

Separation process lab manual

Branch-petroleum engineering

Class-6th semester

Sub.code-6PE4-24



List of experiment

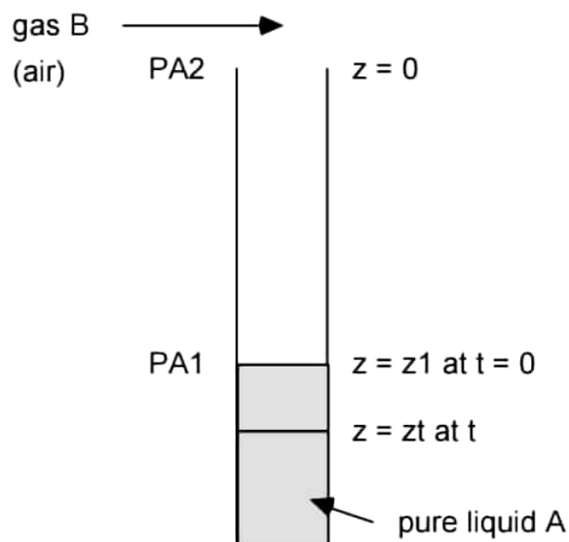
- To study diffusion coefficient of liquid vapour in air.
- To study mass transfer characteristics of a wetted wall column.
- Liquid liquid extraction in a packed column for co current and counter current flow of binary system
- To study the absorption of a gas in packed column and calculation of ntu and htu.
- Study solid-liquid extraction column.study on sieve plate distillation unit.
- Design of distillation tower
- Air fuel ratio in a gas burner.
- Pyrolysis and characterization of pyrolysis products.

Experiment No. 1**GASEOUS DIFFUSION**

We have an experiment in the lab that can be used to measure gaseous diffusion coefficients. As you run the experiment you will find that it is simple to obtain and to analyze the data.

Introduction

We will be determining the gaseous diffusivities of ethyl ether and acetone (or a similar volatile material) in air. The volatile liquid compound will be contained in a capillary tube (ask Jim Rounds for the capillary tubes and for the technique of getting the liquid into the tube). The capillary tube will be contained in a test tube immersed in a constant temperature, water bath. A stream of air, at the same water bath temperature, will pass over the end of the capillary tube in an attempt to reduce the concentration of volatile material, at that point to zero. The experimental procedure is to measure the rate of drop of the liquid level in the capillary with time.

Experimental Setup

For liquids of high volatility, A. Stefan (1879) devised a convenient means of measuring the diffusivity of their vapor through a stagnant gas. If the volatile substance A (e.g. ethyl ether or ethanol) is placed in the lower part of a vertical capillary, then liquid A will evaporate and, by the mechanism of diffusion, travel to the end of the capillary. Maintaining the mouth of the capillary at a given composition automatically establishes the concentration gradient in the capillary, and the falling rate of the meniscus in the capillary provides the rate of transport. The capillary is placed in an envelope through

which air is passed. At the meniscus the gaseous phase composition is specified by the vapor pressure of liquid A, the diffusing constituent. At the mouth of the capillary, the gaseous phase is essentially air. The gradient in the capillary is thus obtained by circulating sufficient air to reduce the substance A concentration at the mouth to a negligible quantity. The air rate should be low, constant, and not turbulent. The falling rate of the meniscus can be observed remotely with a cathetometer. The cathetometer is like a horizontal telescope with cross hairs. It can be moved up and down on a graduated, vertical rod to measure elevation. The rod must be vertical and the cathetometer must be horizontal (use the leveling screws in the base to center the bubble in the circle). It is essential that the temperature of the envelope remain constant and that the envelope not be disturbed during observations; i.e. vibrations will give erroneous data.

In the situation just described, the diffusion process is unsteady-state. Under pseudo-steady-state conditions, the diffusivity of vapor A in gas B (air) is given by

$$D_{AB} = \frac{\rho_{A,L} P_{B,lm} RT}{t P M_A (P_{A1} - P_{A2})} \left(\frac{z_t^2 - z_1^2}{2} \right)$$

where

$$P_{B,lm} = [(P - P_{A1}) - (P - P_{A2})] / \ln [(P - P_{A1}) / (P - P_{A2})]$$

P_{A1} = vapor pressure of liquid A at temperature T.

P_{A2} = partial pressure of vapor A at the mouth of the capillary.

R = gas law constant.

t = time during which the meniscus fall from z_1 to z_t .

z_1 = distance from the mouth of the capillary to the meniscus at $t = 0$.

z_t = distance from the mouth of the capillary to the meniscus at t.

P = ambient atmospheric pressure

$\rho_{A,L}$ = density of liquid A at T.

Procedure

1. Read the barometer. Measure the inside diameters of the capillary and the envelope.
2. After filling the capillary with di-ethyl ether or acetone to within 1 cm of the top, gently lower the capillary into the envelope and submerge the envelope into the thermostat in a vertical position.
3. Initially maintain the thermostat at a convenient (room) temperature. Set an air flow rate through the envelope at about 10 cm reading. After allowing sufficient time for the envelope and its contents to come to the thermostat temperature, mark the position Y_0 of the mouth of the capillary and the position Y_1 of the meniscus with the cathetometer as an arbitrary zero time ($z = z_1 = Y_0 - Y_1$, at $t = 0$).
4. **The air temperature within the envelope must be recorded. This should be the temperature where the diffusivity is measured.**
5. Then follow the fall of the meniscus as a function of time. Remember the precautions about use of the cathetometer. Take three or more readings ($z = z_t = Y_0 - Y_t$ at $t = t$).

Plot your data as you go to evaluate its validity. From the equation for D_{AB} , you see that a graph of $(z_1^2 - z_2^2)$ versus t will be a straight line through the origin. So your second reading will show whether your experimental data conform to a straight line or not. If not, you must take additional readings in the hopes that at least three of the later data points fall on a straight line. When the run is complete, raise the bath temperature to the next level.

6. Repeat the run (steps 2, 3, 4, 5 above) at a higher temperature of about 80-82°F. As the temperature rises the runs can be made more quickly. Make sure that bubbles of vapor do not form in the liquid phase while a run is being made. This indicates that you are exceeding the boiling point of the ether or acetone.
7. Make another run (steps 2, 3, 4, 5, above) at the same temperature of 80-82°F but use a different air flow rate. If the slope of the line representing the lab data is not the same as for (6) above, stop and investigate; especially the flow rate of the air stream.

Note: Experimental technique is very important in this somewhat crude but educational experiment. The following are critical for obtaining good data:

- a. Spend some time in the determining the relative position of the test tube in the water bath and the relative position of the cathetometer, so that you can sight clearly to make good measurements. You should practice making some height readings while getting ready for your first run. Note that there is a vernier scale on the cathetometer so that you can read height to the nearest 0.1 mm.
- b. The test tube should be as fully immersed in the water bath as possible (in the interest of having the contents as close to the water bath temperature as possible)
- c. A thermometer should be placed in the test tube as close to the evaporating liquid as possible.
- d. The water bath stirrer should run as fast as possible (for a constant bath temperature) but slow enough so that vibrations do not interfere with the cathetometer height readings.

Note: The volumetric air flow rate through the flow meter can be estimated from the equation

$$V = 123.6 H^* P/T$$

where V = volumetric flow rate in ft³/hr at T and P

H = flow meter reading in cm

T = ambient air temperature (°R)

P = ambient air pressure (psia)

Analysis

1. Determine the diffusivity from the slope of the least square line of $(z_1^2 - z_2^2)$ vs. t .

Note: You should use the liquid density at the temperature of the system. The specific volume V_s at any temperature can be estimated from the Rackett equation if one experimental volume V_s^R is available at a reference temperature T^R .

$$V_s = V_s^R (Z_{RA})^\alpha$$

where $\alpha = (1 - T_r)^{2/7} - (1 - T_r^R)^{2/7}$, $T_r = \text{reduced temperature} = T/T_{\text{critical}}$
 $Z_{RA} = 0.29056 - 0.08775\omega$, $\omega = \text{acentric factor}$

2. Determine Reynolds number for the flow inside the envelope.
3. Estimate the mole fraction of the volatile species at the mouth of the capillary.

WETTED-WALL COLUMN

(Evaluation of Mass Transfer Co-efficient)

1. OBJECTIVE:

To study the evaluation of mass transfer coefficient in wetted wall column.

2. AIM:

Determination of the Liquid phase mass transfer coefficient in a wetted wall column using Air-water system.

3. INTRODUCTION:

A wetted wall column is essentially a vertical tube with means of admitting liquid at the top and causing it to flow downwards along the inside wall of the tube, under the influence of gravity and means of admitting gas to the inside of the tube, where it flows through the tower in contact with the liquid. In absence of ripple formation at the liquid surface, the interfacial area, A , is known and form drag is absent.

4. THEORY:

The mass transfer coefficient, K_a , can be measured in a wetted wall column in which the area of contact between two phases is known and boundary layer separation does not take place. A wetted wall column also provides useful information on mass transfer to and from fluids in turbulent flow.

Various dimensionless groups that control the phenomenon are:

$$\text{Reynolds number, } NR_c = \frac{dG}{\mu}$$

$$\text{Schmidt Number, } NS_s = \frac{\mu}{\rho D}$$

$$\text{Sherwood Number } NS_h = \frac{K d}{D}$$

G = Gas mass velocity, $\text{kg/m}^2 \text{ s}$

D = diffusivity Coeff., m^2/s

d = tube diameter, m

K_a = mass transfer coeff.

For turbulent flow mass transfer to pipe wall-involving evaporation of liquids in wetted wall towers, Gilliland and Sherwood proposed the correlation:

$$K_G = \left(\frac{D\pi}{Pd} \right) (0.023) \left(\frac{dV\rho}{\mu} \right)^{0.81} \left(\frac{\mu}{\rho \lambda l} \right)^{0.44}$$

For $N_{Re} \Rightarrow 2000 - 3500$

$N_{Sc} \Rightarrow 0.6 - 2.5$

$P \Rightarrow 0.1 \text{ to } 3 \text{ atm}$

The known flow rate of air at a measured humidity is brought into contact with a film of water at a certain temperature and vapor pressure. Moisture is absorbed by the air from water film and the resultant humidity of the exit air and the temperature and vapor pressure of the entry water measured.

5. DESCRIPTION:

In this system the air is firstly heated by passing through a heated chamber and then directed to the column from bottom. Water pumped in from the top and flows down in the form of a thin film. The moisture is absorbed from the wetted wall as the air travels up through the column and then discharged to the atmosphere. Water is recycled in the sump tank through the pump. The temperature of the air dry bulb and wet bulb are measured by RTD 100 are provided.

6. UTILITIES REQUIRED:

1. Electrical supply: Single Phase, 220 V AC, 50 Hz, 0.5 kW with earth connection.
2. Water Supply
3. Drain required.
4. Compressed Air Supply at 2 Bar, 4 CFM.

7. EXPERIMENTAL PROCEDURE:

1. Feed Water to the column from top at a rate at which complete wetting with minimum of ripple formation takes place.
2. Commence the operation with minimum airflow and after 15-20 minutes, the humidity of the inlet air and the outlet air at this flow rate of air is noted and read corresponding vapor partial pressure, from the psychrometric chart. Simultaneously the temperatures of the water in and out are noted and flow rates

measured. From steam tables obtain Vapour pressure of water corresponding to these temperatures.

3. Repeat step 3 for 3-4 airflow rates.
4. Calculate experimental value of K_G by using humidity chart data.
5. Calculate predicted value of K_G using Sherwood-Gilliland correlation, eq. (3).
6. Compare the experimental and predicted Value of K_G

8. OBSERVATION & CALCULATION:

DATA:

d = ----- cm
 L = ----- cm
 P = ----- atm
 W = -----
 ρ = ----- kg/m³

From Psychrometric chart

a = -----
 b = ----- (from data book)

From steam tables, corresponding to water inlet and outlet temperature, calculate the Vapour pressure.

P_1 = ----- bar
 P_2 = ----- bar

OBSERVATION TABLE:

S.no	F. LPM	$T_1, ^\circ\text{C}$	$T_2, ^\circ\text{C}$	BOTTOM SECTION		TOP SECTION	
				$T_3, ^\circ\text{C}$	$T_4, ^\circ\text{C}$	$T_5, ^\circ\text{C}$	$T_6, ^\circ\text{C}$
1							
2							
3							

CALCULATIONS:

ACTUAL VALUE OF K_G ,

$$y_1 = \frac{a/18}{[(a/18)+(1/29)]} = \text{-----}$$

$$y_2 = \frac{(b/18)}{[(b/18)+(1/29)]} = \text{-----}$$

$$(P)_{Av} = \frac{P_1 + P_2}{2}, \text{ bar} = \text{----- bar}$$

$$y_i = \frac{(P)_{Av}}{P} = \text{-----}$$

$$A = \pi d L, \text{ cm}^2 = \text{----- cm}^2$$

$$M = F \times \rho, \text{ kg/hr} = \text{----- kg/hr}$$

$$V = \frac{M}{W}, \text{ g mole/hr} = \text{----- g mole/hr}$$

$$\frac{K_G P A}{V} = \frac{1}{1 - y_i} \ln \left[\left(\frac{y_i - y_2}{y_i - y_1} \right) \left(\frac{1 - y_1}{1 - y_2} \right) \right] = \text{-----}$$

$$K_G = \text{----- g mole/h cm}^2 \text{ atm}$$

THEORETICAL VALUE OF K_G

$$K_G = \left(\frac{D\pi}{Pd} \right) (0.023) \left(\frac{dV\rho}{\mu} \right)^{0.81} \left(\frac{\mu}{\rho D} \right)^{0.44} = \text{----- g mole/h cm}^2 \text{ atm}$$

Then Compare the actual & theoretical value of K_G

9. NOMENCLATURE:

- A = Area, cm^2
- a, b = Inlet & Outlet humidity of air
- D = Diffusivity coefficient
- d = Column diameter, cm
- F = Flow rate of air, m^3/h
- K_G = Mass transfer coefficient, $\text{g mole/h cm}^2 \text{ atm}$
- L = Column effective length, cm
- M = Mass flow rate, kg/hr

P	=	Atmospheric pressure, atm
P_1, P_2	=	Vapor pressure of water at inlet & outlet temperature, bar
$(P)_{AV}$	=	Average vapor pressure, bar
T_1	=	Water inlet temperature, °C
T_2	=	Water outlet temperature, °C
T_3	=	Dry bulb temperature of inlet air, °C
T_4	=	Wet bulb temperature of inlet air, °C
T_5	=	Dry bulb temperature of outlet air, °C
T_6	=	Wet bulb temperature of outlet air, °C
V	=	Mass velocity of air, g mole/hr
W	=	Molecular weight of air
y_1, y_2	=	Mole fraction of water
ρ	=	Density of air, kg/m ³
μ	=	Viscosity of air, kg/m h

10. PRECAUTIONS & MAINTENANCE INSTRUCTIONS:

1. For condenser the cold water supply should be constant.
2. Don't switch on the pump at low voltage.
3. Air should be moisture free.
4. Wet bulb temperature should be properly contacted with water.

11. TROUBLESHOOTING:

1. If any type of suspended particles are come in the Rotameters. Remove the Rotameters clean the tube and fit that at its place.
2. If there is any leakage tight that part or remove that and refix that again after wrapping Teflon tape.
3. If Rotameters fluctuating more than average tight control knob of that.
4. If D.T.I display '1' on display board it means sensors connection are not OK tight that.
5. If switch ON the heater but temperature can't rise but panel LED is ON it means bath heater had burned replace that.

A] Aim: To determine overall height of a transfer unit using liquid - liquid extraction column

Chemicals Required: Benzene-20Lit, Acetic Acid 2 Lit, Sodium Hydroxide - 100 ml

(For Titration), Phenolphthalein indicator.

Procedure:

- 1) Prepare 1 mol of Acetic Acid by adding: 57ml of acetic acid / Lit of Benzene.
- 2) The extraction column is filled with Benzene- Acetic Acid mixture of 1 to 2 kmole / m³.
- 3) The rot meter reading for both phases is adjusted to a prefixed value. The recommended range of velocity is 1 to 4 x 10⁻² m/s.
- 4) The outflow from the column is adjusted in such a way that the liquid -liquid interface is maintained at a suitable height .The interface is maintained at the same height while taking all further observations.
- 5) The Raffinate flow and extract flow are controlled through the Extract rot meter by opening the valve.
- 6) From above either Raffinate or Extract can be controlled at a time through Rota meter while conducting the Experiment.
- 7) The first observation should be taken after two times the mean residence time of dispersed and continuous phase (depending on which is larger).The following measurements are done after allowing sufficient time for steady state to be attained i.e. roughly 600 sec.
- 8) At steady state conditions note the following :
 - i) Flow rate of feed, water, and extract.
 - ii) Density of feed, water, extract and Raffinate.
- 9) Titrate the feed, extract and Raffinate samples using standard NaOH solution.

Observations:

- 1) Flow rate of feed=
- 2) Flow rate of water =
- 3) Flow rate of extract =
- 4) Density of feed=
- 5) Density of water =
- 6) Density of extract =
- 7) Density of Raffinate =

Observation Table:

Sr. No.	Vol. of Sample	Volume of NaOH required
1		
2		
3		

Calculations:

- 1) To find $Y_2 = \text{kg acetic acid} / \text{kg H}_2\text{O}$

$C_2 = \text{Concentration of acetic acid in extract, kmole} / \text{m}^3$.

$P_2 = \text{density of extract, kg} / \text{m}^3$

$Y_2 = \text{Concentration of acetic acid in the extract expressed as kg acetic acid} / \text{kg}$

$$\text{H}_2\text{O} = 60 C_2 / P_2 - 60 C_2$$

- 2) To find $X_2 = \text{kg acetic acid} / \text{kg Benzene}$

$C_2 = \text{Concentration of acetic acid in extract, kmole} / \text{m}^3$.

$P_2 = \text{density of feed, kg} / \text{m}^3$

$X_2 = \text{Concentration of acetic acid in the feed expressed as kg acetic acid} / \text{kg}$

$$\text{Benzene} = 60 C_2 / P_2 - 60 C_2$$

- 3) To find $X_2 = \text{kg acetic acid} / \text{kg Benzene}$

$C_2 = \text{Concentration of acetic acid in extract, kmole} / \text{m}^3$.

$P_2 = \text{density of extract, kg} / \text{m}^3$

$X_2 = \text{Concentration of acetic acid in the extract expressed as kg acetic acid} / \text{kg}$

$$\text{Benzene} = 60 C_2 / P_2 - 60 C_2$$

- 4) $Y_0 = \text{Concentration of acetic acid in the water kg acetic acid} / \text{kg H}_2\text{O}$.

- 5) $F_2 = \text{Mass velocity acetic acid free benzene kg} / \text{m}^2\text{s}$.

$F_1 = \text{Mass velocity acetic acid in water kg} / \text{m}^2\text{s}$.

$$F_2 = V_R P_2 (1 - X_2) / \text{C.S. area}$$

Where

$V_R = \text{Flow rate of Raffinate, m}^3 / \text{s}$

$X_2 = X_2 \text{ kg acetic acid} / 1 + X_2 \text{ kg Raffinate}$

$$F_1 = V_E P_1 (1 - X_1) / \text{C.S. area}$$

Where

$V_E = \text{Flow rate of Extract, m}^3 / \text{s}$

$X_1 = X_1 \text{ kg acetic acid} / 1 + X_1 \text{ kg extract}$

- 6) $Y_1 = F_2 / F_1 \cdot X_2$

7) $(N_{Ox}) = dX / X - X^*$

8) $(N_{Ox}) = dY / Y^* - Y$

9) $(H_{Ox}) = \text{Overall height of a transfer unit (extract phase)}$
 $= Z / (N_{Ox})$

10) $(H_{Ox}) = \text{Overall height of a transfer unit (Raffinate phase)}$
 $= Z / (N_{Ox})$

11) $K_{Ox} = \text{Overall volumetric mass transfer coefficient (Raffinate phase) in}$
 $\text{Kg Benzene} / \text{m}^3$

$= F_x / H_{Ox}$

12) $K_{Ox} = \text{Overall volumetric mass transfer coefficient (extract phase) in}$
 $\text{kg water} / \text{m}^3$

$= F_x / H_{Ox}$

Results:

- 1) Overall height of a transfer unit (extract phase)
- 2) Overall height of a transfer unit (Raffinate phase)
- 3) Overall volumetric mass transfer coefficient (Raffinate phase) in $\text{kg Benzene} / \text{m}^3$
- 4) Overall volumetric mass transfer coefficient (extract phase) in
 $\text{kg water} / \text{m}^3$

A) Aim: To find out rate of absorption of CO₂ in a packed column.

Chemicals:

- 1) NaOH- 1 to 2 N
- 2) HCl standard
- 3) BaCl₂ solution 25% [w/w]
- 4) CO₂ gas
- 5) Phenolphthalein
- 6) Methyl orange

Apparatus/ glassware: Volumetric flask, Burette, Conical flask, measuring cylinder

Theory:

Procedure:

- 1) Feed preparation: 20 lit of approx 1N NaOH solution is prepared and transferred to the supply tank. 50 ml of BaCl₂ of 25% w/w (25% BaCl₂ +75% water) solution is prepared.
- 2) The flow rates of the gas and liquid phases are adjusted to predefined values. The CO₂ flow rate should be such that % of CO₂ in the air -CO₂ mixture is about 8-10%.
- 3) The system is allowed to attain steady state after fixing the liquid level in the bottom section at any height. This level is adjusted by changing valve opening at bottom. The time steady state should be 3-4 times mean residence time of the liquid phase in the column. During this period the liquid level in the bottom section must be maintained at the marked height. Inlet and outlet liquid samples are withdrawn for analysis after steady state is reached.
- 4) A known volume of sample is titrated against std. HCl using methyl orange indicator; end point is yellowish to reddish.
- 5) To another volume of the sample is added a 25% [w/w] solution of BaCl₂ to ppt the Na₂CO₃. Go on adding BaCl₂ solution in the sample till precipitation occurs, add some excess amount. This solution is then titrated against std. HCl solution with phenolphthalein indicator.
- 6) The experiment can be repeated for different flow rates.

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Observation:

- 1) Height of column = 750 mm
- 2) Diameter of tower = 40 mm
- 3) Flow rate of NaOH = _____LPH
- 4) Flow rate of CO₂ = _____LPH
- 5) Flow rate of Air = _____LPH
- 6) Concentration of NaOH (i) = _____ kmole/m³
Concentration of NaOH (i) = $x_1 \cdot \text{Eq. Wt of NaOH} / \text{Mol wt of NaOH}$
- 7) Concentration of NaOH (O) = _____ kmole/m³
- 8) Normality of NaOH = $T_1 \cdot \text{Concentration of HCl} / \text{Volume of sample} = x_2$
- 9) Concentration of Na₂CO₃ in outlet is $c (\text{Na}_2\text{CO}_3 \text{ (O)}) = 0.5 [c (\text{NaOH (I)}) - c (\text{NaOH (O)})]$
- 10) Rate of absorption of CO₂ = $R (\text{CO}_2) = 0.5 [c (\text{NaOH (I)}) - c (\text{NaOH (O)})]$

Scanned with CamScanner

11) Rate of absorption of CO₂ / Unit volume of packed tower = R (CO₂) / V

12) Volume of CO₂ absorbed

$$R_v = (R_{A_0}) \text{ CO}_2 \cdot V \cdot \text{Molar volume at experimental conditions} \\ = \quad \text{m}^3/\text{sec}$$

13) CO₂ flow rate at outlet

$$= \text{inlet flow rate of CO}_2 - R_v = X \text{ m}^3/\text{sec}$$

$$= X \text{ m}^3/\text{sec} / \text{molar volume m}^3/\text{Kmol}$$

$$= \text{Kmol/sec}$$

$$\text{at NTP } V = 22.4136 \text{ m}^3/\text{Kmol}$$

14) CO₂ flow rate at inlet

$$= \text{flow rate at inlet m}^3/\text{sec} / \text{molar volume m}^3/\text{Kmol}$$

$$= \text{Kmol/sec}$$

15) Air flow rate at the inlet

$$= \text{flow rate at inlet m}^3/\text{sec} / \text{molar volume m}^3/\text{Kmol}$$

$$= \text{Kmol/sec}$$

16) Mole fraction of CO₂ entering with gas = y₁

$$= \frac{\text{flow rate of CO}_2 \text{ Kmole/sec}}{\text{Flow rate of CO}_2 \text{ Kmole/sec} + \text{flow rate of air Kmole/sec}}$$

$$\text{Flow rate of CO}_2 \text{ Kmole/sec} + \text{flow rate of air Kmole/sec}$$

17) Mole fraction of CO₂ at outlet = y₂

$$= \frac{\text{flow rate of CO}_2 \text{ Kmole/sec}}{\text{Flow rate of CO}_2 \text{ Kmole/sec} + \text{flow rate of air Kmole/sec}}$$

$$\text{Flow rate of CO}_2 \text{ Kmole/sec} + \text{flow rate of air Kmole/sec}$$

18) $(y - y_e)_m$ = The logarithmic average of the conc. Differences at the end of tower

$$(y - y_e)_m = \frac{y_1 - y_2}{\ln(y_1 / y_2)}$$

$$\ln(y_1 / y_2)$$

19) Overall gas transfer coefficient

$$G_m = \frac{\text{Flow rate of CO}_2 \text{ Kmole/sec} + \text{flow rate of air Kmole/sec}}{\text{Cross sect. Area of column}}$$

$$\text{Cross sect. Area of column}$$

$$= \text{Kmol/sec.m}^2$$

$$P = \text{Operating pressure} = \text{Atmospheric pressure} = 101.3 \text{ KN/ m}^2$$

Simplifying above equation for K_{Ga}

$$K_{Ga} = G_m (y_1 - y_2) / P Z (y - y_e)_m$$

Result:

Conclusion:

Experiment – 5

Sieve Tray Distillation

AIM:-

To determine the overall efficiency of the sieve plate distillation column and to calculate number of plates using experimental data.

APPARATUS:-

- Sieve plate distillation column
- 87 : 13 mixture of propane-1-ol and iso-propanol
- Refractometer

THEORY:-

Distillation is a separation process in which a new phase is generated by heating the mixture. The difference in relative volatility of components is exploited in the process. In our case relative volatility of iso-propanol is more than propane-1-ol. In sieve tray distillation mixture is fed to the bottom of the column and heated through hot oil. The vapour is passed through plates and liquid is been fed from top of the column as a reflux.

LAB SETUP:-

Due to sieves and the liquid hold up over plates there is bubbling of heated vapor phase through liquid phase providing contact area for the mass transfer.

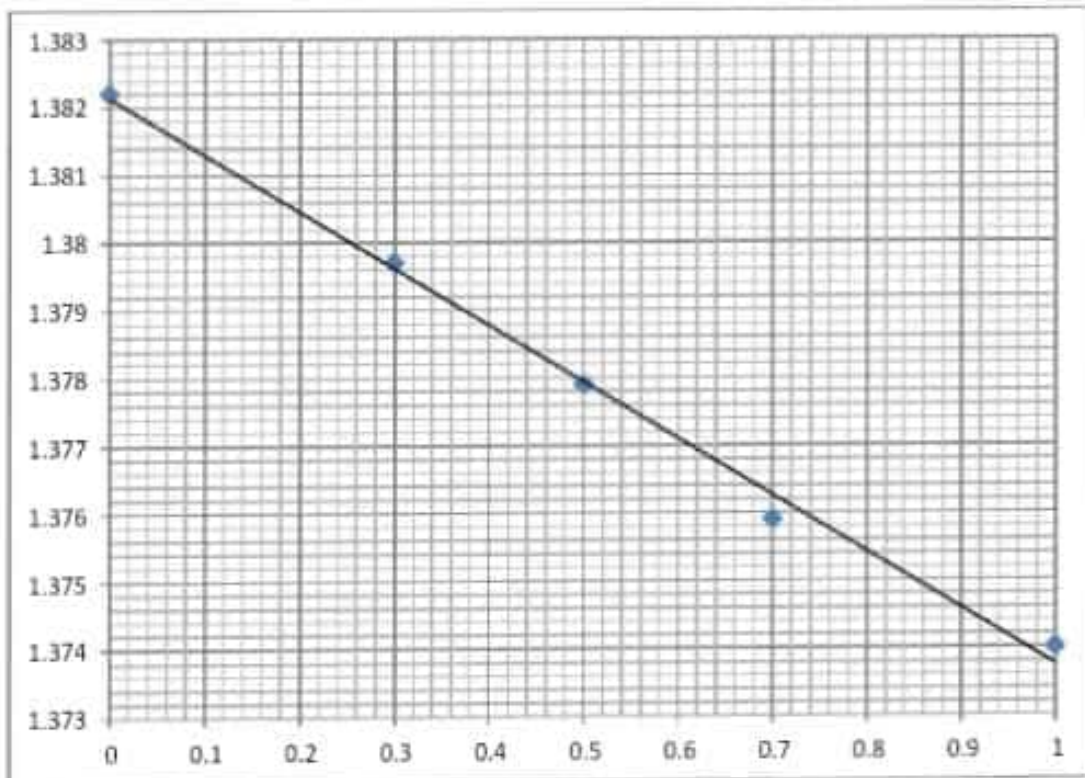
PROCEDURE:-

- Add mixture into reboiler.
- Hot oil is circulated through shell side of the reboiler which work as a heating medium.
- Start the heater.
- Vapors will flow upwards and distillate is been accumulated and after few minutes there will be a reflux coming in.
- Collect the samples of distillate and residue.
- Get the value of the refractive index by refractometer.
- When the values of refractive indexes become constant we can find number of plates using McCabe Thille method.
- Calculate the efficiency

DATA FROM THE LITERATURE:-

- Molecular weight of both A and B
Propane-1-ol = 60 gm/ mol
Iso-propanol = 60 gm/mol
- Refractive index v/s mole fraction data.
Propane-1-ol = 1.3822 D
Iso-propanol = 1.3740 D

Calibration Curve for Propane-1-ol and Iso-propanol mixture ~



Observation Table –

sample No.	RI (w)	RI (D)	X (w)	X (d)
1	1.3805	1.3783	0.190476	0.452381
2	1.3803	1.3782	0.214286	0.464286
3	1.3801	1.3777	0.238095	0.52381
4	1.3799	1.3775	0.261905	0.547619
5	1.3797	1.3771	0.285714	0.595238
6	1.3796	1.377	0.297619	0.607143

CONCLUSION AND DISCUSSION:-

- Total number of plates from McCabe thille method= 2.37
- Efficiency of the tower = $2.37/4 * 100\% = 59.25\%$
- According to experimental setup there are only 3 plates and one reboiler but there can be more number of plates. The reason of not having number of plates having the concentration set up in the critical zone of having equilibrium data is that the contribution of the number of the plates is negligible and composition difference can be easily measured by the calibration curve.
- According to the calibration curve, the RI of iso-propanol is inversely proportional to the concentration. The R.I. of the mixture depends on the components of the mixture and nature of dependence of the curve depends on the composition of the mixture.
- In the case of total reflux the diagonal line will work as an operation line.

I. INTRODUCTION

I.1. Distillation

Distillation is defined as a process in which a liquid or vapour mixture of two or more substances having different vapour pressures is separated into its component fractions of desired purity, by the application of heat [1]. When the vapours of the boiling mixture is cooled and condensed, the condensate will contain more volatile components (low boiling point). As distillation progresses, the original mixture will contain more of less volatile material (high boiling point). Depending on the concentrations of the components present, the liquid mixture will have different boiling point characteristics. Distillation occurs because of vapour pressures of the components in the liquid mixture. If relative volatility is very close to 1, they have similar vapour pressures and hence, it will be difficult to separate the two components by distillation [1]. Distillation is a unit operation capable of producing pure substance from liquid mixtures.

1.1.2 Application of Distillation

- Distillation has long been used as the separation process in chemical and petroleum industries
- Distillation technology is applied in pharmaceutical, chemical, food, and alcohol industries
- Distillation is used in refineries for fractionating crude oil to produce many chemicals
- It is used in distillery for producing alcohol

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There are many types of distillation columns, each designed to perform specific types of separations and each design differs in terms of complexity.

A few common types are given below:

- As batch and continuous processes based on how they are operated.
- As binary or multi-component distillation processes based on the number of components in the feed.
- As tray or packed distillation columns based on the type of column internals.

1.2 Packed Bed Column

Packed bed columns are used most often for absorption, liquid-liquid extraction and distillation. This experiment was conducted to study the operation of packed bed distillation column. The packing provides a large surface area for vapour - liquid contact. The feed mixture contains components of different volatilities, and enters the column approximately at the middle. The liquid flows downward through the packing, and the vapour flows upward through the column [2]. Differences in concentration cause the less-volatile components to transfer from the vapor phase to the liquid phase. After the feed mixture enters the column it flows down the column through the packing counter currently and contacts the rising vapour stream. The liquid at the bottom enters a reboiler. Two streams exit the reboiler; a vapour stream, which returns to the column, and a liquid product stream. The vapour stream flows upward through the packing, picks up the more volatile components, exits the column, and enters a condenser. After the vapour condenses, the stream enters a reflux drum, where it is split into an overhead product stream, known as the distillate, and a reflux stream that is recycled back to the column [2].

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2. MATERIALS AND METHODS

VALVE	
V1	Feed Supply Valve for Funnel
V2	Air Vent Valve of Vessel
V3	Water Flow Control Valve
V4	Air Vent Valve of Condenser
V5	Valve for Distillation Collection
V6	Drain Valve of Vessel

2.1 Starting Procedure

1. Methanol water solution is prepared by mixing known amount of water and methanol
2. The total amount of solution is 15L.
3. The volume of water and methanol was noted down
4. V_1 - V_6 valves was closed
5. All the switches on the panel were ensured to be at OFF position.
6. The water is filled in the cold water tank
7. The reboiler was filled with methanol water solution by opening the valve V_1 and air vent valve V_2
8. Valve V_1 and V_2 was closed
9. The electricity was connected to the set.
10. The process temperature was set at 90°C.
11. The heater and cooling water pump was started.
12. The cold water flow was adjusted by the rotameter valve to a moderate value.
13. The cyclic temperature was set for total reflux.
14. Wait 25-30 minute for the system to achieve steady state.
15. Samples from the bottom & distillate stream were taken out.
16. The samples were cooled to room temperature and the refractive indices were measured

2.2 Closing Procedure

1. The water supply was stopped when the experiment was over.



2. The pump, heater and the main power supply was switched off.
3. Column, reflux drum, receiving tank and water tank were drained.
4. The vessels were drained after the temperature fell down to atmospheric pressure by opening valve V_6 .

2.3 Formulae used

To calculate the moles of methanol in feed

$$\frac{M}{F\rho V} \quad (1)$$

To calculate the moles of methanol in residue

$$B \frac{x_F x_D}{x_B x_D} \times F \quad (2)$$

$$\frac{F x_f}{B x_B} \quad (3)$$

$$a \ln \frac{F 1 x_f}{B 1 x_B} \quad (4)$$

To calculate the error

$$E = \frac{F x_f}{B x_B} - a \ln \frac{F 1 x_f}{B 1 x_B} \quad (5)$$

To calculate minimum number of plates

$$N = \frac{x_D 1 x_B}{x_B 1 x_D} - 1 \quad (6)$$

Figure 1 shows the block diagram of packed bed distillation column. The set-up consists of packed column, which is packed with glass rasching rings. An electrically heated reboiler is installed at the

bottom of the column. A tank is provided to collect the bottom product. Shell and tube type condenser is provided to condensate the vapour. A tank with pump and rotameter is provided for circulation of cooling water. Reflux drum is provided to collect the condensate. Receiving tank is for collecting the distillate. Drain valves are provided for drainage purpose.

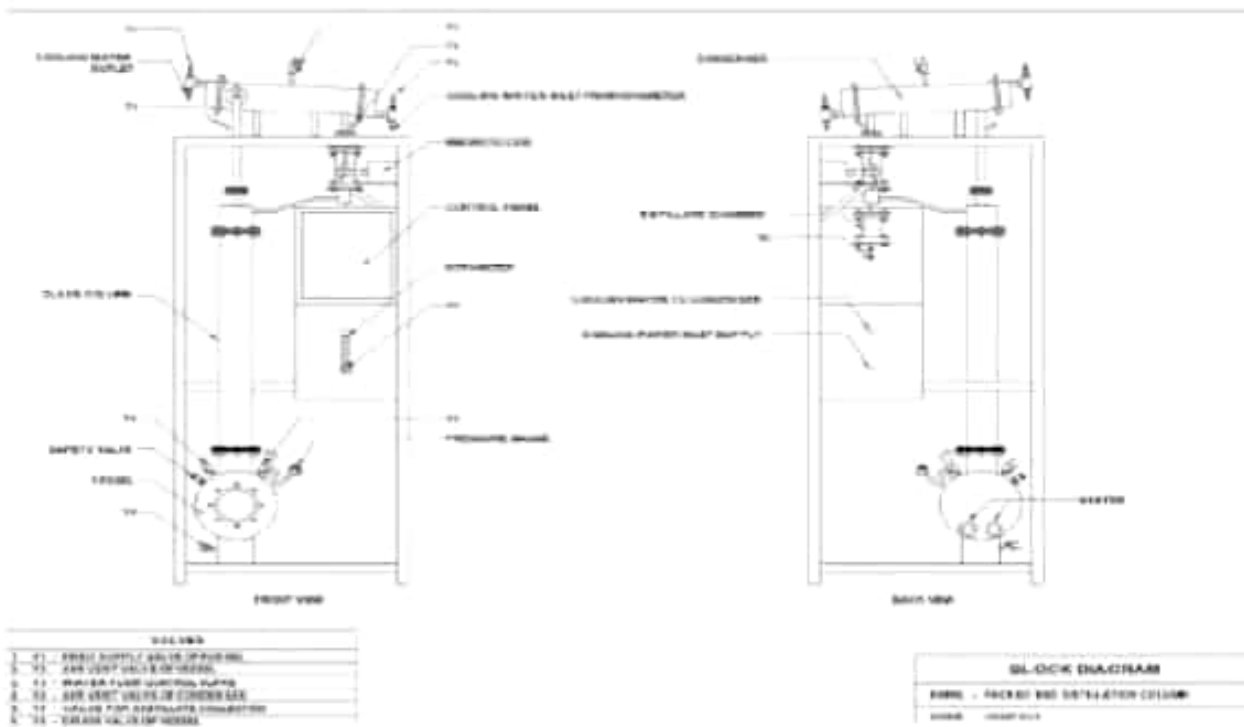


Figure 1: Block Diagram of Packed Bed Distillation Column

3. RESULTS AND DISCUSSION

From the graph shown in Figure 2 as shown in Appendix, the mole fraction of methanol in feed, F was determined to be 0.38 moles. The mole fraction of methanol of residue B was determined to be 0.25 moles. The mole fraction of methanol of distillate D was to be 0.057. Table 1 shows the readings of refractive index of the sample taken for every 5 min interval. From table 1 it can be seen that the temperature increases as the time increase and decrease at the final time taken. The refractive index changes throughout the experiment. This error could be occurred while taking the readings in decimal places through refractometer as the difference between the consecutive readings are very small.



Table 1: Refractive index reading

Total reflux condition		
Time	T °C	R. I
5	85.1	1.3330
10	87.2	1.3348
15	89.6	1.3369
20	90.2	1.3360
25	89.3	1.327

Table 2 shows the theoretical and experimental values of mole fraction of methanol in liquid phase. It can be seen that the values of mole fraction of methanol experimental are higher values than the values of mole fraction in theoretical. This could be due to some errors during the experiment. The error could be while calculating the readings with decimal places to solve the following required variables.

Table 2: Theoretical and Experimental values of mole fraction of methanol in liquid phase

Time	X	X_{the}
10	0.0544	0.225
20	0.0558	0.320
30	0.1856	0.662
40	0.1281	0.444
50	0.0369	0.156
60	0.0538	0.215

4. CONCLUSIONS AND RECOMMENDATION

The experiment was carried out to study the process of packed bed distillation column. The experiment was conducted under total reflux condition. The error that was calculated is 2.61. The value of minimum plates, N_m that was calculated is 0.84. There is little recommendation that should be considered for this experiment. More readings should be taken to see the steady state of the distillation process. A proper source of light should be provided while taking the readings of refractive index through the refractometer.

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Experimental Capabilities

- Familiarisation of the adjustment and operation of a commercial oil or gas burner.
- Assessment of a burner, including:
 - Firing rate
 - Turndown range
 - Flame stability
 - Flame shape
 - Flame radiation
 - Smoke emission
- Using either clean light boiler fuels, or natural gas or LPG
- The effect of air/fuel ratio on:
 - Combustion efficiency as measured by flue gas constituents and temperature.
 - Heat transfer
 - Energy balance
- Comparison of Flue Gas Analysis with theoretical predictions.
- Comparison of Oil and Gas Burners.
(With optional extra Burner)
- Comparative performance of different fuels or fuel additives.

Description

The Combustion Laboratory Unit is mounted on a frame, allowing easy access to the burner, controls and combustion chamber.

The package burner starts with an air purge, ignites its appropriate fuel (oil or gas) and automatically sets to a safe firing condition. Combustion air is provided by the integral fan, and a sensor monitors the flame, shutting the fuel valve in the event of flame failure. Air/fuel ratio and fuel firing rate can then be varied by the user.

Gas from either an LPG cylinder stored outside the building, or from local mains supply, is fed through pipes to the connections on the frame. Oil is supplied from portable tanks provisioned with the oil burner.

The flame burns within a stainless steel combustion chamber which is water cooled and of sufficient size to prevent flame impingement under normal conditions. Observation windows on the side of the chamber allow the flame to be observed.

A water cooled gas sampling probe and thermocouple probe allow conditions within the flame to be investigated

The unit allows similar locally supplied automatic package burners of up to 150kW to be operated and analysed.

Experimental Results

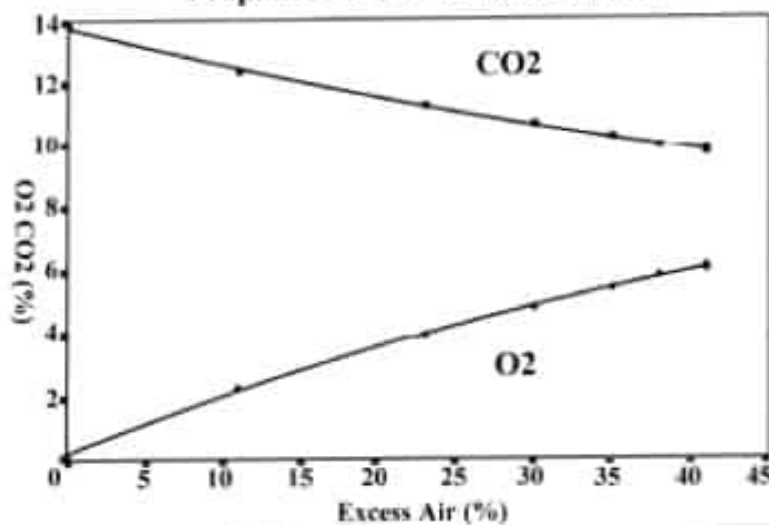
The combustion products leave the combustion chamber through a duct in the end remote from the burner, and are turned to discharge vertically upward. Due to high flue temperatures, an air break transition piece is supplied between the chamber duct and the main flue, to entrain cool air and so reduce flue temperature. A flue system is available as an optional extra.

The unit must be installed in a well ventilated area, with access to the burner end and at least one of the two sides.

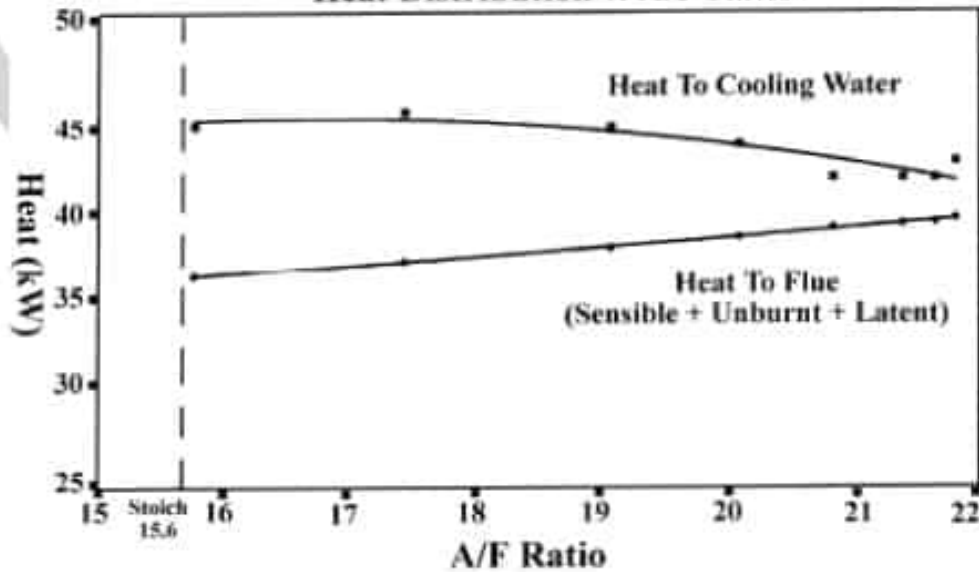
Electrical safety is provided by overload and earth leakage circuit breakers. The cooling water system has a flow switch and high temperature cut-out.

The standard instrumentation provides measurement of flue temperature, O_2 content, excess air, combustion efficiency, cooling water inlet and outlet temperatures, water and fuel flow, thereby allowing energy balances to be determined.

Propane CO_2 & O_2 v. Excess Air



Heat Distribution v. A/F Ratio



AIM-To study pyrolysis and characterization of pyrolysis products

1. Introduction

Pyrolysis of biomass is a promising method for simultaneous production of activated carbon, bio-oil and gaseous fuels and other valuable chemicals, while the almost simultaneous pyrolysis and gasification of the fuel result in formation of solid product with high surface area and well-developed porous structure (Nickolov & Mehandjiev, 1995; Mehandjiev et al., 1997). Pyrolysis is the thermal destruction of organic macromolecules in the absence of oxygen in small molecules. The destructed portion comprises a high energy content and significant organic content, which leads to the possibility of energy extraction as well as the production of activated carbon and chemicals from biomass (Prakash & Karunanithi, 2008).

Pyrolysis is discussed here to improve the valorisation of two Mexican typical agricultural wastes for energy and carbon activated production. The product characteristics, their relative proportions in the gas/liquid/solid phases and the process energy requirements depend upon the input material and the process conditions. Therefore, the goal of this chapter is to describe the conversion of waste biomass into activated carbon. Waste biomass like orange peel and pecan nut shell is converted thermally in one step. First, the biomass undergoes a pyrolysis process at 600 °C in nitrogen atmosphere. The gaseous and liquid pyrolysis products were collected as bio-oil, and then they can be used as fuel either for heating the facilities or for electricity production.

2. Experimental method for biomass pyrolysis

Bench scale experiments were carried out in a pyrolysis system with controlled temperature and a semi-batch stainless steel reactor. The schematic diagram of the process is illustrated in Figure 1. The reactor has a volume of three liters, and is externally heated by an electrical furnace. Pyrolysis experiments are normally performed with approximately 400 g of feedstock. The sample was placed inside the reactor and heated at 600 °C for one hour. The gases and vapours generated during pyrolysis pass through a condensation train, which consists of four Pyrex traps. The remaining non-condensable gases are collected and stored in a plastic sampling bag with a valve for future chromatography analysis. Pyrolysis product yields

determined by weighing the char and bio-oil. Non-condensable gases yield is calculated by the mass difference. Pyrolysis end temperatures were fixed at 600, 700 and 750 °C. All experiments were performed under nitrogen atmosphere using a flow of 60 ml/min.

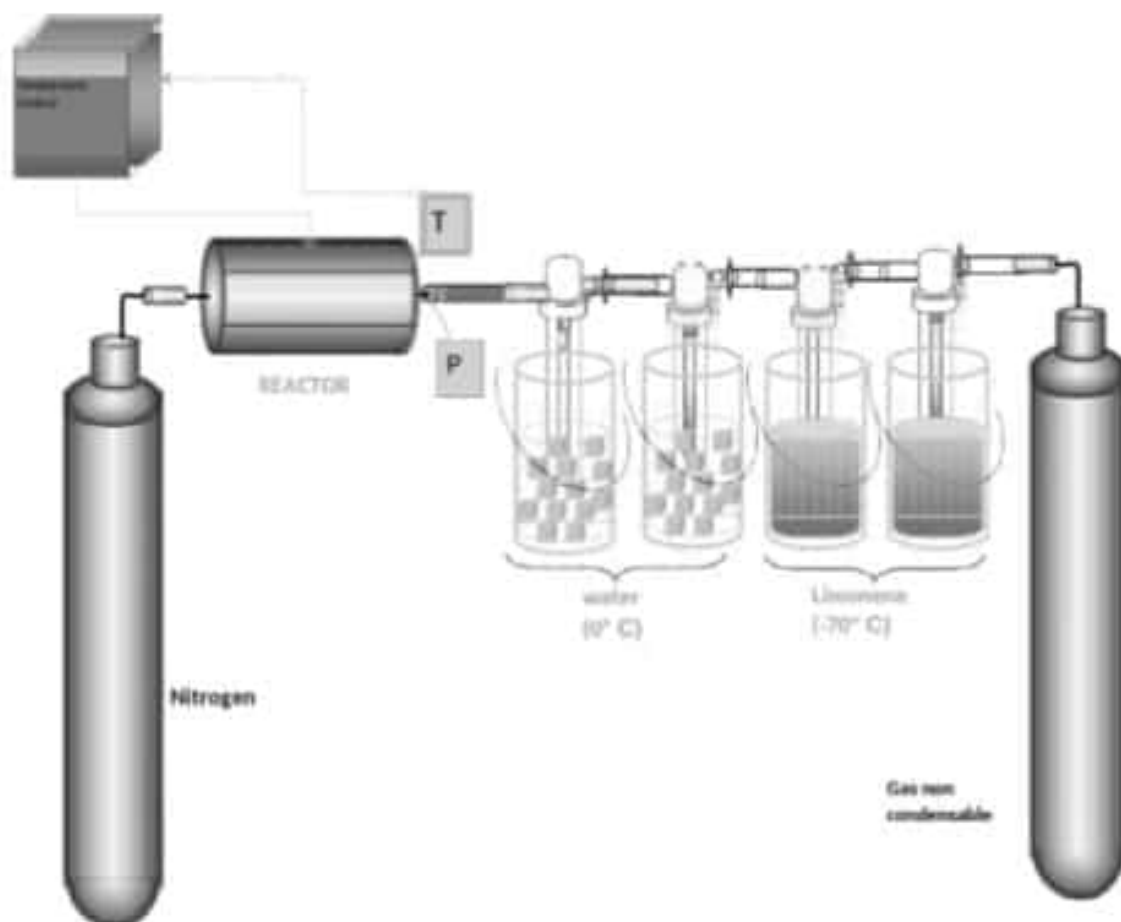


Figure. 1. The schematic diagram of the pyrolysis batch system. T—Thermocouple and P—Pressure transducer.

2.1 Raw material

Two types of biomass were used in the present study. Specifically, pecan nut shell was used as feedstock, obtained from a trading industry of nut located in the city of Torreon, Coahuila in the northeast of México. The orange peel sample was obtained from a juice processing factory in Monterrey, N.L., México. For easy storage and management, the samples were cut into small pieces of an average area of 2 cm² and dried in an air-tunnel at room temperature for the orange peel and in a stove for the case of nut shell.

The elemental analysis of the major components was carried out in a Perkin-Elmer 2400. Moisture was determined by the weight loss at 105 °C for 12 h and is expressed as a weight percentage of the dry matter. The calorific value was obtained in a calorimetric bomb from Parr (model 1341) and it can be expressed in two forms: the gross or higher heating value (HHV) and the net calorific value or lower heating value (LHV). HHV was determined using the calorimeter bomb, through the determination of the temperature difference before

and after the occurrence of the sample combustion. LHV is obtained by subtracting the latent heat of vaporization of the water vapor formed by the combustion from the gross or higher heating value.

Ash residue was obtained by thermogravimetry (ASTM D5630 method). Proximate analysis was performed using a thermogravimetric analyzer. The sample was heated under an inert atmosphere at 850 °C and the weight loss during this step is the volatile matter (VM). The gas atmosphere is then switched to air to burn off fixed carbon (FC), while the temperature is reduced to 800 °C. Finally, any residue left after the system is cooled to room temperature and is considered ash.

2.2 Product Characterization

All the collected liquid fractions were characterized by GC/MS using an Agilent Technologies 6890 GC coupled to a 5973 MS. The capillary column was a HP-1, 30 m large, 0.025 mm ID, and helium UHP as the carrier gas. A NIST library in the GC-MS chemstation is used as reference to identify the components of the bio-oil. The last solid residue from the different pyrolysis runs was analysed by elemental analysis. Figure 1 illustrates the schematic diagram of the process, as well as the places where the temperature and pressure were measured and recorded every 30 seconds by means of a data acquisition system.

3. Results of biomass pyrolysis

3.1 Biomass Properties

3.2 Yield of pyrolysis products

3.3 Characterization of bio-oils

4. Conclusion

Pecan nut shell and orange peel are excellent feedstocks for production of energy and value-added products. Biomass residues store a large amount of energy, which can be converted to several forms of usable energy through a number of commercially available processes. Pyrolysis is believed to be the reasonable choice to convert orange peel and pecan nut shell residues to liquid fuels, biochar, and activated carbons. The biomass solid waste in the form of pecan nut shell and orange peel is successfully converted into liquid, char and gas by fixed bed pyrolysis system. The heating value of the pyrolysis oil is found to be 32.98 MJ/kg for pecan nut shell and 42.70 MJ/kg for orange peel bio-oil, which is higher than other biomass-derived pyrolysis oils and also significantly higher than that obtained for the original waste. The maximum liquid yield is found to be 55.3 %, wt. and 54.5 wt% of dry biomass feedstock at the temperature range of 700-750°C and 600-750°C for orange peel and pecan nut shell, respectively. The oil from the biomass under study may be considered as an important potential source of alternative fuel. A lot of research and development process will be necessary in this topic. However, this will occur with greater economic incentive and the climate change regulation will promote research activities in this direction.