August, 2025

University of Jordan
Faculty of Engineering
Department of Chemical Engineering

Separation Processes Lab.

(0905563)

August, 2025

Experiment Number -1-

Gas absorption

Objectives

1. To study the hydrodynamic characteristics of a packed column.
2. To establish a mass balance on the absorbed gas in the column.
3. To determine mass transfer coefficients, the number of transfer units and the effective height equivalent to a transfer unit.

Apparatus

The equipment consists of a packed column of 2 meters in height and 7.5 cm in diameter, packed with 10mm glass Raschig rings. Air compressor is supplied with a flow meter. Ammonia cylinder is also available with an ammonia flow meter. Water supply tank is available with water pump, and a flow meter to indicate the flow of water. Mercury manometer and water manometer present to indicate the pressure drop across the column.

Procedure

1. Fill the water supply tank with water.
2. Make sure that valves V1, V2, and V7 are closed and that V4 and V5 are open.
3. Check that the manometers show zero pressure difference across the column.
4. Switch on air compressor. Adjust valve V2 and take readings of manometers at different values of air flow rates.
5. Repeat again with packed column in wet.
6. Repeat again with different values of water flow rates.
7. To determine mass transfer coefficient, adjust flow rates of water and air so that the condition in the absorption column between flooding point and loading point.
8. Allow Carbon Dioxide to enter the column at certain flow rate, noting the inlet pressure and temperature of Carbon Dioxide.
9. At steady state, take a sample of the liquid going to drain and find the concentration of Carbon Dioxide in it by titrating against NaOH solution.

Theory

When a liquid is flowing counter currently to the gas, for low gas flow rates, the effect is as for the wet packing:

The pressure drop increases, and the pressure drop follows a square law characteristic. Further increase in gas flow rate or liquid flow rate makes the liquid fails to drain from some sections of the tower.

Pressure drop for dry packing (kg.m^{-2}) is given by:

$$\Delta P_d = \frac{C.g_o^2}{\rho_g}$$

Where:

g_o= mass velocity of the gas based on the total cross section of the tower (kg.m^{-2}.s^{-1})

ρ_g= gas density (kg.m^{-3})

C= constant which equals to 264 for 10 mm Raschig rings.

Pressure drop for wetted packing (kg.m^{-2}) is given by:

$$\Delta P = \Delta P_d . A_l$$

$$log A_l = \frac{B.g_l}{\rho_l}$$

Where: g_l= liquid flow rate based on the total cross section of the tower (kg.m^{-2}.s^{-1})

ρ_l= liquid density (kg.m^{-3})

B= constant which equals to 0.084 for 10 mm Raschig rings.

For mass transfer characteristic

Consider a packed tower of cross section sectional area A; assume that the effective interfacial area for mass transfer of liquid film on the packing is *a* ($m^2/_{m^3}$) of the tower volume.

The area for mass transfer in small increment of height *dh* will be *a(A.dh)* m^2, and the quantity of solute in the gas passing into solution in this incremental height is *d(Gy).* If K_{og} is the overall mass transfer coefficient based on the gas phase, then:

$$d(Gy) = K_{og}.a.A.(y^* - y).dh$$

$$H = \int_{y_2}^{y_1} \frac{d(Gy)}{K_{og}.a.A.(y^* - y)}$$

The height of transfer unit H_{og} is

$$H_{og} = \frac{G}{K_{og}.a.A}$$

The number of transfer unit N_{og} is

$$N_{og} = \int_{y_b}^{y_t} \frac{dy}{y^* - y}$$

And $H = N_{og}.\, H_{og}$

Similarly, in terms of the liquid phase;

$H = N_{ol}.\, H_{ol}$

Calculations

1. draw the hydrodynamic characteristic curves of the packed column and find the loading point and the flooding point.
2. Establish a total mass balance on Ammonia and discuss the difference between the inlet and outlet Ammonia amounts.
3. Calculate the overall mass transfer coefficient, the number of transfer unit, and the height of transfer unit.

References

1- J. M. Coulson, and J. L. Richardson, “Chemical Engineering” vol.2, Pergamon Press.

2- Treybal, R. E; “Mass Transfer Operations”, McGraw Hill.

3- Foust, A.S; Wenzel, L.A; Clump, C. W; Mans, L; and Anderson, L.B; Principles of Unit Operations”, Wiley-Mass Transfer, chapter 14, 16.

4- Nrman, A.S; “Absorption, Distillation and Cooling Towers’’, Longman, London-1962.

Gas Absorption

Raw Data Sheet part (1)

Dry Packing		**Wet Packing**		**Water flow Rate=**		**Water flow Rate=**		**Water flow Rate=**	
Air flow rate	ΔP	Air flow rate	ΔP	Air flow rate	ΔP	Air flow rate	ΔP	Air flow rate	ΔP

Raw Data Sheet part (2)

Run No.					
Inlet Water Flow Rate (ml/min)					
Inlet Air Flow Rate (ml/min)					
Inlet Carbon Dioxide Flow Rate(ml/min)					
Concentration of NaOH solution(mol/l)					
Volume of sample(ml)					
Volume of NaOH titrant (ml)					
Temperature of outlet solution (ºC)					

Instructor’s Signature:
Date:

Experiment Number -2-

Distillation

Apparatus

The Distillation apparatus allows the study of both batch and continuous distillation, tray column operation, operation under atmospheric pressure and manual or computer control of the process. Data logging of the process is also possible.

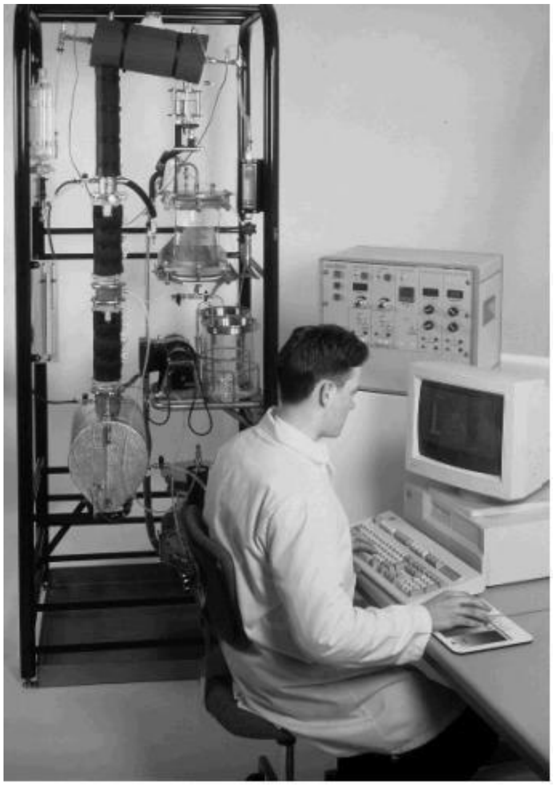


Fig. (1): Distillation Column with computer control.

As shown in Fig.(1), the Distillation Column is a self-contained distillation facility consisting of two interconnected units: A floor standing process unit and a bench mounted control console.

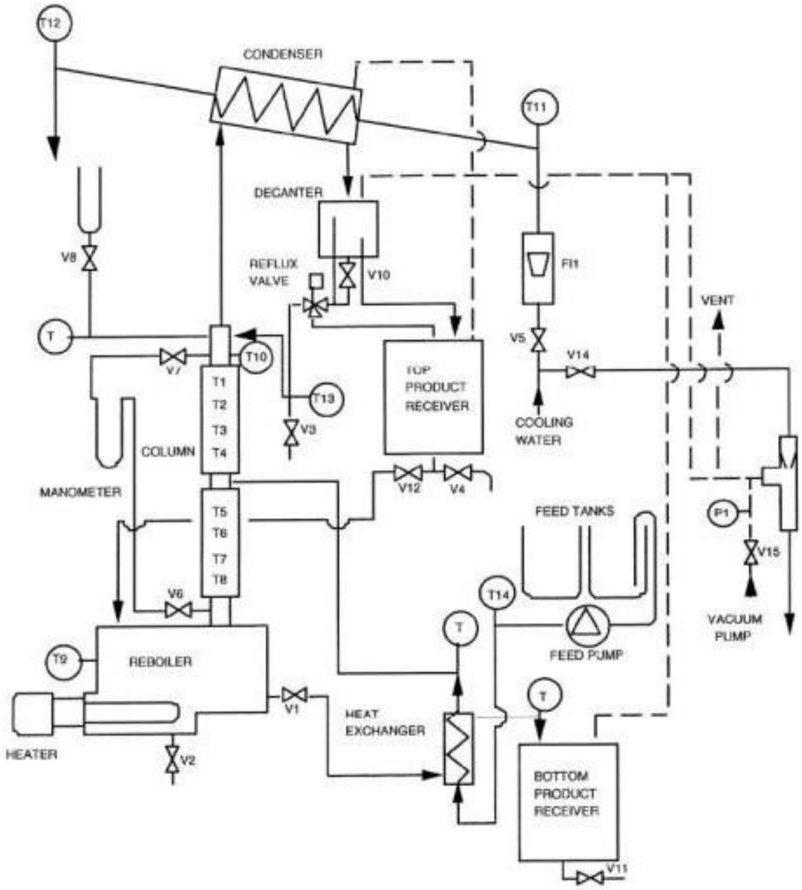


Fig. (2): Schematic Diagram for distillation column.

Fig. (2) Shows a schematic Diagram for the apparatus which consists mainly of:

Distillation column: which is made up of two glass sections, separated by a central feed section and arranged vertically for counter-current vapor/liquid flow, each section containing four sieve plates with 50mm diameter, the column is insulated to minimize heat losses.

Reboiler: the reboiler is situated at the base of the column and manufactured from stainless steel, it incorporates a flameproof immersion type-heating element. Either batch or continuous distillation can be carried out using this reboiler. In continuous operation, valve (V1) is open and bottom product flows to the bottom product tank, while for batch operation, valve (V1) remains closed so that the reboiler can be filled with the initial charge (10 to 12 litres) of binary mixture. A sight glass allows the level in the reboiler to be observed.

Condenser: Vapor from the top of the column passes to a water-cooled, coil-in-shell condenser. cooling water enters the condenser at a regulated rate through a variable area flowmeter and the flowrate is controlled by diaphragm valve (V5).

Decanter: Condensate is collected in a glass decanter (phase separator) which is bypassed for normal distillation experiments by opening valve (V10). Condensate from the condenser outlet passes directly through the decanter to the inlet of the reflux ratio control valve (12) which is a 3-way solenoid operated valve. Depending on the setting of the reflux timers, condensate is directed by the reflux valve either back to the top of the column or to the top product collecting vessel. When directed to the column, the reflux passes through a U-seal where a valve (V3) can be used for measuring boil-up rate or for draining the Useal. The contents of the top product tank can be drained into the reboiler for re-use via valve (V12).

Thermocouples: Temperatures within the system are monitored by fourteen thermocouple sensors (T1 to T14) located at strategic positions in the system. T1 to T8 are located in the column and measure the temperature of the liquid on each sieve plate.

Manometer: The total pressure drop across the column is indicated on a U-tube manometer fitted with isolating valves (V6) and (V7).

Product Receivers: for top and bottom products.

Control Console: The console is attached to the process.

Theory

The distillation column available in the laboratory is an enriching section of a distillation column. The relationship between vapor (y) and liquid (x) as they pass each other between theoretical plates is given by (Numbering of plates increase from top to bottom).

$$y_{n=1} = \frac{R}{R+1} X_n + \frac{X_D}{R+1}$$

Where:

R= Reflux ratio

X_D = composition of distillate at the top of the column.

That is the equation of the operating line for McCabe and Thiele method, and passes the points (y1, D) and has an intercept of $\frac{X_D}{R+1}$.

The column overall efficiency is given by:

$$E \frac{\text{number of theoritical plates}}{\text{number of actual plates}} * 100\%$$

Distillation – Exercise A - Variation of Column Pressure Drop

Objective

To determine the variation of pressure drop over the distillation column with boil-up rate.

Procedure

1- Put 10 liters of water and Ethanol (2:1) in the reboiler through the filler cap provided.

2- Make sure that:

- Initially at start up the power of reboiler is switched off.
- All valves on the equipment are closed except valve V10 on the reflux pipe.
- Reflux ratio timer on the console is switched off (to operate at total reflux).
- The filler cap on the top of the reboiler is firmly closed.

3- Turn on the power to the control panel and Turn the power controller clockwise until a reading of approximately 0.5kW is obtained on the digital wattmeter.

4- Set the temperature selector switch to T9 (the temperature in the reboiler).

5- Open valve V5 until the cooling water flow rate FI1 to the condenser is approximately (1.5-3) liters/min.

6- Switch the temperature selectors onT9, T8, T7, T6, T5, T4, T3, T2 and T1 to observe the warm up of reboiler content and then vapour rising up in the column.

7- When the liquid distillate is observed on the top of the column, let the apparatus running for at least 10 minutes and then take the readings of pressure drop and boil up rate.

8- Measure the boil-up rate by performing a timed volume collection: partially open valve V3 and drain the condensate (in a separate measuring cylinder) from the reflux system until a steady flow is obtained. (Ensure that liquid remains in the flexible reflux tube to prevent vapour from escaping.) Start sample collection and timing at the same time, and collect a sizeable amount. Repeat the measurement three times and take an average value to determine the boil-up rate.

9- After taking the samples, take reading of pressure drop over both the rectifying (top) and the stripping (bottom) sections by opening the valves V6 and V7 on the manometer. When opening the valves, make sure always to open valve V6 then V7 to prevent vapour from the column entering the manometer.

Close the valves in the same order after taking the pressure drop reading.

10- Repeat steps (3-10) at different values of power.

11- After finishing the experiment:

- Switch off the reboiler heater.
- Keep the cooling water on running for approximately five minutes.

Calculations of Exercise A

1- Show graphically the relationship between the manometer readings and the boil-up rate.

Distillation Exercise A

Raw Data Sheet

Power (kw)	Boil-up rate (ml/sec)	Pressure drop (cm H2O)
0.5		
0.75		
1.0		
1.25		
1.5		
1.75		

Instructor’s Signature:
Date:

Distillation – Exercise B – Overall Column Efficiency

Objective

To determine the overall column efficiency at different reboiler power or different reflux ratio.

Procedure

1- Put 10 liters of water and Ethanol (2:1) in the reboiler through the filler cap provided.

2- Make sure that:

- Initially at start up the power of reboiler is switched off.
- All valves on the equipment are closed except valve V10 on the reflux pipe.
- Reflux ratio timer on the console is switched off (to operate at total reflux).
- The filler cap on the top of the reboiler is firmly closed.

3- Turn on the power to the control panel and Turn the power controller clockwise until a reading of approximately 1kW is obtained on the digital wattmeter.

4- Set the temperature selector switch to T9 (the temperature in the reboiler).

5- Open valve V5 until the cooling water flow rate FI1 to the condenser is approximately (1.5-3) liters/min.

6- Switch the temperature selectors onT9, T8, T7, T6, T5, T4, T3, T2 and T1 to observe the warm up of reboiler content and then vapour rising up in the column.

7- When the liquid distillate is observed on the top of the column, let the apparatus running for at least 30 minutes so that the system can reach an equilibrium condition.

8- Take a sample from overhead condensate using valve V3 and a sample from the bottom using valve V2 at the bottom of the reboiler.

9- Measure and record the refractive index of the collected samples after cooling them to room temperature.

10- Using the refractive index calibration curve, read the compositions of the overhead and bottom samples.

11- Measure the boil-up rate by performing a timed volume collection: partially open valve V3 and drain the condensate (in a separate measuring cylinder) from the reflux system until a steady flow is obtained. (Ensure that liquid remains in the flexible connecting tube to prevent vapour from escaping.) Start sample collection and timing at the same time, October, 2021 14 and collect a sizeable amount. Repeat the measurement three times and take an average value to determine the boil-up rate.

12- Repeat steps (3-10) at different values of power.

13- After finishing the experiment:

- Switch off the reboiler heater.
- Keep the cooling water on running for approximately five minutes.

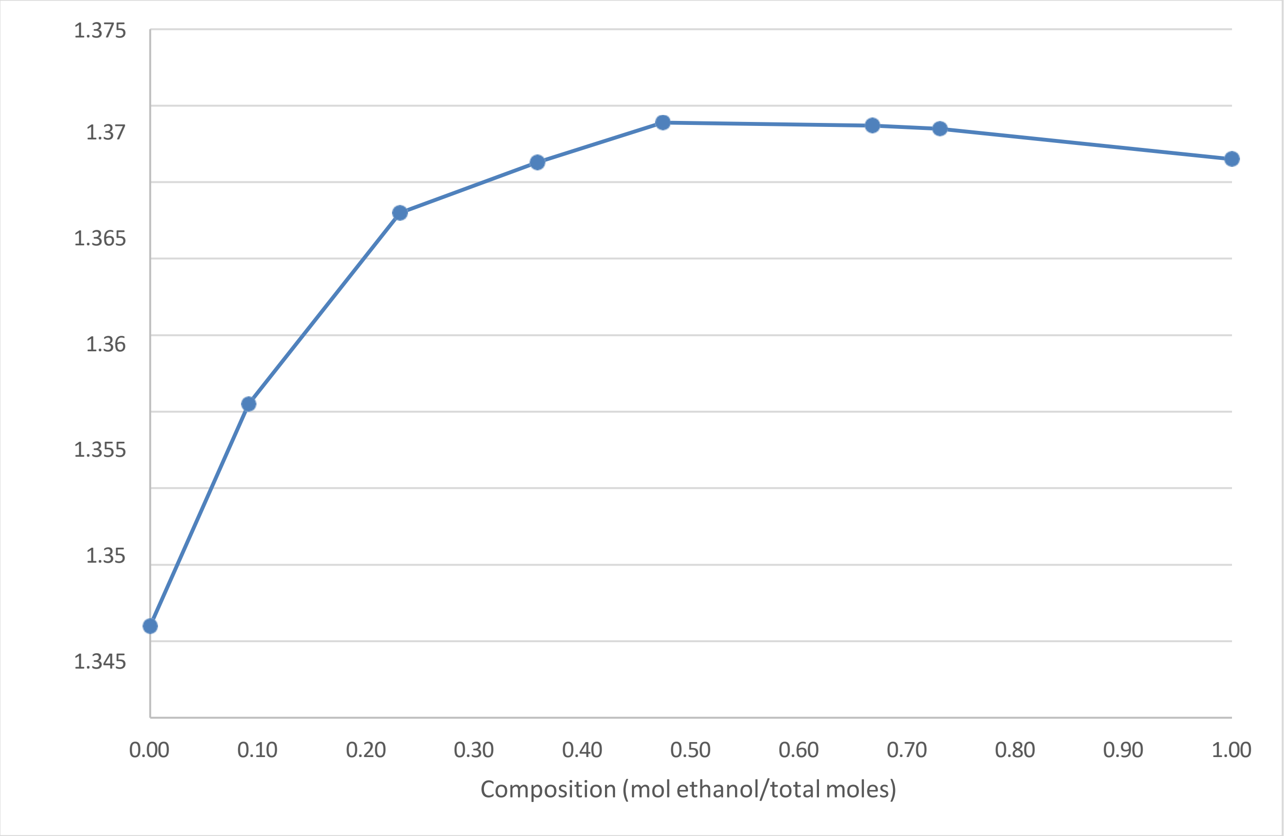


Fig.(3): Calibration curve of Ethanol-Water system using Refractometer instrument at 20 C^o.

Calculations of Exercise A

1- Calculate the number of theoretical plates using the methods of McCabe and Thiele.

2- Calculate the efficiency of the column at different boil-up rates or at different reflux ratios.

Distillation Exercise B

Raw Data Sheet

Total Reflux

Power (kw)	Boil-up rate (ml/sec)	Top Composition (mol/mol)	Bottom Composition (mol/mol)
0.75			
1.00			
1.25			

Constant Reboiler Power

Reflux Ratio	Top Composition (mol/mol)	Bottom Composition (mol/mol)
Total Reflux		
5:1		
3:1		

Instructor’s Signature:
Date:

Experiment Number -3-

Water Cooling Tower

Objective

To study the mass transfer characteristics of a packed water cooling tower.

Apparatus

A forced draught laboratory packed tower is used. The tower is mounted in the Chemical Engineering main laboratory, pilot plants area. The total depth of packing (z) is 1.27m. Dry and wet bulb temperatures of all streams could be measured by mercury thermometers located at different point in the column. Air flow rate can be measured by an orifice meter. Water flow rate is measured by a flow meter.

Calculations

1.For each run, calculate the overall mass transfer coefficient ky.a using the relation:

$$Z = H_{tOG} . N_{tOG}$$

Z = total depth of packing=1.27m

H_{tOG} = height of transfer unit. (m)

N_{tOG} = number of transfer unit .

$$H_{tOG} = \frac{Gs}{ky.a}$$

Gs= mass flow rate of inert gas. (Kg/hr)

$$N_{tOG} = \int_{H1}^{H2} \frac{dH}{H^* - H} \text{ == area under the curve of } (1/(H^*\text{-}H) \text{ vs. } H)$$

H =Enthalpy of air at wet and dry temperatures. (KJ/kg)

H^*=Enthalpy of air at equilibrium. (KJ/kg)

The integral is to be evaluated using a graphical integration method.

2. The values of ky.a at different gas and liquid flow rates are correlated by the relation:

$$\text{Ky.a} = \alpha\left(\frac{Gs}{L}\right)^{\beta}$$

α, β = constants

References

1. Treybal; R.E., ‘Mass Transfer Operations’, McGraw Hill.
2. J.M.Coulson, and J.L. Richardson, “Chemical Engineering”, vol. 1, Pergamon press.

Water Cooling Tower

Raw Data Sheet

Run No.	1	2	3	4	5
Inlet water Flow Rate(kg/hr)					
Outlet water Flow Rate(kg/hr)					
Inlet air Flow Rate (m^3/hr)					
Inlet air dry bulb temperature (ºC)					
Inlet air wet bulb temperature (ºC)					
Outlet air dry bulb temperature (ºC)					
Outlet air wet bulb temperature (ºC)					
Inlet water temperature (ºC)					
Outlet water temperature (ºC)					

Instructor's Signature:
Date:

Experiment Number -4-

Liquid-Liquid Extraction

Objectives

To examine the mass transfer coefficient's dependence on liquid flow rate for counter current liquid-liquid extraction in a packed column.

Procedure

1. Prepare approximately 20 liters of 0.5 N solution of benzoic acid in toluene and place in feed tank.
2. Fill water storage tank with approximately 20 liters of distilled water.
3. Start water pump and set water flow rate to 200ml/min.
4. Once the extraction column is filled with water, start the feed tank pump.
5. Keep the interface between the organic and aqueous phases constant so that steady state could be achieved.
6. Operate for thirty minutes and then take a sample of each outlet stream and toluene-benzoic acid inlet stream.
7. Titrate the aqueous phase sample with 0.05 M sodium hydroxide solution and the organic phase samples with 0.20 M ethanolic sodium hydroxide solution.
8. Repeat sampling after 5 minutes and the run to be stopped if two successive readings are the same.
9. Repeat for different flow rates of water.

Calculations

Calculate the mass transfer coefficient and find its dependence on liquid flow rate.

References

1. J. M. Coulson, and J. L. Richardson, "Chemical Engineering" vol. 2, Pergamon Press.
2. Treybal, R. E., "Mass transfer Operations", McGraw Hill.
3. T. J. Appel and J. C. Elgin, Counter Current Extraction of Benzoic Acid between Water and Toluene, Ind. Eng. Chem. Vol. 29, No. 4, pg 451-459.

Liquid-Liquid Extraction

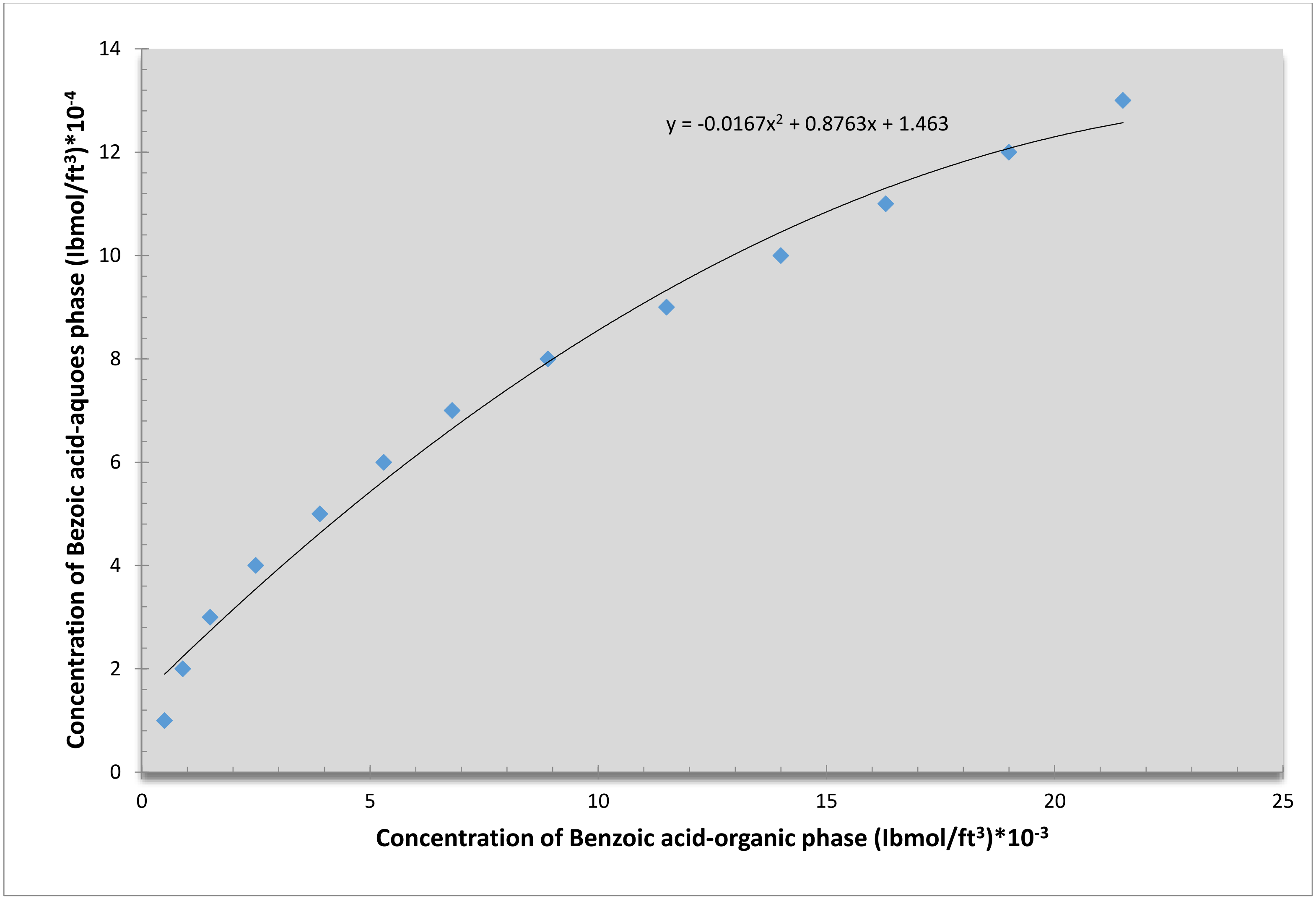
Data Sheet

Concentration of aqueous NaOH: …………………………..

Concentration of ethanolic NaOH: …………………………

Run No.	1	2	3	4	5
Water flow rate					
Volume of ethanolic NaOH titrant needed for feed solution					
Volume of ethanolic NaOH titrant needed for Raffinate					
Volume of aqueous NaOH titrant					

Instructor’s Signature:	
Date:	



y = -0.0167x² + 0.8763x + 1.463
Concentration of Bezoic acid-aquoes phase (Ibmol/ft³)*10⁻⁴
0
2
4
6
8
10
12
14
0
5
10
15
20
25
Concentration of Benzoic acid-organic phase (Ibmol/ft³)*10⁻³

Experiment Number -5-A

Determination of Diffusion Coefficient in Gases

Objectives

1. To determine the diffusion coefficient of acetone vapor into a stagnant non-diffusion air film.
2. To determine the convective mass transfer coefficient kc of the evaporation of pure acetone into air film.

Apparatus

The following figure is the overview experiment unit:

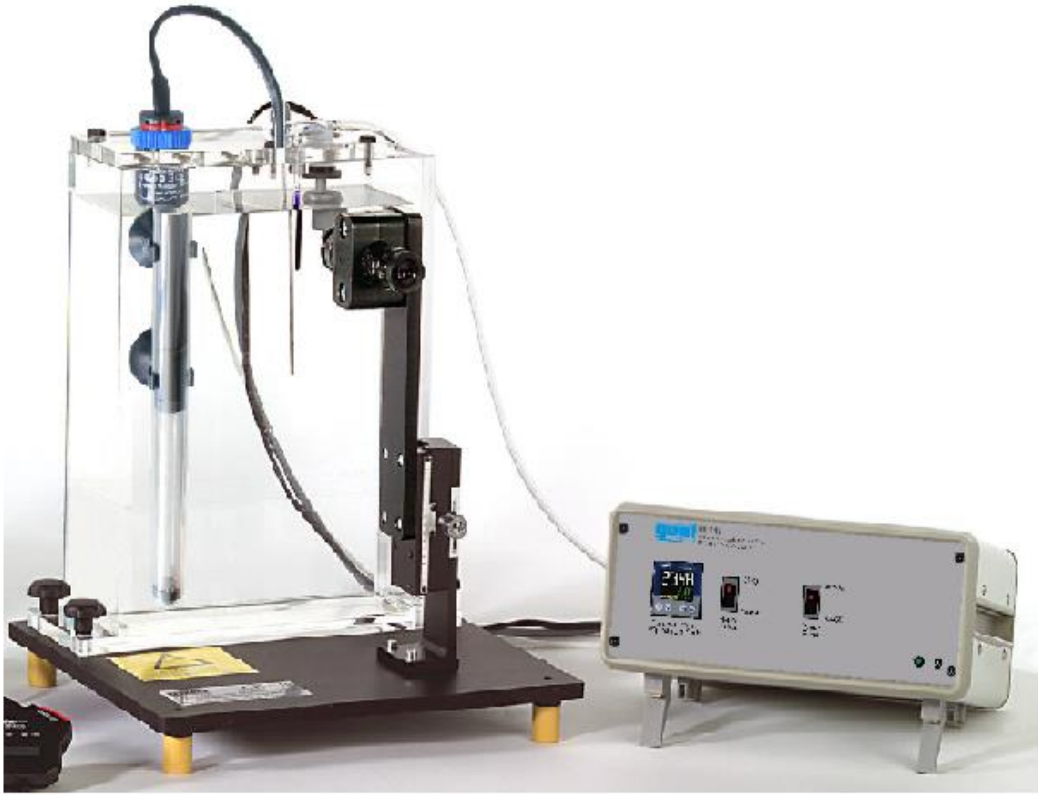
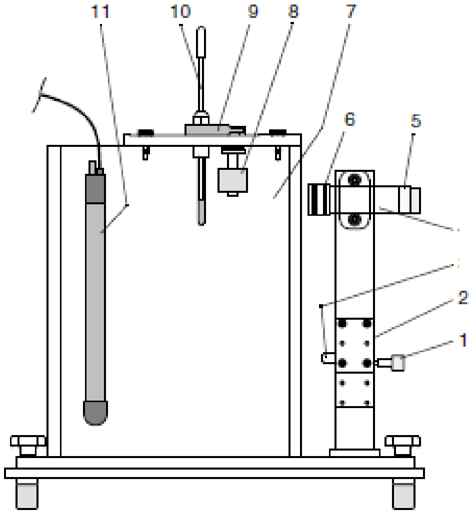


Figure (1): Overview experiment unit

And the following figure shows the parts of the device:



	Experimental unit
1	Mechanism for moving the lens
2	Scale with 0,1 mm vernier for measuring the level
3	Locking screw
4	Lens for viewing the level
5	Scale adjusting ring in the lens
6	Focus ring to focus the level
7	Water bath tank
8	Float switch
9	Diffusion tube
10	Temperature sensor
11	Electric heater

Figure (2): Experimental unit diffusion in gases
(The lens for viewing is shown 90° rotated here.)

The experimental unit essentially consists of diffusion capillary tube (Fig.2) with test liquid (acetone).A temperature-controlled, electrically heated water bath keeps the test liquid at a constant temperature. The temperature sensor in the water bath measures the temperature of the water. A float switch in the water bath ensures that the heating can only be switched on if there is sufficient water. Crosswise to the diffusion direction a constant air flow flows through the upper horizontal tube from the T-piece of the diffusion tube. A lens that can be moved vertically allows the level of the test liquid to be viewed. A scale on the movable lens allows the loss (acetone level) caused by diffusion to be measured.

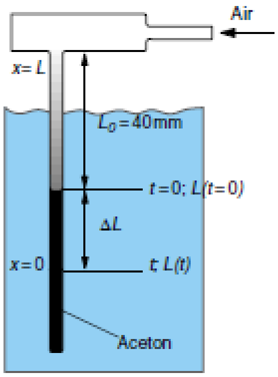


Figure (3): Diffusion tube in water bath

Theory

The rate of diffusion of a vapor through a stationary gas layer, which is assumed to have ideal behavior, can be described by n(t) formula which describes the time-dependent flow of the vapor through the gas:

$$n(t) = -\frac{D.P}{R.T.L(t)}.ln\left[\frac{1-y_0}{1-y_L}\right] \qquad \ldots\ldots (1)$$

Where:
n: Flow of vapor (particle flow) through the gas in g.mol/(m^2.s)
t: Time in s
D: Coefficient of diffusion of vapor in gas in m^2/s
P: Pressure in system in Pa
R: Gas constant J/(g.mol.k)
T: Temperature in K
L: Length of diffusion path in m
y: Molar component (molecular proportion) of vapor in gas phase
0: Index for lower end of diffusion path
L: Index for upper end of diffusion path

To derive the relationship between n(t) and L(t), we equate the above equation with evaporation equation:

$$n(t) = \frac{\rho}{M}.\frac{dL(t)}{dt}$$

Where:
ρ: Density of vapor in kg/m^3
M: Molar mass of vapor in kg/(g.mol)

We thus obtain differential equation:

$$\frac{dL(t)}{dt} == -\frac{M.D.P}{\rho.R.T.L(t)}.ln\left[\frac{1-y_0}{1-y_L}\right]$$

To simplify the calculation, a variable K is introduced, which includes all of the variables that are not dependent on time:

$$K = -\frac{M.D.P}{\rho.R.T}.ln\left[\frac{1-y_0}{1-y_L}\right]$$

Can be rearranged for D:

$$D = -\frac{\rho.K.R.T}{M.P.ln\left[\frac{1-y_0}{1-y_L}\right]}$$

Inserting the variable K into the differential equation:

$$\frac{dL(t)}{dt} = \frac{K}{L(t)}$$

The differential equation can be rearranged and integrated by inserting the conditions gives us the specific limits, then a linear function results:

$$\Delta L = 2.K.\frac{t}{\Delta L} - 2.L_0$$

The measured data can be used to calculate K using linear regression; which then can be used to calculate D.

Procedure

1. Preheat the water bath to the required temperature (35°C).
2. Fill the diffusion capillary tube with acetone until the distance from the lower edge of the
3. T-piece to the acetone level is 40mm (starting condition).
4. Connect the diffusion pipe to the air on the control unit. And insert the diffusion capillary tube into the unit in such a way that the diffusion capillary tube with the acetone is exposed to the temperature in the water bath.
5. Turn on the air pump using the control unit.
6. Record the level of acetone in the diffusion capillary tube with time.

Calculation

1. Plot the change in acetone level against the difference quotient t/ΔL in a graph.
2. Determine the K factor.
3. Calculate the coefficient of diffusion D.

References

Diffusion in Liquid and Gases experiment instruction manual, Gunt

Determination of Diffusion Coefficient in Gases

Raw Data Sheet

Temperature: ____________________

Density of Acetone: _____________________

Atmospheric Pressure: __________________

Time	**Height**

Instructor’s Signature:
Date:

Experiment Number -5-B

Determination of Diffusion Coefficient in Liquid

Objectives

Determination of the diffusion coefficient for the mass transport in liquid

Apparatus

Figure (1): Experimental unit diffusion in Liquids

The main components for this part of the experiment are a diffusion cell and a flask.The diffusion cell is a U-tube, which is closed with a capillary disc on one side. For the experiment, this diffusion cell is filled with a salt solution. During the experiment, salt ions will diffuse out of this solution through the capillaries. The flask is filled with distilled or demineralised water and represents the sink for the ions. They diffuse into it. To achieve this, the diffusion cell is positioned in the flask in such a way that the capillary disc is vertical and is below the water level.

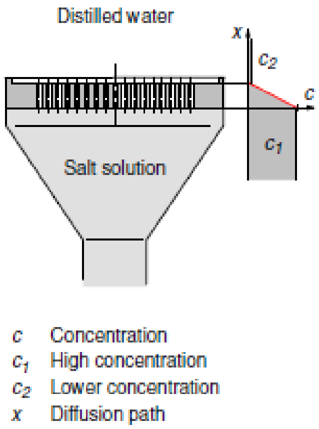


Figure (2): Concentration gradient at capillary disc

This arrangement means that the salt solution is at the lower side of the capillary disc, with clear, demineralised water on its upper side. This results in a defined concentration gradient dc/dx along the length of the vertical capillaries. As a result of the concentration gradient, the ions diffuse through the capillaries from the salt solution into the water. The capillaries restrict this motion to a single direction. To enable us to draw conclusions about the number of ions diffused through the capillaries, the conductivity of the water in the flask is constantly measured. Even tiny quantities of ions increase the conductivity of water significantly. The change in conductivity dκ/dt in the flask is a direct linear measure for the change in the quantity of ions in the flask. The water is constantly agitated by the magnetic stirrer to evenly distribute the diffused ions to give as accurate a conductivity measurement as possible and prevent local concentration of the capillary disc. Due to the low absolute number of diffusing ions, the concentrations in the salt solution and in the distilled water can be assumed to be constant.

Theory

From FICK‘s 1st law

$$J = -D.\frac{\partial c}{\partial x}$$

and the formula for calculating the molar concentration c2 (large volume with low concentration

$$c_2 = \frac{J.t.A}{V}$$

Resolved for J:

$$J = \frac{c_2 . V}{t . A}$$

Then:

$$-D . \frac{\partial c}{\partial x} = \frac{c_2 . V}{t . A}$$

From the 3-dimensional partial differential $\partial c/\partial x$one dimensional view becomes dc/dx with c2 ≈ 0 and c1 >> c2 thus:

$$-\frac{c_1 - c_2}{x} = -\frac{c_1}{x}$$

For c2 the following relationship also applies:

$$c_2 = \frac{\Delta \kappa}{\Delta t} . \frac{1}{c_s}$$

to enable the molar concentration to be recorded using a conductivity meter.

Then the Coefficient of Diffusion can be expressed by this formula:

$$D . \frac{c_1}{x} = \frac{\Delta \kappa}{\Delta t} . \frac{1}{c_s} . \frac{V}{A}$$

or

$$D = \frac{\Delta \kappa}{\Delta t} . \frac{x . V}{c_s . A . c_1}$$

Where:

A: Diffusion area in m^2

V: Volume of solution with lower concentration (sink) in m^3

$\frac{\Delta \kappa}{\Delta t}$: Change in conductivity of sink over duration of experiment in µS/(m.s)

c_1: Molarity value of solution in flask with higher concentration in mol/m^3

D: Coefficient of diffusion in m^2/s

t: Time in s

x: Diffusion path (capillary length) in m

c_s: Conductivity of solution per mol in large volume $\Delta\kappa/\Delta c = c_s$ in $115.m^2/mol$

J: Specific diffusion flow (particle flow density) in mol/ (m^2.s)

FICK‘s 1st law can then be applied as follows for an experiment with a solution

$$D = \frac{\Delta \kappa . V . x}{\Delta t . N . \frac{\pi . d_c^2}{4} . c_1 . c_s} = \frac{4 . V . x}{N . \pi . d_c^2 . c_1 . c_s} . \frac{\Delta \kappa}{\Delta t}$$

Where:

A: Diffusion area in m^2 (area of all capillaries)

$$A = N . \frac{\pi . d_c^2}{4}$$

N: number of capillaries (253)

d_c: Capillary diameter (1mm)
x: Capillary length (5mm)

Procedure

1. Fill the measuring flask with 900mL of the distilled water.
2. Place the filled measuring flask on the magnetic stirrer.
3. Prepare 1 N (NaCl) solution: weigh out 11,5g of NaCl and dissolve it completely in 200mL distilled water.
4. Fill the diffusion cell with the (NaCl) solution (approx. 30mL) until the solution escapes at the capillary disc. The capillary disc has a small step on its edge. This ensures that some of the experimental solution can remain above the capillaries.
5. Using a pipette or syringe, remove any air bubbles that may remain in the capillaries until as many of the capillaries as possible are filled with solution.
6. Then carefully wipe excess (NaCl) solution from the capillary disc. If necessary, add slightly more (NaCl) solution
7. Now position the diffusion cell in the measuring flask and attach it to the bracket in such a way that the upper edge of the capillary disc is around 5mm above the water level.
8. Extremely carefully allow the rest of the distilled water to flow into the measuring flask, if possible without creating any swirling or turbulence. To do this, allow the water to flow along a rod or a laboratory spoon. The capillary disc should now be covered by approx. 5mm of water. The setup is now prepared for an experiment. The experiment begins immediately after completing the preparation described above.
9. Set the magnetic stirrer to a speed of 3 to max. 5 revolutions per second.
10. Each 5 min record the conductivity and the temperature in measuring flask.

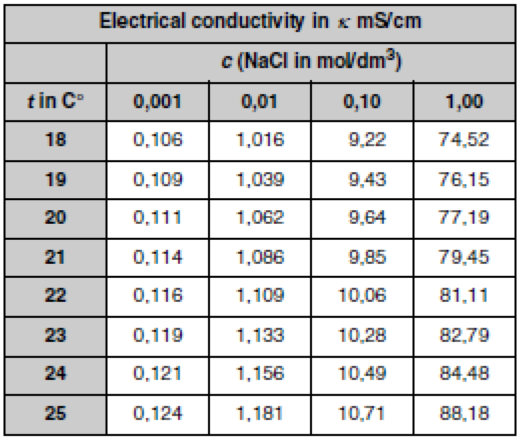
Calculation

1. Plot the measured values obtained for the conductivity in the flask in a graph over time.
2. Calculate the difference quotient $t/\Delta L$ from the conductivity and experiment time from the linear area of the measuring curve.
3. Use this data to calculate the coefficient of diffusion D

References:

Diffusion in Liquid and Gases experiment instruction manual, Gunt

Table (1): Electrical conductivity in mS/cm of NaCl solutions depending on temperature.



Electrical conductivity in κ mS/cm				
	c (NaCl in mol/dm^3)			
t in C°	**0,001**	**0,01**	**0,10**	**1,00**
18	0,106	1,016	9,22	74,52
19	0,109	1,039	9,43	76,15
20	0,111	1,062	9,64	77,19
21	0,114	1,086	9,85	79,45
22	0,116	1,109	10,06	81,11
23	0,119	1,133	10,28	82,79
24	0,121	1,156	10,49	84,48
25	0,124	1,181	10,71	88,18

Determination of Diffusion Coefficient in Liquid

Raw Data Sheet

NaCl Molarity: ____________________

Distilled water volume: _____________________

Conductivity of distilled water: __________________

Time t in min	Conductivity κ in	Temperature T in ⁰C

Instructor’s Signature:
Date:

Experiment Number -6-

Wetted Wall column

Objectives

To determine the power-law relationship between the liquid film mass transfer coefficient and the mass flow rate of water and to compare the results with theoretical predictions.

Apparatus

The wetted wall column consists mainly of:

- Supporting framework (30) stands on four adjustable feet (10) so that the apparatus can be set level on a hard floor surface.
- Water supply storage tank (8).
- De-oxygenator feed pump (15).
- De-oxygenator column (13).
- The absorption column feed pump (16).
- Water flowmeter (28).
- Water absorption column (3).
- Absorption column bearing and knife-edge gimbal (2).
- Absorption column knurled screws in the support ring (31).
- Dissolved oxygen probe (12) situated at absorber inlet.
- Dissolved oxygen probe (9) situated at the absorber outlet.
- Dissolved oxygen meter (19).
- Air compressor (17).
- Air flowmeter (27).
- Absorption column sintered metal disc (5).
- Nitrogen regulator (26).
- Nitrogen inlet valve (14).
- De-oxygenator column sintered metal disc (18).

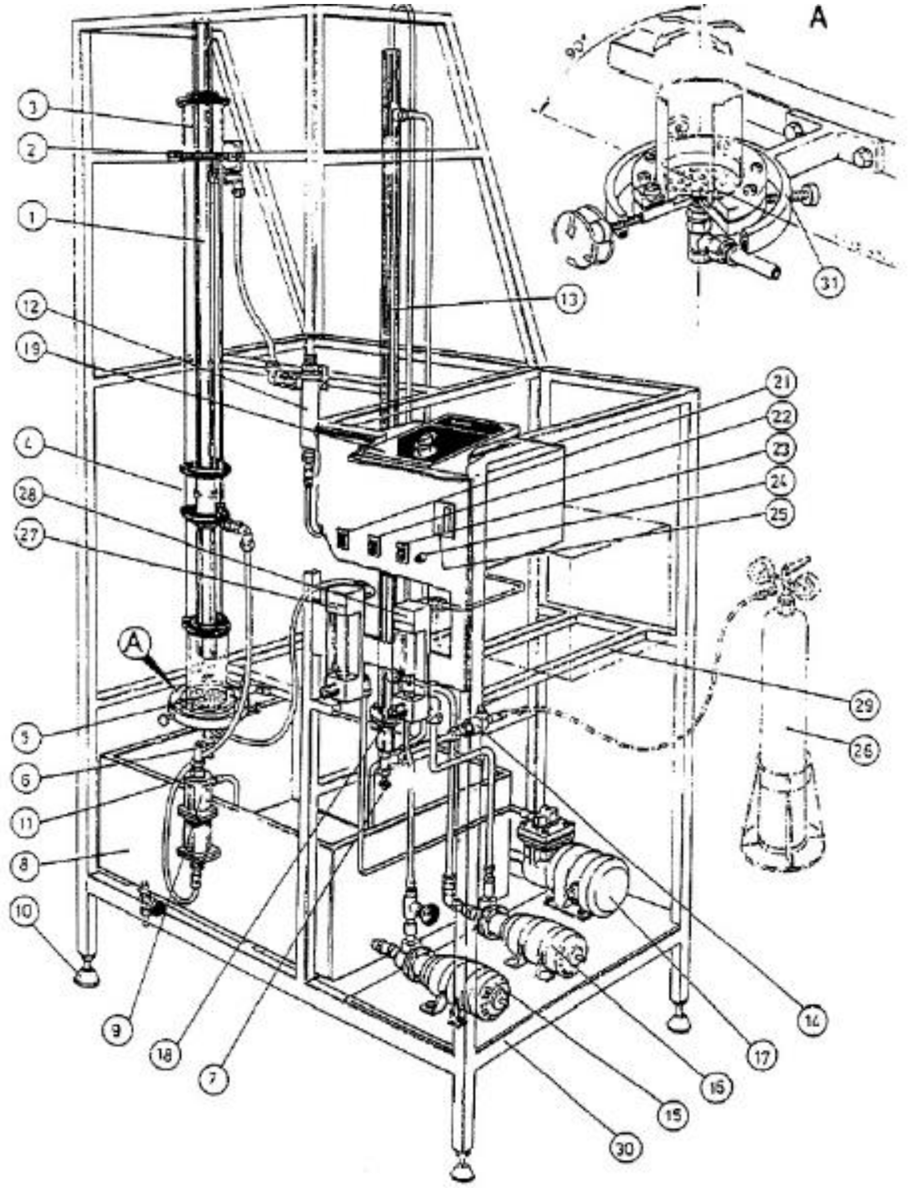


Fig.(1): Wetted Wall Gas Absorption Column

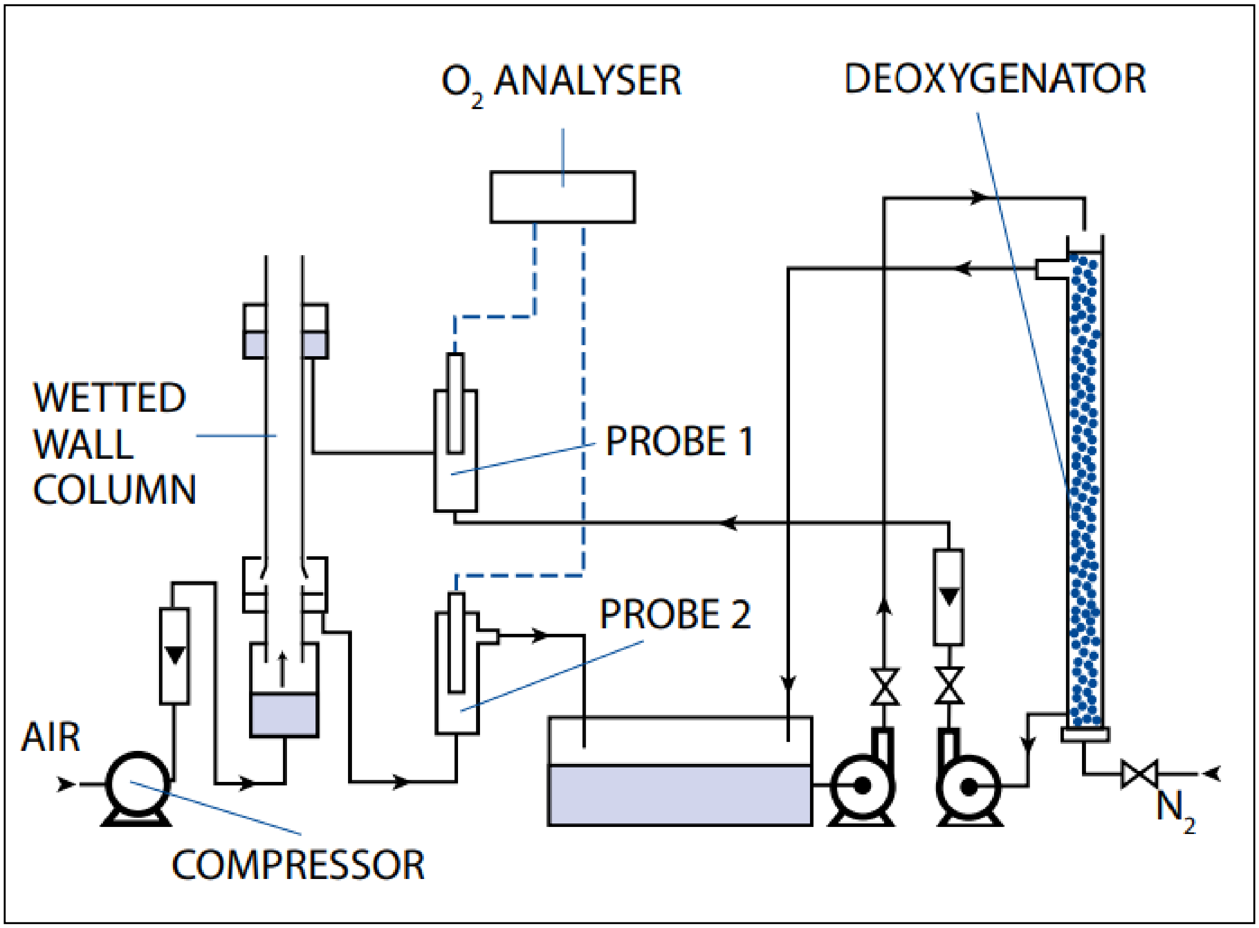


Fig.(2) : Set Up Flow Chart

Theory

The system chosen for the experiment is the absorption of oxygen into oxygen-free water. In this system the solubility and enthalpy of solution are small and by saturating the inlet air with water, humidification effects are eliminated. Thus it is possible to maintain reasonably isothermal conditions throughout the column. Experimental procedure allows a power law relationship to be calculated and comparison is made between this and published relationships such as:

The absorption of oxygen in water is an example in which most of the resistance to transfer lies in the liquid phase. Many measurements of the mass transfer coefficient have been developed for comparison between the coefficient and the mass flow rate.

$$(Sh)_L = 0.724(Sc)_L^{1/2}(Ga)_L^{1/6}(Re)_L^{0.333} \ldots\ldots\ldots\ldots\ldots\ldots\text{(a)}$$

$$(Sh)_L = 0.433(Sc)_L^{1/2}(Ga)_L^{1/6}(Re)_L^{0..4} \ldots\ldots\ldots\ldots\ldots\ldots\text{(b)}$$

Where

$$(Sh)_L = \frac{K_L\, Z}{D_L}$$

$$(Sc)_L = \frac{\mu_L}{\rho_L D_L}$$

$$(Ga)_L = \frac{\rho_L^2 g_Z^3}{\mu_L^2}$$

$$(Re)_L = \frac{4\ \ \Gamma}{\mu_L}$$

And

k_L = liquid film mass transfer coefficient (m/s)
Z = height of column (m)
D_L = diffusivity of oxygen in water (m^2/s)
μ_L = viscosity of water (Ns/m^2)
ρ_L = density of water(kg/m^3)
Γ = mass flow rate of water per meter of wetted perimeter (kg/s.m)

It is therefore possible to determine the power-law relationship between (Sh)L and (Re)L by experiment and compare it with the above formulae. This will require calculation of kL from the following formula:

$$K_L = \frac{j}{A.\Delta C_{LM}}$$

Where

j = change in concentration of oxygen in the water passing through the column x volumetric flow rate of water
A = Area for mass transfer
ΔC_{LM} = Log mean concentration difference

Procedure

1. Fill the water supply tank with ordinary tap water until the level is about 80mm above the pump suction connections.
2. Start the de-oxygenator vessel feed pump and regulate the flow until the vessel is maintained full to the overflow.
3. Admit nitrogen to this vessel and regulate the nitrogen flow until a constant stream of gas bubbles fills the vessel.

Note: If a plug flow pattern occurs in this vessel there is too high nitrogen flow rate.

4. Start the compressor and regulate the air flow rate until it reads 1000 cm^3/min on the flowmeter.
5. Start the wetted wall column feed pump and set the flow rate to 60 cm3/min on the flowmeter.
6. Measure the oxygen concentration in the ingoing water with the appropriate oxygen meter. The oxygen content of this water will fall to below 10.0% as the deoxygenator takes effect.
7. The water will gradually fill the top section of the wetted wall column and overflow down the inside wall.
8. A complete wetted wall round the whole perimeter can be achieved by cleaning the inside of the tube with the special long handled brush provided. At this point, fine adjustments can be made in the positioning of the column to achieve the required all round water film.
9. Oxygen will be absorbed into the water and readings can be taken from both inlet and outlet by using the two oxygen meters.
10. Increase the water flow rate to 100 cm^3/min. Now sufficient time must be allowed for the water at the new flow rate to pass through the inlet probe. This will take a maximum of 15 minutes so it is best to leave 15 minutes between readings after each change in flow rate.
11. Repeat for flow rates of 120, 150, 200, 250 and 280 cm^3/min. When this series of readings has been taken, reduce the flow rate back to 60 cm^3/min and reset the air flow rate at 2000 cm^3/min.

Calculations

For various flow rates of oxygen (in the form of air) a power law can be determined and compared to published values. In order to plot the graph, the student must calculate log mean concentration difference, mass flux and transfer coefficients. Also, a full error analysis can be carried out

The mass transfer coefficient in the liquid phase could be calculated from:

$$K_L = \frac{j}{A.\Delta C_{LM}}$$

Where

K_L = liquid film mass transfer coefficient (m/s)

j = Change in concentration of oxygen in the water passing through the column x volumetric flow rate of water (Kg/s)

A = Area for mass transfer (m^2)

ΔC_{LM} = Log mean concentration difference (Kg/m^3)

References

1. R.E. Treybal , Mass Transfer Operation , 3 rd Ed. McGraw – Hill Book Co.
2. W.L. Badger and J.T. Banchero , Introduction to Chemical engineering , McGraw – Hill Book Co.
3. O.C , Bennet and J.C Myero, Momentum Heat and Mass Transfer , 3 rd Ed.McGraw – Hill Book Co.

Wetted Wall Column
Raw Data Sheet

Air Flow Rate =1000 cm^3/min

Water Flow Rate cm^3/min	%-sat O_2 IN	%-sat O_2 OUT
60		
100		
120		
150		
200		
250		
280		

Air Flow Rate =2000 cm^3/min

Water Flow Rate cm^3/min	%-sat O_2 IN	%-sat O_2 OUT
60		
100		
120		
150		
200		
250		
280		

Air Flow Rate =3000 cm^3/min

Water Flow Rate cm^3/min	%-sat O_2 IN	%-sat O_2 OUT
60		
100		
120		
150		
200		
250		
280		

Instructor's Signature:	
Date:	

Appendix (A)

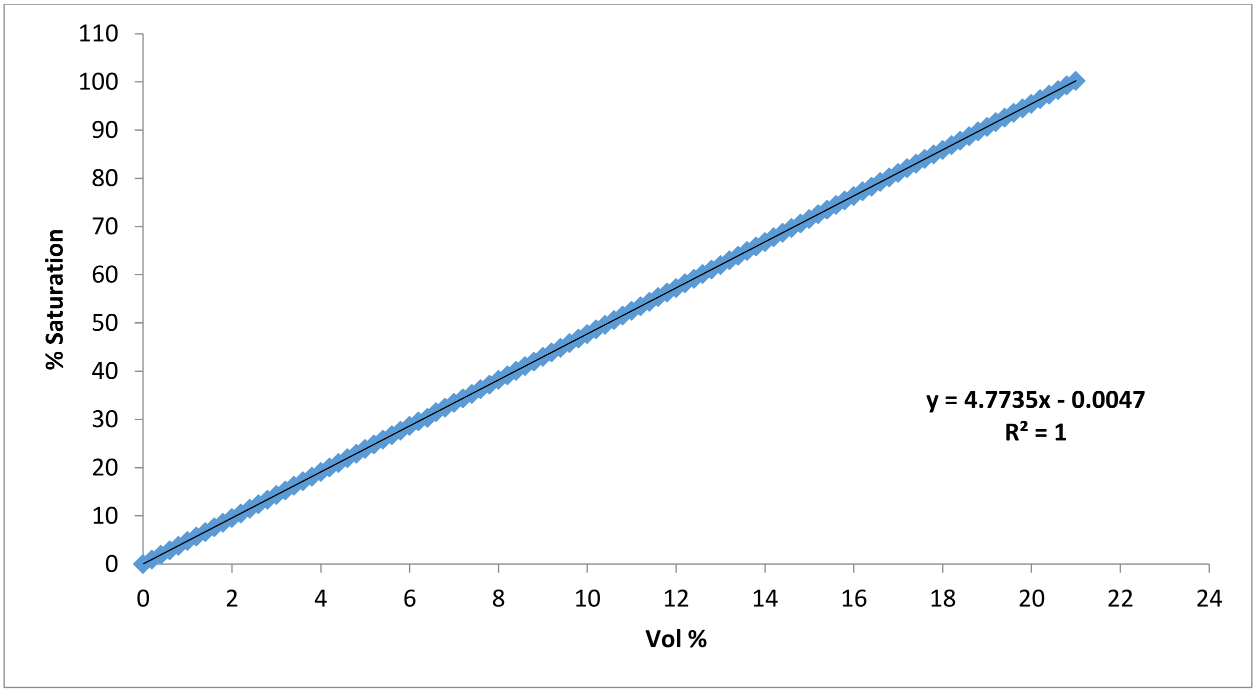


Fig.A (1): Conversion chart from %- Volume to %-Saturation for dissolved oxygen meter

Experiment Number -7-

Adsorption of Dye Solution on Activated Carbon

Objectives

1. To produce Concentration –Time curve for the adsorption of dye solution by activated carbon.
2. To investigate the effect of initial dye concentration and speed of agitation on the Concentration- Time curves.
3. To construct the equilibrium isotherm for the adsorption of solute on adsorbent.

Introduction

Activated carbon adsorption is one of the physical purification techniques which offer one of the most efficient processes available for removing certain organics and in-organics from waste water.

Physical adsorption results from the action of Vander Waals forces comprised of London dispersion forces and classical electro-static forces between adsorbate and adsorbent.

Apparatus

The apparatus comprises of 3×2 L beakers, 3 variable motors, 50 small bottles (about 10-15 ml), and visible spectrophotometer.

Materials

Activated carbon and dye solutions

Procedure

a) Isotherms
   1. Make up 2 liters dye solution by dissolving exactly 1g dye in 2 liters of distilled water.
   2. Using the small bottles, weigh out 0.001, 0.002, 0.004, 0.008, 0.012, 0.016, 0.020, 0.030, 0.040, 0.080, 0.100,and 0.15 (g carbon).

b) Batch studies
   1. Fill the beaker to 1.7 liter of dye solution; adjust the speed of agitator to three different values in the beakers (100,200,300rpm).
   2. Add .085 g of carbon to each beaker. Time sample should be taken for the three beakers after (1, 2, 3,4, 6, 9, 15, 25, 35, 49, 64, and80 min).
   3. Repeat step 1 and 2, but change the amount of carbon to 1.2g.

Calculations

1. Plot (Cf/C0) vs. time for the three different speeds.
2. Plot the equilibrium isotherm curve and find the Freunlich equation constants

References

1. Treybal, R.E, “Mass Transfer Operation”, McGraw Hill.
2. Perry and Chilton, “Chemical Engineer’s Handbook”, McGraw Hill.

Adsorption of Dye Solution on activated Carbon

Raw Data Sheet (1)

a. Isotherms

Volume of Solution	
Initial Concentration of Solution	

Mass of Carbon (g)	**Reading of (Absorbance OR Transmittance) at equilibrium**
0.001	
0.002	
0.004	
0.008	
0.012	
0.016	
0.020	
0.030	
0.040	
0.080	
0.100	
0.150	

Raw Data Sheet (2)

b. Batch Studies

Volume of the Solution	
Mass of Carbon	
Speed of Agitator	
Initial Concentration of Solution	

Time	Reading of (Absorbance OR Transmittance)		
(min)	Run#1	Run#2	Run#3
1			
2			
3			
4			
6			
9			
15			
25			
35			
49			
64			
80			

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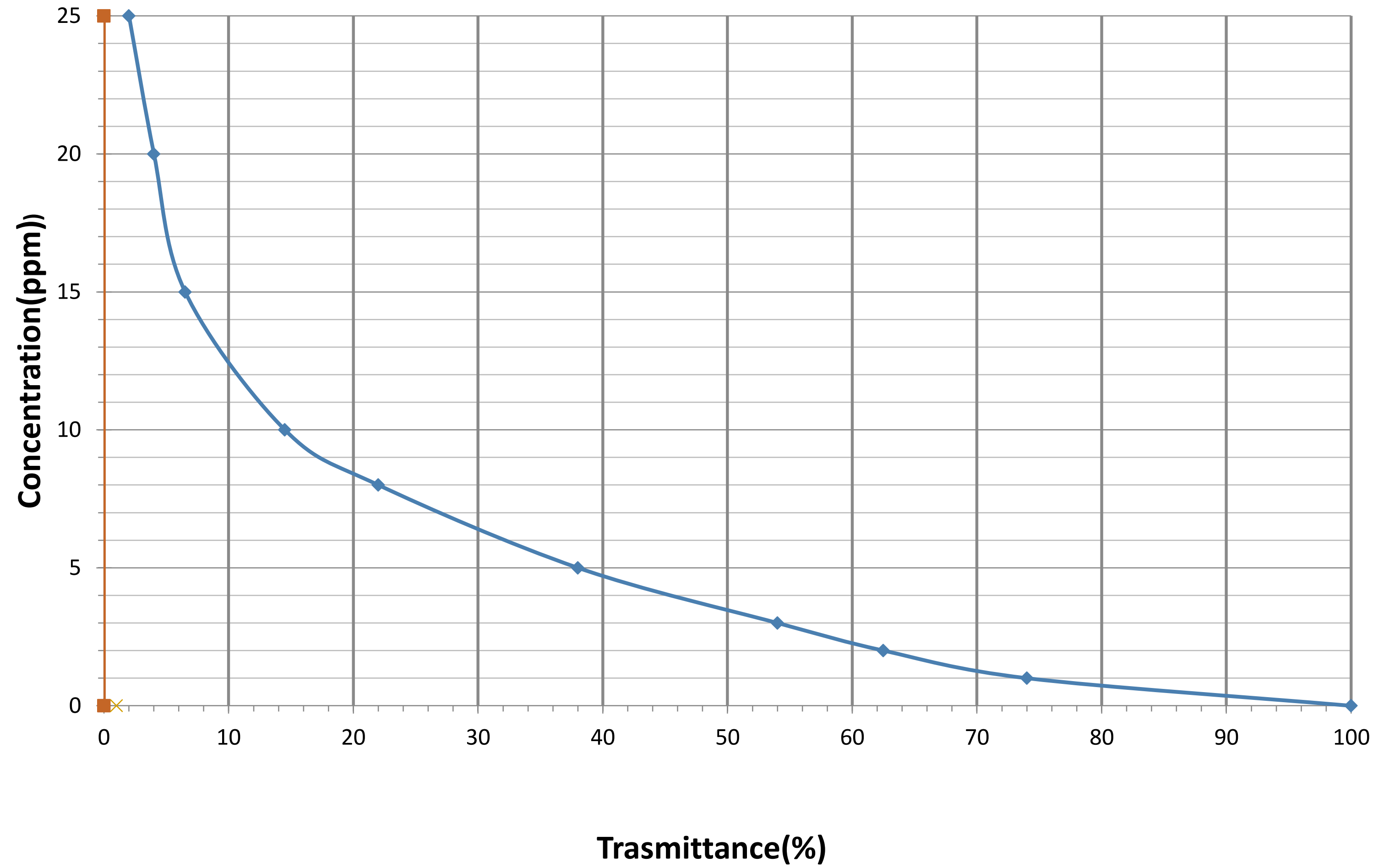
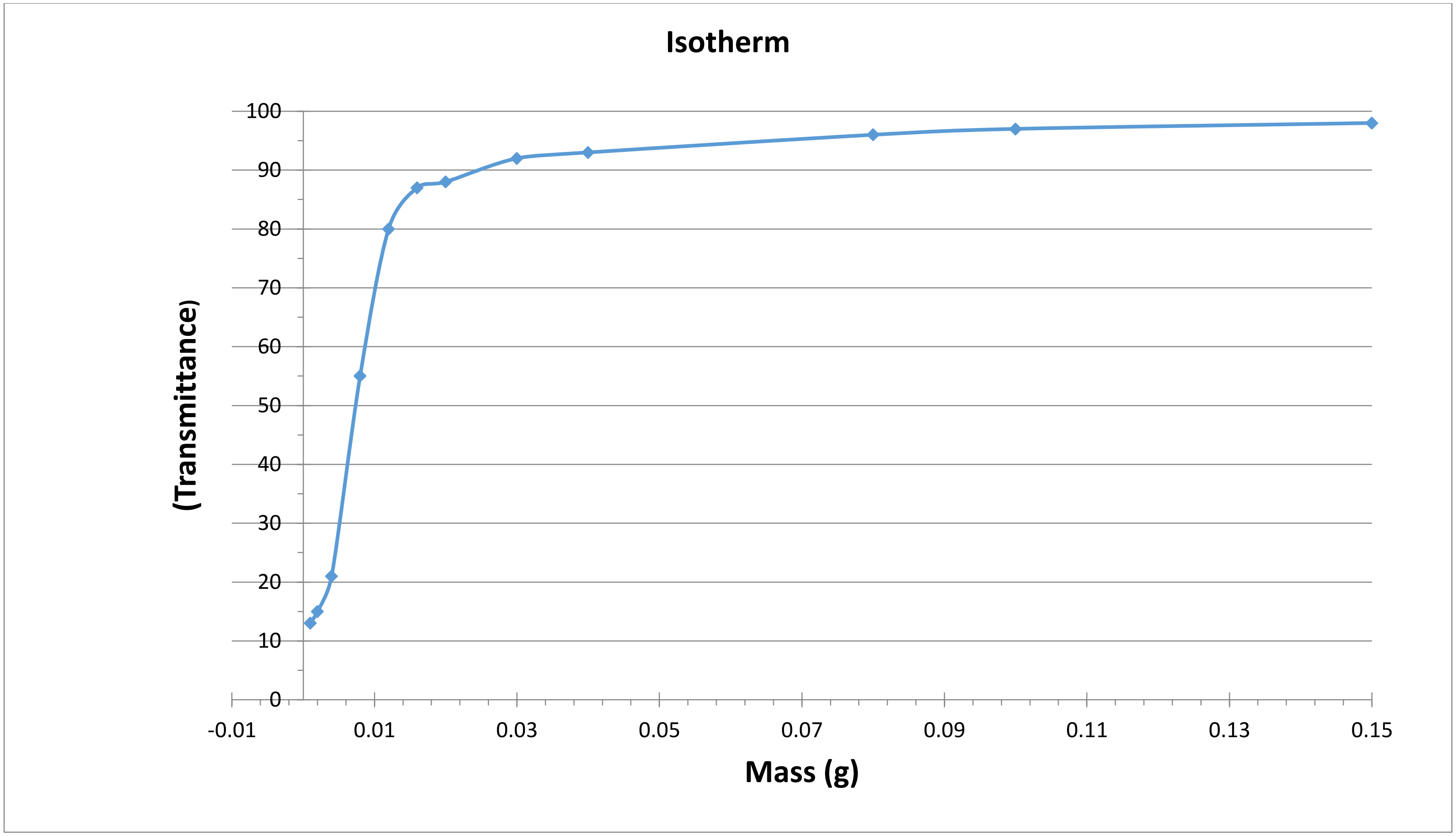
Date:


25
20
15
10
5
0
0
10
20
30
40
50
60
70
80
90
100
Concentration(ppm))
Trasmittance(%)


Isotherm
(Transmittance)
100
90
80
70
60
50
40
30
20
10
0
-0.01
0.01
0.03
0.05
0.07
0.09
0.11
0.13
0.15
Mass (g)

Experiment Number -8-

Soxhlet Extraction

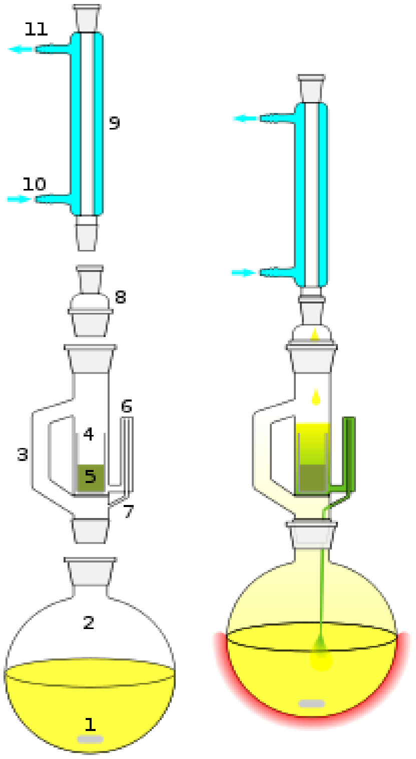
Objectives

1. To study Solid-Liquid extraction process using Soxhlet extractor.
2. To investigate the effect of residence time and solvent type on the extraction of oil from Olive cake.
3. To calculate the Soxhlet extraction efficiency.

Apparatus

The experiment apparatus (Figure (1)) consists of:

- Soxhlet extractor - 40 mm ID, with 500-mL round bottom flask.
- Thimble
- Condenser
- Heating mantel



1. Solvent
2. Still pot
3. Distillation arm
4. Thimble
5. Solid
6. Siphon top
7. Siphon exit
8. Expansion adapter
9. Condenser
10. Cooling water in
11. Cooling water out

Figure (1): A schematic diagram of a Soxhlet extractor

The solid material containing some of the desired compound is placed inside a thimble made from thick filter paper, which is loaded into the main chamber of the Soxhlet extractor. The extraction solvent to be used is taken into a distillation flask and the Soxhlet extractor is then equipped as shown in fig (1). The solvent is heated to reflux. The solvent vapor travels up the distillation arm and floods into the chamber housing the thimble of solid. The condenser ensures that any solvent vapor cools, and drips back down into the chamber housing the solid material.

The chamber containing the solid material is slowly filled with warm solvent. Some of the desired compound will then dissolve in the warm solvent. When the Soxhlet chamber is almost full, the chamber is automatically emptied by a siphon side arm, with the solvent running back down to the distillation flask. This cycle may be allowed to repeat many times, over hours or days.

After extraction the solvent is removed, typically by means of a rotary evaporator, yielding the extracted compound.

Theory

Solid liquid extraction (leaching) means the removal of a constituent from a mixture of solids by bringing the solid material into contact with a liquid solvent that dissolves this particular constituent. In this experiment oil is extracted from olive cake.

The amount of oil extracted (oil percentage) can be calculated as follow:

$$\text{Oil percentage} \quad = \frac{\text{Weight of solid before extraction} - \text{Weight of solid after extraction}}{\text{Weight of solid before e}} * 100\%$$

$$\text{Extraction Efficiency} = \frac{\text{Experimental oil percentage}}{\text{Theoretical oil percentage}} * 100\%$$

Procedure

Part 1: Residence Time effect

1. Weigh about 10 grams of olive cake sample.
2. Put the solid sample into a previously weighed thimble and then put into the Soxhlet apparatus.
3. Add 100 ml of Hexane solvent to the extraction chamber.
4. Turn the heat on and start the experiment.

5. End the experiment after 3 extraction cycles (3 siphons).
6. Turn off the heater and wait until it cools down. Remove the thimble and leave it in the fume hood to dry out.
7. Repeat steps (1-6) three times doing 6, 9 and 12 cycles.
8. The next day, when the thimbles containing the olive cake are completely dry, record the weight of each thimble.

Part 2: Solvent Type effect

1. Perform the experiment as in (Part 1) using (100 ml Hexane) and end it after 8 cycles.
2. Repeat the experiment using (100 ml Isopropanol) and end it after 8 cycles.

Note: Either Part 1 or Part 2 is done in the lab.

Calculations

1. Calculate oil percentage for each run.
2. Draw the percentage of oil as a function of number of cycles done.
3. Calculate the extraction efficiency.
4. What are the optimum conditions (number of cycles and solvent type) for Soxhlet extraction of oil from olive cake, why?

Soxhlet Extraction Data Sheet

Part 1: Residence Time effect

Type of solvent	No. of cycles	Thimble's weight (g)	Olive cake weight (g)	Final weight (g)

Part 2: Solvent Type effect

Type of solvent	No. of cycles	Thimble's weight (g)	Olive cake weight (g)	Final weight (g)

Instructor's Signature:
Date:

Experiment Number -9-

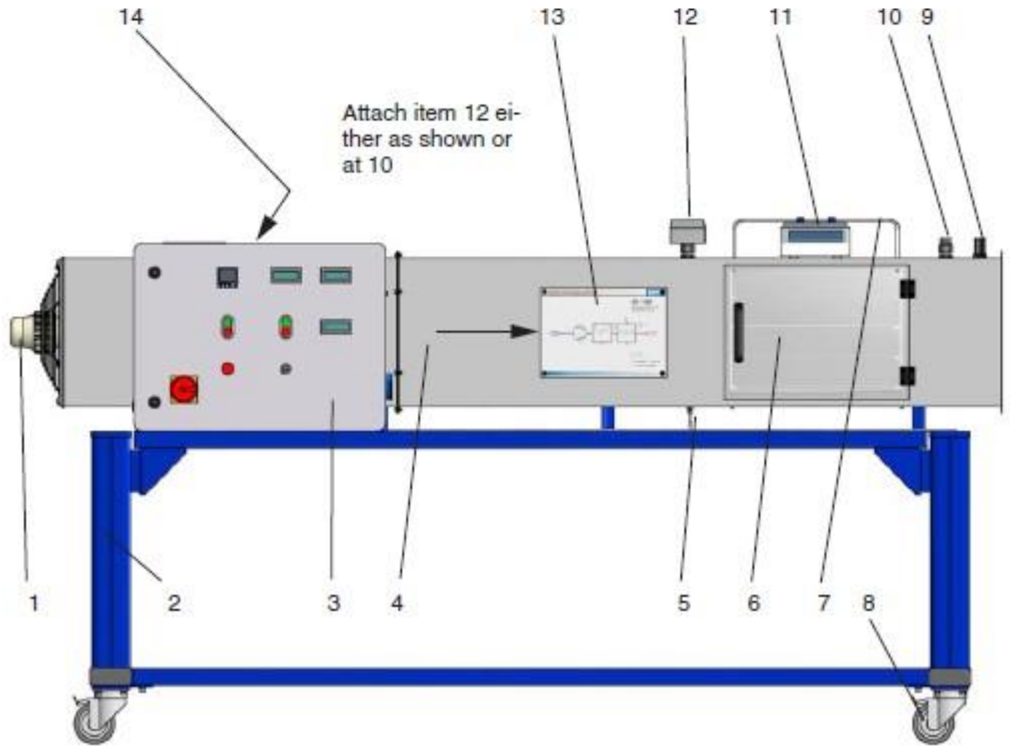
Convection Drying

Objectives

1. To determine the reduction in weight for a wet solid during drying process.
2. To produce a drying rate curve for the wet solid in air of fixed temperature and humidity.
3. To investigate the effect of air temperature and air velocity on drying rate.
4. To calculate the total heat transfer coefficient, and mass transfer coefficient for a wet solid in air.

Apparatus

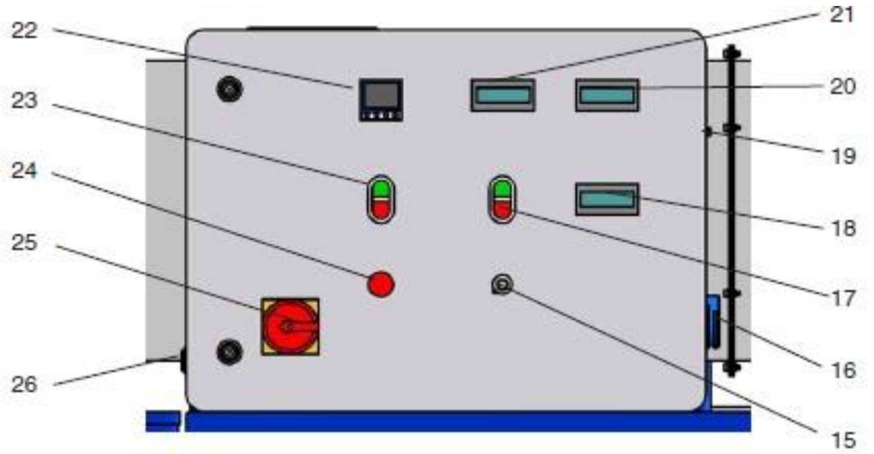
The apparatus consists of a flow channel with transparent section where the material to be dried can be observed on a holder inside the flow channel during the experiment. The drying air is fed into the flow channel by way of an adjustable fan and is passed over the material to be dried. To improve the drying process, the air can be warmed before hand, using a heater where the power can be adjusted to various levels; both fan and heater are controlled from the control cabinet. The quantitative evaluation is performed using a mobile combined temperature/moisture sensor for measuring the temperature and relative humidity of air before and after the material holder, a digital scale for taking the weight of material and an anemometer for taking air speed. See Figures (1, 2 &3).



- 1 Fan
- 2 Support
- 3 Control cabinet
- 4 Flow channel
- 5 Temperature sensor of the controller
- 6 Transparent flap
- 7 Material holder with support
- 8 Locking wheels
- 9 Anemometer
- 10 Holder for the Temperature T - humidity sensor φ
- 11 Digital scale
- 12 Temperature T - humidity sensor φ
- 13 System diagram
- 14 Heating element on the rear

Fig.1: Overview of Convection Drying Apparatus.

Fig.2: Fan at air inlet.



15 Fan speed control
16 Power socket
17 Fan on / off switch
18 Relative humidity φ
19 Connection for scale
20 Temperature T
21 Air speed in channel in m/s
22 Temperature controller of the Heater
23 Heater on / off switch
24 Temperature monitor display
25 Master switch
26 USB port

Fig.3: Control Cabinet

Procedure

1. Remove drying plates from support frame.
2. Tare scale to zero.
3. Insert drying plate into support frame and record the weight of the drying plate on the digital scale.
4. Switch on the fan, set the speed using flow rate gauge (e.g. 1.3 m/s) and record the value.
5. Switch on heater, set the temperature and record the value.
6. Run air conditioning duct until it is warmed up evenly all along its length.
7. Record the readings of Air temperature T1 and Relative humidity(1) before material to be dried.
8. Spread material to be dried in a thin layer on the drying plates.
9. Insert drying plate in the support frame and record the total weight.
10. Tare scale to zero.
11. Add some water to the material and record the weight.
12. Start drying experiment, start time measurement using stopwatch.

13. At regular intervals time record the following measurements and enter on the worksheet:
    - Weight of water in the wet material using the digital scale.
    - Air temperature T2 after material to be dried.
    - Relative humidity2 after material to be dried.
14. The drying experiment is complete when either:
    - The drying time specified in advance has expired.
    - The mass of the material to be dried is no longer falling.
15. Once drying is complete, switch off the unit under the supervision of your supervisor.
16. The procedure can be repeated with another variable such as air speed, air temperature or different material.

Theory

Immediately after contact between the wet solid and drying medium (air), the solid temperature adjusts until it reaches a steady state. The drying rate becomes constant and remains constant until it reaches certain critical moisture content.

The constant rate of drying is given by:

$$(\mathcal{H}_2 - \mathcal{H}_1)\, R_c = M.A$$

$$M \,.\, A = V.\, \rho \,.\, A$$

V = air velocity. (m/hr)

A_d = cross-sectional area of duct. (m^2)

ρ = air density. (kg/m^3)

$\mathcal{H}_1$, $\mathcal{H}_2$ = specific humidity of air at outlet and inlet of trays. .(Kg/Kg)

** Use humidity chart or equations from your references to obtain specific humidity.

M = air mass flow rate per unit area (Kg/hr. (m^2)

The rate of drying is also given by:

$$= \frac{h}{\lambda}\left(T_{dry} - T_{wet}\right).A\, R_c$$

Rc =constant rate of drying (kg/hr)

h = heat transfer coefficient ($KJ/hr.m^2.k$).

λ = latent heat of evaporation of water. (KJ/Kg)

T_d,T_w = temperature of inlet dry &wet air.(K)

** Use humidity chart or equations from your references to obtain Tw at experiment conditions

A = area of drying plate (m^2)

The rate of drying (R_c) also depends upon the rate of diffusion of vapor into the bulk gas stream and is given by:

$$= h_d \,.\, A.\rho\,(\mathcal{H}_w - \mathcal{H})\,R_c$$

h_d = Mass transfer coefficient. (m/hr)

A =Area of tray/ trays. (m^2)

ρ =Density of the gas. (kg/m^3)

$\mathcal{H}_w$ = Humidity of the gas saturated with vapor at the inlet wet bulb temperature. (Kg/Kg)

$\mathcal{H}$ = Humidity of the inlet gas. (Kg/Kg).

Calculations

1. Draw graph of moisture contents vs. time.
2. From the above curve, draw graph of drying rate vs. moisture contents and locate the critical Points.
3. Calculate the mass transfer coefficient and heat transfer coefficient.
4. Perform a mass balance to get rate of drying and compare the results with the difference in Weights

Convection Drying Raw Data Sheet

Air speed (m/s)		Weight of empty tray (g)	
Heater set point (^{0}C)		Weight of dry solid (g)	
Air temperature (^{0}C)		Weight wet solid (g)	
Relative humidity (1) of inlet air (Kg/Kg)		Weight of added water (g)	

Time (min)	Weight of sample(g) (Weight of water evaporated)	Outlet Air Temperature	
		Temperature (2)(^{0}C)	Relative humidity (2) (Kg/Kg)
0			
2			
4			
6			
8			
10			
13			
16			
19			
22			
25			
30			
35			
40			
45			
50			
55			
60			
70			
80			
90			

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