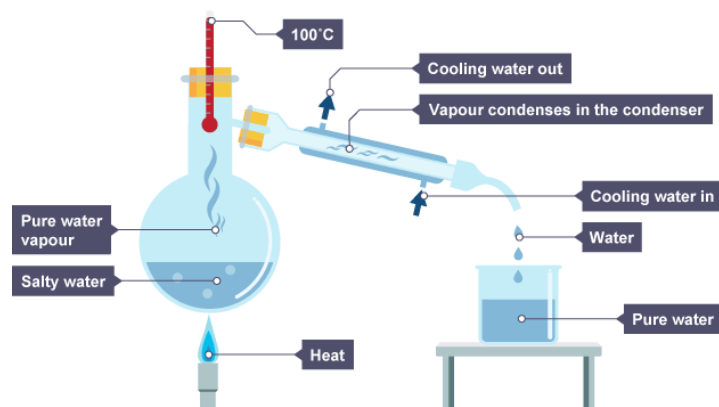


Laboratory Manual

for Mass Transfer Lab (CHC305)

AY: 2021-22



Department of Chemical Engineering
IIT(ISM)Dhanbad

Mass Transfer Lab (CHC305)

Sl. No.	Name of the Experiment	Page No.
1.	CALIBRATION OF REFRACTOMETER	05
2.	DETERMINATION OF GAS DIFFUSIVITY	10
3.	DETERMINATION OF MASS TRANSFER COEFFICIENT	17
4.	UNDERSTANDING SIMPLE DISTILLATION	20
5.	LIQUID-LIQUID EXTRACTION	24
6.	PACKED BED ADSORPTION STUDY	29
7.	LEACHING STUDY	33
8.	TRAY DRYER	36



Mass Transfer Lab (CHC305)

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

CALIBRATION OF REFRACTOMETER

AIM

To get the calibration curve for the Mettler Toledo refractometer for ethanol – water mixtures at different temperatures.

REQUIREMENTS

Mettler Toledo refractometer, heating plate, thermometer, Ethanol, Distilled water, 50 ml beakers (# 10), pipette, cleaning tissues.

INTRODUCTION

The Mettler Toledo refractometer is used for the simple determination of the refractive index of liquid samples. It has the following characteristics:

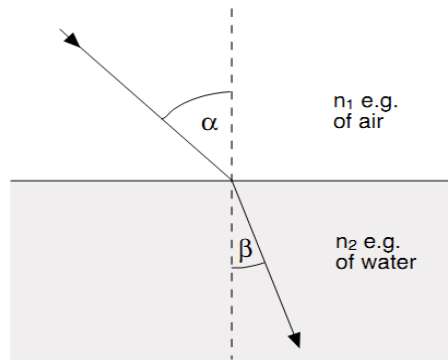
1. Measures samples with a refractive index in the range 1.3200 to 1.5000.
2. Needs a minimum amount of sample (min. 0.4 ml) for measurement.
3. Keeps the temperature of the sample being measured constant between 15 to 40 °C.
4. The light source for the measurement is an LED and prism is made of sapphire, which is strongly resistant to corrosion, very rigid and of high thermal conductivity.

REFRACTIVE INDEX

The refractive index 'n' of a substance is the ratio of the velocity of a ray of light in a vacuum to its velocity in the medium. If a ray of light at a particular angle passes from optically less dense (air) to optically more dense (water) medium, it changes its direction except when the incident light is vertical. According to Snell's law of refraction, the ratio of the refractive indices of the two media is proportional to the ratio of the angle of refraction and angle of incidence of the ray of light:

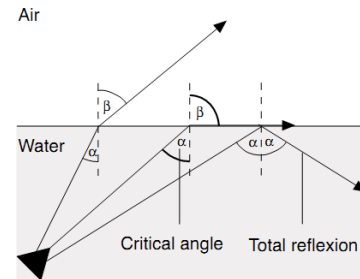


$$\frac{n_1 \sin(\beta)}{n_2 \sin(\alpha)}$$



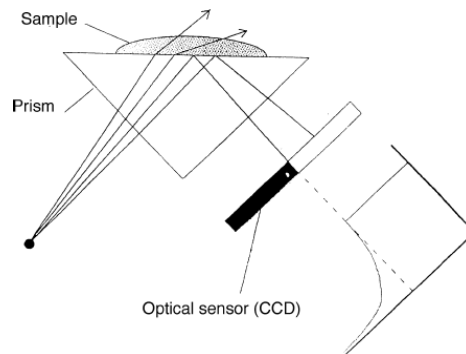
If a ray of light passes into an optically less dense medium from an optically denser medium, it also changes its direction. If the angle of incidence α is increased, it reaches a critical value (angle of refraction $\beta = 90^\circ$) at which the ray of light no longer passes into the optically less dense medium. If this "critical angle" is exceeded, total reflection occurs. The critical angle α is used to calculate the refractive index:

$$\frac{n_1}{n_2} = \frac{1}{\sin \alpha}$$



MEASUREMENT PRINCIPLE

The light emitted passes through the prism and encounters the sample. It is partially refracted (angle of incidence < critical angle) and partially reflected (angle of incidence > critical angle). The reflected light is recorded using an optical sensor (CCD). The boundary between the dark and light areas represents the critical angle needed to calculate the refractive index.



PROCEDURE

1. Prepare 10 different mixture samples of the given liquids with known mole fraction.
2. Place the refractometer on a table and place one or two drops of the liquid mixture sample on the prism.
3. Press the OK button on the meter and note down the refractive index and temperature from the screen.
4. Clean the prism with cleaning tissues and repeat the above steps for measuring the next liquid mixture sample.
5. Repeat steps 1 to 4 for different temperatures.

Mass Transfer Lab (CHC305)

OBSERVATIONS

Refractive Index of Ethanol-Water mixtures at 30 °C

Serial number	Volume of ethanol (ml)	Volume of water (ml)	Mole fraction of ethanol	Refractive index

Refractive Index of Ethanol-Water mixtures at 35 °C

Serial number	Volume of ethanol (ml)	Volume of water (ml)	Mole fraction of ethanol	Refractive index

Refractive Index of Ethanol-Water mixtures at 40 °C

Serial number	Volume of ethanol (ml)	Volume of water (ml)	Mole fraction of ethanol	Refractive index

RESULT

These measured refractive indexes were plotted against to their mole fraction of the mixtures.



Mass Transfer Lab (CHC305)

Note:

1. Compare these refractive index data of samples at different temperatures with the refractive index values in the literature (taking into consideration errors in observation and measurements, and the refractometer).
2. Write the report in your own words, mention the conclusions of the experiment and validate your conclusions by stating proper references.



Experiment NO 02

DETERMINATION OF GAS DIFFUSIVITY

AIM

To determine the diffusion coefficient of acetone vapour in the air at different temperatures and comparison with literature data.

REQUIREMENTS

Stefan tube apparatus, compressor, thermometer, stop watch, acetone.

THEORY

The principles of momentum transfer and heat transfer are applied in most branches of engineering but the application of the principles of mass transfer have traditionally been the province of the chemical engineer. Mass transfer refers to the tendency of a component of a fluid to flow from a region of high concentration to one of low concentrations.

Molecular diffusion is the transfer or movement of individual molecules through a fluid by random molecular movements. In the diffusion process, the molecules of interest flow from regions of high concentration to low concentration. Molecular diffusion can occur in both directions with the system. The diffusivity or diffusion coefficient (D_{AB}) is a property of the system, dependent upon the temperature, pressure and nature of the components.

Fick's law:

The rate of mass transfer is expressed by Fick's law which for molecular diffusion in the direction x in a fluid of constant molal density is expressed below.

$$J_A = -D_{AB} \frac{dC_A}{dx}$$

Where: J_A = flux of constituent A relative to the average molar velocity in units of moles/m²s

D_{AB} = diffusivity of constituent A in solution B in units of m²/s



Mass Transfer Lab (CHC305)

C_A = concentration of constituent A in units of moles/m³

The flux of the constituent is the rate of mass transfer per unit cross sectional area. The flux can be expressed as J_A relative to the average molar velocity or as N_A relative to a fixed location in space. For diffusion of A through stagnant B the flux is given by (for gases)

$$N_A = \frac{(D_{AB} P)}{(RTX \bar{P}_{BM})} (\bar{P}_{A1} - \bar{P}_{A2})$$

Where

$$\bar{P}_{BM} = \frac{(\bar{P}_{A1} - \bar{P}_{A2})}{\ln \frac{\bar{P}_{A2}}{\bar{P}_{A1}}}$$

A number of empirical correlations have been derived from experimental studies to predict the diffusivity of different substances diffusing through a liquid or a gas. For gases the following correlations give more reliable predictions.

1. Fuller – Schetler – Giddings (1966)

$$D_{AB} = \frac{10.13 \times 10^{-3} T^{1.75} (1/M_A + 1/M_B)^{1/2}}{P \left[\left(\sum_A V_i \right)^{1/3} + \left[\left(\sum_B V_i \right)^{1/3} \right]^2 \right]}$$

Where: D_{AB} = diffusivity of units m²/s

T = temperature of units °K

M_A, M_B = molecular weights of components A and B

P = absolute pressure of units N/m²



$$\sum v_i, \sum v_i$$

$\sum v_i$ = the summation of special diffusion volume coefficients for components A and B.

2. The Wilke – Lee modification of the Hirschfelder – Bird – Spatz method

$$D_{AB} = \frac{10^{-4} \left[1.084 - 0.249 \sqrt{\frac{1}{M_A} + \frac{1}{M_B}} \right] T^{3/2} \left[\sqrt{\frac{1}{M_A} + \frac{1}{M_B}} \right]}{P_t r_{AB}^2 f \left(\frac{kT}{\epsilon_{AB}} \right)}$$

Where: D_{AB} = diffusivity of units cm^2/s

T = temperature of units $^{\circ}\text{K}$

M_A, M_B = molecular weights of components A and B

P = absolute pressure of units Atmospheres

r_{AB} = molecular separation at collision, Angstroms = $(r_A + r_B)/2$

ϵ_{AB} = energy of molecular interaction, Ergs. = $(\epsilon_A \epsilon_B)^{1/2}$

$f \left(\frac{kT}{\epsilon_{AB}} \right)$ = Collision function for diffusion given in figure 1.

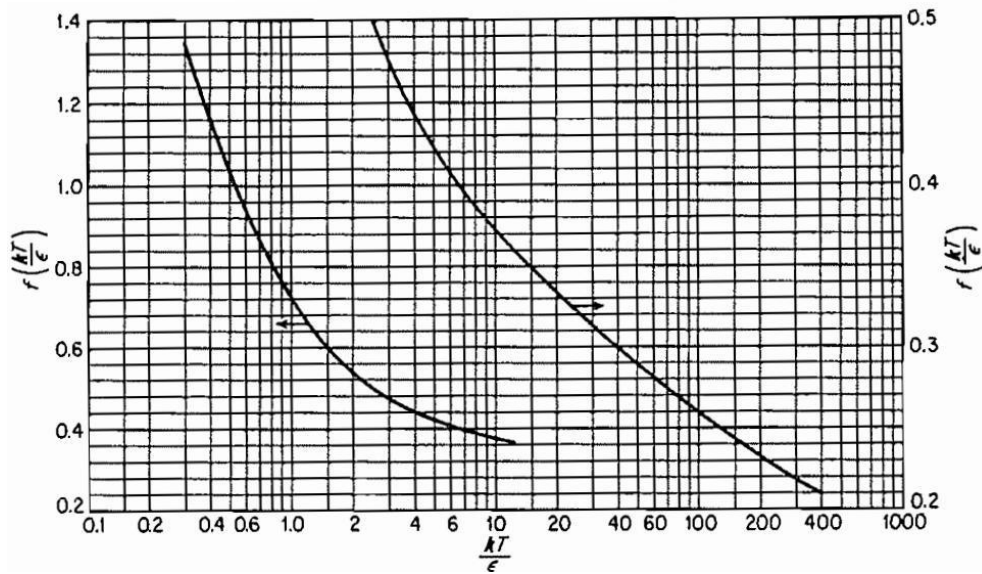


Figure 1. Collision function for diffusion

The values of r and ϵ are calculated from the properties of gases such as viscosity.

$$r = 1.18v^{1/3} \quad \text{where } v = \text{molal volume of liquid at normal boiling point, m}^3/\text{kmol}$$

$$\epsilon / k = 1.21T_b \quad T_b = \text{normal boiling point, K}$$

EXPERIMENTAL PROCEDURE

The apparatus employed to estimate the diffusivity of acetone vapour in air is shown in figure 2 below.

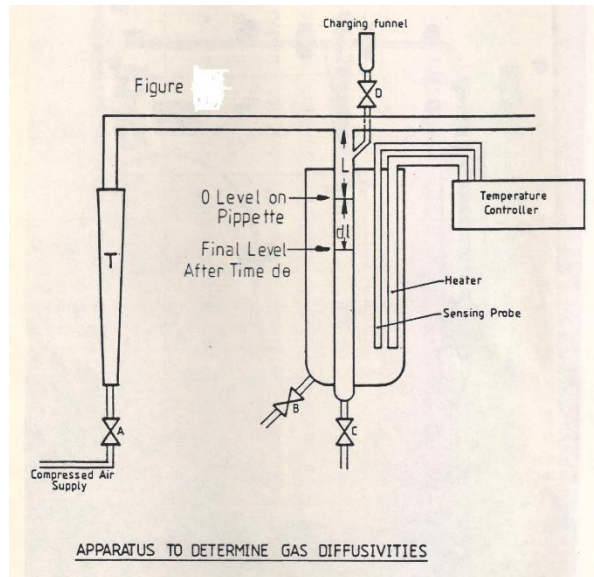


Figure 2. Apparatus to determine gas diffusivities

1. The method involves passing air over the open top of a graduated tube partially filled with liquid acetone which is maintained at a constant temperature.
2. The air flow rate is maintained at a rate sufficient to sweep away acetone vapour from the top of this tube, but, at the same time keeping the turbulence at the junction to a minimum.
3. Under these conditions the concentration of acetone vapour at the top of the graduated tube may be taken to be zero and the air above the acetone in the tube can be considered to be stagnant.
4. Before carrying out the experiment measure the distance from the '0' graduation of the pipette to the junction of the tube carrying the stagnant gas. Record this as L .
5. The pipette holds 10 ml and is graduated in 0.1 ml divisions, the distance between divisions is 2 mm (this should also be confirmed).

Mass Transfer Lab (CHC305)

OBSERVATIONS:

Sl.No	Temperature (°K)	L (m)	dl (m)	dθ (s)

CALCULATIONS:

In this experiment the flux N_A is created by drop in acetone level 'dl' in time $d\theta$

$$N_A = \frac{\text{mass transfer}}{\text{time} \times \text{molecular weight} \times \text{crosssectional area}}$$

$$N_A = \frac{dl \times S \times \rho_A}{d\theta \times M_A \times S} = \frac{dl \times \rho_A}{d\theta \times M_A} \text{-----(1)}$$

Where: S = cross sectional area

ρ_A = density of fluid

M_A = molecular weight

For diffusion of A through stagnant B in the liquid phase, the flux N_A is given by

$$N_A = \frac{CD_{AB}}{(y_2 - y_1)X_{BM}} (X_{A1} - X_{A2}) \text{-----(2)}$$

$$\text{Where } C = \frac{P}{RT}, X_A = \frac{P_A}{P_t}$$



Mass Transfer Lab (CHC305)

Where in the experiment:

y_1 is the level of acetone liquid in the pipette

y_2 is the junction of the air pipe, above the acetone carrying the stagnant gas

Equation (2) can be re-written as:

$$D_{AB} = \frac{N_A X_{BM} (y_2 - y_1)}{(X_{A1} - X_{A2})C}$$

From equation (1) & (2)

$$D_{AB} = \frac{(L + dl/2) \times X_{BM} \times dl \times \rho_A}{d\theta \times M_A \times C \times (X_{A1} - X_{A2})}$$

RESULT:

Diffusivity of acetone into air (experimental and theoretical) values at different temperature are calculated.



Experiment NO 03

DETERMINATION OF MASS TRANSFER COEFFICIENT

AIM

1. To calculate the mass transfer coefficient.
2. Compare the effect of temperature on the mass transfer coefficient

THEORY AND PRINCIPLES

Mass transfer occurs when a component in a mixture migrates in the same phase or from phase to phase because of a difference in concentration (Chemical potential) between two points e.g. liquid in an open pail of water evaporates into still air because of the difference in concentration of water vapor at the water surface and the surrounding air. This is a driving force from surface to air. There are two different modes of mass transfer, namely diffusion and convection. Diffusion is the movement under the influence of an individual component through a mixture and the physical mechanism is that of random atomic or molecular activity. The convection mass transfer mode is comprised of two mechanisms as free and forced convection. Free convection mass transfer involves thermally driven free convection flows which enhance free evaporation or sublimation occurring at a surface. In many cases, the rate of diffusion is slow, and more rapid mass transfer is desired. In the case of forced convection mass transfer, the flow is induced by external means such as a fan, pump or atmospheric winds etc.

Many chemical engineering problems involves transfer of mass from bodies such as solid particles or liquid droplets to a main fluid stream. A common starting point in investigating such problems is to consider the transfer mechanism from a single sphere in a flowing fluid. A typical plot for the mass transfer of a dissolving solid from a surface to a turbulent fluid in a conduit is given in figure 1. The concentration drop from C_{A1} adjacent to the surface is very abrupt close to the surface and then levels off and gradually decreases to C_{A2} .



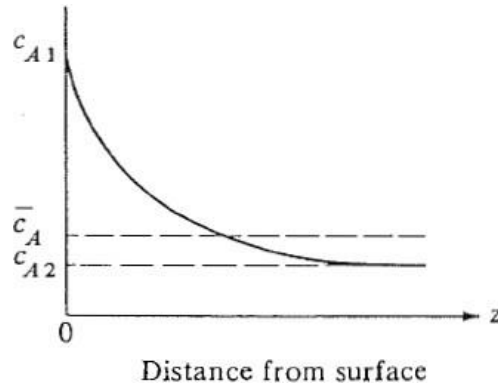


Figure 1

Correlations for mass transfer from single sphere are represented as addition of terms representing transfer by purely molecular diffusion and transfer by forced convection, in the form:

$$\text{Sh} = 2 + 0.552 \text{Re}^{0.53} \text{Sc}^{1/3} \quad (1) \quad \text{Ref: [3]}$$

For very low Reynold's number, the Sherwood number should approach a value of 2.

EXPERIMENTAL PROCEDURE

1. Take a naphthalene ball and measure its weight and diameter (at least in four directions and take average) decorously.
2. Switch on the equipment and set a fixed temperature and wait till it reached. Now switch on the blower and set a fixed flowrate.
3. Place the naphthalene ball in the space provided such that the hot air is flow past the ball and start the timer. After 30 mins remove the naphthalene ball and measure its weight and diameter.
4. Take manometer reading to find the velocity of the fluid.
5. Repeat the experiment for different temperatures and flowrates to observe the influence/effect of temperature and velocity of the fluid on the mass transfer coefficient.

CALCULATIONS

1. Let 'A' denote naphthalene and 'B' denote air. Since the mole fraction of naphthalene is very small, the physical properties of air at 45°C and 1 atm will be used for the gas mixture.

$$\mu = \mu_B = 1.93 \times 10^{-5} \text{ Pa.s} \quad \rho = \rho_B = 1.113 \text{ kg/m}^3$$

2. Now evaluate the dimensionless numbers:

$$Sc = \frac{\mu}{\rho D_{AB}}, \quad Re = \frac{\rho V D}{\mu}$$

3. Now calculate the Sherwood number from the equation (1) and calculate the theoretical mass transfer coefficient using the definition. i.e.

$$Sh = \frac{k_c D}{D_{AB}}$$

4. Calculate the theoretical flux and actual flux using measured data.
5. Report the actual/practical mass transfer coefficient obtained and compare with the theoretical value.

REFERENCES:

1. Incropera F.P, De Witt D. P, "Fundamentals of Heat and Mass Transfer", 4th Edition, John Wiley & Sons Inc., 1996.
2. Perry J.H, "Chemical Engineering Handbook", 4th Edition, Kogakusha Company, Tokyo, 1963.
3. Transport Processes and Separation Process Principles (Includes Unit Operations) 4th Ed. Christie J. Geankoplis



Experiment NO 04

UNDERSTANDING 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 form to a condenser, where the distillate is collected. The vapors which form as soon as the 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 $\frac{2}{3}$ rd 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 & x_w).
5. Plot $1/(y^* - x)$ versus x by using equilibrium data given. (See last page)
6. Determine the 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, $T_R =$ _____ °C

Density of water at $T_R = \rho_w =$ _____ gm/cc

Density of ethanol at $T_R = \rho_e =$ _____ 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, $x_F = \frac{(V_A * \rho_e)/M_A}{(V_A * \rho_e)/M_A + (V_B * \rho_w)/M_B}$

Refractive index of ethanol = $\mu_e =$ _____



Mass Transfer Lab (CHC305)

Refractive index of water = $\mu_w =$ _____

Weight of a beaker = $W_4 =$ _____ gm

Weight of a beaker + distillate $W_5 =$ _____ gm

Volume of Distillate = $V_D =$ _____ cc

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

Mole fraction of distillate, $x_D =$ _____ (from calibration chart)

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

Moles of distillate, $D = (V_D * \rho_D) / M_D =$ _____ gm moles

Weight of a beaker + Residue $W_6 =$ _____ gm

Volume of Residue = $V_r =$ _____ cc

Density of distillate = $\rho_r = [(W_6 - W_1) / (W_2 - W_1)] * \rho_w$, gm/cc

Mole fraction of residue, $x_w =$ _____ (from calibration chart)

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

$$\text{RHS} = \int_{x_W}^{x_F} \frac{dx}{y^* - x} = \text{_____ from graph of } x \text{ vs } 1/(y^* - x)$$

$$\text{LHS} = \ln(F/W) = \text{_____}$$

LHS \approx RHS, Thus the Rayleigh equation is verified.



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 05

LIQUID – LIQUID EXTRACTION

AIM

To determine LLE data for the given system (1-butanol – water – acetic acid).

THEORY

It is sometimes necessary to know the mutual solubilities of liquids in a two-phase system. For example, you may need to know how much water is dissolved in an organic liquid with which it is in contact, and also the amount of the organic compound that is in the aqueous phase. In this experiment you will consider a three-component mixture (1-butanol – water – acetic acid at 25 °C and barometric pressure) and construct the corresponding ternary phase diagram.

THE PHASE RULE:

Information regarding phase equilibria can be predicted by Gibbs phase rule.

$$f = c - p + 2$$

Where c is the number of components and p is the number of phases present in the system. The degrees of freedom f , or variance, gives the number of variables (e.g., pressure, temperature, composition, etc.) that must be given to completely describe the system, or to locate the state of the system on the phase diagram.



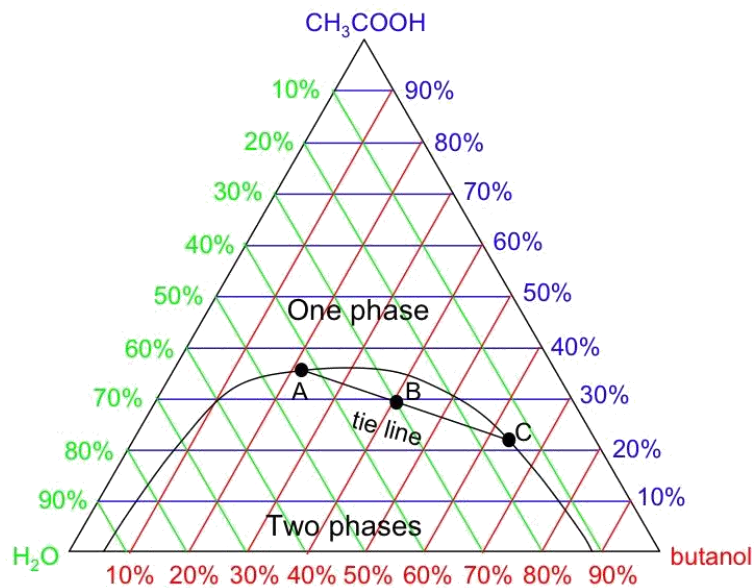


Figure 1: A triangular phase diagram

In figure 1 it is showing the representation of the mass fractions for ternary systems. The colors indicate how concentrations for different species should be read from the diagram. The point marked in the diagram (•) represents 30% 1-butanol, 10% water and 60% acetic acid. The one- and two-phase regions have been separated by a black line. This graph accounts for the fact that only two variables are required and along the phase boundary only one variable is required.

Regions where one or two phases appear have also been indicated in figure 1. Note that the line drawn is hypothetical, the real curve will be determined in this experiment. When the solution is stirred, the transition from one region to another can be observed by appearance (or disappearance) of cloudiness or turbidity in the solution. The turbidity results from scattering of light by the large number of very small “oily” droplets of the second phase that are produced when the system is stirred. Sometimes it is easier to see this when stopping the stirring briefly.

If the three components are mixed to give an overall system composition that falls in the 2-phase region, the system will separate into two phases: a phase rich in water and another rich in 1-butanol. The compositions of the phases that form are given by the intersections of a tie line with the phase boundary. The tie line must also contain the point describing the overall system composition. A graphical demonstration and an interpretation of a tie line are given in figure 1.

Note that in the figure 1 only the mass fraction of 1-butanol must be given when the system remains on the phase boundary line. This determines the mass fractions for water and acetic acid. Hence the phase rule holds with $f = 5 - p = 3$ (i.e., mass fraction for 1-butanol, temperature and pressure). If the system was initially in the two-phase region, the tie line uniquely connects the points along the phase separating line. Given the point 'A' in figure 1 (depending on the 1-butanol mass fraction in phase 1), the points 'B' and 'C' are uniquely determined. Thus only the 1-butanol mass fraction in phase 1, temperature and pressure are required for complete description of the system, which had separated into two phases. This is again in accordance with the phase rule. The point 'A' denotes the composition of phase 1, 'B' denotes the initial composition of the system, and 'C' the composition of phase 2.

EXPERIMENTAL PROCEDURE

The total experiment is divided into three parts. In the first part of the experiment, solubility of 1-butanol in water and solubility of water in 1-butanol will be determined. The switch to the two-phase region can be observed as appearance of the turbidity in the stirred solution. This gives the first two points in the phase diagram that lie along the horizontal axis (see Fig. 2; “the starting points for the arc”). In the second part, the points defining the arc will be determined by starting from the two-phase region and adding 17.5M acetic acid (molecular weight 60.05 g mol⁻¹) until the system switches into one phase. This transition can be observed by disappearance of the turbidity in the stirred solution. In the third part one of the tie lines is determined experimentally. This will be done by choosing a point from the two-phase region and determining the compositions of the two phases formed.

Part 1:

1. Place 20 mL of water in a 50 mL Erlenmeyer flask. Cover the flask with Parafilm. Poke the burette containing 1-butanol through the Parafilm. Add 1-butanol to the water drop by drop with continuous stirring until the turbidity (“small oily droplets form”) appears and remains for at least 5 min. It will take less than 2 mL of 1-butanol to reach this point. Record the amount 1-butanol required.



Mass Transfer Lab (CHC305)

2. Place 20 mL of 1-butanol in a 50 mL Parafilm-covered Erlenmeyer flask. Add water to the 1-butanol drop by drop with continuous stirring until the turbidity appears and remains for at least 5 min. It will take more than 2 mL but less than 5 mL to reach this point. Record the amount of water required.

Part 2:

1. Place 20 mL of 1-butanol and 5 ml of water in a 200 mL Parafilm-covered Erlenmeyer flask. Add acetic acid drop by drop while continuously stirring the solution until the turbidity disappears. Record the volume of acetic acid added.
2. Add an additional 5 mL aliquot of water to the mixture from the previous step. Again titrate the stirred solution with acetic acid until the turbidity disappears. Record the volume of acetic acid added.
3. Continue adding water in 5 mL aliquots and titrating to the turbid point with acetic acid until a total of 30 mL water has been added. After this add 10 mL aliquots and keep titrating until a total of 110 mL of water has been added. Record the volumes of acetic acid added at each stage.

Part 3:

1. Mix 25 mL of water and 25 mL of 1-butanol. Stir for 5 min and let the solution settle into two phases.
2. Add 3 mL of acetic acid and stir again for 5 min and let the solution settle. Place the solution in a separating funnel and separate. Determine the mass and volume of each phase.
3. Titrate the bottom phase with standardized 1.0 M NaOH. Obtain an average of three runs. Use three drops of 1% phenolphthalein solution as indicator.

Note: The standardization of NaOH must be done during the period that the bottom phase titration is done since the NaOH concentration changes rapidly when carbon dioxide is absorbed from the atmosphere. Use 0.1 N solution of oxalic acid for standardization. Use the same indicator for standardization.



CALCULATIONS

Calculate mass fractions X_i (in %) for each component in every solution (i.e., parts 1 and 2 in previous section) using the following equation:

$$X_i = \frac{V_i \rho_i}{\sum_{i=1}^3 V_i \rho_i} \times 100\%$$

Where V_i is the volume and ρ_i the density of the component i (water, acetic acid, 1-butanol). The densities of water, 1-butanol and acetic acid are 0.9970 , 0.8098 and 1.046 kg L^{-1} , respectively.

From the results obtained in part 3, determine the weight percent of the three components in each phase. Note that in part 3 just by knowing the mass percentage of acetic acid allows you to determine the 1-butanol and water concentrations as well (use the phase diagram previously determined and note that the points in part 3 must land on the phase boundary arc). However, there are two possibilities (i.e., the acetic acid mass fraction line can intersect the phase boundary arc at two different points) but you know that the densest liquid will reside in the bottom phase and this allows you to discard one of the points. The same applies for the top phase. The mass fraction for the top phase can be calculated as: “the total amount of acetic acid” – “the amount of acetic acid in the bottom phase”. Put the previously calculated two points on the triangular graph along with a point for the initial composition and draw the tie line between the three point

Experiment NO 06

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.



Mass Transfer Lab (CHC305)

- Carefully measure and transfer 50 ml of acetic acid solution of known concentration (0.1N) to each reagent bottle.
- Close the lids and keep the bottles in a shaker. Switch on the shaker and operate it for about 30 minutes.
- At which time the equilibrium is expected to reach between the liquid and solid phases.
- 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.
- 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	



Mass Transfer Lab (CHC305)

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

$C_0 = N_0 \times$ equivalent weight of acetic acid,

Equilibrium concentrations of filtrate after adsorption,

$C_1^* = N_1 \times$ equivalent weight of acetic acid,

Kg acid / m^3 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					



Mass Transfer Lab (CHC305)

GRAPH

The Freundlich adsorption isotherm over the small 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 $\text{Log } C^*$ Vs $\text{Log } [V (C_0 - C^*)]$. 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 07

LEACHING STUDIES

Aim

To study the principles governing leaching and compare the results obtained by conducting the batch leaching test and a mixture of sodium hydroxide and sand.

Apparatus

Five stoppered bottles, five conical flasks, burette stand, funnel, pipette, and measuring jar.

Chemical required

Std. solution of Oxalic acid, Sodium hydroxide solution, Sand, and Phenolphthalein indicator.

Procedure

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	

Mass Transfer Lab (CHC305)

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	

Leaching Studies

Take five clean and dry 500ml beakers and number them 1 to 5. To each beaker add 50g of sand and 10g of sodium hydroxide. Mix both the solids thoroughly. Add exactly 400ml, 200ml, 133ml, 100ml, and 80ml of distilled water in the beakers and note them. Stir the contents. Pipette out 10ml, 8ml, 6ml, 4ml, and 2ml of supernatant liquid from each beaker respectively and titrate it with std. HCl using phenolphthalein indicator.

Leaching:

Bottle No	Weight of sand taken (g)	Weight of Sodium hydroxide (g)	Volume of water added (ml)	Volume of supernatant liquid (ml)	Volume of HCl Consumed
1	50	10	400	10	
2	50	10	200	8	
3	50	10	133	6	
4	50	10	100	4	
5	50	10	80	2	



Mass Transfer Lab (CHC305)

Amount of sodium hydroxide present in supernatant liquid=

$$\frac{\text{Vol. (HCl)} * \text{N. (HCl)} * 40 * \text{volume of water added}}{1000 * \text{volume of supernatant liquid}}$$

Percentage of sodium hydroxide recovered

$$= \frac{\text{Amount of sodium hydroxide present} * 100}{\text{Amount of sodium hydroxide added to beaker}}$$

RESULT

The % of sodium hydroxide extract was found to be =



Experiment NO 08

TRAY DRYER

AIM

To determine the drying characteristics of a given sample by drying in a tray dryer.

Theory

Drying is removing relatively small amounts of water from material. In drying water is usually removed as a vapor by air. In evaporation large amounts of water is removed from material when the water reaches its boiling point.

If the wet stock is kept in a compartment over a tray/plate and air is passed over it by a blower, the dryer is known as atmospheric tray dryer. When a slab of wet stock is placed in contact with air, water evaporates from its surfaces and diffuses as vapor through the air film that covers the surface. The concentration of the moisture on the surface decreases. A concentration difference is setup, which makes them moisture in the interior to diffuse to the surface or it moves by capillary action. The drying rate at this point is constant and independent of moisture content of the slab as there is continuous supply of moisture to the surface, provided the air velocity, temperature, and humidity do not change. During this *constant-rate* period the rate of drying is controlled by the rate of vapor diffusion through the air film and is, therefore, depends upon the humidity across the film and the thickness of the film.

When the moisture content become less than a certain value known as the critical moisture content, the rate of diffusion of moisture from interior of the slab to the surface begins to fall hence the rate of drying decreases. This is known as the *falling rate*, which continues till equilibrium moisture content is reached, after further drying is not possible under the particular drying conditions.

PROCEDURE

1. Take about 150 gm of the given sample (sawdust) in the tray, spread it uniformly.
 2. Add about 30 ml of water to the sample.
 3. Set the temperature at desired level (say 25 °C i.e. room temperature) switch on the blower and start the stop clock.
 4. Note down the initial weight of the sample.
 5. Note down the time taken for the digital balance for every 2 gm loss in weight.
-

Mass Transfer Lab(CHC211)

OBSERVATIONS AND CALCULATION

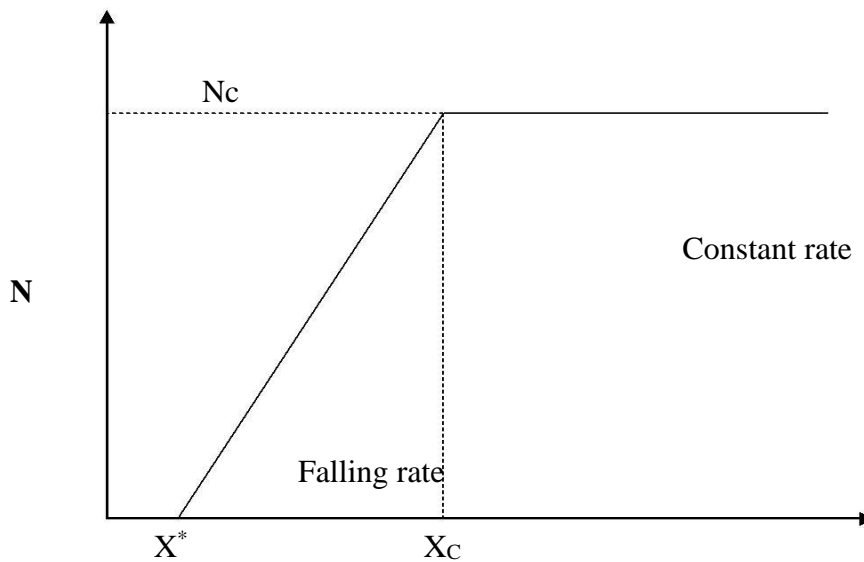
Moisture content in dry basis (X) = weight of water remaining / weight of dry solid X

$$= X_2 - X_1 \quad dt = t_2 - t_1$$

Drying area, $A = \frac{\pi}{4} d^2 = \underline{\hspace{2cm}} \text{m}^2$

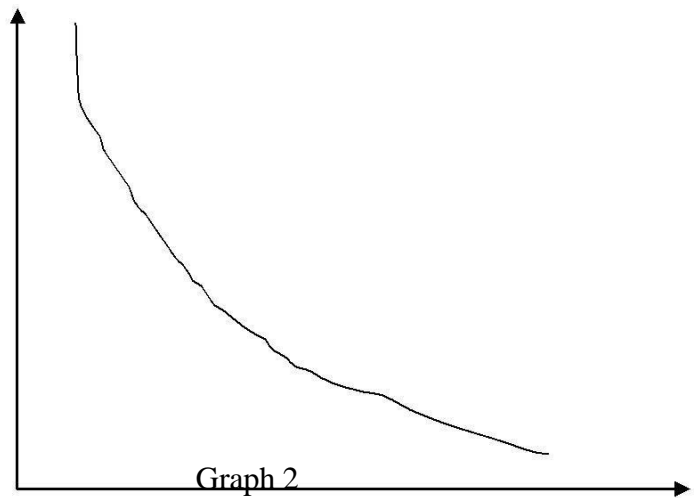
Serial no.	Weight change, dw (kg)	Time taken, (sec)	Moisture content, X (kg moisture / kg dry solid)	dX / dt, Kg/sec	N, kg/m ² sec	1/ N, m ² sec /Kg

I. Graph of N vs X



II. Graph of 1/N vs X

From Graph get the values of X_c , N_c , X_e



By Graphical method

Time of constant rate period, $T_C = \text{Area under the curve from } X_{\text{initial}} \text{ to } X_c / (A N_C) =$
_____sec

Time of falling rate period, $T_F = \text{Area under the curve from } X_c \text{ to } X_{\text{final}} / A N_C =$
_____sec

RESULT

1. Equilibrium Concentration (X_e^*) =
2. Critical Moisture Content (X_c) =
3. Time period of Drying = Sec (By graphical method)