

rheological properties of materials; rheology being defined as the study of the flow and deformation of matter, which describes the interrelation between force, deformation and time.

Unlike a viscometer, which can only measure the viscosity of a fluid under a limited range of conditions, a rheometer is capable of measuring viscosity and elasticity of non-Newtonian materials under a wide range of conditions. Some of the most important properties that can be measured using a rheometer include viscoelasticity, yield stress, thixotropy, extensional viscosity, creep compliance and stress relaxation behaviour, as well as process-relevant parameters such as die swell, melt fracture.

In general, either the fluid remains stationary and the object moves through it. or the object is stationary, and the fluid moves past it. the drag caused by the relative motion of fluid and surface is a measure of viscosity. The flow conditions must have viscosity value of Reynolds number less than 2100 for laminar flow. Rotational viscometer uses the idea that torque required to turn on an object in a fluid is a function of the viscosity of that fluid. They measure the torque required to rotate a disk or bob in a fluid at a known speed.



Block diagram of Rheometer.

PRINCIPLE:

The fluid is sheared in a narrow gap between concentric cylinders, parallel plates or a cone and a plate. The viscosity is defined as the ratio of the shear stress and the shear rate. The stress is related to the torque and the shear rate to the angular velocity. One of these quantities is preselected the other is measured, depending on the set-up of the instrument (controlled stress / controlled strain). A great variety of tools is available to cover a large viscosity and shear rate range. Elastic material-functions can be obtained from axial forces acting perpendicular to the plane of shear in cone-plate or plate-plate geometry. Rotational rheometers allow for numerous experimental modes controlling either shear rate, shear deformation or shear stress.

PROPERTIES WHICH CAN BE MEASURED:

1) Dynamic viscosity	6) temperature
2) Shear rate	7)time
3) Shear stress	8)kinematic viscosity
4) Speed	9)fixed point
5) Torque	10)deformation



Rotational Rheometer

PROCEDURE:

- Turn on the rheometer/viscometer.
- Turn on the thermostat water bath. The setpoint should be between 20 to 30°C (the bath is used only as a thermal reservoir).
- Turn on computer, start the rheoplus program on computer.
- In toolbar press the icon “device”, which is a symbol of “rheometer”.
- In the control panel tab in the new window, press initialize to start.
- Put your desired tool.
- In control panel tab, press “set zero gap”.
- Set “default program”.
- Press “start” to begin adjustment.
- In the control panel tab, lift position high enough to put your sample (60 mm recommendable).
- Put your sample on plate.
- Set temperature.
- In the control panel tab, lift position.
- Press measuring position to go to measuring distance.
- Remove entered sample.
- The status column in your data table is good indication of reliability of measurement.

After measurements are done:

- ✓ Lift the position to clean the plate.
- ✓ Clean the plate with distilled water or organic solvents (if needed).
- ✓ Exit rheoplus program.
- ✓ Take off the tool and clean it with water.
- ✓ Turn off device, thermostat bath and the computer.

OBSERVATION TABLE

S.NO	Time	Viscosity	Shear rate	Shear stress	torque

RESULT/DISCUSSION:

OBSERVATION:

QUESTIONNAIRE:

Q1. What is the function of Rheometer?

Q2. What are rheological flow behaviour fluids?

Q3. Describe the working procedure of Rheometer?

EXPERIMENT NO. 2: MEASUREMENT OF FLUID FLOW

INTRODUCTION:

Flow measurement is an important topic in the study of fluid dynamics. It must be made in chemical plants, refineries, power plants, and any other place where the quality of the product or performance of the plant depends on having a precise flow rate. Flow measurements also enter into our everyday lives in the metering of water and natural gas into our homes and gasoline into our cars.

There are many instruments used in flow measurements. In this experiment, we are going to use the following devices:

1. Venturi.
2. Orifice plate.
3. Rotameter.

OBJECTIVES:

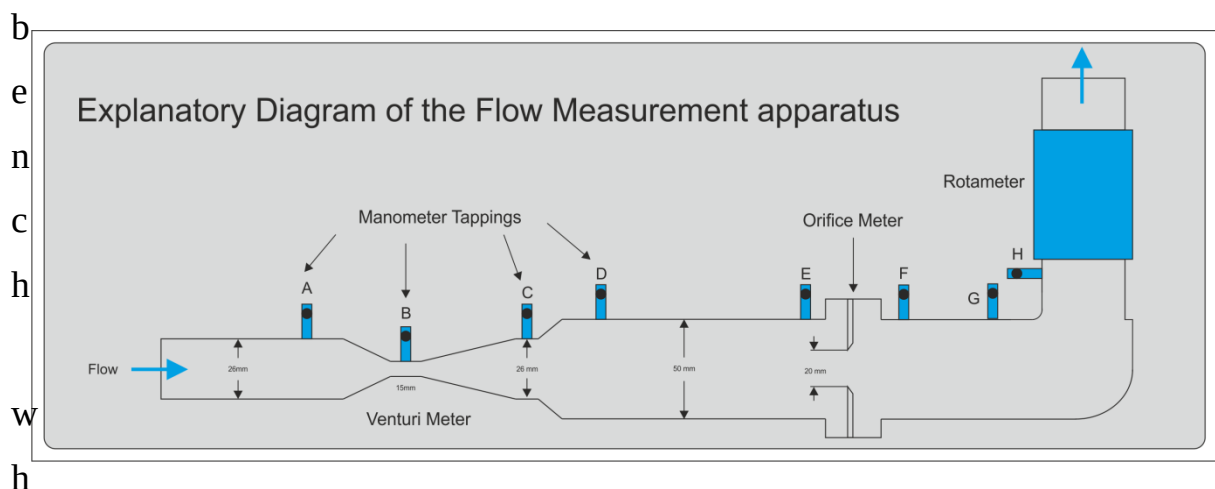
This experiment aims to:

1. Familiarize students with some common devices and methods used in measuring flow rate.
2. Each flow measurement device will be compared to the standard method of using the catch-tank and stopwatch to measure the flow rate.
3. Determine the energy loss incurred by each of these devices.
4. Determine the energy loss arising in a rapid enlargement and a 90° elbow

APPARATUS:

The Flow Measurement apparatus. Water from the Hydraulic Bench enters the equipment through a Venturi meter, which consists of a gradually converging section, followed by a throat, and a long gradually diverging section. After a change in cross-section through a rapidly diverging section, the flow continues along a settling length and through an orifice meter. This is made from a plate with a hole of reduced diameter through which the fluid flows. The water then continues around a bend and up through a rotameter-type flowmeter. The rotameter is a transparent tube of gradually diverging cross section in which the “float” takes an equilibrium position; the vertical position of the float is a measure of the flow rate.

After the rotameter, the flow returns via a control valve to the hydraulic



ere the flow rate can be measured using the catch-tank and a stopwatch.

THEORY:

For steady, adiabatic flow of an incompressible fluid along a stream tube, as

shown in figure (3), Bernoulli's equation between points 1 and 2 can be written in the form:

$$\frac{P_1}{\rho g} + \frac{V_1^2}{2g} + z_1 = \frac{P_2}{\rho g} + \frac{V_2^2}{2g} + z_2 + \Delta H_{12} \text{-----(1)}$$

Where:

$$\frac{P}{\rho g} = \text{Hydrostatic Head}$$

$$\frac{V^2}{2g} = \text{Kinetic Head, (V is the mean velocity; } V = \frac{\text{volumetric flow rate}}{\text{cross sectional area of tube}} = \frac{Q}{A}$$

$$z = \text{Potential Head}$$

$$\frac{P}{\rho g} + \frac{V^2}{2g} + z = \text{Total Head}$$

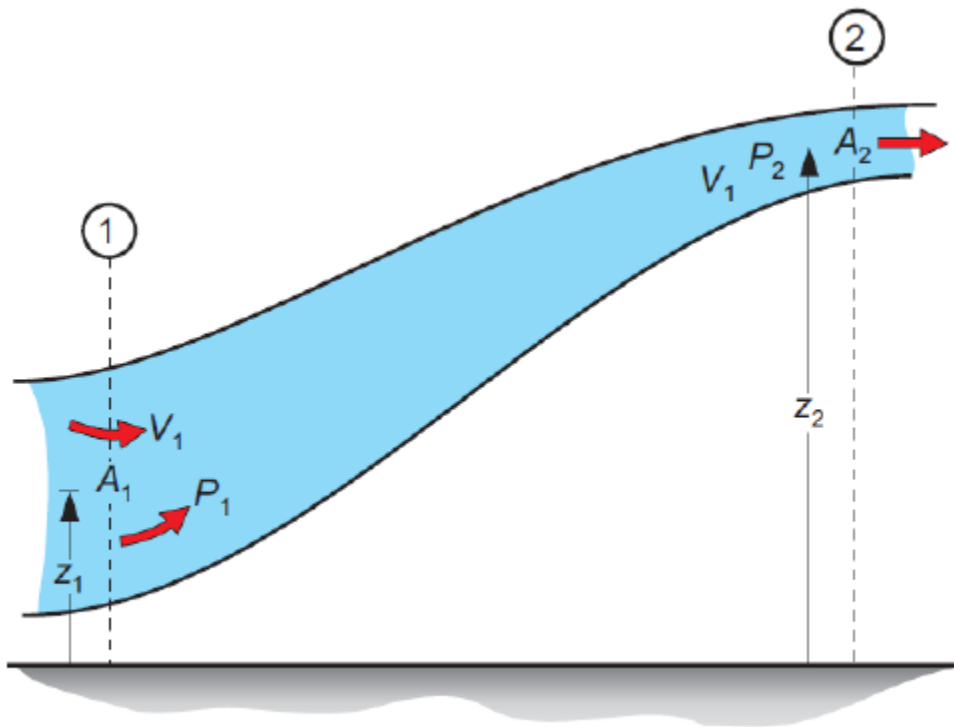


Figure 3: The steady flow energy equation

The head loss H_{12} may be assumed to arise as a consequence of the vortices in the stream. Because the flow is viscous a wall shear stress exists and a pressure force must be applied to overcome it. The consequent increase in flow

work appears as an increase in internal energy, and because the flow is viscous, the velocity profile at any section is non-uniform.

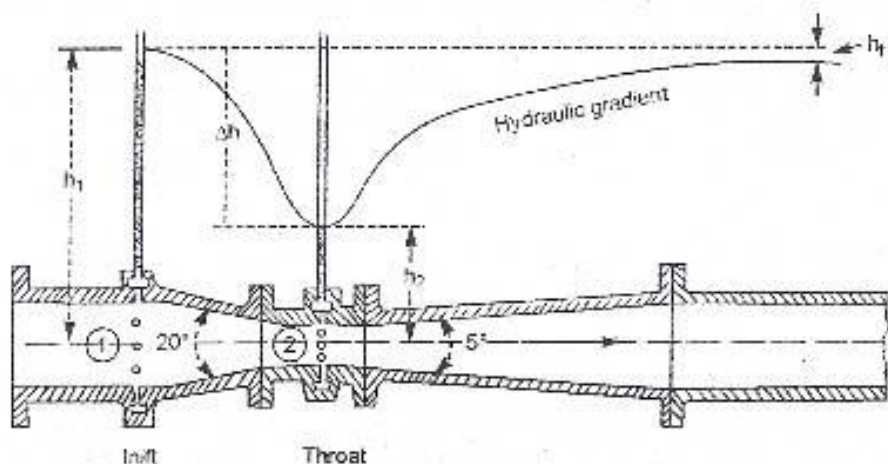
FLOW MEASUREMENT:

1. Ventury meter:

A Venturi Meter is a device used for measuring the rate of a flow of a fluid flowing through a pipe. It consists of three parts:

- (i). A short converging part
- (ii). Throat
- (iii). Diverging part

A Venturi Meter apparatus is based on Principle of Bernoulli's equations. The principle of apparatus is that, by reducing the cross section area of flow passes, pressure difference is created and the measurement of pressure difference enables the determination of the discharge through the pipe.



A Venturi meter consists of an inlet section followed by a convergent cone, a cylindrical throat and a gradually divergent cone.

By knowing the pressure difference across the inlet section and the throat, discharge can be calculated by the formula:

$$Q_a = C_d * \frac{a_1 a_2 \sqrt{2gh}}{\sqrt{a_1^2 - a_2^2}}$$

Consider a venturimeter fitted in a horizontal pipe through which a fluid is flowing (say water), as shown:

Let d_1 = diameter at inlet

p_1 = pressure at inlet

v_1 = velocity of fluid at inlet

a_1 = area at inlet section = $\frac{\pi}{4} d_1^2$

And d_2, p_2, v_2, a_2 are corresponding values at throat section (2).

Applying Bernoulli's equation at both the inlet and throat section, and considering the pipe to be horizontal,

The theoretical discharge Q is,

$$Q_t = \frac{a_1 a_2 \sqrt{2gh}}{\sqrt{a_1^2 - a_2^2}}$$

$$Q_{act} = C_d * Q_t$$

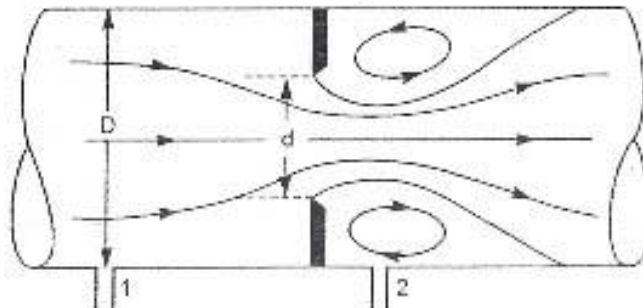
Where, C_d = Co-efficient of venturimeter and its value is less than 1.

Such that the diameters (bores) of the meter at (A) and (B) are 26 mm and 15 mm respectively.

2. Orifice meter:

An Orifice Meter is also a device used for measuring the rate of flow of a fluid through a pipe. It also works on the same principle as that of venturi meter. It consists of a flat circular plate which has a circular sharp edged hole called orifice, which is concentric with the pipe. The orifice diameter is kept generally 0.5 times the diameter of the pipe, though it may vary from 0.3 to 0.8 times the pipe diameter.

The principle of apparatus is that, by reducing the cross section area of flow passes, pressure difference is created and the measurement of pressure difference enables the determination of the discharge through the pipe.



An orifice meter is another device used for measuring the discharge through pipes. Orifice meter works on the same principle as that of venturimeter i.e. by reducing the cross sectional area of the passes a pressure difference between the two sections is developed and the measurement of the pressure difference enables the determinations of the discharges through the pipe.

An orifice meter consists of a flat circular plate with a circular hole called orifice, which is concentric with the pipe axis. The discharge through the orifice meter can be calculated by the formula

$$Q_{act} = C_d * \frac{a_1 a_2 \sqrt{2gh}}{\sqrt{a_1^2 - a_2^2}}$$

$$Q_{act} = C_d * Q_t$$

Consider an orificemeter fitted in a horizontal pipe through which a fluid is flowing (say water), as shown:

Let d_1 = diameter at inlet

p_1 = pressure at inlet

v_1 = velocity of fluid at inlet

a_1 = area at inlet section = $\frac{\pi}{4} d_1^2$

And d_2, p_2, v_2, a_2 are corresponding values at downstream section (2).

Applying Bernoulli's equation at both the upstream and downstream section, and considering the pipe to be horizontal,

The theoretical discharge Q_t is,

$$Q_t = \frac{a_1 a_2 \sqrt{2gh}}{\sqrt{a_1^2 - a_2^2}}$$

$$Q_{act} = C_d * Q_t$$

Where, C_d = Co-efficient of orificemeter and its value is less than 1.

With the apparatus provided, the diameter (bore) at (E) is 50mm. At (F), the water diameter (not the bore) is 20 mm

3. Wide-angled diffuser:

The inlet to the diffuser may be considered to be at (C) and the outlet at (D).
Applying equation (1):

$$\frac{P_G}{\rho g} + \frac{V_G^2}{2g} = \frac{P_H}{\rho g} + \frac{V_H^2}{2g} + \Delta H_{GH} \text{ -----(10)}$$

4. Right-angled bend:

The inlet to the bend is at (G) where the pipe bore is 51.9 mm and outlet is at (H) where the bore is 40 mm. Applying equations (1):

$$\frac{P_G}{\rho g} + \frac{V_G^2}{2g} = \frac{P_H}{\rho g} + \frac{V_H^2}{2g} + \Delta H_{GH} \text{ -----(11)}$$

The outlet kinetic head is now 2.8 times the inlet kinetic head.

STANDARD DATA:

Venturimeter upstream diameter	26mm
Venturimeter converging Angle	14 degree
Venturimeter Throat diameter	15mm
Venturimeter diverging Angle	21 degree
Pipe diameter	26mm
Sudden enlargement pipe diameter	50mm
Orifice diameter	20mm
Density of water ρ_w	1000 kg/m ³

EXPERIMENTAL PROCEDURE:

Part 1: Flow measurements and head losses

1. Make sure the air purge valve is closed.
2. Close the control valve of the flow measurement apparatus fully, then open it by about 1/3.
3. Switch on the hydraulic bench pump
4. Slowly open the hydraulic bench valve until water starts to flow. Allow the flow measurement apparatus to fill with water
5. Open the bench valve fully, and then close the control valve of the flow measurement apparatus
6. Open the valve of the flow measurement apparatus (**1/4 turn ONLY**)
7. Connect the hand pump to the air purge valve and pump until all the manometers read approximately 330 mm

8. Dislodge any entrapped air from the manometers by gentle tapping with the fingers
9. Check that the water levels are constant. The levels will rise slowly if the purge valve is leaking
10. Open the apparatus valve until the rotameter shows a reading of approximately 10 mm.
11. When a steady flow is maintained measure the flow with the hydraulic bench as follows:
 - a. Direct the outlet of your experiment to the volumetric tank
 - b. Start your stopwatch as soon as the water level in the volume indicator reaches 0.
 - c. Stop your stopwatch when the level in the volume indicator reaches 10
12. Record the readings of the manometers in table (1)
13. Repeat steps 10-12 for 10 tests. Increase the opening of the apparatus valve such that the rotameter reading is increased in steps of 10 mm.

OBSERVATION TABLE:

		1	2	3	4	5	6	7	8
	A								
	B								
Manometer Levels	C								
	D								
	E								
	F								
	G								
	H								
Rotameter (cm)									
Water W (kg)									
Time T (seconds)									
Mass Flow Rate m (kg/s)	Venturi								
	Orifice								
	Rotameter								
	Weight Tank								
ΔH /Inlet Kinetic Head	Venturi								
	orifice								
	Diffuser								
	Elbow								

CALCULATIONS

- Actual discharge,

$$R = \frac{R_1 - R_2}{100}, m = \text{-----}m$$

$$Q = \frac{A \times R}{t} m^3 / \text{sec} = \text{-----}m^3/\text{sec}$$

- Head losses m of water, (H_f)

$$h = \frac{h_1 - h_2}{100}, m = \text{-----}m$$

$$H_f = h \left(\frac{\rho_m}{\rho_w} - 1 \right), m = \text{-----}m$$

- Cross- section area, m^2

$$a = \frac{\pi}{4} d^2, m^2 = \text{-----}m^2$$

- Velocity of fluid/s

$$V = \frac{Q}{a}, m / \text{sec} = \text{-----}m/\text{sec}$$

NOMENCLATURE:

A	=	Area of measure tank, m ²
V	=	velocity of fluid/s
R	=	rise of water level in measuring tank, m
R ₁	=	final level of water in measuring tank, cm
R ₂	=	initial level of water in measuring tank, cm
h	=	manometer difference, m
h ₁ , h ₂	=	man metric reading at both points, cm
h _f	=	head losses, m of water.
Q	=	discharge, m ² /s
T	=	time taken for r, sec
ρ _w	=	density of water,

PRECAUTION AND MAINTENANCE INSTRUCTIONS:

1. Never run the apparatus if power supply is less than 180 volts & above 230volts
2. Never fully close the delivery valve & by-pass valve at a time.
3. To prevent clogging of moving parts, run pump at least once in a for night.
4. Always use clean water.
5. If apparatus is not in use for more than one month, drain the apparatus completely.
6. Always keep the apparatus free from dust.

TROUBLESHOOTING:

1. If pump gets jam, open the back cover of pump & rotate the shaft manually.
2. If pump gets heat up, switch OFF the main power for 15 min & avoid closing the flow control valve at a time during operation.

REFERENCES:

1. Ychengel j. cembala, “fluid mechanics”, 2nd ed. Mc grew hill, ND,2007, page 329.
2. Dr.P.N.Modi & P.N.seth, “Hydraulics & fluid mechanics including mechanics,”15th ed., rajinder Kumar Jain, ND, 2005, page 458-459.

EXPERIMENT NO. 3: DETERMINATION OF THE HEAT TRANSFER COEFFICIENT IN DROP WISE & FILM WISE CONDENSATION

OBJECTIVE: To study heat transfer in the process of condensation.

AIM: To find heat transfer coefficient for drop-wise & film-wise condensation process.

INTRODUCTION:

In all applications, the steam must be condensed as it transfers heat to a cooling medium, e.g. Cold water in the condensers of a generation station, hot water in a heating clarifier, sugar solution in a sugar refinery etc. During condensation very high heat fluxes are possible and provided the heat can quickly transferred from the condensing surface to the cooling medium, heat exchangers using steam can be compact and effective.

THEORY:

Steam may condense on a surface in two distinct modes, known as film wise and drop wise condensation. For same temperature between the steam and surface dropwise condensation is much more effective than film wise condensation and for this reason, the former is desirable although in practical plants, it rarely occurs for prolonged period.

Film wise condensation: Unless specially treating, most materials are wet table and as condensation occurs as film condensate spreads over the surface. The thickness of the film depends upon a number of factors, e.g. the rate of condensation, the viscosity of the fluid, the surface orientation and nature.

Fresh vapor condenses on to the outside of the film and heat is transferred by conduction through the film to metal surface beneath, as the film thickness increases it flows downward drips from the low points leaving the film intact and at an equilibrium thickness. The film of liquid is a barrier to the heat transfer and

its resistance accounts for most of the difference between the effectiveness of the film wise and drop wise condensation.

Drop-wise condensation: By specially treating the condensing surface, the contact angle can be changed and the surface become not wettable. As steam condense, a large number of generally spherical beads cover the surface. As condensation proceeds, the beads become larger, coalesce, and then strike downwards over the surface. The moving bead gathers on the static beads along its downward in its tail. The bare surface offers very little resistance to the transfer of heat and very high heat fluxes are therefore possible. Unfortunately, due to nature of material used in construction of condensing heat exchangers, filmwise condensation is normal.

EXPERIMENTAL SETUP:

The equipment consists of a metallic container in which steam generation takes place. The lower portion houses suitable electric heater for steam generation. A cover is provided for filling water. The glass cylinder houses two water cooled copper condensers, one of which is chromium coated to promote drop wise condensation and other is in its natural state to give film wise condensation. A pressure gauge is provided to measure steam pressure. Separate connections of two condensers for passing water are provided. One Rota meter is provided to measure flow rate of water through the condenser. A multi-channel digital temperature indicator is provided to measure temperature of steam, condensers, and water inlet and outlet from condenser.

SPECIFICATIONS:

1. Condensers- one chromium plated for drop wise condensation and one having natural finish for film wise condensation. Both condensers are identical in construction. Dimensions:

Outer diameter: 20.4 mm

Inner diameter: 20 mm

Length: 150 mm

Material: Copper

2. Main unit: M.S. fabricated construction comprising of test section and steam generator. Test section is provided with glass cylinder for visualization of the process. It also houses Rota meter and necessary piping and valves to connect any one condenser in operation
3. Steam generator: Suitable for above experiment with electric heater.
4. Instrumentation:
 - a) Multy-channel digital temperature indicator with cold junction compensation with thermocouples. Range 0 to 300°C.
 - b) Rota meter to measure flow rate of water flowing through condenser. Range 10 to 100 Lph.
 - c) Pressure gauge to measure steam pressure. Dial type, range 0 to 2.1 kg/cm²
5. Control panel: Separate control panel to house temperature indicator, necessary switches, steam generator etc.

PROCEDURE:

- 1) Fill up the water in steam generator by opening the valve
- 2) Switch ON the heater switch.
- 3) After filling the water close to the top cover of steam generator. Start water flow through one of the condensers which is to be tested and note down the water flow rate in Rota meter. Ensure that during measurement, water is flowing only through the condenser under test.
- 4) Slowly steam generation will start in the steam generator of the unit and as the steam rises to the test section, gets condensed and falls down in the cylinder.

- 5) Depending upon type of condenser under test drop wise or film wise condensation can visualized.
- 6) If the water flow rate is low then steam pressure is changed then condensation will occur at more or less atmospheric pressure.
- 7) Observations like temperature, water flow rate, pressure, are noted down in the observation table at the end of each test.

FORMULAE:-

Heat loss from steam : $Q_s = M_s \cdot \theta$

Heat taken by cold water: $Q_w = M_w \cdot C_p \cdot (T_5 - T_4)$

Average heat transfer: $Q = (Q_s + Q_w) / 2$

Inside heat transfer coefficient: $H_i = Q / (A_i \cdot \Delta T_m)$

Outside heat transfer coefficient: $H_o = Q / (A_o \cdot \Delta T_m)$

Experimental overall heat transfer coefficient: $1/U_{ex} = 1/h_i + (D_i/D_o) \cdot (1/h_o)$

Nusset number: $Nu = 0.023 (Re)^{0.8} (Pr)^{0.4}$

Theoritically heat transfer coefficient: $1/U_{th} = 1/h_i + (D_i/D_o) \cdot (1/h_o)$

OBSERVATION TABLE:

	Sl.No.	Water flow rate (lph)	Steam condensed (ml)	Time (min)	Temperature						
					T ₁	T ₂	T ₃	T ₄	T ₅	T ₆	T ₇
Film wise	1										
	2										

Drop	1										
wise	2										

RESULTS: Do calculations and report all the results that you have obtained in your experiment and write down the discussions in your journal.

CONCLUSIONS: Compare the experiment heat transfer coefficient that you have obtained for different conditions.

PRECAUTIONS:

1. Do not start heater supply unless water is filled in the steam generator.
2. Operate all the switches and controls gently.
3. Never allow steam to enter the cylinder unless the water is flowing through condenser.
4. Always ensure that the equipment is earthed properly before switching on the supply.

QUESTIONS:

1. What is condensation? How does it occur?
2. What is the difference between film and drop wise condensation? Which is a more effective mechanism of heat transfer?
3. Consider film condensation on a vertical plate. Will the heat flux be higher at the top or at the bottom of the plate? Why?

EXPERIMENT NO. 4: PERFORMANCE OF A CALANDRIA EVAPORATOR

OBJECTIVE: To concentrate the solution carbonate using Calandria evaporator.

AIM: To determine an overall heat transfer coefficient for the evaporator.

INTRODUCTION:

Evaporation deals with the concentration of a non-volatile solute from a solution by the removal of required amount of volatile solvent. Usually the solvent is water. By vaporizing a part of the solvent, useful product i.e. the concentrated solution or thick liquor is produced and the vapor is discarded. This is usually done before crystallizing or drying the material, or to reduce the volume for transport or storage.

THEORY:

For heat transfer from the condensing steam to the boiling solution, the overall heat transfer coefficient is found from the equation:

$$Q = UA\Delta T$$

The temperature difference ΔT is the difference between the temperature at which the steam condenses at the shell side pressure, and the temperature at which the solution boils at the tube side atmospheric pressure.

$$\Delta T = T_s - T_w$$

The area could be taken as either the inside or outside surface area of the tubes. The inside area is chosen because the greatest resistance to heat transfer is on this side. The area of the central downcomer is included, but the area of the tube plates is excluded.

$$A = n(\pi D l)_{tube} + (\pi D l)_{downcomer}$$

Heat flow from the steam to solution:

The heat flow is expressed in Watts (W) and calculated from an energy balance on

the feed and the two outgoing streams which are:

Product stream i.e. a saturated hot liquid phase leaving the evaporator.

Condensate i.e. a saturated vapor leaving the evaporator, then being condensed and cooled.

Then the steady state energy balance (neglecting the losses) is:

$$Q = w_p h_p + w_v h_v - w_f h_f$$

Q will be in Watts and corresponding units of overall heat transfer coefficients will be, $W/m^2 K$

Material balance:

At steady state, you would expect: $w_f = w_p + w_v$ (this should check out within $\pm 5\%$)

EXPERIMENTAL SETUP:

Present evaporator is a vertical short tube type evaporator with stainless steel tubes. A central downcomer is provided. The feed solution (to be evaporated) is fed near the top end of the tubes, flows down through the downcomer and rises through the tubes due to density difference. It circulates up through the tubes by natural convection, and some of the water is vaporized in the tubes. The liquid and vapor separate in the vapor space above. The vapor is passed through the condenser and collected as vapor condensed at the end of the condenser, while liquid returns through the central downcomer to the bottom space before again rising through the tubes. The water level in the evaporator is maintained nearly at constant level by supplying a continuous feed and adjusting the product flow at the bottom of the evaporator. The amount of water evaporated is estimated by measuring the rate of water collected at the base of the rate of water collected at the base of the condenser.

The heat used to evaporate the water, comes from steam from the steam

boiler provided at the base of the assembly. It enters the (shell side) space around the tube bundle where it loses heat, condenses and collects at the bottom of the shell space. The condensate drains out through a steam trap (which permits condensed steam but not the live steam to pass through it). Flow rate of feed is recorded from the readings of Rota meter in LPH. Bottom product flow can be obtained with the help of measuring cylinder and stop watch. Densities of feed and product are measured by using specific gravity bottles at ambient conditions.

Sensor positions are as follows:

T_1 = Steam inlet temperature

T_2 = Feed inlet temperature

T_3 = Liquid temperature in vessel

T_4 = Temperature of vapor entering to condenser

T_5 = Condensed steam temperature

T_6 = Water inlet temperature

T_7 = Water outlet temperature

UTILITIES REQUIRED:

Water supply 5 LPM and drain

Electricity supply: 1 Phase, 220 V AC, 6 kW

Chemicals: Na_2CO_3

EXPERIMENTAL PROCEDURE:

1. Fill the feed tank with solution (~ 5 wt %) up to $\frac{3}{4}$ height.
2. Switch on the power supply to the steam generator (keep steam supply closed) and attain the desired steam pressure.
3. Introduce the feed in to the evaporator by opening the Rota meter needle valve at the maximum. Fill the evaporator.

4. Direct the steam into the shell side of the evaporator by opening the steam valve. Heat the liquid in the evaporator till observes boiling in the glass-viewing window. Adjust the steam rate at the desired level in the steam jacket. Now allow fresh feed to enter by opening the feed valve and adjusting the feed rate at the desired level.
5. Open the product valve and adjust the flow rate of product such that the liquid level in the evaporator remains steady by observing it through front window glass.
6. Wait till steady state is achieved. (Temperature and flow rates do not vary with time). It may take about one hour.
7. During this one hour record the following at 15 minutes interval:
Product density, product concentration, weight of water vapor evaporated (w_v), feed rate, product rate, boiling temperature of liquid in the evaporator (T_w), condenser water outlet temperature (T_{wo}), cooling water flow rate to the condenser (F_{wc}), shell side steam pressure gauge reading (P_s).

CLOSING PROCEDURE:

1. Close the inlet feed valve.
2. Close the steam supply by switching off the electric supply to the steam generator.
3. Wait till residual vapor is condensed.
4. Drain Calandria, concentrated and condensate receivers.
5. At last close the cooling water supply to the condenser.

SPECIFICATION:

Evaporator: Material Stainless Steel, OD-30cm, with central down take of

10cm Dia,

Feed Tank: Material Stainless Steel, Capacity 20 Ltrs

Flow measurement: Rotameter 2 Nos. (One each for feed and cooling water)

Feed Circulation: By compressed air (optional: using mini compressor)

Steam Generator: Made of Stainless Steel provided with Pressure & Level Gauge, Safety valve and drain etc. & insulated with ceramic wool and cladding with Aluminum foil.

Piping: Stainless Steel and PVC, size 1/4"

Condenser: Shell & Tube type made of Stainless Steel

Product tank: Made of Stainless Steel, capacity 10 Ltrs.

Heater: Nichrome wire heater.

Water supply tank: Made of Stainless Steel, capacity 100 Ltrs. (for condenser).

Pump: FHP capacity

Temperature Sensors: RTD PT-100 type

Control panel of:

Digital Temperature Controller: 0-199.9°C (For Steam Generator)

Digital Temperature Indicator: 0-199.9°C with multi-channel switch

On/Off switch, Mains Indicator etc.

FORMULAE:

1. Mass flow rate of feed: $W_F = w_f \times \rho_f$, g/s
2. Mass flow rate of product: $W_P = w_p \times \rho_p$, g/s
3. Mass flow rate of condensed vapor: $W_V = w_v \rho_v$, g/s
4. Heat transfer area: $A = n(\pi D l)_{tube} + (\pi D l)_{downcomer}$, m²

5. Heat transfer coefficient: $U = \frac{Q}{A \times \Delta T}, W/m^2 \text{ } ^\circ\text{C}$

6. Heat balance: $Q = w_p h_p + w_v h_v - w_f h_f$

7. Mass balance: $w_f = w_p + w_v$

8. Economy = Amount of water vaporized (kg) / Amount of steam used (kg)

OBSERVATIONS:

Data:

Number of tubes in Calandria, n =

Outer diameter of tube =

Inner diameter of tube =

Tube length =

Downcomer outer diameter =

Calibration curve for sugar solution at 30 °C

Conc. (% w/v)	0	10	12	14	16	18	20	25	30
Density g/ml									

Plot density vs concentration on a simple graph and obtain a straight line.

Feed density =

Feed concentration from the calibration curve =

OBSERVATION TABLE:

SI. No	Time (min)	Product density	P, lph	W_{ev} lph	T_w °C	T_{wi} °C	T_{wo} °C	F_{wc} lph	T_s °C
1	0								
2	15								
3	30								

4	45								
5	60								

RESULTS: Report all the results that you have obtained in your experiment and write down the discussions in your journal.

CONCLUSIONS: Using the results obtained from your experiment comment on the heat transfer coefficient of the evaporator.

Precaution:

- i. Use the stabilize AC single phase power supply only.
- ii. Do not disturb any electrical connections of the experimental setup.

QUESTIONS:

1. Define evaporator capacity and economy.
2. Write down the enthalpy balances for the single effect evaporator
3. Explain enthalpy – concentrations diagram.

EXPERIMENT NO. 5: ISOTHERMAL BATCH REACTOR (DETERMINATION OF ACTIVATION ENERGY)

AIM: To study of a non catalytic homogeneous reaction in an isothermal batch reactor.

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

To determine the effect of temperature on k and determine the activation energy.

THEORY:

A batch reactor is a closed system with no input and output streams. A batch reactor can operate under the following conditions:

Isothermal (temperature of reaction mass remains constant)

Perfectly mixed (composition of the reaction mixture is uniform throughout)

Constant volume (volume of the reaction mixture within the reactor remains constant; there is no appreciable change in the density of reaction mass)

In a homogeneous reaction, all the reactants remain in single phase. The rate of reaction for such a reaction is expressed as:

Moles of Product formed per unit volume of reaction mixture per unit time.

For a first order unidirectional reaction $A \rightarrow \text{Product}$

Rate of reaction can also be expressed as moles of A disappearing per unit volume per unit time

$$\text{Also - } r_A = KC_A = K N_A / V$$

Combining these two equations yield.

$$\ln N_A = \ln N_{A0} - kt$$

$$N_A = N_{A0} e^{-kt}$$

Or, in terms of Concentrations

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

And

N_{A0} = No of moles of A at $t = 0$

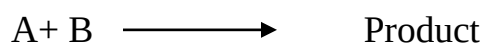
N_A = No of moles of A at $t = t$

For second order unidirectional reaction



If we plot $1/C_A$ Vs t we must get a straight line with slope = k and intercept = $1/C_{A0}$

For second order reaction:



(If $N_{A0} = N_{B0}$ then $N_A = N_B$ for all t)

It reduces to $2A \longrightarrow \text{product}$

Degree of conversion X is defined as:

Equation

And $N_B = N_{B0} - N_{A0} X$ for $N_{A0} \leq N_{B0}$

Equation

The effect of concentration on the rate of reaction is generally determined experimentally in a batch reactor by studying the rate of reaction at constant temperature

The interpretation of the kinetic data involves a trial and error procedure. A kinetic model is first assumed and the experimental observed conversion – time rate is matched with the selected model. The reaction rate constant is a strong function of reaction temperature. The temperature dependence of k is expressed in terms of Arrhenius equation: $k = A \exp(-E_A/RT)$

A = frequency factor of Arrhenius constant

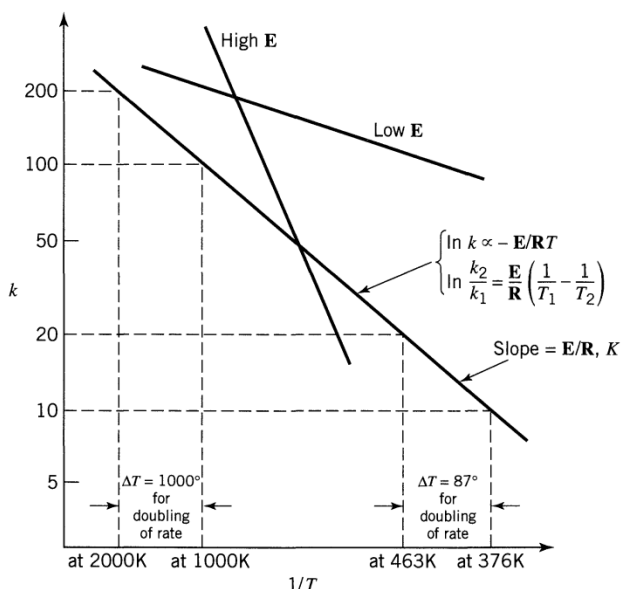
E_A = Activation Energy

R = gas law constant

This equation can be written in the form:

$$\ln k = \ln A - E_A/(RT)$$

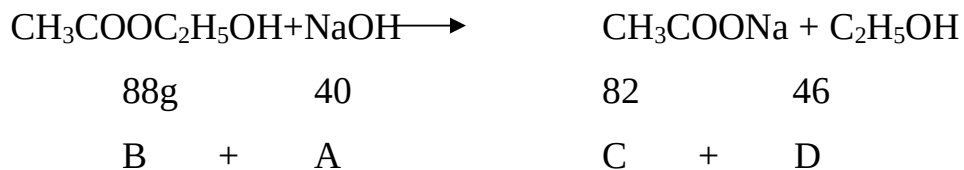
A plot of $\ln k$ Vs $1/T$ shall yield E_A and A .



In case of second order reversible reaction with $C_{A0} = C_{B0}$ Equation

Equation

X_{Ae} = fractional conversion at equilibrium
Saponification of Ethyl acetate & NaOH



Description

A batch reactor is closed system with no input and output streams. A batch reactor can operate under conditions like isothermal (Temperature of reaction Mass remains constant), perfectly mixed (composition of reaction mixture is

uniform throughout), and constant volume (volume of the reaction mixture within the reactor remains constant, there is no appreciable change in the density of reaction mass). This set – up is used to study a non – catalytic homogeneous reaction under isothermal condition. The set up consists of a reactor fitted in a constant temperature water bath. 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. The temperature of bath can be maintained from ambient to 90°C with the help of Digital temperature indicator cum controller. Samples can be taken out with the help of a sampling pipette.

Volume of reactor = 2.815

Volume of bath = 14.3lt

Reactor stirrer = 100rpm

Bath stirrer = 100rpm

Temperature controller – ambient to 200°C with 1°C

resolution Utilities required

1. Water supply
2. drain
3. electricity supply 1 phase 220V AC, 1.5kw
4. Instruments, laboratory glass ware and chemicals required for analysis as per the system adopted.

Chemicals:

1. N/10 NaOH
2. N/10 HCl
3. N/10 Ethyl acetate (8.8gms of ethyl acetate in 1 liter of water), indicator (phenolphthalein)

EXPERIMENTALPROCEDURE:

1. Prepare a solution of N/10 ethyl acetate by mixing 8.8gms (or 9.8ml at 20°C) of ethyl acetate in 1L of solution.
2. Prepare a solution of N/10 NaOH by dissolving 4gms / 1L of solution
3. Prepare a solution of N/10 HCl
4. Use phenolphthalein as indicator
5. Take 6Nos of 250ml beakers and put 20ml of N/10HCl in each beaker
6. Take N/10 NaOH in the burette
7. Adjust the temperature of water bath at 25°C
8. If the volume of reactor is 1L then take 400ml each of N/10 $\text{CH}_3\text{COOC}_2\text{H}_5$ and N/10 NaOH in two separate flasks and keep these in the water bath for about 15min
9. Transfer these solutions quickly in the batch reactor. Start immediately the mixer and the stopwatch
10. At regular intervals of 3-5min withdraw 100ml of reaction mixture and put it in the marked beakers containing N/10HCl. Take at least 6 samples at regular intervals of time.
11. Transfer the solution from beakers to the conical flasks, and titrate the excess N/10 HCl in each flask using N/10 NaOH from burette and phenolphthalein as indicator
12. Record the reaction temperature
13. Equilibrium conversion can be determined at reaction time of ½hr
14. In order to study the effect of temperature on k the above steps may be repeated at temperature = 35°C and 50°C. all the reactants must be

preheated in the water batch before mixing in thereactor.

Standard Data

Reactor : Material stainless steel 304 grade, Volume 2.1 Lts(Approx)

Water Bath : Material stainless steel 304 grade, double wall, insulated with ceramic wool

Heater : Nichrome wireHeater

Stirrers (2Nos) : stainless steel 304 grade impeller and shaft coupled with FHPmotor

Stopwatch : electronic

Temperature sensor : RTD PT – 100type

Control panel comprises of Digital Temperature controller cum – indicator (For Batch) = 0 – 1999.9°C, RTD PT – 100 type

With standard make on/off switch, mains indicator fuse etc

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

FORMULAE:

Degree of conversion $X_A = (C_{A0} - C_A)/C_{A0}$

Amount of HCl taken as quench, $HCl = V_{HCl} * N_{HCl} / 1000$, gmol

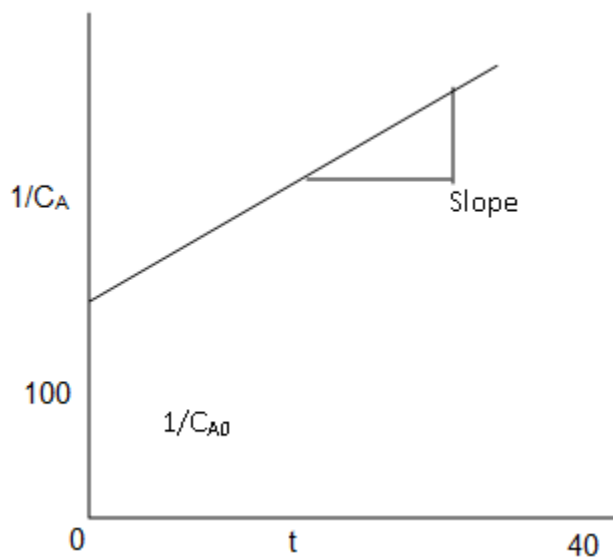
Amount of HCl reacted with the un-reacted NaOH of the reaction sample = $HCLR = HCL_0 - (V * N_{NaOH})/1000$, gmol

Concentration of unreacted NaOH in the reaction mixture, $C_A = (\text{HCLR} / V_{\text{Samp}}) * 1000$, gmol/L

Measurement data:

Time, t (min)	C_A Concentration of NaOH (gmol/L)	$1/C_A$ (L/gmole)
0		
-		
-		
-		

Plot t Vs $1/C_A$



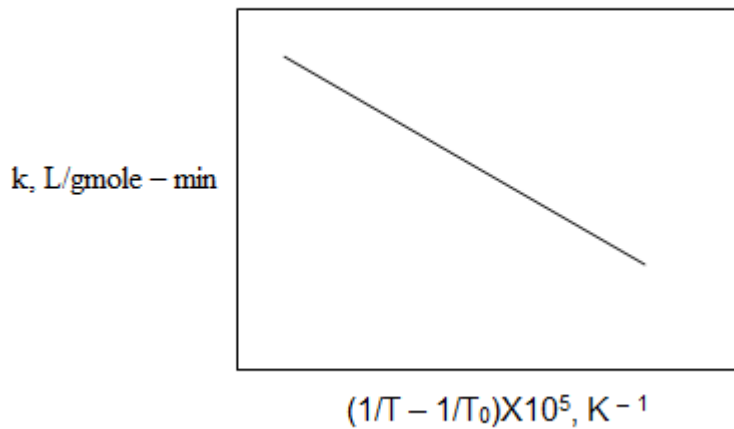
Slope = k , L/gmole – min

C_A = concentration of NaOH in
gmole/L

t = time in min

Plot k Vs $(1/T - 1/T_0)$, k^{-1} on a semi log graph

Slope = $-E_A/Rk$, (L/gmole –min)



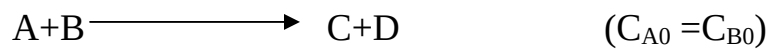
Intercept = K_0

Express

$$K = k_0 e^{-E_A/RT}$$

Observations and Calculation Reaction:

k



Reaction temperature = 0°C = $^\circ\text{K}$

Concentration of Ethyl acetate = $N/10$

Concentration of NaOH = $N/10$

Volume of Ethyl acetate taken
=400ml

Volume of Sodium Hydroxide = 400ml

Initial concentration of Ethyl acetate (B) in the mixture = 0.01N = C_{B0}
(gmole/L) Initial concentration of NaOH (A) in the mixture = 0.01N = C_{A0}
(gmole/L)

S. No	Time, t Min.	Concentration of unreacted NaOH in the reaction mixture, C_A (gmole/L)	$1/C_A$

Tabulate the time concentration data (t Vs C_A)

Determine the rate constant k by assuming the reaction to be of second order and plotting $1/C_A$ Vs t

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

Draw a smooth curve through all the points

If the data points yield a straight line, the assumed order of 2 is confirmed Slope = k (L/gmole –min)

Intercept = $1/C_{A0}$ (L/gmole)

Tabulate k values at 3 or 4 different temperatures

S. NO	k	T, K	T ₀ , K	(1/T – 1/T ₀), K ⁻¹
1		T ₁	T ₀	
2		T ₂		
3		T ₃		
4		T ₄		

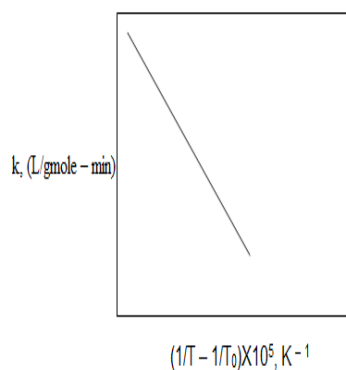
$$T_0 = (T_1 + T_2 + T_3 + T_4)/4$$

Plot k Vs (1/T – 1/T₀), K⁻¹ on a semi log graph

$$\text{Slope} = \frac{E_A}{R}$$

intercept = k₀

Express, $k = k_0 e^{-E_A/RT}$



PRECAUTIONS & MAINTANANCE INSTRUCTION:

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 filling the reactant in feed tanks
 5. Flow should not be disturbed during the experiments
 6. Handle the chemicals carefully
 7. Don't ON heater switch before filling the water in the bath
- There should be no air in the flow during experiment

TROUBLESHOOTING:

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 fluctuating more than average tight control knob of that (Procedure: two nuts are there lose first nut and tight the second lightly, and then first also, both nuts are on rotameter)
4. if D.T.C display '1' on display board it means sensor connection is not OK tight that
5. if switch ON the heater but temperature can't rise but panel LED is ON it means bath had burned replace that

REFERENCE: Chemical Reaction Engineering by Octave Levenspiel, Chapter 2.

EXPERIMENT 6: RTD IN PACKED BED REACTOR (PBR)

OBJECTIVE: Study of a Non-Catalytic Homogeneous reaction in Packed Bed Reactor.

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

INTRODUCTION:

A packed-bed reactor shown in figure. When a reactor is packed with catalyst, the reacting fluid usually does not flow through the reactor uniformly. Rather, there may be sections in the packed bed that offer little resistance to flow, and as a result a major portion of the fluid may channel through this pathway. Consequently, the molecules following this pathway do not spend as much time in the reactor as those flowing through the regions of high resistance to flow. We see that there is a distribution of times that molecules spend in the reactor in contact with the catalyst.

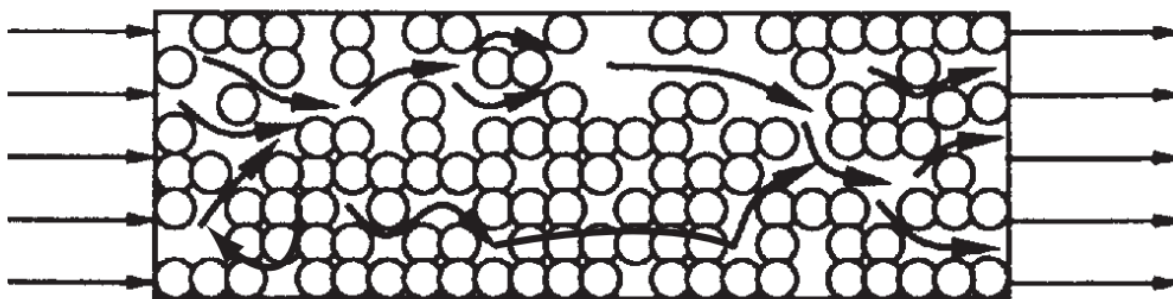


Figure. Packed-bed reactor.

RTD in Batch and Plug-Flow reactors

The RTDs in plug-flow reactors and ideal batch reactors are the simplest to consider. All the atoms leaving such reactors have spent precisely the same amount of time within the reactors. The distribution function in such a case is a spike of infinite height and zero width, whose area is equal to 1; the spike occurs at $t = V/v$,

or $\Theta = 1$. Mathematically, this spike is represented by the Dirac delta function:

$$E(t) = \delta(t - \tau)$$

The Dirac delta function has the following properties:

$$\delta(x) = \begin{cases} 0 & \text{when } x \neq 0 \\ \infty & \text{when } x = 0 \end{cases}$$

$$\int_{-\infty}^{\infty} \delta(x) dx = 1$$

$$\int_{-\infty}^{\infty} g(x) \delta(x - \tau) dx = g(\tau)$$

To calculate τ the mean residence time, we get $g(x) = t$

$$t_m = \int_0^{\infty} t E(t) dt = \int_0^{\infty} t \delta(t - \tau) dt = \tau$$

But we already knew this result. To calculate the variance we set, $g(t) = (t - \tau)^2$, and the variance σ^2 , is

$$\sigma^2 = \int_0^{\infty} (t - \tau)^2 \delta(t - \tau) dt = 0$$

All material spends exactly a time τ in the reactor, there is no variance.

The cumulative distribution function $F(t)$ is

$$F(t) = \int_0^t E(t) dt = \int_0^t \delta(t - \tau) dt$$

The $E(t)$ function is shown in figure (a), and $F(t)$ is shown in figure (b).

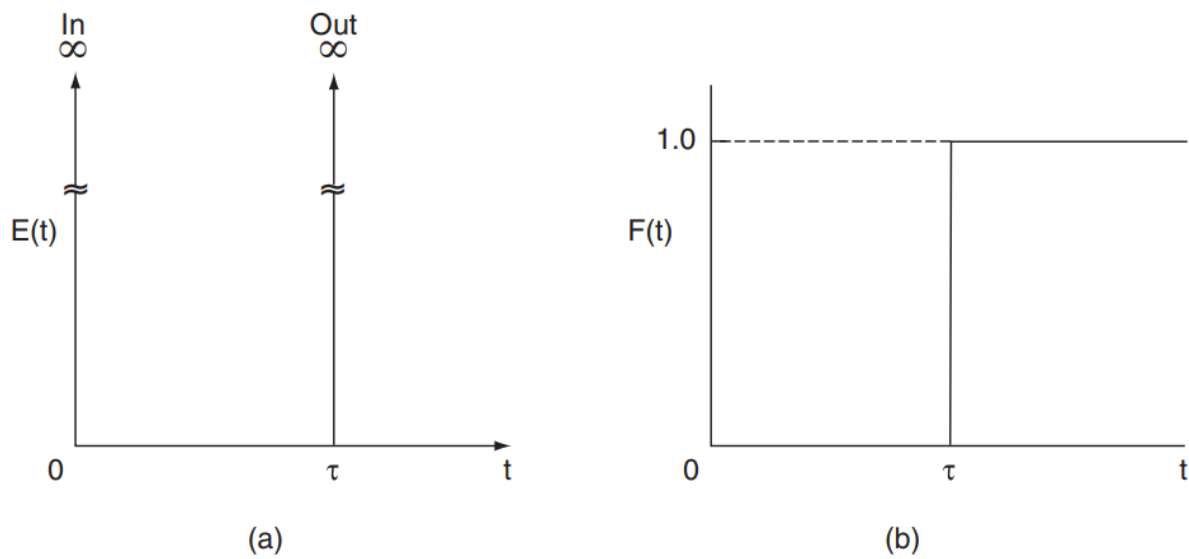


Figure. Ideal plug-flow response to a pulse tracer input.

RTD in CSTR

In an ideal CSTR the concentration of any substance in the effluent stream is identical to the concentration throughout the reactor. Consequently, it is possible to obtain the RTD from conceptual considerations in a fairly straightforward manner. A material balance on an inert tracer that has been injected as a pulse at time $t = 0$ into a CSTR yields for $t > 0$

In – Out = Accumulation

$$0 - vC = V \frac{dC}{dt}$$

Because the reactor is perfectly mixed, C in this equation is the concentration of the tracer either in the effluent or within the reactor. Separating the variable and integrating with $C = C_0$ at $t = 0$ yields

$$C(t) = C_0 e^{-t/\tau}$$

This relationship gives the concentration of tracer in the effluent at any time t .

To find $E(t)$ for an ideal CSTR,

$$E(t) = \frac{C(t)}{\int_0^\infty C(t) dt} = \frac{C_0 e^{-t/\tau}}{\int_0^\infty C_0 e^{-t/\tau} dt} = \frac{e^{-t/\tau}}{\tau}$$

Evaluating the integral in the denominator completes the derivation of the RTD for an ideal CSTR given by Equations,

$$E(t) = \frac{e^{-t/\tau}}{\tau}$$

$$E(\Theta) = e^{-\Theta}$$

Recall $\Theta = t/\tau$ and $E(\Theta) = \tau E(t)$.

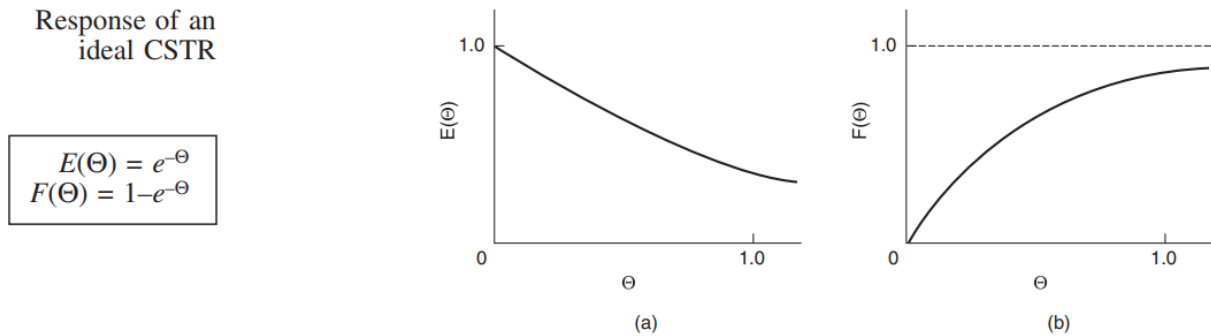


Figure. $E(\Theta)$ and $F(\Theta)$ for an Ideal CSTR.

The cumulative distribution $F(\Theta)$ is

$$F(\Theta) = \int_0^{\Theta} E(\Theta) d(\Theta) = 1 - e^{-\Theta}$$

The $E(\Theta)$ and $F(\Theta)$ functions for an Ideal CSTR respectively.

Earlier it was shown that for a constant volumetric flow rate, the mean residence time in a reactor is equal to V/v , or τ . This relationship can be shown in a simpler fashion for the CSTR. Applying the definition of the mean residence time to the RTD for a CSTR, we obtain

$$t_m = \int_0^{\infty} tE(t)dt = \int_0^{\infty} \frac{1}{\tau} e^{-t/\tau} dt = \tau$$

Thus, the nominal holding time (space time) $\tau = V/$ is also the mean residence time that the material spends in the reactor.

The second moment about the mean is a measure of the spread of the distribution about the mean. The variance of residence times in a perfectly mixed tank reactor

is (let $x = t/\tau$)

$$\sigma^2 = \int_0^\infty \frac{(t - \tau)^2}{\tau} e^{-t/\tau} dt = \tau^2 \int_0^\infty (x - 1)^2 e^{-x} dx = \tau^2$$

Then $\sigma = \tau$. The standard deviation is the square root of the variance. For a CSTR, the standard deviation of the residence-time distribution is as large as the mean itself!!

UTILITIES REQUIRED

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

CHEMICALS REQUIRED

5. 1N NaOH
6. N/20 HCl
7. Phenolphthalein indicator

EXPERIMENTAL PROCEDURE

- Fill the feed tank with water and connect compressed air line to the apparatus.
- Place the syringe containing known amount of 1N NaOH at Tracer inlet
- Start the supply of water to the reactor at particular flow rate with the help of rotameter
- Allow water to flow through the PFTR and attain steady state.
- Inject 1N NaOH into the system as a pulse signal from the tracer inlet.

- At regular time intervals (say 1 minute), collect the samples at the outlet in pre-marked beakers/measuring cylinders (about 20ml) until all tracer leaves the vessel.
- Analyze these samples with N/20 HCl using phenolphthalein as indicator.
- Repeat the experiment for different flow rates.

SPECIFICATIONS

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/cm²

Pressure Gauge : Bourdon type 0-2 Kg/cm²

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 rest are painted with auto paints.

FORMULAE

1. Concentration of NaOH in sample,

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

2. Experimental mean residence time,

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

3. Theoretical mean residence time,

$$\tau_t = \frac{V_R}{V_o}$$

4. $\sigma_\theta^2 = 2 (D/UL) - 2(D/UL)^2(1 - e^{-UL/D})$

OBSERVATIONS AND CALCULATIONS

For calculating the concentration of NaOH in the exit stream, take 10 ml of the solution in 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

$$V_1 N_1 = V_2 N_2$$

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

Normality of NaOH in the solution, N_2 = $V_1/200$ -gram eq/L

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 10ml of Solution	Conc. Of NaOH gm/L, C_i	$t_i \times C_i$	$t_i^2 \times C_i$
		$\Sigma C_i =$	$\Sigma t_i \times C_i =$	$\Sigma t_i^2 \times C_i =$

$$\Sigma C_i =$$

$$\sum t_i \times C_i =$$

$$\sum t_i^2 \times C_i =$$

$$\tau = [\sum t_i^2 C_i \times \sum C_i]$$

$$\sigma^2 = [\sum t_i^2 C_i \times \sum C_i] - \tau^2$$

$$\sigma_\theta^2 = \sigma^2 / \tau^2$$

using the dispersion model:

$$\sigma_\theta^2 = 2(D/UL) - 2(D/UL)^2(1 - e^{-UL/D})$$

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

$$2(D/UL) = \sigma_\theta^2$$

By hit and trial method (improve upon the previous values exact values 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 mean 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	$E_i = \sum C_i$ Δt	$\theta = t / \tau$	$E_\theta = \tau E_i$

Plot E curve, θ vs E_θ

NOMENCLATURE

θ	=	reduced time
E	=	activation energy
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 AND MAINTENANCE INSTRUCTIONS

- Always use clean water, good quality chemicals and standard solutions for titration.
- Keep close all the drain valves. Vent valve should open while filling the water in feed tank
- Air pressure should not more than 1 Kg/cm²
- Flow should not be disturbed during the experiments
- Handle the chemicals carefully
- If any types of suspended particles are in the rotameter, stop the flow, the drain the water tank and reactor. Fill the tank with clean water after proper cleaning of feed tank and reactor.

EXPERIMENT NO. 7: SIMPLE DISTILLATION

AIM:

To verify '*Rayleigh Equation*' by conducting simple distillation of given binary mixture (Ethanol + Water)

THEORY: Simple distillation is a process in which a binary mixture is distilled batch-wise without giving any reflux. It is also known as differential distillation because the composition of the liquid in the distillation still changes with time. The liquid is heated slowly and the vapors are withdrawn as rapidly as they are formed into a condenser, where the distillate is collected. The vapors which form as soon as vaporization begins will be richest in the more volatile component. Eventually, the vapors become leaner in more volatile component.

$$\ln\left(\frac{F}{W}\right) = \int_{x_W}^{x_F} \frac{dX}{Y - X}$$

Where F = original moles charged

W = moles left in the still

x_F = original composition &

x_W = final composition of liquid

y^* = composition of the component in vapor phase in equilibrium with the liquid.

The above equation is known as Rayleigh equation. The integration part of the equation is obtained graphically by plotting $1/(y^* - x)$ versus x .

PREPARING CALIBRATION CHART:

Use the data from the experiment No: 01

EXPERIMENTAL PROCEDURE

1. Fill the Round bottom (RB) flask with known volumes of water and ethanol (100 ml + 100 ml). Allow running water to flow through the condenser.
2. Heat the still at a low heating rate till $2/3$ of the liquid is distilled.
3. Cool the distillate and residue to room temperature and determine their refractive indices and volume.
4. Using calibration chart note down the mole fractions of distillate and residue (x_D).
5. Plot $1/(y^* - x)$ versus x by using equilibrium data given. (See last page)
6. Determine area under the curve between x_w and x_f which gives the RHS part of the Rayleigh equation.

OBSERVATIONS AND CALCULATIONS

Weight of empty sp. gravity bottle, $W_1 =$ _gm

Weight of empty sp. gravity bottle + water, $W_2 =$ _____gm

Weight of sp.gr. bottle + ethanol, $W_3 =$ __gm

Room temperature, $T_R = \text{___}^\circ\text{C}$

Density of water at $T_R = \rho_W = \text{___ gm/cc}$

Density of ethanol at $T_R = \rho_e = \text{___ gm/cc}$

Moles of feed, $F = (V_A * \rho_e)/M_A + (V_B * \rho_w)/M_B$

M_A and M_B are molecular weight of methanol and water respectively

V_A and V_B are volume of ethanol and water added in the still.

Mole fraction of ethanol in the feed

$$x_f = \frac{V_A \rho_g / M_A}{\left(\frac{V_A \rho_g}{M_A} \right) + (V_B \rho_W) / M_B}$$

Refractive index of ethanol = $\mu_e = \text{___}$

Refractive index of water = $\mu_W = \text{___}$ Weight of a beaker = $W_4 = \text{___ gm}$

Weight of a beaker + distillate $W_5 = \text{___ gm}$

Volume of Distillate = $V_D = \text{___ cc}$

Density of distillate = $\rho_D = [(W_5 - W_1) / (W_2 - W_1)] * \rho_W$, gm/cc

Mole fraction of distillate, $x_D = \text{(from calibration chart)}$

Average molecular wt. of distillate, $M_D = x_D M_A + (1 - x_D) M_B$

Moles of distillate, $D = (V_D * \rho_D) / M_D = \text{___ gm}$

Weight of a beaker + Residue $W_6 = \text{___ gm}$

Volume of Residue = $V_R = \text{___ cc}$

Density of distillate = $\rho_R = [(W_6 - W_1) / (W_2 - 1)] *$

ρ_W , gm/cc Mole fraction of residue, x_W = (from calibration chart)

Moles of Residue, $W = F - D =$ _____ gm
moles

$$rhs = \int_{x_W}^{x_F} \frac{dX}{Y - X}$$

EQUILIBRIUM DATA

x	y*	$1/(y^* - x)$
0	0	
0.019	0.17	
0.0721	0.3891	
0.0966	0.4375	
0.1238	0.4704	
0.1661	0.5089	
0.2337	0.5445	
0.2608	0.558	
0.3273	0.5826	
0.3965	0.6122	
0.5079	0.6564	
0.5198	0.6599	
0.5732	0.6841	
0.6763	0.7385	
0.7472	0.7815	
0.8943	0.8943	

RESULT

Plot the graph between $1/(y^* - x)$ versus x and find the area under the curve between x_F to x_W

EXPERIMENT NO. 8: ADSORPTION STUDIES

AIM

To Verify the Freundlich Equation by an adsorbing acetic acid on activated carbon from an acetic acid solution and estimation of the constants of the equation.

APPARATUS

Five stoppered bottles, conical flask, burette with stand, funnel, pipette, measuring Jar.

CHEMICALS REQUIRED

Standard solution of Oxalic acid, Sodium hydroxide solution, acetic acid, activated Charcoal and Phenolphthalein indicator.

THEORY

Freundlich adsorption isotherm:- (for dilute solutions)

At a given temperature, the mass of a solute adsorbed by a solid adsorbent at various amounts are given by the following empirical relation.

$$C^* = K [V(C_0 - C^*)]^n$$

Where C_0 = initial concentration of acetic acid solution
 C^* = equilibrium concentration after adsorption
 V = Volume of given solution / mass of solid

K , n are constants at a given temperature for a given solute and adsorbent.

PROCEDURE

1. Prepare exactly 0.1 N Oxalic acid solution.
2. Take 5 reagent bottles and label them 1 to 5.
3. Weight separately and accurately 0.5, 1, 1.5, 2, 2.5 gm of activated carbon and transfer it in to 5 reagent bottles in the same order.
4. Prepare solution of 0.1 N HCl and 0.1 N NaOH solutions.
5. Verify the Normality of NaOH by taking 10 ml of standard Oxalic solution and titrating against it.
6. From this NaOH solution, find exactly the normality of HCl solution by titration.
7. Carefully measure and transfer 50 ml of acetic acid solution of known concentration (0.1N) to each reagent bottle.
8. Close the lids and keep the bottles in a shaker. Switch on the shaker and operate it for about 30 minutes.
9. At which time the equilibrium is expected to reach between the liquid and solid phases.
10. Remove the bottles from the shaker, filter the contents of each bottle and collect the clear solution in to 5 separate 250ml conical flasks also numbered 1 to 5.
11. From each of these solutions take 10 ml with pipette and titrate against the previously standardized NaOH solution using phenolphthalein indicator.

STANDARDISATION OF 1N NaOH:

Standard solution of Oxalic acid (1N) was prepared by dissolving 6.3g of Oxalic acid in 100ml of water.

Wash 50ml burette and rinse with 1N NaOH. Pipette out 10ml of std. Oxalic acid into conical flask and titrate it with NaOH in burette using phenolphthalein indicator. The end point has been reached when the pale pink color of the phenolphthalein persists for 30 seconds. There should be a one-drop difference between when the solution is colourless and when it is pink. If too much NaOH is added (that is, if you "over-shoot" the endpoint), discard the solution and repeat the titration. A white piece of paper placed under the flask will aid in the color detection.

Sl. No.	Volume of Sodium hydroxide(ml)	Burette reading		Volume of Oxalic acid, (ml)
		Initial	Final	

STANDARDISATION OF 1N HCl:

Wash 50ml burette and rinse with 1N NaOH. Pipette out 10ml of HCl into conical flask and titrate it with NaOH in burette using phenolphthalein indicator. The end point has been reached when a very light pink color persists for at least 30 seconds.

Sl. No.	Volume of Sodium hydroxide(ml)	Burette reading		Volume of HCl, (ml)
		Initial	Final	

SAMPLE CALCULATION

Normality of initial acetic acid, $N_0 = 0.1 \text{ N}$

Normality of the bottle 1 filtrate, $N_1 = (\text{Normality of NaOH} \times \text{burette reading}) / 10$

N = Initial concentration of Acetic acid

$C_0 = N_0 \times \text{equivalent weight of acetic acid}$,
Equilibrium concentrations of filtrate after adsorption, $C_1^* = N_1 \times \text{equivalent weight of acetic acid}$,
 $\text{Kg acid} / \text{m}^3 \text{ solution}$

Bottle No.	Weight of adsorbent gm	Volume of 0.1 N acetic acid, ml	V, m ³ /Kg	C^* , Kg / m ³	log C^*	$V(C_0 - C^*)$	log[V (C ₀ - C*)]
1	0.5	50					
2	1	50					
3	1.5	50					
4	2.0	50					
5	2.5	50					

GRAPH

The Freundlich adsorption isotherm over the small

$$\text{concentration range is } C^* = K [V(C_0 - C^*)]^n$$

$$\text{Log } C^* = \text{Log } K + n \text{ Log } [V(C_0 - C^*)]$$

Plot a graph between $\log C^*$ Vs $\log [V (C_0 - C^*)]$. A straight line is drawn fitting all the five points. Calculate the slope (n) and intercept ($\log K$).

RESULT

1. A straight line obtained in the plot shows the validity of the Freundlich adsorption isotherm over the concentration range studied.
2. The values of n and K are _____ & _____

EXPERIMENT NO. 9: STUDY OF PROPORTIONAL (P) CONTROLLER USING COMPUTER-CONTROLLED LEVEL TRAINER

AIM: To study the effect of proportional band (PB) on the response of the given level system

OUTCOMES: After completing this experiment students are expected to know

- basic feedback control configuration using P-controller
- concept of PB or proportional gain and its effect on output
- hardware involve in direct digital control (DDC) system

EQUIPMENT: Level control trainer (DDC type)

THEORY:

An automatic control is essential in industrial operations for controlling pressure, temperature, liquid level, concentration humidity, viscosity, and flow in the process industries. Feedback control systems represent the control schemes encountered most often in a chemical plant. Fig.1 represents a schematic of basic feedback loop in which feedback is taken from output and fed in to input. It comes between the measuring device and the final control element and generates actuating signal, CO using error signal $e(t)$ as input. There are three basic types of feedback controllers

- i. Proportional (P controller)**
- ii. Proportional- Integral (PI controller)**
- iii. Proportional- Integral -Derivative (PID controller)**

Actuating output of P- controller is proportional to current error $e(t)$:

$$CO(t) = K_c e(t) + C_s$$

and

$$G_C(s) = K_c$$

where K_c = proportional gain of the controller and C_s = controller's bias signal (i.e., its actuating signal when $e = 0$). If the error value is zero, then this controller output is zero.

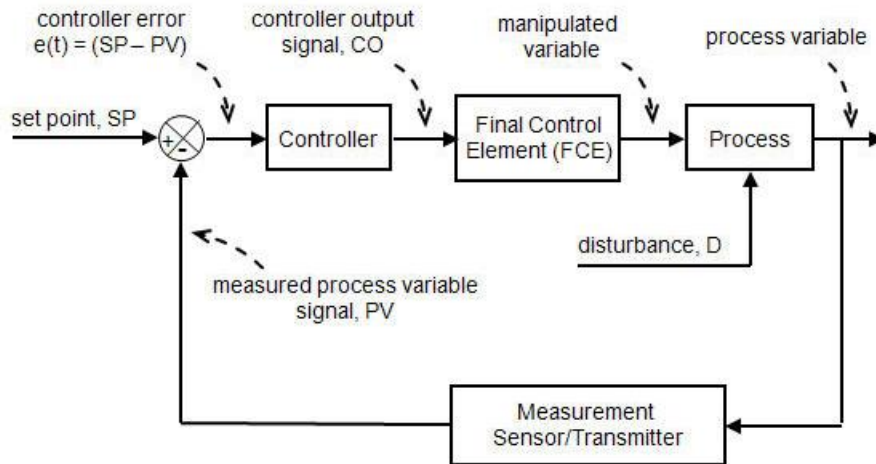


Fig. 1 Schematic of Basic Feedback Control Loop

A proportional controller is described by the value of its proportional gain K_c , or equivalently by its proportional band PB, where $PB = 100/K_c$; The proportional band characterizes the range over which the error must change in order to drive the actuating signal of the controller over its full range. The larger the gain K_c or equivalently, the smaller the PB value, the higher the sensitivity of controller's actuating signal to deviations $e(t)$ will be.

This controller requires biasing or manual reset when used alone. This is because it never reaches the steady state condition. It provides stable operation but always maintains the steady state error between the process variable and set point, called 'offset'. It is defined as the ultimate response, after $t \rightarrow \infty$, never reaches the desired new set point. There is always a discrepancy called offset:

$$\text{Offset} = (\text{new set point}) - (\text{ultimate value of the response})$$

The offset is characteristic effect of P-controller (see Fig. 2). It decreases as K_C becomes larger and theoretically: $\text{Offset} \rightarrow 0$ when $PB \rightarrow 0$

Speed of the response is increased when the proportional constant K_C increases. However, we will never use extremely large values of K_C (why?)

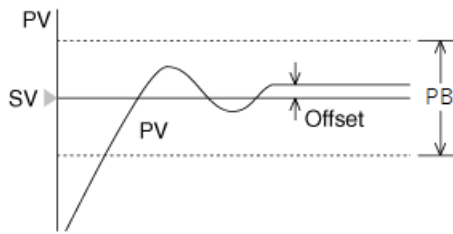


Fig. 2 Unit-step response for set point change

Description of Setup:

The role of the level controller is to determine the desired control action by sensing the level of water column in the process tank with the help of a level transmitter by producing current in the range of 4 to 20 mA. As per the desired level to be attained in the process tank, a pneumatic control valve is opened or closed (manipulated) by an I/P controller to minimize any error to maintain a steady level in the process tank with the help of supervisory control and data acquisition (SCADA) system.

The process setup consists (see Fig. 3 and 4) of supply water tank fitted with centrifugal pump for water circulation. The rate of flow is regulated by the pneumatic control valve. A level transmitter used for level sensing produces current in the range of 4 to 20 mA proportional to the height of water column in the water tank. The difference in desired level and current water level is computed. Based on this information the controller determines the desired water level. The process parameter (level) is controlled by microprocessor based digital indicating

controller which manipulates pneumatic control valve through I/P converter.

Specification:

- **Level Transmitter:** Type electronic, two wire, range 0-250 mm, output 4-20 mA
- **I/P converter:** Input 4-20 mA, output 3-15 psig
- **Control valve:** Pneumatic (*Air to close*); **Size:** ¼ inch; **Input:** 3-15 psig
- **Valve Characteristics:** Linear
- **Rotameter:** 10-100 LPH
- **Pump:** Type submersible
- **Process tank:** Transparent, MOC: Acrylic, with 0-100 % graduated scale
- **Air filter regulator:** Range 0-2.5 kg/cm²

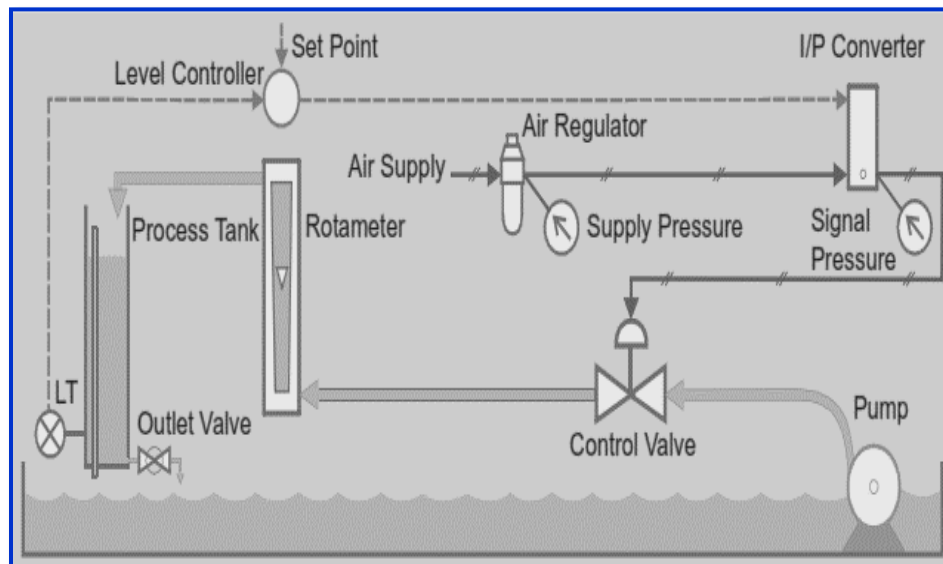


Fig. 3 A Schematic of the Level Control Trainer

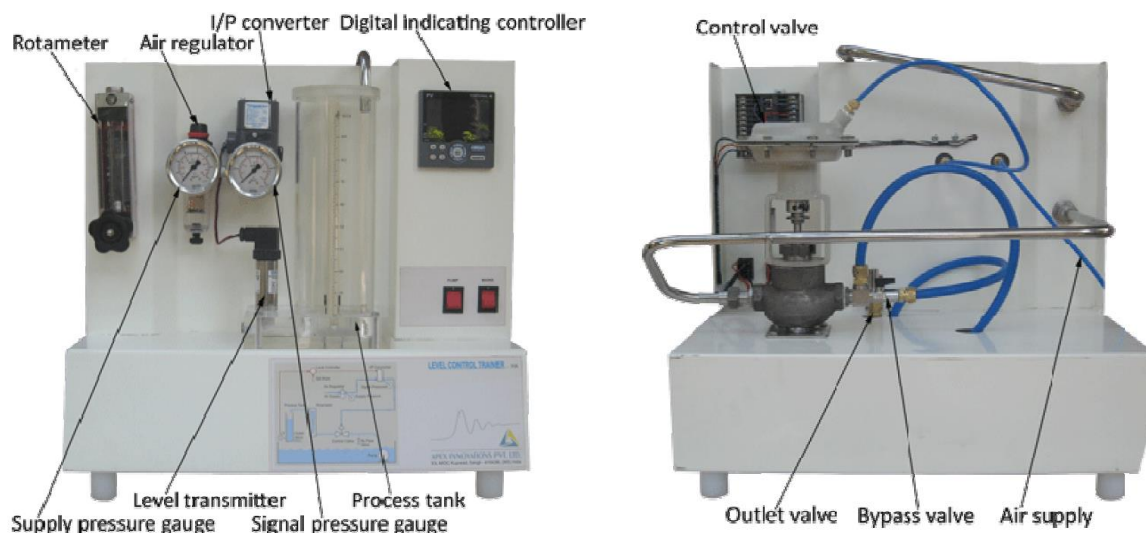


Fig. 4 (a) Front View and (b) Back View of the Setup

PROCEDURE:

- Fill supply tank with distilled water and keep the pump inside the water.
- Switch on electric supply. Switch on Mains.
- Switch on the pump and ensure that flow through rotameter is above 100 LPH.
- Adjust the tank drain valve such that the tank level remains between 90 and 100%
- Confirm the reading on digital indicating controller is within $\pm 2\%$.
- Switch on the compressed air source and adjust the air regulator to set supply air pressure at 2 kg/cm^2
- Set the controller to manual mode by pressing the A/M key.
- Increase output of controller from 0 to 100% in steps of 25%. Check the pressure on pressure gauge at the output of I/P converter is varying from 3-15 psig and ensure that control valve operates from full open to fully close position.

Start-up of software:

- Click on Start|All programs|Wonderware|InTouch|Yes to open InTouch Application manager.
- Open “Level Control Trainer” under InTouch Application 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

FOR STUDY OF P-CONTROLLER

- Select P Mode option for control from software. (click on “Change Expt.” Button, click on “Change”, Click on “P Mode” button.)
- Adjust the process value by switching the controller to manual mode to a particular level (say 50 %) on the screen and apply output of the controller as bias value. Change the proportional band to 100%. Switch the controller to auto mode.
- Apply step change of 10% to set point.
- Switch the controller to manual mode. Decrease proportional band to half of the previous value. With each decrease, obtain a new response of the step change.
- Ensure that the set point changes are around the same operating point (say 50%).

OBSERVATIONS:

- From the data file stored observe the output response of the process and note down the output and plot the response
- Observe the effect of proportional band values

CALCULATIONS:

Plot PV vs time for various PB values

Sl. No.	Value of PB	Offset amount
1.		
2.		
.		
.		

Offset =

RESULTS:

Report the effect of proportional band values

Discuss the characteristics of the closed-loop response of the process.

QUESTIONS:

- i. Define proportional band (PB) of a P-controller. Explain its physical significance.
- ii. What are the relative advantages and disadvantages of the proportional control actions? What are its characteristic effects on the closed-loop response of a process?
- iii. Where can we use a P-controller in process industries?
- iv. What are the weaknesses of a P-controller?

EXPERIMENT NO. 10: STUDY OF PROPORTIONAL INTEGRAL (PI) CONTROLLER USING COMPUTER-CONTROLLED FLOW TRAINER

AIM: To study the effect of integral time on the response of the given process flow system

OUTCOMES: After completing this experiment students are expected to know

- basic feedback control mechanism using PI-controller
- concept of integral time and its effect on response
- hardware involve in direct digital control (DDC) system

EQUIPMENT: Flow control trainer (a direct digital controller)

THEORY:

Feedback control systems represent the control schemes encountered most often in a chemical process plant. Fig.1 represents a schematic of a basic feedback loop in which feedback is taken from output and fed in to input. It comes between the measuring device and the final control element and generates actuating signal, CO using error signal $e(t)$ as input. There are three basic types of feedback controllers

- i. **Proportional (P controller)**
- ii. **Proportional- Integral (PI controller)**
- iii. **Proportional- Integral -Derivative (PID controller)**

Actuating output of PI- controller is proportional to current error $e(t)$:

$$CO(t) = K_c e(t) + \frac{K_c}{\tau_I} \int_0^t e(t) dt + C_s$$

and

$$G_c(s) = K_c \left(1 + \frac{1}{\tau_I s} \right)$$

where K_C = proportional gain of the controller, τ_I = is the integral time constant or reset time in minutes, and C_s = controller's bias signal (i.e., its actuating signal when $e = 0$). Here,

the reset time is an adjustable parameter and is sometimes referred to as minutes per repeat. The integral control action has "repeated" the response of the proportional action. This repetition takes place every τ_I minutes.

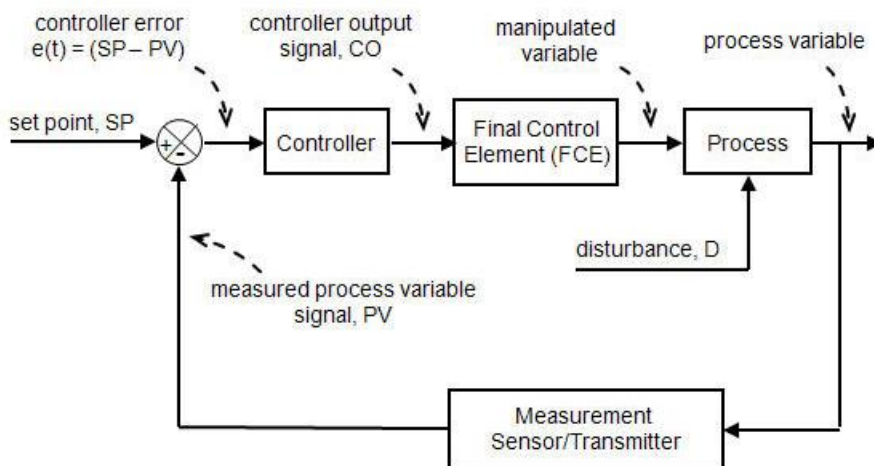
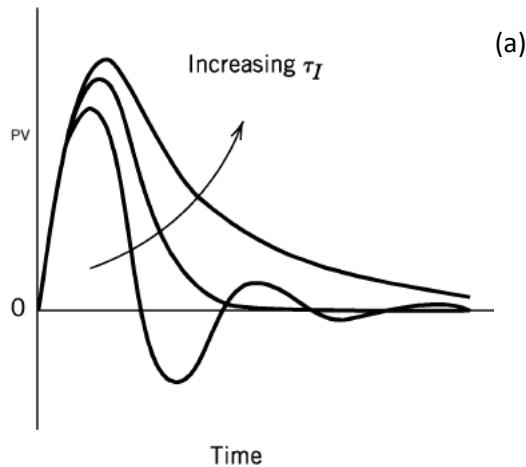


Fig. 1 Schematic of Basic Feedback Control Loop

The integral action causes the controller output $CO(t)$ to change as long as a non-zero error exists in the process output. Therefore, such a controller can eliminate even small errors or offset but it can cause the response to become more oscillatory to set point changes. The qualitative effects of changing individual controller settings are shown in Fig.2.



g.2. Proportional-integral control: (a) effect of integral time, (b) effect of controller gain

Description of Setup:

The role of the level controller is to determine the desired control action by sensing the level of water column in the process tank with the help of a level transmitter by producing current in the range of 4 to 20 mA. As per the desired level to be attained in the process tank, a pneumatic control valve is opened or closed (manipulated) by an I/P controller to minimize any error to maintain a steady level in the process tank with the help of supervisory control and data acquisition (SCADA) system.

The process setup (see Fig. 3 and 4) consists of supply water tank fitted with centrifugal pump for water circulation. The rate of flow is regulated by the pneumatic control valve. A level transmitter used for level sensing produces current in the range of 4 to 20 mA proportional to the height of water column in the water tank. The difference in desired level and current water level is computed. Based on this information the controller determines the desired water level. The process parameter (level) is controlled by microprocessor based digital indicating controller which manipulates pneumatic control valve through I/P converter.

Specification:

- **Differential pressure transmitter:** Type capacitance, two wire, range 0-200mm, output 4-20 mA sq. root.
- **I/P converter:** Input 4-20 mA, output 3-15 psig
- **Control Valve:** Pneumatic type, **Size:** ¼ inch, **Input:** 3-15 psig; **Air to close;** **Characteristics:** Linear
- **Rotameter:** 10-100 LPH
- **Pump:** Fractional horse power, type submersible
- **Supply Tank:** SS304
- **Flow Measurement:** Orifice meter
- **Air filter regulator:** range 0-2.5 kg/cm²
- **Pressure gauge:** range 0-2.5 kg/cm², range 0-7 kg/cm²

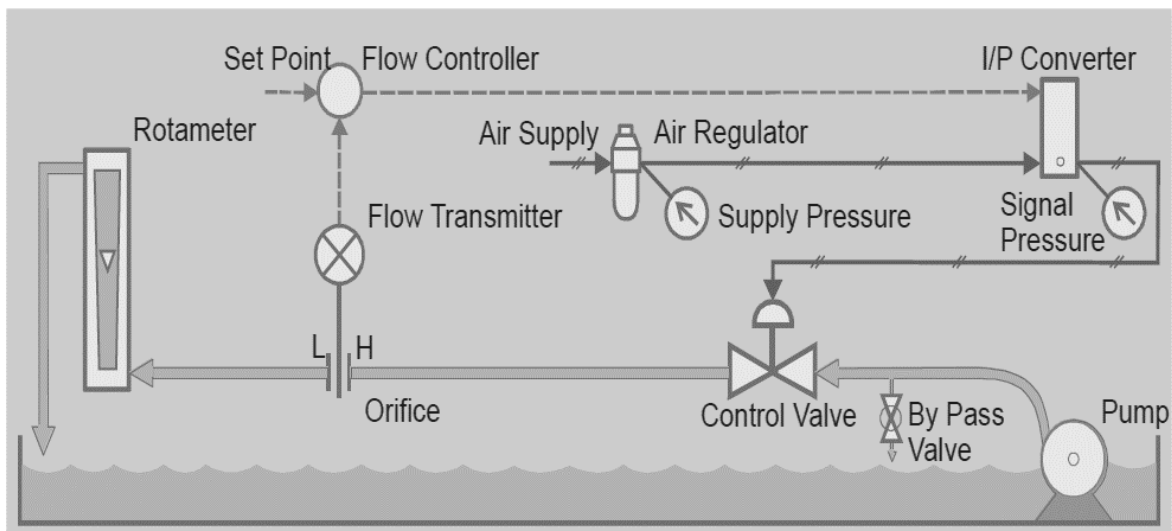


Fig. 3. A Schematic of the Flow Control Trainer

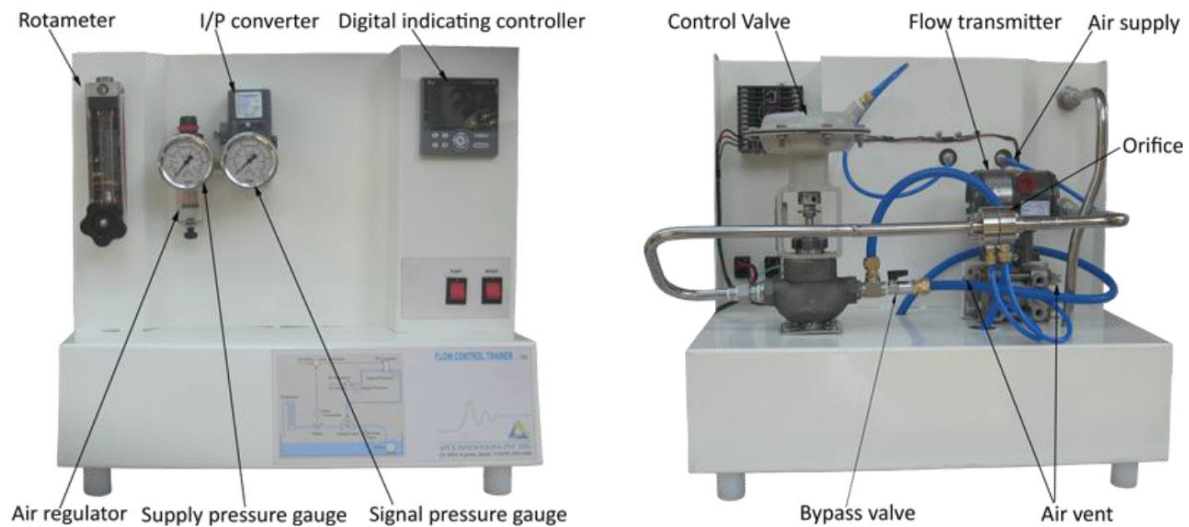


Fig. 4 (a) Front View and (b) Back View of the Setup

PROCEDURE:

- Fill supply tank with distilled water and keep the pump inside the water.
- Switch on electric supply. Switch on Mains.
- Switch on the pump and ensure that flow through rotameter is above 100 LPH.
- Confirm that flow signal reading is displayed at controller (For 100 LPH flow the reading should be @100%. If not, remove air from the pressure signal connections of Flow transmitter by loosening vent plugs provided on it).
- Ensure that air regulator is fully open by rotating anticlockwise. Switch on the compressed air source and adjust the Air regulator to set supply air pressure at 25 psig.
- Set the controller to manual mode by pressing the A/M key.

- Increase output of controller from 0 to 100% in steps of 25%. Check the pressure on pressure gauge at the output of I/P converter is varying from 3-15 psig and ensure that Control valve operates from full open to fully close position.
- Switch on the computer and execute the software.

START-UP OF SOFTWARE:

- Click on Start|All programs|Wonderware|InTouch|Yes to open InTouch Application manager.
- Open “Flow Control Trainer” under InTouch Application 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

FOR STUDY OF PI-CONTROLLER

- Select PI Mode option for control from software. (click on “Change Expt.” Button, click on “Change”, Click on “PI Mode” button.)
- Select PID controller. Set the proportional band. Set derivative time to 0 sec 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 flow value (@70%). Allow the process to reach at steady state. Record the steady state error.

- Switch the controller to manual mode. Reduce the integral time to half of the previous value. Switch to Auto mode and apply step change (+/- 10%) to the set point. Note the response of the system.
- Repeat above step to observe the effect of changes in Integral setting.

OBSERVATIONS:

- From the data file stored observe the output response of the process and note down the output and plot the response
- Observe the effect of reducing integral time on offset and on the response of the process.

CALCULATIONS:

Plot PV vs time for various τ_I

Sl. No.	Value of PB	Value of τ_I	Offset amount, if any	Oscillation/Overshoot	Rise Time
				Increase/decrease	Faster/slower/no effect
				Increase/decrease	Faster/slower/no effect
				Increase/decrease	Faster/slower/no effect

RESULTS:

Report the effect of integral time values for a fixed PB value.

Discuss the characteristics of the closed-loop response of the process.

QUESTIONS:

- i. What is the transfer function of a PI- controller?
- ii. Define reset time and reset rate, and explain their importance.
- iii. What are the relative advantages and disadvantages of the integral control actions? What are its characteristic effects on the closed-loop response of a process?
- iv. Where can we use a PI-controller in process industries?

EXPERIMENT NO. 11: STUDY OF PROPORTIONAL-INTEGRAL-DERIVATIVE (PID) CONTROLLER USING COMPUTER-CONTROLLED TEMPERATURE TRAINER

AIM: To study the effect of derivative time on the response of the given process system

OUTCOMES: After completing this experiment students are expected to know

- basic feedback control configuration using PID controller
- concept of various feedback parameters and their effect on output
- hardware involve in direct digital control (DDC) system

EQUIPMENT: Temperature control trainer (DDC type)

THEORY:

Feedback control systems can be designed to achieve specific behavior of the output variable, for example (i) to keep the output variable within a tightly constrained range, irrespective of changes in the environment, and (iii) to reduce the influence of process changes, etc. It allows a system dynamic response to be modified without changing any system components. Fig.1 represents a schematic of a basic feedback loop in which feedback is taken from output and fed into input. It comes between the measuring device and the final control element and generates actuating signal, CO using error signal $e(t)$ as input. There are three basic types of feedback controllers:

- i. **Proportional (P controller)**
- ii. **Proportional- Integral (PI controller)**
- iii. **Proportional- Integral -Derivative (PID controller)**

The PID controller is by far the most common control algorithm. With the presence of the derivative term, a PID controller anticipates what the error will be in the immediate future and applies a control action which is proportional to the current rate of change in the error.

Actuating output of PID- controller is related to current error $e(t)$ as:

$$CO(t) = K_c e(t) + \frac{K_c}{\tau_I} \int_0^t e(t) dt + K_c \tau_D \frac{de(t)}{dt} + C_s$$

and

$$G_C(s) = K_c \left(1 + \frac{1}{\tau_I s} + \tau_D s \right)$$

where K_C is proportional gain of the controller, τ_I is the integral time constant or reset time in minutes, τ_D is the derivative time and C_s is controller's bias signal (i.e., its actuating signal when $e = 0$). In this case, all three values K_c , τ_I , and τ_D can be adjusted in the controller.

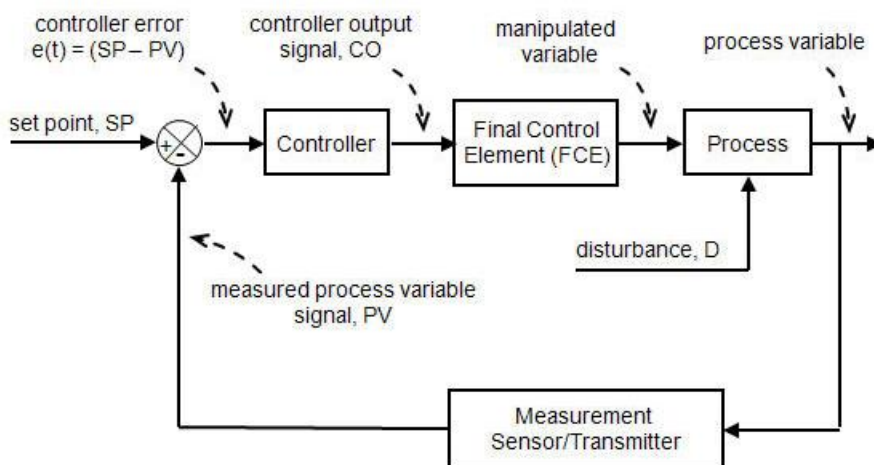
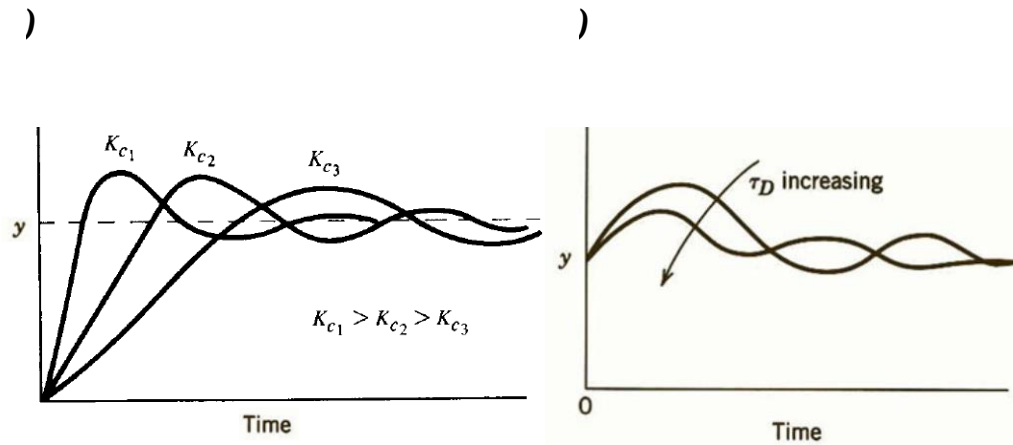


Fig. 1 Schematic of Basic Feedback Control Loop

The qualitative effects of changing proportional gain and derivative time settings are shown in Fig.2.



g.2. PID control: (a) effect of gain (b) effect of derivative time

Description of Setup:

Temperature control trainer (Fig. 3 & 4) is designed for understanding the basic temperature control principles using PID controller. The process setup consists of heating tank fitted with solid state relay (SSR) controlled heater for on-line heating of the water. The flow of water can be manipulated and measured by rotameter. Temperature sensor (RTD) is used for temperature sensing. The process parameter (Temperature) is controlled by microprocessor based digital indicating controller which manipulates heat input to the process. These units along with necessary piping and fitting are mounted on support frame designed for tabletop mounting. The controller can be connected to computer through ethernet port for monitoring the process in SCADA mode.

Specification:

- ***Temperature sensor: Type RTD, PT100***

- **Heating control Proportional power controller:** Solid state relay (SSR), Input 4-20 mA, Capacity 50 A.
- **Heater Type:** Electrical 2 coil, Capacity 3 KW
- **Rotameter:** 6-60 LPH
- **Process tank:** SS304, Capacity 0.5 lit, insulated

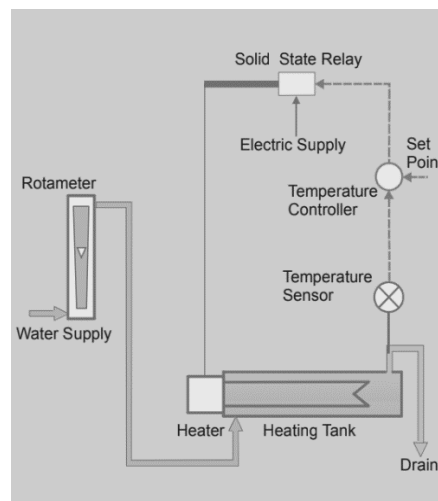


Fig. 3. A Schematic of the temperature control trainer



Fig. 4 (a) Front View and (b) Back View of the Setup

PROCEDURE:

- Switch on electric supply. Switch on Mains.
- Adjust rotameter flow rate to 40 LPH.
- Switch on the heater.
- Double click on Apex_Process_Trainers icon on the desktop
- Click Select Experiment, select PID mode and click Start
- Switch the controller to manual mode.
- Set the proportional band, integral time and derivative time.
- Change the controller to Auto mode. Apply step change by 2 to 3% to the set point and observe the response of the process.
- Keeping the proportional band, PB and integral time constant, τ_i change the derivative time, τ_D and observe the response of the process for step change for each change in setting.

OBSERVATIONS:

- From the data file stored observe the output response of the process and note down the output and plot the response
- Compare the steady state response of the PID controller for various derivative time

CALCULATIONS:

Plot PV vs time for various τ_D values

Sl. No.	Value of PB	Value of τ_I	Offset amount, if any	Oscillation/Overshoot	Rise Time
				Increase/decrease	Faster/slower/no effect
				Increase/decrease	Faster/slower/no effect
				Increase/decrease	Faster/slower/no effect

RESULTS:

Plot the steady state responses of the PID controller for various derivative time but for fixed PB and τ_I values.

Discuss the characteristics of the closed-loop response of the process.

QUESTIONS:

- What is the transfer function of a PID controller?
- Define derivative time and explain their importance.
- What are the relative advantages and disadvantages of the derivative control actions? What are its characteristic effects on the closed-loop response of a process?
- Discuss the effects of K_c , τ_I and τ_D on the closed-loop response of a process controlled with PID.
- Where can we use a PID-controller in process industries?