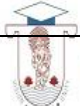




AVIT
AARUPADAI VEEDU INSTITUTE OF TECHNOLOGY



VINAYAKA MISSION'S
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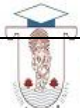
DEPARTMENT OF BIOTECHNOLOGY

B.TECH CHEMICAL ENGINEERING LABORATORY

(17BTCC86)

A. H.

HOD



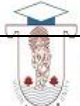
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STANDARD OPERATING PROCEDURES (SOP) FOR LABORATORY SAFETY

One should remember that, in the laboratory there is no such thing that can be regarded as a harmless substance. Following are the guidelines that are commonly practiced as SOP in the laboratory.

- (i) Lab coats or ankle length aprons must be worn while handling toxic, corrosive and flammable materials.
- (ii) Long hair, neckties, or loose clothing should be tied or otherwise secured.
- (iii) Gloves should be worn, while handling corrosive and highly toxic chemicals.
- (iv) Open shoes are not worn in the laboratory.
- (v) Bare legs are not acceptable, while handling hot, cold, toxic, corrosive or sharp materials.
- (vi) Appropriate eye protection should be worn at all times in laboratories.
- (vii) Always wash hands with soap after working with chemicals, even though gloves have been used.
- (viii) Do not mouth pipette or siphon toxic chemical reagents, corrosive liquids, organic solvents, strong acids and alkalies
- (ix) Do not directly smell, sniff or taste any chemical. Avoid inhalation.
- (x) Containers should be closed when not in use.
- (xi) When working with flammable chemicals, make sure that there are no sources of ignition near by, in order to avoid fire or explosion.
- (xii) Handle toxic, corrosive chemicals and flammable solvents in a chemical safety hood or a fume hood.
- (xiii) No smoking in any area of a laboratory.
- (xiv) No eating, drinking of beverage or application of cosmetics in the laboratory, except in designated areas in which no chemicals are used or stored.



PARALLEL, COUNTER FLOW HEAT EXCHANGER

SPECIFICATIONS

Length of the heat exchanger, L	=	ft
Inner copper tube, I. D.	=	mm
Inner copper tube, O. D.	=	mm
Outer steel tube, I. D.	=	mm
Outer steel tube, O. D.	=	mm

VENTURIMETER

SPECIFICATIONS

Diameter of the pipes, D_p	=	
Diameter of venturi throat, D_v	=	m
Area of the collecting tank, A	=	m^2
Density of manometric fluid	=	$13600 \text{ Kg} / m^3$
Density of flowing fluid	=	$1000 \text{ Kg} / m^3$

ORIFICEMETER

SPECIFICATIONS

Diameter of the pipes, D_p	=	m
Orifice diameter, D_o	=	m
Area of the collecting tank	=	m^2

PACKED BED COLUMN

SPECIFICATION

Diameter of particles (Packings), D_p	=	mm
Diameter of pipe, D	=	mm
Length of the column	=	ft
Diameter of the column	=	m
Area of the collecting tank	=	m^2
Cross sectional area of the column	=	m^2

FLUIDIZED BED COLUMN

SPECIFICATIONS

Density of glass beads	=	Kg / m^3
Diameter of glass beads	=	m
Diameter of the column	=	m
Length of the column	=	ft
Cross sectional area of the column	=	m^2

PLATE AND FRAME FILTER PRESS

SPECIFICATIONS

Number of plates	=	
Number of frames	=	
Area of the collecting tank	=	m^2
Filtration area	=	m^2
Cross sectional area of the frame	=	m^2
Air compressor		
N	=	RPM
HP	=	
Three phase induction motor		
N	=	RPM
Power	=	KW.

JAW CRUSHER

SPECIFICATIONS

ENERGY METER CONSTANT :

N	=	REV / KWHR
Power requirements	=	HZ, A, V

EXPT. NO. :

DATE :

CALIBRATION OF VENTURIMETER

AIM

To calibrate the given venturimeter for a whole range of Reynolds number and to determine the coefficient of discharge.

MATERIALS REQUIRED

- (i) Venturimeter
- (ii) Stopwatch
- (iii) Meter scale

THEORY

A venturimeter shown is usually inserted directly into a pipeline. A short conical inlet section leads to a throat section, then to a long discharge cone. Pressure taps at the start of the inlet section and at the throat are connected to a manometer, which measures the pressure difference ($P_1 - P_2$) between points 1 and 2.

In the upstream cone the fluid velocity is increased and its pressure decreased. The pressure drop in this cone is used to measure the flow rate. In the discharge cone the velocity is decreased and the original pressure largely recovered in the downstream cone.

To make pressure recovery large, the angle of discharge cone is made small between 5° and 15° to prevent boundary layer separation and to minimize friction. Since there is no separation in a contracting cross section, the upstream cone can be made shorter than the downstream cone and space and materials are conserved.

Venturi can be applied to the measurement of gas flow rates; they are most commonly used with liquids especially large flows of water where, because of the large pressure recovery, a venturi requires less power than other types of meters.

The basic equation for the venturimeter is obtained by writing the Bernoulli's equation for incompressible fluids between two pressure stations. Friction is neglected and the pipe is assumed horizontal, turbulent flow. If v_1 and v_2 are the average velocity at 1 and throat velocity at point 2 with diameter D_1 and D_2 ,

The Bernoulli's equation reduces to

$$v_1^2 + P_1/\rho = v_2^2/2 + P_2/\rho \quad (1)$$

The continuity equation for constant p is

$$v_1 [\pi D_1^2/4] = v_2 [\pi D_2^2/4] \quad (2)$$

Combining (1) and (2) and eliminating v_1

$$v_2 = \sqrt{1 \times [2(P_1 - P_2) / \rho (1 - (D_2/D_1)^4)]} \quad (3)$$

To account friction loss, the equation (3) is corrected by introducing the correction factor called C_v . Then

$$v_2 = C_v \times \sqrt{[2(P_1 - P_2) / \rho (1 - (D_2/D_1)^4)]} \quad (4)$$

For $N_{Re} > 10^4$, C_v is about 0.98 for pipe diameters below 0.2 m and 0.99 for larger sizes. This may vary and individual calibration is needed for every meters.

FORMULAE USED

Difference in Manometer Reading

R_m	$= h_1 - h_2, \text{ cm}$
ΔP	$= P_1 - P_2 = R_m(\rho_1 - \rho_2)g, \text{ N/m}^2$
g	$= \text{Acceleration due to gravity} = 9.81 \text{ m/s}^2$
Where ΔP	$= \text{Pressure drop at stations a and b}$
Pressure head ΔH_{H_2O}	$= R_m (\rho_1 - \rho_2) / \rho_2, \text{ m of water}$

Volumetric flow rate Q = Volume of water collected / Time taken for 5 cm level of water, m^3/sec

Volume of water collected = Area of the collecting tank x Height of the tank, m^3

Actual velocity

$$u_v = Q/A_v$$

Theoretical velocity or velocity

$$u_{th} = \sqrt{2\Delta P / (1-\beta^4)\rho_2} \text{ , m/s}$$

Where ρ_2 = Density of water, kg/m^3

ρ_1 = Density of mercury, kg/m^3

β = D_v / D_p

C_o = Coefficient of discharge.

D_v = Diameter of the venturi throat, m

D_p = Pipe diameter, m

Cross sectional area of the venturi throat $A_v = \pi D_v^2 / 4$, m^2

Reynolds number

$$[N_{Re}]_{exp} = D_p u_v \rho_2 / \mu$$

$$C_v = \frac{\text{Actual velocity}}{\text{Theoretical velocity}}$$

PROCEDURE

- (i) Switch on the pump. Maintain a constant head in the tank by having a continuous over flow of water.
- (ii) Open the inlet or control valve fully so that levels of the manometric fluid in the two limbs of the manometer are equal.
- (iii) Note the initial manometer reading for zero flow rate.
- (iv) Slightly open the delivery valve or discharge valve for minimum possible flow rate so that there is a slight difference in the levels of the manometric fluid in the manometer.
- (v) Note down the manometer reading. Close the outlet valve of the collecting tank and record the time taken for approximately 10 cm rise in the water level.
- (vi) Repeat the above procedure for different flow rates to cover the full range of manometer.

GRAPHS

Plot the Graphs : C_o vs. N_{Re} , u_0 vs. u_{th} , ΔH_{Hg} vs. Q

OBSERVATIONS

Density of water	=	Kg / m^3
Density of Mercury	=	Kg / m^3
Diameter of the pipe D_p	=	m
Diameter of the venturi throat, D_v	=	m
Cross sectional area of the venturi throat A_v	=	$\pi D^2 / 4 = \text{m}^2$
Area of the collecting tank	=	m^2

Tabular Column 1

S.No.	Manometer Reading, m		R_m m	ΔH_{H_2O} , m of water	Volume of Water Collected, m^3	Time sec	ΔP N/m^2
	h_1	h_2					

Tabular Column 2

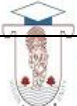
Volumetric Flow Rate Q , m^3/s	Actual Velocity u_v , m/s	Theoretical Velocity u_{th} , m/s	N_{Re}	C_v

MODEL GRAPH

u_o Vs u_{th}

C_v Vs N_{Re}

ΔH_{Hg} Vs Q



EXPT. NO. :

DATE :

CALIBRATION OF ORIFICEMETER

AIM

To calibrate the given Orificemeter for a whole range of Reynolds number and to determine the Coefficient of discharge.

MATERIALS REQUIRED

- (i) Orificemeter
- (ii) Stopwatch
- (iii) Meter scale

THEORY

Orifice is a device, which is used for measuring the flow of fluids. A orifice meter is considered to be a thin plate containing an aperture through which the fluid flows. It may be placed in the side or bottom of a container.

An orificemeter consists of an accurately machined and drilled plate having hole of diameter D_o mounted between two flanges in a pipe of diameter D_1 . The opening in the plate may be level on downstream side. Pressure taps at point 1 upstream and 2 downstream measure $p_1 - p_2$. The taps are installed about 1 pipe diameter upstream. And 0.3 to 0.8 pipe diameter.

The reduction of cross-section of the flowing stream while passing through the orifice plate increases the velocity head of the flowing stream at the expense of pressure head (forms a vena contracta or free-flowing jet) and the reduction in the pressure between the taps is measured by manometer, Bernoulli's equation provides a basis for correlating the increase in velocity head with the decrease in pressure head.

Theoretical velocity or velocity through orifice is given by

$$u_o = \sqrt{2\Delta P / (1 - \beta^4) \rho_2} \text{ ,m/s}$$

Since the pressure difference between the tapping is a function of rate of flow, the flow meter can be calibrated. The orifice coefficient C_o is almost constant and is independent of β provided if $N_{Re, o}$ is above 30,000, and D_o/D_1 is less than about 0.5. The value of C_o is 0.61. The coefficient 0.61 is not accurate when N_{Re} is less than about 30,000.

One important application appears in orifice meter that is not found in the venturi is because of the sharpness of the orifice, the fluid stream separates from the downstream side of the orifice plate and forms a free flowing jet in the downstream fluid. A vena contracta forms. The jet is not under the control of solid walls, as in the case of venturi, and the area of jet varies from that of the opening in the orifice to that of the vena contracta.

FORMULAE USED

Difference in manometer reading

R_m	$= h_1 - h_2, \text{ m}$
ΔP	$= P_1 - P_2 = R_m (\rho_1 - \rho_2) g, \text{ N/m}^2$
g	$= \text{Acceleration due to gravity} = 9.81 \text{ m/s}^2$
Where ΔP	$= \text{Pressure drop at stations 1 and 2}$
Pressure head developed ΔH_{H_2O}	$= R_m (\rho_1 - \rho_2) / \rho_2, \text{ m of water}$
Volumetric flow rate Q	$= \text{Volume of water collected} / \text{Time}, \text{ m}^3/\text{sec}$
Volume of water collected	$= \text{Area of the collecting tank} \times$ $\text{Height of the tank}, \text{ m}^3$

Actual velocity through orifice $u_o = Q/A_o, \text{ m/s}$

Theoretical velocity or velocity through orifice

$$u_{th} = \sqrt{2\Delta P / (1 - \beta^4) \rho_2} \text{ ,m/s}$$

Where ρ_2 = Density of water, kg/m^3
 ρ_1 = Density of mercury, kg/m^3
 β = D_o/D_1
 C_o = Coefficient of discharge.
 D_o = Orifice diameter, m
 D_1 = Pipe diameter, m
Cross sectional area
of the orifice A_o = $\pi D^2/4$, m^2

Reynolds number

$$N_{Re} = D_o u_o \rho_2 / \mu$$

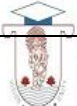
$$C_o = \frac{\text{Actual velocity}}{\text{Theoretical velocity}}$$

PROCEDURE

- (i) Switch on the pump. Maintain a constant head in the tank by having a continuous over flow of water.
- (ii) Open the inlet or control valve fully so that levels of the manometric fluid in the two limbs of the manometer are equal.
- (iii) Note the initial manometer reading for zero flow rates.
- (iv) Slightly open the outlet or discharge valve for minimum possible flow rate so that there is a slight difference in the levels of the manometric fluid in the manometer.
- (v) Note the steady level in the manometer. Close the outlet valve of the collecting tank and record the time taken for approximately 10 cm rise in the water level. Repeat the above procedure for different flow rate to cover the full range of manometer.

GRAPHS

Plot the Graphs of C_o Vs. N_{Re} , u_o Vs. u_{th} , R_m Vs. Q (Calibration graph)



OBSERVATIONS

Density of water	=	1000 Kg / m ³
Density of Mercury	=	13600 Kg / m ³
Diameter of the pipe D ₁	=	m
Diameter of the orifice D _o	=	m
Cross sectional area of the orifice A _o	=	$\pi D_o^2/4 =$ m ²
Area of the collecting tank	=	m ²
Viscosity of water	=	Kg / m s

Tabular Column 1

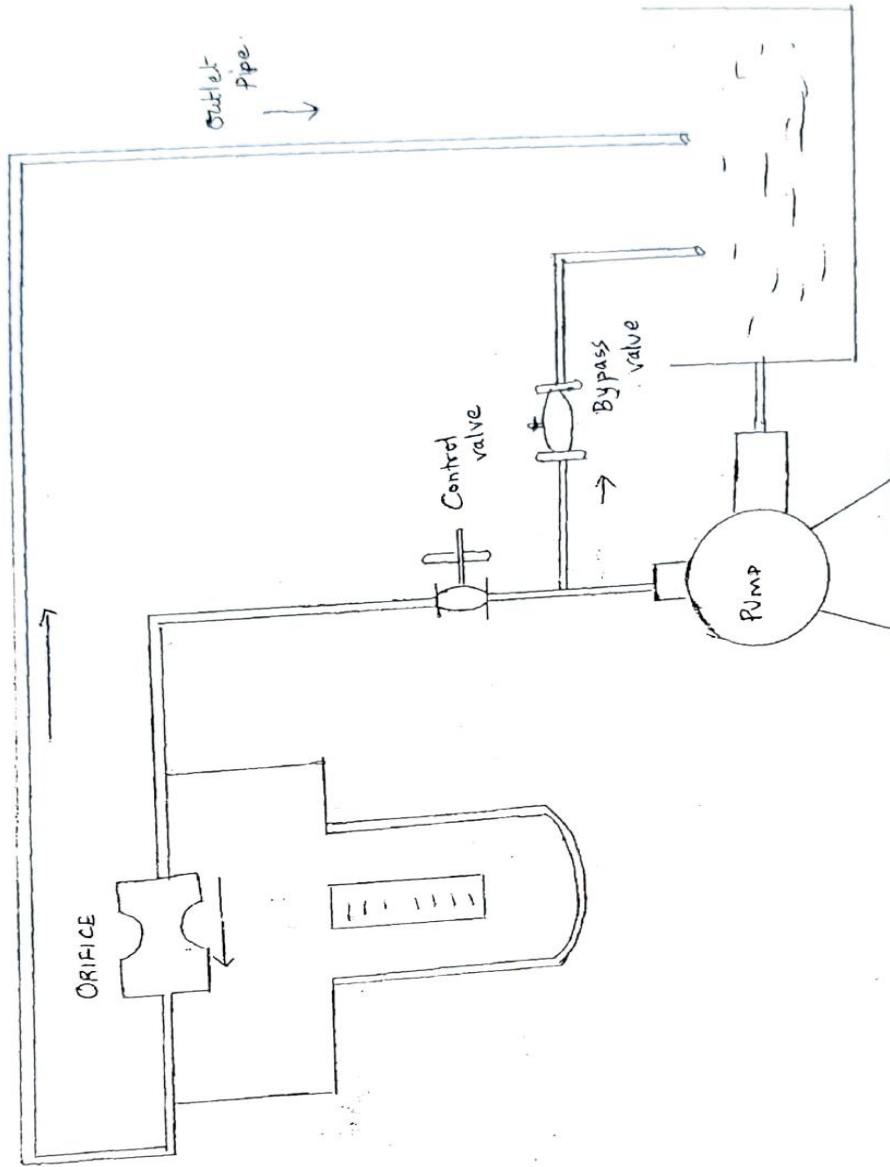
S. No.	Manometer Reading, cm		R _m , m	ΔH_{H_2O} , m of Water	ΔP N/m ²	Volume of Water Collected, V, m ³	Time sec
	h ₁	h ₂					

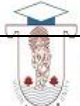
Tabular Column 2

S. No	Volumetric Flow Rate Q, m ³ /s	Actual Velocity u _o , m/s	Theoretical Velocity u _{th} , m/s	N _{Re}	C _o

MODEL GRAPHS

CALIBRATION OF ORIFICEMETER





EXPT. NO. :

DATE :

PARALLEL FLOW HEAT EXCHANGER

AIM

To determine the overall heat transfer coefficient of a double pipe heat exchanger in parallel flow condition

MATERIALS REQUIRED

- (i) Double pipe exchanger
- (ii) Thermometers
- (iii) Stop clock
- (iv) Measuring cylinders.

SPECIFICATIONS

Length of the heat exchanger	=	ft		
Inner copper tube I. D.	=	mm, O.D.	=	mm
Outer steel tube I. D	=	mm, O.D	=	mm

THEORY

In the process industries the transfer of heat between two fluids is generally done in heat exchangers. Heat exchangers are classified as i. Transfer type heat exchanger ii. Storage type heat exchanger iii. Direct contact type heat exchanger. The most common type (transfer) is one in which the hot and the cold fluid do not come into contact with each other but are separated by a tube wall or flat or curved surface. The transfer of heat is accomplished from the hot fluid to the wall or tube surface by convection, through the tube wall or plate by conduction, and then by convection to the cold fluid.

The simplest exchanger is the double pipe or concentric pipe exchanger. The one fluid flows inside on pipe and the other fluid in the annular space between the two pipes. The flow of fluids can be in parallel or cocurrent or counter current. The exchanger can be made from a pair of single length of pipe with fittings at the ends or from a number of pairs interconnected in series. This type of exchanger is useful mainly for small flow rates. If larger flows are involved, a shell and tube exchanger is used, which is most important type of exchanger in use in the process industries.

FORMULAE

(i) **Heat transfer from hot water**

$$Q_h = m_h C_{ph} (T_{hi} - T_{ho}), \text{ Watts}$$

Where Q_h = Heat transfer from hot water, W

m_h = Mass flow rate of hot fluid, Kg / s

C_{ph} = Specific heat capacity of hot fluid at constant pressure, J / Kg.

K

T_{hi} = Inlet temperature of hot water, °C

T_{ho} = Outlet temperature of hot water, °C

(ii) **Heat absorbed by cold water**

$$Q_c = m_c C_{pc} (T_{co} - T_{ci}), \text{ Watts or J/s}$$

Where Q_c = Heat transfer from cold water, W

m_c = mass flow rate of cold fluid, kg/s

C_{pc} = Specific heat capacity of cold fluid at constant pressure, J/kg

K

T_{co} = outlet temperature of cold fluid, °C

T_{ci} = inlet temperature of cold fluid, °C

(iii) **Logarithmic mean temperature difference**

$$\text{LMTD or } \Delta T_{\ln} = \frac{\Delta T_1 - \Delta T_2}{\ln (\Delta T_1 / \Delta T_2)}$$

Where

$$\Delta T_1 = T_{hi} - T_{ci}$$

$$\Delta T_2 = T_{ho} - T_{co}$$

(iv) **Overall Heat transfer coefficient**

$$Q = U_{\exp} A_o \Delta T_{\ln}$$

Where U_{\exp} = Overall heat transfer coefficient, $W/m^2 K$
 A_o = Area of heat exchanger (Outside area of inner tube) in m^2
 $= \pi d_o L, m^2$
 d_o = outer diameter of inner tube, m.

(v) **Effectiveness ε** =
$$\frac{\text{Actual heat transfer rate in a given exchanger}}{\text{Maximum possible amount of heat transfer}}$$

$$= Q_{\min} / Q_{\max}$$

Maximum possible amount of heat transfer

$$Q_{\max} = (mC_p)_{\min} (T_{hi} - T_{ci})$$

If hot fluid is minimum fluid i.e. the fluid which has the minimum value of mC_p or C_{\min}

$$\varepsilon_h = \frac{m_h C_{ph} (T_{hi} - T_{ho})}{(m_h C_{ph} (T_{hi} - T_{ci}))}$$

If cold fluid is minimum

$$\varepsilon_c = \frac{m_c C_{pc} (T_{ci} - T_{co})}{m_c C_{pc} (T_{hi} - T_{ci})}$$

PROCEDURE

- Start the Unit and check the water flow direction
- Adjust the valve in the exchanger to parallel flow condition
- Adjust the flow to some known quantity
- Record the outlet and inlet temperatures of hot and cold fluid
- Repeat the experiment for different flow rates.

GRAPH

Plot ϵ Vs. NTU

RESULT

The Overall heat transfer coefficients for a double pipe heat exchanger in parallel flow condition for different flow rates are determined and the graph of ϵ vs. NTU is drawn.

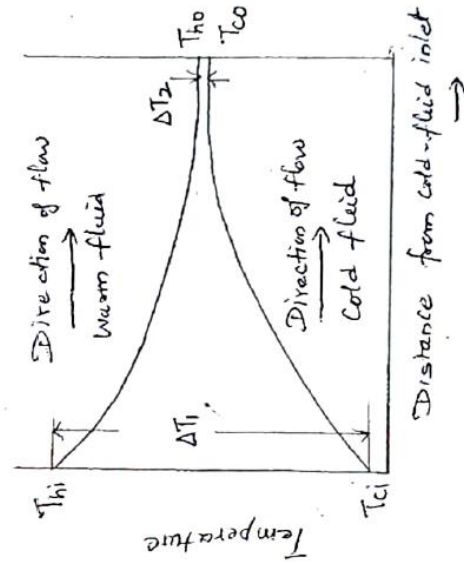
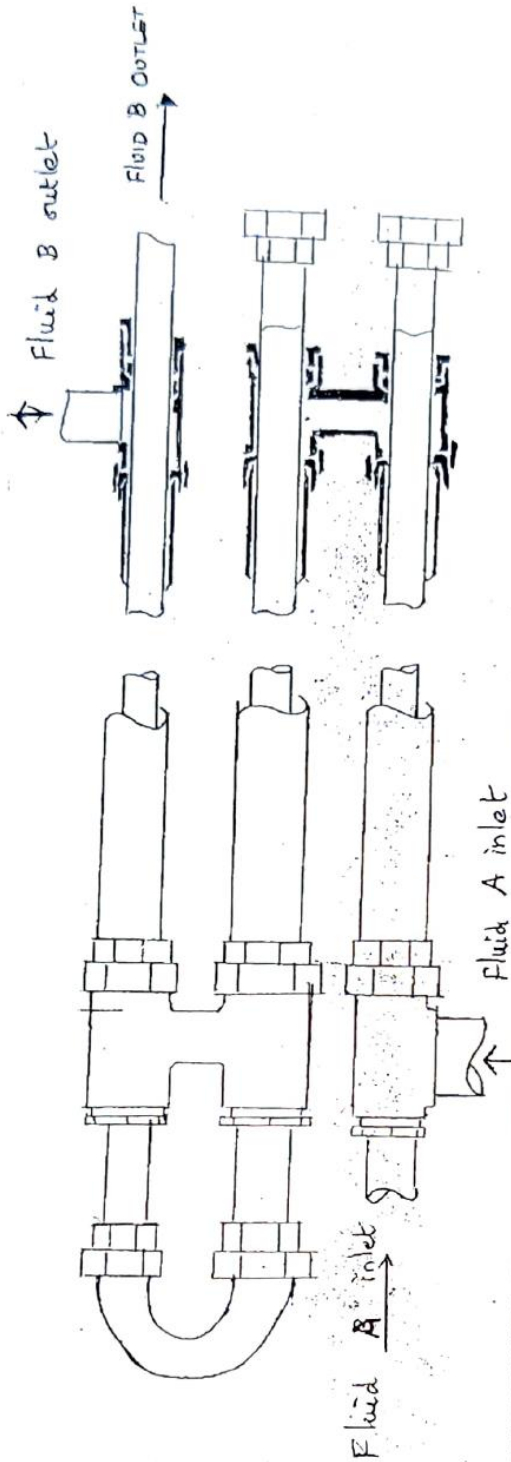
U_{exp} W/m^2K	Effectiveness ϵ	NTU = Number of Transfer Units

OBSERVATIONS

Tabular Column 1

S. No.	Hot Fluid			Cold Fluid		
	Mass Flow Rate m_h , kg / s	Inlet Temp. T_{hi} , °C	Outlet Temp. T_{ho} , °C	Mass Flow Rate m_c , kg / s	Inlet Temp. T_{ci} , °C	Outlet Temp. T_{co} , °C

PARALLEL FLOW HEAT EXCHANGER



EXPT. NO. :

DATE :

COUNTER FLOW HEAT EXCHANGER

AIM

To determine the overall heat transfer coefficient of a double pipe heat exchanger in counter flow condition

MATERIALS REQUIRED

- (i) Double pipe heat exchanger
- (ii) Thermometers
- (iii) Stop clock
- (iv) Measuring cylinders.

SPECIFICATIONS

Length of the heat exchanger	=	ft
Inner copper tube I.D.	=	mm, O.D. = mm
Outer steel tube I.D.	=	mm, O.D. = mm

THEORY

In the process industries the transfer of heat between two fluids is generally done in heat exchangers. Heat exchangers are classified as i. Transfer type heat exchanger ii. Storage type heat exchanger iii. Direct contact type heat exchanger. The most common type (transfer) is one in which the hot and the cold fluid do not come into contact with each other but are separated by a tube wall or flat or curved surface. The transfer of heat is accomplished from the hot fluid to the wall or tube surface by convection, through the tube wall or plate by conduction, and then by convection to the cold fluid.

The simplest exchanger is the double pipe or concentric pipe exchanger. The one fluid flows inside on pipe and the other fluid in the annular space between the two pipes. The flow of fluids can be in parallel or cocurrent or counter current. The exchanger can be made from a pair of single length of pipe with fittings at the ends or from a number of pairs interconnected in series. This type of exchanger is useful mainly for small flow rates. If larger flows are involved, a shell and tube exchanger is used, which is most important type of exchanger in use in the process industries.

FORMULAE

(i) Heat transfer from hot water

$$Q_h = m_h C_{ph} (T_{hi} - T_{ho}), \text{ Watts}$$

Where Q_h = Heat transfer from hot water, W

m_h = Mass flow rate of hot fluid in Kg / s

C_{ph} = Specific heat capacity of hot fluid at constant pressure

T_{hi} = Inlet temperature of hot water °C

T_{ho} = Outlet temperature of hot water °C

(ii) Heat absorbed by cold water

$$Q_c = m_c C_{pc} (T_{co} - T_{ci}), \text{ Watts}$$

Where Q_c = Heat transfer from cold water, W

m_c = Mass flow rate of cold fluid in Kg / s

C_{pc} = Specific heat capacity of cold fluid at constant pressure

T_{co} = Outlet temperature of cold fluid °C

T_{ci} = Inlet temperature of cold fluid °C

(iii) Logarithmic mean temperature difference

$$\text{LMTD or } \Delta T_{lm} = \frac{\Delta T_1 - \Delta T_2}{\ln (\Delta T_1 / \Delta T_2)}$$

Where $\Delta T_1 = T_{hi} - T_{co}$

$\Delta T_2 = T_{ho} - T_{ci}$

(iv) **Overall heat transfer coefficient**

$$Q = U_{\text{exp}} \cdot A_o \cdot \Delta T_{\text{ln}}$$

Where U_{exp} = Overall heat transfer coefficient, $\text{W/m}^2 \text{K}$

A_o = Area of heat exchanger (outside area of inner tube) in m^2

$$= \pi d_o L, \text{m}^2$$

d_o = Outer diameter of inner tube in m.

(v) **Effectiveness ε** =
$$\frac{\text{Actual heat transfer rate in a given exchanger}}{\text{Maximum possible amount of heat transfer}}$$

$$= Q_{\text{min}} / Q_{\text{max}}$$

Maximum possible amount of heat transfer

$$Q_{\text{max}} = (mC_p)_{\text{min}} (T_{\text{hi}} - T_{\text{ci}})$$

If cold is minimum fluid i.e the fluid which has the minimum value of mC_p

$$\varepsilon_c = \frac{m_c C_{pc} (T_{\text{co}} - T_{\text{ci}})}{(m_c C_{pc} (T_{\text{hi}} - T_{\text{ci}}))}$$

If hot fluid is minimum

$$\varepsilon_h = \frac{m_h C_{ph} (T_{\text{hi}} - T_{\text{ho}})}{m_h C_{ph} (T_{\text{hi}} - T_{\text{ci}})}$$

(vi) **NTU** = $UA_o / (mC_p)_{\text{min}}$

Where NTU = Number of Transfer Units.

PROCEDURE

- (i) Start the Unit and check the water flow direction
- (ii) Adjust the valve in the exchanger to counter flow condition
- (iii) Adjust the flow to some known quantity.
- (iv) Record the inlet and outlet temperatures of hot and cold fluid
- (v) Repeat the experiment for different flow rates.

GRAPH

Plot ϵ Vs. NTU

RESULT

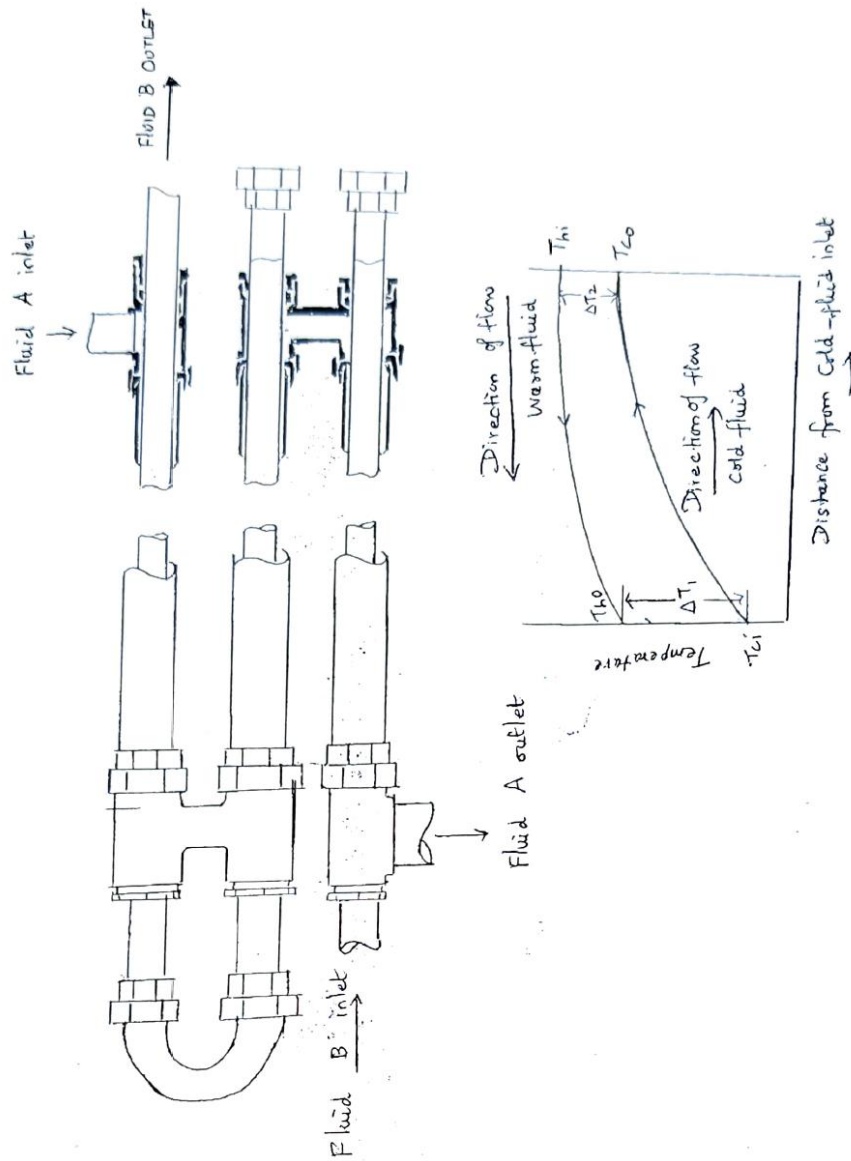
The Overall heat transfer coefficients for a double pipe heat exchanger in parallel flow condition for different flow rates are determined and the graph of ϵ vs. NTU is drawn.

U_{exp} W/m^2K	Effectiveness ϵ	NTU = Number of Transfer Units

OBSERVATIONS

S. No.	Hot Fluid			Cold Fluid		
	Mass Flow Rate $m_h, Kg / s$	Inlet Temp. $T_{hi}, ^\circ C$	Outlet Temp. $T_{ho}, ^\circ C$	Mass Flow Rate $m_c, kg / s$	Inlet Temp. $T_{ci}, ^\circ C$	Outlet Temp. $T_{co}, ^\circ C$

COUNTER FLOW HEAT EXCHANGER



EXPT. NO. :

DATE :

SIMPLE DISTILLATION

AIM

To verify the Rayleigh equation for the given system by Simple distillation.

MATERIALS REQUIRED

- (i) Simple distillation set up
- (ii) Weighing balance
- (iii) Measuring cylinders
- (iv) Weighing bottle.

SYSTEM

PRINCIPLE

Simple distillation is the process consisting of infinite number of successive steps of flash vaporization where in each step at infinite small portion of the liquid is vaporized and the resulting vapor, which is in the equilibrium with the liquid, is removed. Simple distillation is the batch operation and this operation is used to separate the liquid whose compounds have fairly large difference in their boiling points. This type of distillation is frequently employed in laboratory and in pilot plants to concentrate one compound in the distilled or residue. It is also used for analytical evaluation of boiling rate of mixtures. It is the simplest form of batch still consists of a heated vessel (pot or boiler) a condenser, and one or more receiving tanks. No trays or packing are provided. Feed is charged into the vessel and brought to boiling. Vapours are condensed and collected in a receiver. No reflux is returned. The rate of vaporization is sometimes controlled to prevent bumping the charge and to avoid overloading the condenser, but other controls are minimal. This is often called

Rayleigh distillation. Rayleigh developed the mathematical equation for simple distillation, which is given by

$$\ln[F / W] = \int_{x_w}^{x_F} dx / (y^* - x)$$

Where F = Feed, Kg / mole
W = Residue, Kg / mole
 x_F = Mole fraction of more volatile compound in feed
 x_w = Mole fraction of more volatile compound in residue.
 y^* = Equilibrium mole fraction of more volatile compound in vapour
 x = Equilibrium mole fraction of more volatile compound in liquid.

Rayleigh equation holds well under the following assumption

- (i) Composition of liquid is uniform
- (ii) Process is carried out slowly, so that the vapours leaving the still are in equilibrium with liquid in still.
- (iii) There is no entrainment in the liquid
- (iv) There is no condensation with the liquid vapour before it reaches the condenser.

PROCEDURE

- (i) Prepare a standard plot of density vs. mole fraction for the given system
- (ii) Take equal volume of benzene and toluene in the distillation flask after determining its density and mole fraction and fit it with condenser.
- (iii) Note down the initial temperature of the mixture.
- (iv) Heat the mixture and note down the temperature at which the first drop condense.
- (v) The mixture is distilled till the sufficient amount ($1/3^{\text{rd}}$ of total benzene charged) of distillate is obtained.
- (vi) Stop heating the mixture. Note down the final temperature of residue
- (vii) Collect the distillate and residue separately
- (viii) Note down the density of distillate and residue.

GRAPHS:

- (i) Density vs. mole fraction of benzene
- (ii) $1 / (y^* - x)$ Vs. x

RESULT

The Rayleigh equation for -----system is verified.

OBSERVATIONS

Volume of benzene	= 100 mL
Volume of toluene	= 100 mL
Density of benzene	= g / mL
Density of toluene	= g / mL
Room temperature	= °C
Temperature at first drop condense (vapor distillate)	= °C
Temperature at which half the liquid vaporized (residue)	= °C

Tabular Column 1

Equilibrium data for

Equilibrium Mole Fraction of More Volatile Component in Liquid, x	Equilibrium Mole Fraction of More Volatile Component in Vapour, y	$1 / (y^* - x)$

S. No.	Volume of mL	Volume of mL	Weight g	Density G / mL	Mole Fraction of -----

CALCULATION

Volume of distillate collected	=	mL
Volume of residue collected	=	mL
Weight of residue	=	g
Weight of distillate	=	g
Density of distillate	=	g / mL
Density of residue	=	g / mL
Temperature at first drop condense (vapor distillate)	=	°C
Temperature at which half the liquid vaporized (residue)	=	°C

To find F and x_F

S. No.	Component	Volume mL	Density g / mL	Weight g	Moles gmol	Molecular Weight	Mole Fraction
1.		100					
2.		100					

To find W and D

Overall balance:

$$F = D + W$$

Material balance of benzene

$$F x_F = D x_D + W x_w$$

$$F = \text{Moles of feed} = \text{g / mol}$$

$$x_F = \text{Mole fraction of more volatile compound () in the feed}$$

$$x_D = \text{Mole fraction of in the distillate}$$

$$x_w = \text{Mole fraction of in the residue}$$

$$D = \text{Moles of distillate, g / mol}$$

$$W = \text{Moles of residue, g / mol}$$

Verification of Rayleigh's equation

$$\text{From the graph } 1 / (y^* - x) \text{ Vs. } x, \text{ Area under the curve} = m^2$$

Rayleigh equation

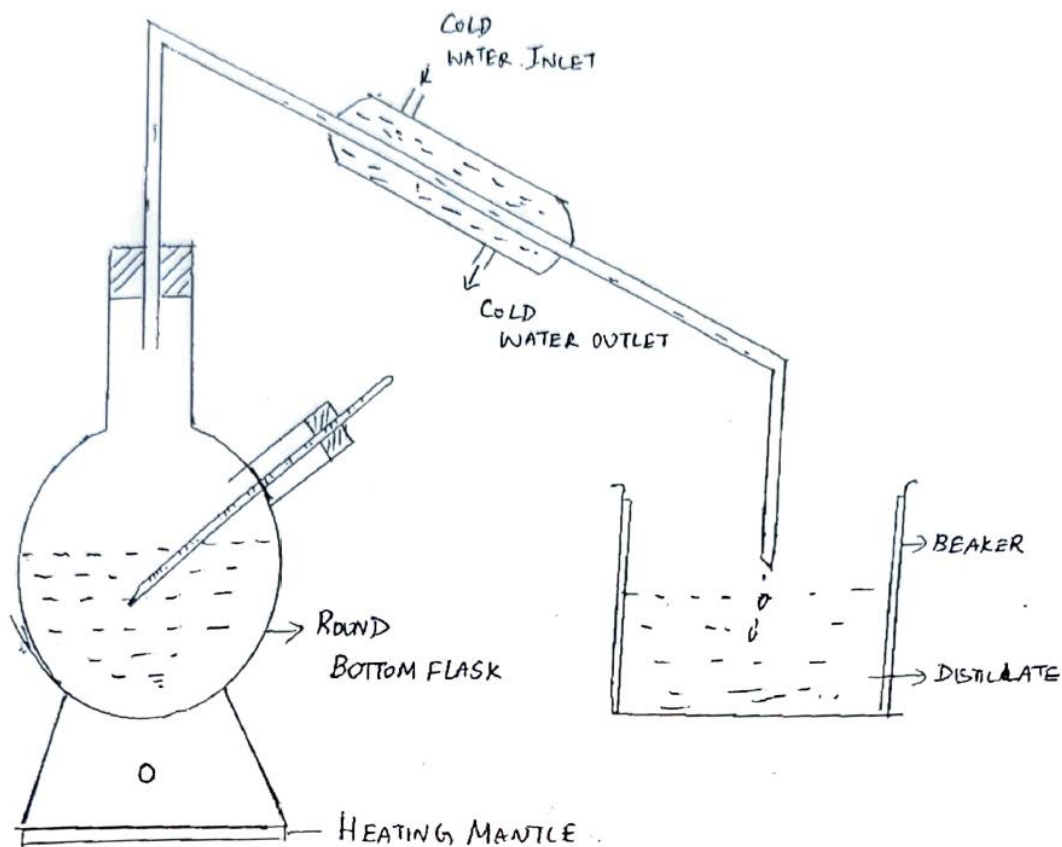
$$\ln[F / W] = \int_{x_w}^{x_F} dx / (y^* - x)$$

MODEL GRAPH

$1 / (y^* - x)$ Vs. x

Density g / mL vs. x (Mole fraction of -----)

SIMPLE DISTILLATION



EXPT. NO. :

DATE :

LIQUID-LIQUID EQUILIBRIUM

AIM

To determine the liquid-liquid equilibrium for the given tertiary system (Benzene-Acetic acid- water) and to draw the bimodal diagram for the above system

MATERIALS REQUIRED

- (i) Conical flasks
- (ii) Burette
- (iii) Pipette
- (iv) Measuring cylinders
- (v) Benzene
- (vi) Acetic acid
- (vii) Distilled water.

THEORY

Liquid-Liquid extraction also called solvent extraction is the separation of constituents of liquid solution by contact with another insoluble liquid. If the substances' consisting of the original solution distributes themselves differentially between the two liquid phases, a certain degree of separation will result.

Extraction involves the use of system composed of at least three substance and two phases are chemically quiet different which leads to a separation of the components according to physical or chemical properties. Generally all three components appear to some extent in both phases. In all such operation, the solution which is to be extracted is called feed and the liquid with which feed is contacted is the solvent. The solvent rich product of the operation is extract and the residual liquid from which the solute has been removed is called raffinate. Solvent extraction can

sometimes used as an alternative to separation by distillation. For example, Acetic acid can be removed from water by liquid-liquid extraction using organic solvent. The resulting organic solvent and acetic acid solution is the distilled.

Equilibria and Phase Composition

One of the mostly common type of system in extraction whose one pair is partially soluble, we observe that liquid C dissolves in A and B. A and B pair is partially soluble.

PROCEDURE

- (i) Clean all conical flasks and dry. In first five conical flasks add 25 mL of water.
- (ii) Add 4,8,12, 16 and 20 mL of acetic acid in each flask containing water.
- (iii) Agitate the flask containing the solution for half an hour.
- (iv) Titrate each solution against benzene taken in a burette.
- (v) In the remaining five conical flasks add 25 mL of benzene.
- (vi) Add the acetic acid in the same manner in each flasks containing benzene solution.
- (vii) Agitate the sample and titrate against water taken in a burette.
- (viii) The end point is turbidity.

RESULT

The liquid - liquid equilibrium for tertiary system is determined and the binodal diagram is drawn.

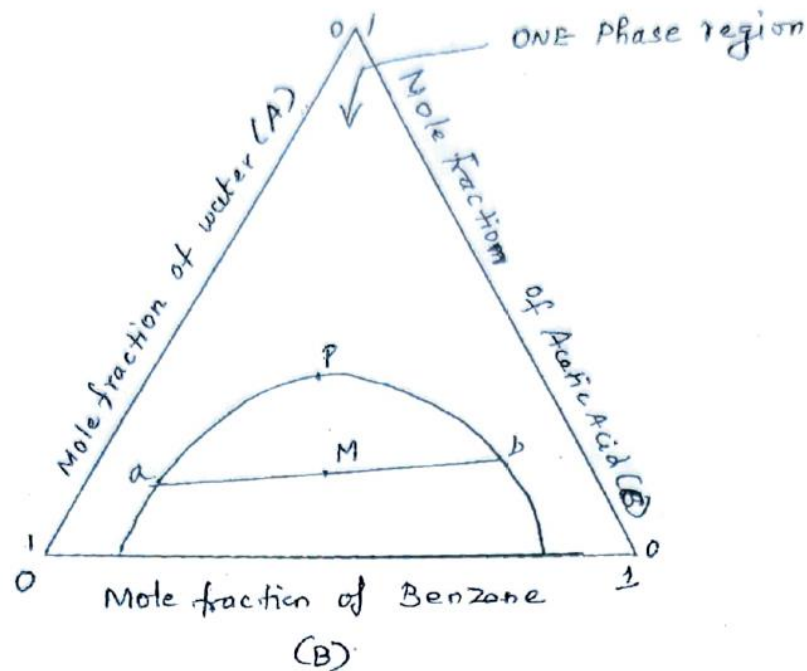
OBSERVATION

Density of acetic acid = g / mL
Density of water = g / mL
Density of benzene = g / mL

Tabular Column

S. No.	Volume of Water, mL	Volume of Acetic Acid, mL	Volume of Benzene, mL	Weight Fraction of Water	Weight Fraction of Acetic Acid	Weight Fraction of Benzene
S. No.	Volume of Benzene, mL	Volume of Acetic Acid, mL	Volume of Water, mL	Weight Fraction of Water	Weight Fraction of Acetic Acid	Weight Fraction of Benzene

LIQUID-LIQUID EQUILIBRIUM



A and B — Partially miscible system

Component C: Soluble in both A & B

P: plait point

EXPT. NO. :

DATE :

BATCH ADSORPTION

AIM

- (i) To study the adsorption of oxalic acid from aqueous solution on activated carbon and to draw the adsorption isotherms
- (ii) To determine the constants of Freundlich and Langmuir isotherm equations.

MATERIALS REQUIRED

- (i) Burette
- (ii) Conical flasks
- (iii) Pipette
- (iv) Measuring cylinders
- (v) Standard flasks
- (vi) Oxalic acid
- (vii) Activated carbon
- (viii) Sodium hydroxide.

THEORY

In adsorption processes one or more components of a gas or liquid stream are adsorbed on the surface of a solid adsorbent and a separation is accomplished.

Adsorption is a surface phenomenon; the adsorption processes may be physical or chemical dependent on the binding force involved. Another classification is reversible

adsorbed material (adsorbate) is thus recovered and the solid adsorbent is ready for another cycle of adsorption. Adsorption from a solution is usually monomolecular (i.e.) adsorption ceases when the surface is completely covered. The amount of adsorption varies with the concentration of the solution. An expression representing the variation of the amount adsorbed with equilibrium concentration is known as adsorption isotherm. Numerous expressions have been proposed to reproduce experimental isotherms. The linear law can be expressed by an equation

$$q = Kc, \quad q = \text{kg adsorbate (solute)} / \text{kg adsorbent (solid)}$$

$$c = \text{kg adsorbate}/\text{m}^3 \text{ of fluid.}$$

The Freundlich isotherm equation often approximates data for physical adsorption and is given by

$$q = Kc^n$$

Where K and n are constants

The plot of q vs. c is practically a straight line at low concentration there by indicating a direct proportionality of the amount of adsorption, with the concentration of low values. At high values of concentration the curve becomes convex towards the axis of q, which shows that at the higher concentration the amount adsorbs increases less than proportionality occurring to the gradual saturation of the surface. On plotting $\log q$ vs. $\log c$, a straight line is obtained, slope of the line gives n and intercept will be $\log K$ from which constant K is obtained.

A more significant isotherm for physical adsorption derived on theoretical basis, is Langmuir isotherm and is given by

$$q = abc / (1 + ac)$$

a and b are constants.

The constant a is proportional to the heat of adsorption. Constant b is amount of adsorption. The equation was derived assuming there are only a fixed number of active sites for adsorption that only a monolayer is formed and that the adsorption is

reversible and reaches an equilibrium condition. By plotting c/q vs. c , a straight line is obtained with intercept $1/(ab)$ and slope $1/b$ from which a and b are calculated.

Uses

Applications of liquid phase adsorption include removal of organic compounds from water or organic solution, coloured impurities from organics and fermentation products from fermentor effluents.

Gas-phase adsorption includes removal of water from hydrocarbon gases, sulfur compounds from natural gas, etc.

PROCEDURE

- (i) Prepare 1 N solution of sodium hydroxide and 1 N solution of oxalic acid.
- (ii) Weigh 2 gm of activated carbon.
- (iii) Take five cleaned conical flasks (reagent bottles), and in the first flask add
i. 10mL of oxalic acid and 90mL of water
- (iv) Fill the rest of the flasks with varying proportions of the oxalic acid and water.
- (v) In each flask, add 2 gm of accurately weighed activated carbon
- (vi) Agitate the flasks in the bottle shaker for 10 min.
- (vii) After 10 min. of shaking, stop the shaker and leave for half an hour. Filter the contents of the flasks.
- (viii) Titrate 20mL of filtrate against standard NaOH solution using phenolphthalein indicator.
- (ix) Calculate the normality of oxalic acid c (filtrate) and x/m or q , weight of oxalic acid adsorbed by 2 gm of activated carbon.

GRAPHS

Freundlich isotherm

Plot q Vs. c to find slope and intercept which is given by

Slope = n

Intercept = $\log K$

K, n – Freundlich constants

Langmuir isotherm

Plot c/q vs. c
Slope = $1/b$; intercepts = $1/(ab)$

RESULT

Freundlich isotherm constants n =
 K =
Langmuir isotherm constants a =
 b =

OBSERVATIONS

Tabular Column 1

S. No.	Volume of Titrate Solution, mL	Volume of Sodium Hydroxide, mL	Normality of Oxalic acid c_f , N	Normality of Oxalic acid (Filtrate), c , g Oxalic acid / m ³ Solution

Tabular Column 2

Bottle No.	m g	x g	c , N	$q = x /$ m	c / q	$\log c$	$\log q$

CALCULATION

$$\text{Volume of NaOH } V_1 = \text{ mL}$$

$$\text{Normality of NaOH } N_1 = N$$

$$\text{Volume of filtrate } V_2 = \text{ mL}$$

$$\text{Normality of filtrate or oxalic acid } N_2 = \{V_1 \times N_1\} / V_2, N$$

$$\text{g of oxalic acid adsorbed, } x = \frac{(c_f - c) \times V \times \text{Equivalent weight}}{1000}$$

1000

Freundlich constants

$$x / m \text{ or } q = Kc^n$$

$$q = \text{g oxalic acid / g of activated carbon.}$$

$$x = \text{g of oxalic acid adsorbed, g}$$

$$m = \text{g of activated carbon used} = 2 \text{ g}$$

Take log on both sides

$$\log q = \log x + n \log c$$

$$\text{Slope} = n; \text{ intercept} = \log K, \text{ Calculate } K$$

Langmuir constants

$$q = abc / (1 + ac)$$

$$c / q = 1/(ab) + (c/b)$$

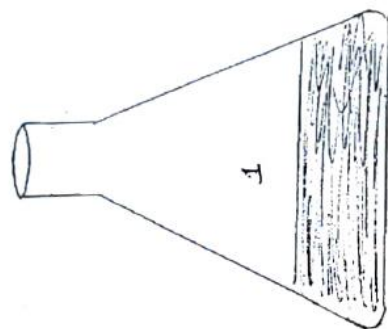
$$\text{Slope} = 1/b; \text{ intercepts} = 1 / ab$$

MODEL GRAPHS

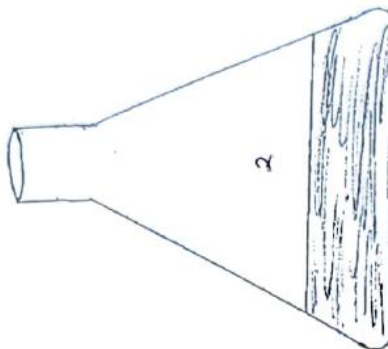
$\log q$ Vs. $\log c$

c/q Vs. c

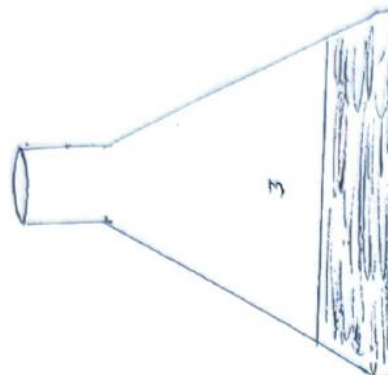
BATCH ADSORPTION



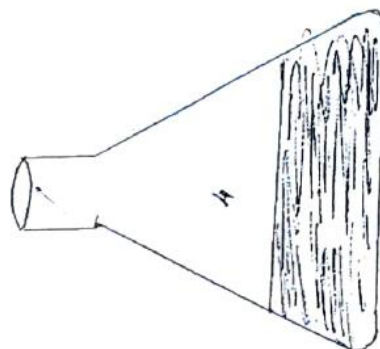
10 mL : OXALIC ACID ; 90 mL DISTILLED WATER
2 g : ACTIVATED CARBON



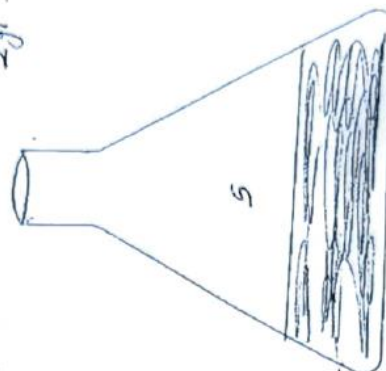
20 mL : OXALIC ACID
80 mL : DISTILLED WATER
2 g : ACTIVATED CARBON



30 mL : OXALIC ACID
70 mL : DISTILLED WATER
2 g : ACTIVATED CARBON



40 mL : OXALIC ACID
60 mL : DISTILLED WATER
2 g : ACTIVATED CARBON



50 mL : OXALIC ACID
50 mL : DISTILLED WATER
2 g : ACTIVATED CARBON

EXPT. NO. :

DATE :

BATCH SEDIMENTATION**AIM**

To determine the minimum area of a continuous thickener required to concentrate the feed of 5% CaCO_3 slurry at the rate of 175 tons / day and solids to give an underflow concentration 40% by carrying out batch sedimentation.

MATERIALS REQUIRED

- (i) Graduated measuring jar
- (ii) CaCO_3 slurry
- (iii) Stop clock.

THEORY

Sedimentation is a process of separation of dilute slurry by gravity settling into a clear fluid and the slurry of higher solid content. When the particle is at sufficient distance from the boundaries of the container and from other particles so that it falls without being effective then, the process is called free settling. If the motion is impeded by other particles, it is called hindered settling.

For lower N_{Re} , drag force on the particle obey's stroke's law. The law is valid at low velocities when the particle moves through the fluid by deforming it. The wall shear is the result of viscous force only. In gravitational settling 'g' is a constant and drag force increases with velocity, acceleration decreases with time and approaches zero. The particle then reaches a constant velocity called terminal settling velocity.

Newly prepared slurry having uniform concentration is taken in a cylinder as shown in fig. As the process begins the particles start settling. Different Zones of varying concentration are obtained. Zone B consists of heavier particles that settle

faster. Zone C is called transition layer consisting of variable size distribution and non uniform concentration. The layers are present as channels through which the fluid raises upward and the particles settles down. Zone B is of uniform concentration. Zone A the topmost layer is of clear liquid. As the sedimentation progresses, Zone A and Zone D grow larger at the expense of Zone B and the Zone C disappears which is known as critical point.

At this stage, solids present in these layers stops the settling process when the force of compression is equal to the weight of solid particle.

PROCEDURE

- Prepare 5% CaCO_3 slurry by taking 50gm of solid in a beaker. Minimum amount of H_2O was added and the contents were transferred to the graduated cylinder.
- Stir the content in a vertical manner until the concentration is uniform throughout the cylinder. Start the stop clock after stirring. Note down the time for each centimeter traveled by the solids.
- Measure the height using a scale fixed to the sides of the beaker. Note the time for every 1 cm, for about half the length after which the time is recorded for 0.5 cm until it reaches the ultimate bed height.

RESULT

The minimum thickener area of continuous thickener is m^2

OBSERVATIONS

Z_o	= Initial height of interface	=	cm
C_f	= Initial solid concentration	=	
C_u	= Concentration of underflow	=	
L_{cL}	= Rate of dry solids	=	

Tabular Column 1

S. No.	Z, cm	T, sec

Tabular Column 2

S. No.	Z _i , cm	T, sec	Rate of Sedimentation $V = (dz / dt) \times 10^{-3}, m / s$	$C_L = C_F \times (Z_o / Z_i)$

Tabular Column 3

S. No.	$V \times 10^{-3}, m / s$	C_L g / cc	$L_{cL} / A = (V \times 10^{-3}) / (1 / C_L - 1 / C_u), g / cm^2 s$



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