Mass Transfer Operations-I

Chemical Engineering Department

L.E.COLLEGE-MORBI

List of Experiments

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Date:

DIFFUSIVITY OF LIQUID IN STAGNANT AIR

Objective

To evaluate the diffusivity of Carbon Tetrachloride in stagnant air at temperature T.

<u>Apparatus</u>

Beaker, Scale, Measuring cylinder, thermometer.

Chemicals

Carbon Tetrachloride

Theory: Student should write theory their own.

Procedure

- Take clean 250 ml beaker.
- Add 100 ml _____ in the beaker.
- Measure the height of the given sample in the beaker Z₁ and keep the beaker for ______ hour for diffusion of ______ in air.
- Determine the loss of _____ by marking the height.
- Measure the height Z₂.

Observation

Initial volume of in the beaker =
Final volume of in the beaker =
$Temp = \underline{\qquad}^0 C$
Mass = Density x volume
Vapour pressure of =
N _A =
$D_{AB} = \frac{N_A \times R \times T \times Z}{p_t \ln \frac{p_t - P_{A2}}{p_t - P_{A1}}}$
= m ² / sec

1) Initial height of liquid in beaker $Z_1 = _ ____ cm$ Diameter of beaker $= _ ____ cm$ $V_1 = Pi /4 (d)^2 (Z_1) = _ ____ (cm)^3$ $= _ ____ (ml)$ 2) Final height of liquid in beaker $Z_2 = _ ___ cm$



Calculation

1) Total concentration

$$C_{T} = (1*273.15)/(22.4*T)$$

Concentration of Carbon tetrachloride,
 $C_{A} = C_{T}*V/P$
 $C_{B1}=273.15 / (22.4*T)$
 $C_{B2}=C_{B1}*(P-V) / P$
 $C_{BM} = (C_{B1}-C_{B2}) / \ln(C_{B1}/C_{B2})$
 $D_{AB}= (P * C_{BM}) / (2*MA* C_{A}* C_{T}*S)$
 $= _ ____ m^{2}/sec$
Experimental $D_{AB} = _ ___ m^{2}/sec$

Result:

Experimental $D_{AB} = \underline{\qquad} m^2$ /sec Theoretical $D_{AB} = \underline{\qquad} m^2$ /sec

Conclusion:

Marks:

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Date:

DIFFUSIVITY WITH RESPECT TO TEMPERATURE

Objective

To evalaute the diffusivity of Carbon Tetrchloride at elevated temperature. Analyse the results for room temperature and elevated temperature.

Apparatus: Beaker, Scale, Measuring cylinder.

Chemical: Toluene, Acetone, Benzene

Theory: Student should write theory their own.

Procedure:

- Take clean 250 ml beaker.
- Take 100 ml_____ in beaker. Measure the initial height (Z₁).
- Heat it up to 50° C & maintain the temperature of 50° C for 20 min.
- Measure the volume & height (Z_2) of in beaker after 20 min. •

Observation:

- Initial volume of _____ in the beaker = _____ ml 1)
- 2) Initial height $Z_1 = _$ cm
- Final height $Z_2 = _ cm$ Temperature = = $^0 C$ 3) 4)
- Temperature = = $__{0}^{0}$ C Difference in height of _____ in beaker due to diffusion 5) $=Z_1-Z_2 = ____ cm$
- Vapour pressure of ______at _____mmHg 6)
- Density of _____ at ____ Temp. ____ Kg/m³ 7)
- 8) Time duration =
- **Calculation:**
 - 1) Total concentration $C_{T} = (1*273.15)/(22.4*T)$

Concentration of Carbon tetrachloride, $C_A = C_T * V/P$

$$C_{B1}=273.15 / (22.4*T)$$

$$C_{B2}=C_{B1}* (P-V) / P$$

$$C_{BM} = (C_{B1}-C_{B2}) / \ln(C_{B1}/C_{B2})$$

$$D_{AB}= (P*C_{BM}) / (2*MA*C_{A}*C_{T}*S)$$

$$= \underline{\qquad m^{2}/sec}$$
Experimental $D_{AB} = \underline{\qquad m^{2}/sec}$

<u>Result</u>

Experimental $D_{AB} = \underline{\qquad} m^2$ /sec Theoretical $D_{AB} = \underline{\qquad} m^2$ /sec (From Perry's handbook)

Conclusion

Marks:

Date:

MASS TRANSFER COEFFICIENT

Objective

To evaluate the mass transfer co-efficient of Carbon tetrachloride and air system

<u>Apparatus</u>

Beaker, heating mantle, Thermometer.

Chemical

Water

Theory

The rate at which a component is transferred from one phase to the other depends up on the mass-transfer rate or mass transfer coefficient. The mass transfer coefficient for the various components in a given phase will differ from each other, but the difference is not really large, where molecular diffusion prevails. Under conditions of turbulence, the mass transfer coefficients for all components become nearly alike.

Thus, due to difference in mass transfer coefficients of the components the degree of separation obtained is small. The separation depends entirely upon the difference concentration, which exist at equilibrium and not upon the difference in transfer co efficients.

F-type mass transfer coefficient is defined for particular location on the Phase interface, or boundary and flux N_A is defined as

$$N_{A} = \frac{N_{A}}{N_{A} + N_{B}} \cdot F \cdot \ln \left[\frac{N_{A} / N_{A} + N_{B} - C_{A_{2}} / C}{N_{A} / N_{A} + N_{B} - C_{A_{1}} / C} \right]$$

Where, $F = \frac{D_{AB} \cdot C}{Z}$

Flux = (coefficient) (concentration difference)

Procedure

- Rinse and clean all the glassware with water. Now measure the diameter of the beaker.
- Take 100 ml water in the beaker and put it in the heating mantle. Measure the initial height of the water level.
- Heat the water for 30 minutes at constant temperature (i.e. 60° C) and during heating note down the dry bulb and wet bulb temperature.
- After 30 minutes note down the final height of the water level in the beaker.
- Also measure the volume of water.

Observations

- 1. Diameter of beaker = _____ cm.
- 2. Volume of water (before heating) = ____ ml
- 3. Height of water level in beaker $Z_1 = _$ cm

After heating

- 1. Height of water level in beaker = _____ cm
- 2. Volume of water = _____ cm
- 3. Dry bulb Temperature=____⁰ C
- 4. Wet bulb Temperature=____⁰ C

Calculations

$$N_A = \frac{D_{AB}Pt}{RTZp_{BM}} (P_{Ai} - P_{AG})$$

1)

 $= F/p_{BM} (p_{BG} - p_{Bi})$

Where,

 p_{Ai} = Vapour pressure of water (A) at interface = _____ p_{AG} = Partial pressure of water vapour (A) in bulk air_____ P = Vapour pressure at 60 ⁰ C Pt = 1 atm., p_{BG} = Pt - p_{AG} , p_{Bi} = Pt - p_{Ai}

$$p_{BG} = Pt - p_{AG}$$

$$p_{Bi} = Pt - p_{Ai}$$

$$p_{BM} = \frac{(p_{BG} - p_{Bi})}{\ln \frac{p_{BG}}{p_{Bi}}}$$

$$N_A = \frac{(V_1 - V_2) \times \rho_{H_2O}}{M_{H_2O} \times Area \times time}$$

$$= kmol/m^2.sec$$

4)

5)
$$N_A = K_G (p_{Ai} - p_{AG})$$

 $\therefore K_G = \underline{\qquad} kmol/m^2.sec.KPa$

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$$N_A = \frac{F(P_{Ai} - P_{AG})}{P_{BM}}$$

F = _____

7) **Result:**

K_G =_____ F= _____

Marks:

Date:

SINGLE STAGE EXTRACTION

Objective

To evaluate efficiency for single stage liquid-liquid extraction of acetic acid from the mixture of acetic acid and water.

Apparatus

Glass beaker (500 ml), Glass rod, Measuring Cylinder, Wt. Box, Separating funnel, Titration set, etc.

Chemicals

Acetic Acid, NaOH, Water, Phenolphthalein Indicator, Ethyl Acetate.

Procedure

- Rinse and clean all the glass apparatus with water.
- Note down room Temp.
- Take 60ml water & 10 ml acetic acid as a feed in Separating funnel & mix well.
- Add 30-ml Ethyl Acetate as a solvent (S_1) in first stage & shake the mixture vigorously.
- Allow the mixture to settle for 30 min. by keeping on stand.
- Separate the two layers in different beakers.
- Measure volume & mass of both the layers.
- Analyze diluted solution with std. NaOH using phenolphthalein as indicator.

Observation

- 1. Room temperature: _____ °C
- 2. Volume of Feed: _____ ml (Water 60 ml (A) and Acetic acid-10 ml (C))
- 3. Volume of solvent: _____ ml (Ethyl Acetate-30 ml (B))

Density at room Temp.

Ethyl Acetate	=	gm/cc
Acetic acid	=	gm/cc
Water	=	gm/cc

Observation table :

Sr.No.	Solvent	Extract Layer	Raffinate Layer	Burette
	Added ml	ml	ml	Reading ml

Calculation

- 1. Volume of sample = _____ ml.
- 2. Extract phase analysis

Normality = (Normality of NaOH* burette rediang)/ (volume of sample) =_____N

Moles of acetic acid= normality * volume of extract

=_____ gm moles Wt. Of acetic acid= moles of acetic acid* molecular weight.

=_____ gm

- 3. Wt. of Acetic acid in feed = _____ gm
- % Extraction = (Wt. of Acetic acid in extract / Wt. of A.A in feed) * 100 4. =_____%

Result

Recovery of acetic acid=

Conclusion

Marks:

Date:

MULTI STAGE CROSS CURRENT EXTRACTION

Objective

- i. To evaluate efficiency for multi- stage liquid-liquid extraction of acetic acid from the mixture of acetic acid and water.
- ii. Compare and Analyse the results for single stage and multi-stage operation.

<u>Apparatus</u>

Glass beaker (500 ml), Glass rod, Measuring Cylinder, Wt. Box, Separating funnel, Titration set, etc.

Chemicals

Ethyl Acetate, Acetic acid, NaOH, Water, Phenolphthalein Indicator, Oxelic Acid.

Procedure

- Rinse and clean all the glass apparatus with water.Note Room Temp.
- Take 60-ml water & 10 ml Acetic Acid as a feed in separating funnel & mix well.
- Add 30-ml Ethyl Acetate as a solvent (S_1) , in first stage & shake the mixture vigorously.
- Allow the mixture to settle for 30 min. by keeping on stand.
- Separate the two layers in different beakers.Measure volume & mass of both the layers.
- Analyzed diluted solution with std. NaOH using phenolphthalein as indicator.
- Take raffinate of stage in separate funnel as feed to repeat the whole procedure up to total 3 stages.

Observation

1.	Room temperature:	^{0}C	
2.	Volume of Feed:	ml	
	Water $: 60 \text{ ml}(A)$		
	Acetic Acid 10 ml	(B)	
	Ethyl Acetate 30 ml	(C)	
3.	Density at room temp.		
	Ethyl Acetate	-	0.875 gm/cc
	Water	-	1.0 gm/cc;
	Acetic acid	- 1.04	8 gm/cc

Observation table

Stage	Feed(ml)	Solvent Added ml	Extract Layer ml	Raffinate Layer ml	Burette Reading ml
1					
2					
3					

Practical calculation

1ST Stage

- 1.Volume of raffinate= _____ ml.Volume of extract= _____ ml.
- 2. Extract layer analysis Density of Extract = _____ gm/cc Wt. of extract (E) = _____ gm Final burette reading = _____ ml Normality of water layer = _____ ml

Mass of A.A. in water layer = (Normality x Eq. Wt. of A.A. x Vol. of Layer) /1000

- = _____ gm
 3. Raffinate layer analysis:
 Density of raffinate = _____ gm/cc
 Wt. of raffinate (R) = _____ gm/cc
- 4. Wt. of Acetic acid in feed = _____ gm
- 5. % Extraction = Wt. of Acetic acid in extract / Wt. of A.A in feed * 100 = $_$ %
- Follow same calculation steps for 2nd and 3rd stage and find out % extraction and % overall efficiency.

<u>Result</u>

Stage	% Extraction of Aectic Acid
1	
2	
3	

Conclusion:

Marks:

Date:

Wetted Wall Column

Objective: To evaluate mass transfer coefficient of the given absorption system.

Theory:

A thin film of liquid falling down the inside of a vertical pipe through which the gas flows, constitutes a wetted wall column. Wetted wall columns have been used as absorbers for Hydrochloric acid, Ammonia, Acetone, Benzene and other volatile liquids. They have also been studied for theoretical studies for mass transfer because the interfacial surface between the phases is kept under control and is measurable.

The height of wetted wall column required for mass transfer operations is excessive and consequently this is not widely used, where large quantities of liquid or gas have to be handled, it would be necessary to arrange many vertical pipes in parallel and this leads to difficulties in the distribution of liquid into the inner surface of the tubes. The gas pressure drop for this is confined to skin friction effects, with few or no expansion or contraction losses.

Mass transfer rates for fluids flowing through pipes have been studied more completely than other cases. The rates of diffusion into gases flowing through pipes have been studied in wetted wall columns. A volatile liquid is submitted to flow down the inside surface of a circular tube, while a gas flows upward or downward through the center of the pipe. Measurement of the rate of evaporation of a liquid into the gas stream over known surface permits calculation of mass transfer coefficient for the diffusion of vapor into gas stream. Since the liquid is pure, the concentration gradient for diffusion exists entirely within the gas phase, the mass transfer coefficient Kg may be calculated.

Procedure:

- 1. Feed 10 liter of 0.1N NaOH solution in the feed tank.
- 2. Start the pumps.
- 3. Maintain the flow rate of NaOH at 1LPM.
- 4. Start the Air flow and maintain the air flow rate about 80LPM.
- 5. Similarly maintain CO₂ Flow rate about 4LPM.
- 6. System is allowed to run at steady state and flow rates of NaOH, air and CO₂ are obtained
- 7. Samples of Liquid at bottom and air + CO₂ mixtures are obtained for the analysis.
- 8. Same experiment is repeated for various flow rate keeping one parameter fixed and other variable.

Observation:

- 1. Concentration of NaOH feed
- 2. NaOH flow rate = ____LPM
- 3. Flow rate of $CO_2 = __LPM$
- 4. Flow rate of Air = _____LPM
- 5. Flow rate of outlet (Air + CO₂)mixture=_____
- 6. Flow rate of NaOH (inlet) = $__LPM = __m^3/sec$
- 7. d = Column diameter = 7.62mm
- 8. T =thickness of film = 2mm
- 9. L=Length of column=1 m
- 10. A= area of film inside the column= π x d x l= 23.93 x 10⁻³
- 11. V = total volumne of film inside the column = $\pi x d x l x T = 4.78 x 10^{-5} m^3$

Sr. No.	Air Flow Rate(LPM)	Buretter reading	Conc. of NaOH in Outlet

calculations:

To calculate Molar Flow rate of NaOH Initial concentration of NaOH (calculated as under) Titrating 0.1N HCL against 10ml NaOH sample

Normality of NaOH in feed = _____N Initial concentration of NaOH, Ci= _____ Initial concentration of NaOH (calculated as under) Titrating 0.1N HCL against 10ml NaOH sample

Similarly at outlet N of NaOH = ____N Final concentration of NaOH Co=____ Molar flow rate of NaOH feed = Ci = Initial concentration of NaOH x Flow rate = ___Kmol/m³ x ____m³/sec $= \underline{\qquad} Kmol/sec$ Molar flow rate of NaOH outlet = Co = Final concentration of NaOH x Flow rate $= \underline{\qquad} Kmol/m^3 x \underline{\qquad} m^3/sec$ $= \underline{\qquad} Kmol/sec$ Molar Flux of NaOH,
N_A= Molar flow rate of NaOH/ Area of column

Now, N_A= K_{La} * (Ci- Co)

Mass Transfer Coefficient is given by $K_{La} = N_A / (Ci-Co)$

Result : The Liquid side Mass Transfer Coefficient, $K_{La} =$

Marks:

Date:

SINGLE STAGE LEACHING

Objective

To evaluate the stage efficiency and overall recovery of NaOH for single stage cross current leaching operation for leaching NaOH from mixture of NaOH and calcium carbonate using water as a solvent.

<u>Apparatus</u>

Glass beaker (500 ml), Glass rod, Measuring Cylinder, Wt. Box, Separating funnel, Titration set, etc.

Chemicals

Calcium carbonate powder (B), NaOH (C), Water (A), Std.HCl solution, Phenolphthalein Indicator.

Procedure

- Rinse and clean all the glass apparatus with water.
- Take a mixture of 5 gm of NaOH and 35 gm of CaCO₃ in glass beaker.
- Add 200 ml water to the beaker.
- Stir the solution for about 5 minute continuously.
- After 20 min. collect the over flows & titrate it against 0.5N HCl.
- Separate the clear solution from settled sludge and measure its volume.
- Notedown all readings & calculate % recovery ,stage efficiency using standard data available.

Observation

Room temperature: $____0^C$ Feed: CaCO₃ powder + NaOH flakes F = $___gm$ + $__gm$ = $__gm$. Solvent: Water, R = $___ml$

Analysis of solution

V1 = Sol. Taken for titration = _____ ml N2 = Normality of HCL = _____ ml V2 = Vol. Of HCL consumed = _____ ml

Calculations

Analysis of clear solution: Sample volume, V1 = ____ ml Burette reading, V2 = ____ ml Normality of Std. HCl solution, N2 = ____N

$$N2 \times V2$$

• Normality of clear solution = -----

=_____N

• Amount of NaOH in clear solution = Normality of clear solution × Volume of the clear solution × Eq. Weight of NaOH

$$= (_) \times (_) \times (_) \times (_) /1000$$

W1 = ____ gm
W = Amount of NaoH in feed = ____ gm
Percentage Recovery = ------ × 100
W
$$= ------ \times 100$$

(____)
= ____ %

Result

% Recovery (Practically) = _____%

Conclusion

Marks:

Date:

MULTI STAGE CROSS CURRENT LEACHING

Objective

- i. To evaluate the stage efficiency & overall recovery of NaOH for multistage cross current leaching operation for leaching NaoH from mixture of NaoH & CaCO₃ using water as solvent.
- ii. Compare and Analyse the results for single stage and multi-stage operation

<u>Apparatus</u>

Glass beaker (500 ml), Glass rod, Measuring Cylinder, Wt. Box, Separating funnel, Titration set, etc.

Chemicals

Calcium carbonate Powder (B), NaOH (C), Water (A), Std.HCl solution, Phenolphthalein Indicator.

Procedure

- Rinse and clean all the glass apparatus with water.
- Take a mixture of 5 gm of NaOH and 35 gm of CaCO3 in glass beaker.
- Add 195 gm water to the beaker.
- Stir the solution for about 5 minute continuously.
- After 20 min. collect the over flows & analyze.
- Separate the clear solution from settled sludge and measure its volume.
- Notedown all readings & calculate % recovery, stage efficiency using standard data available .
- Add water as a solvent to 2nd stage with same volume as clear solution withdraw from 1st stage.

Observation

- Room temperature: _____ °C
- Feed: CaCO3 powder + NaOH flakes
- F = gm + gm = gm.
- Solvent: Water, R = ____ gm

Analysis of solution

- V1= Sol. Taken for titration = _____ ml
- N2= Normality of HCL = ____ ml
- V2= Vol. Of HCL consumed= ____ ml

Practical calculation for stage-I:

Analysis of clear solution:

Clear liquid volume =	_ ml
Sample volume V1 =	_ ml
Burette reading V2 =	ml
Normality of Std. HCL solution, $N2 = $	N

$$\frac{N_2 \times V_2}{V_2}$$

- Normality of clear solution = V_1 = _____ N
- Amount of NaoH in clear solution = Normality of clear solution x Volume of the clear solution x Eq. Weight of NaoH

$$= \underline{\qquad x \qquad x \qquad 1000}$$

W1 = gm
W = Amount of NaoH in feed = gm
% Recovery = $\frac{W1}{W} \times 100$
= %

• Follow same calculation steps for 2nd stage and find out % recovery and overall efficiency.

<u>Result</u>

% Recovery of NaoH

Stage	Practically
1	
2	
Over all efficiency for 2 stages	

Conclusion

Marks:

Date:

CRYSTALLIZATION (Without Seeding)

Objectives : To determine crystal yield without seeding.

Apparatus: Beaker, Heating mental, Stirrer, Conical flask, Filter Paper, Pipette, Thermometer, Weight Box, Burette.

Chemicals: Water, Boric acid Acid, .

Theory:

Crystallization is the formation of solid particles within a homogeneous phase. It may occur as the formation of solid particles in a vapor as in snow, as solidification from a liquid melt, as in manufacture of large single, crystals or as crystallization from liquid solution. Crystallization from solution is important industrially because of the variety of materials that are marketed in the crystalline form. Its wide use has a two-fold basis.

(a) Crystal formed from an impure solution is itself pure (unless mixed crystals occur).

(b) Crystallization affords a practical method of obtaining pure chemical substances in satisfactory condition for packing & storing.

Crystallization may be analyzed from the standpoints of purity, yield, energy requirements and rates of nucleation and growth. Crystal growth is a diffusion process modified by the effect of the solid surfaces on which the growth occurs. By adding the component self-crystal growth can be accelerated and also amount of crystals will be increased.

Procedure:

1. Clean all the Glassware and take 250 ml water in beaker.

2.Heat the water up to 60 °C on a electric heater equipped with a magnetic stirrer and regulate temperature of water at 60 °C the thermostat provided.

3. Add Boric acid acid gradually in water up to extent so solution becomes saturated.

4.Note down the amount of Boric acid Acid added in the water.

5.Take 200 ml saturated solution as a feed and cool it slowly up to the room temp. due to which crystals will be formed. Using the filter paper separate the crystals, dry it and weight it.

Observation Table:

1	Amount of Boric acid dissolve in feed = gm.
2	Volume of feed= ml.
3	Weight of crystals =gm

Calculations:

1. Crystal without seeding = Wt. Of acid crysatl collected/ Wt. Of acid in feed

Result: Crystal without seeding =_____.

Conclusion:

Marks :

Date:

CRYSTALLIZATION (With Seeding)

Objectives: To determine crystal yield with seeding.

Apparatus: Beaker, Heating mental, Stirrer, Conical flask, Filter Paper, Pipette, Thermometer, Weight Box, Burette.

Chemicals: Water, Boric Acid,.

Theory:

Crystallization is the formation of solid particles within a homogeneous phase. It may occur as the formation of solid particles in a vapor as in snow, as solidification from a liquid melt, as in manufacture of large single, crystals or as crystallization from liquid solution. Crystallization from solution is important industrially because of the variety of materials that are marketed in the crystalline form. Its wide use has a two-fold basis.

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Procedure:

- 1. Clean all the Glassware and take 250 ml water in beaker.
- 2. Heat the water up to 60 °C on a electric heater equipped with a magnetic stirrer and regulate temperature of water at 60 °C the thermostat provided.
- 3. Add Boric acid acid gradually in water up to extent so solution becomes saturated.
- 4. Note down the amount of Boric Acid added in the water.
- 5. Add 5 gm of Boric acid as a seed in saturated solution of Boric acid and shake the solution well.
- Take 200 ml saturated solution as a feed and cool it slowly up to the room temp.
 due to which crystals will be formed. Using the filter paper separate the crystals, dry it and weight it.

Observation Table:

1	Amount of Boric acid dissolve in feed = gm.
2	Volume of feed= ml.
3	Weight of crystals =gm

Calculations:

Crystal yield = Wt. Of acid crysatl collected/ Wt. Of acid in feed

Result: Crystal yield with seeding =_____.

Conclusion:

Marks :