JEET BINDRA UNIT OPERATIONS & INNOVATION LAB
DEPT. OF CHEMICAL ENGINEERING, I.I.T. KANPUR

Date: 19/3/24

LAB DATA SHEET

Experiment Name: Batch distillation

ChE -	213	Name of students	Roll No.
Exp. No. -	M-4		
Group No. -	10	1. Sidharth Budania	221057
Name & Sign of T.A.		2. Satvik Pratap Singh	220981
Kartavya Maurya		3. Ujjawal Bisaria	221154
(KH Maurya)		4. Ponaem	220835
		5) Vipin kumar	211172

Objective:

a) To observe the operation of batch distillation

b) To determine the no of Theoretical plates; to obtain an overall ~~heat~~ mass Balance.

Relevant Equation(s):

$$N+1 = \frac{\log\left(\frac{x_p(1-x_s)}{x_s(1-x_p)}\right)}{\log \alpha}$$

α = relative volatility x_s = final still mole fraction/concentration

N = theoretical plates

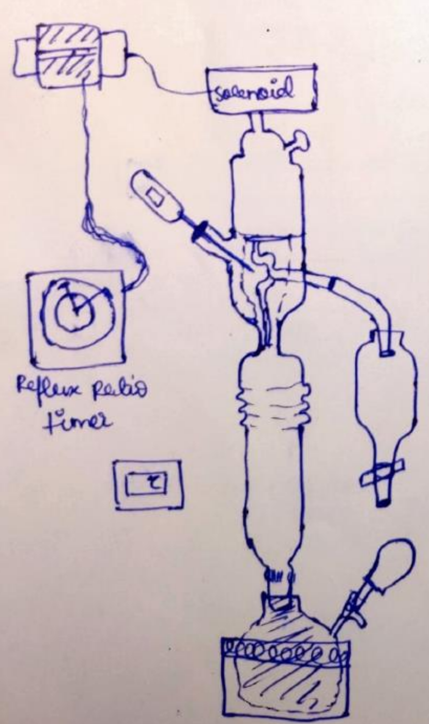
x_p = product concentration

$$\ln \frac{S_1}{S_2} = \int_{x_{S_2}}^{x_{S_1}} \frac{dx_s}{(x_p - x_s)}$$

S_1 = Initial batch charge, moles

S_2 = residue in the kettle at the end, moles

Line Diagram:



* Inferences:-

1) The temperature of bottom as well as top was somewhat constant which depict the concept of latent heat of vapourisation.

2) The temperature of system was between the boiling point of ethanol and water.

3) Reflex system was installed at the top of the column to enhance the purity of ethanol obtained in the distillate.

4) the distillate (mixture of ethanol-water obtained) when kept in open condition the concentration of ethanol must decrease because evaporation will come into effect and ethanol being more volatile would evaporate faster as compared to water.

Batch Distillation Data Sheet

Date: 19/3/24

Binary Component Mixture: ....

Reboiler **Feed Volume** = 1800ML......	Time of heating **start:**
Reboiler **Feed RI** =...1.35988	Time of heating **stop:**
Cooling water: **Inlet** temp = ... **Outlet** temp.=	Energy meter reading: **Initial :** .... KWH . **Final :** .... KWH
Cooling water **Flow Rate :**	Empty wt of sp. Gr bottle : 14.459gm
Sp. Gravity of **Water:**	Volume of SP. Gravity Bottle :

Total Reflux:

Sr. No.	Time of Interval	~~Top~~ bottom Temp	~~Bottom~~ Top Temp	RI Bottom	Sp. Gr. Top	TOP RI	(mL) Vtop	(mL) Vbott
1	30	82	77.2	1.35910	0.7733		25	11
2	40	82	76.6	1.35900	0.7723		26	12
3	50	82	77.6	1.35893	0.7769		28	13.5

Partial Reflux : Ratio = L/D = 2.6

Sr. No.	Time Interval	~~Top~~ Bottom 82 Temp	~~Bottom~~ Top Temp	RI Bottom	Sp. Gr. Top	RI TOP	Volume of distillate collected	(mL) Vtop	(mL) Vbott
1	0	[illegible]	77.6	1.35893	0.7769			28	13.5
2	10	83	78.9	1.35837	0.7771			72	18
3	20	83	79.0	1.35800	0.7772			72	13.5
4	30	83	79.1	1.35750	0.7773			60	17

Total Distillate collected		Sp. Gr. :
Total Bottom collected		RI :

Plot Calibration Curve = 1. **RI Vs Mole Fraction**

2. **Sp. Gravity Vs Mole Fraction**

KH Maurya

Equipment and Materials Required:

Distilled water– Ethanol mixture, distillation column setup, refractometer, weighing machine, stopwatch, sample bottles, gravity specific bottle and thermocouples.

Experiment Procedure:

1. Obtain the calibration curve for refractive index vs percent ethanol.

2.Charge a known amount of ethanol and water mixture in the still and start heating.

3. Operate under total reflux (keeping the timer off) until steady state is reached obtained distillate and bottoms (xp, xs) samples.

4.Take the necessary data – refractive index and specific gravity of reflux and residue, cooling water rate, reflux rate and temperature of inlet and outlet water stream in order to be able to obtained a heat balance.

5.Record the weight (or volume) distilled vs time and record the weight of the still sample.

6.Operate under a known fixed reflux ratio. Obtain samples at various times and measure total amount of distillate collected.

7. Obtain RI and Specific Gravity of both Distillate collected and RI of bottoms.

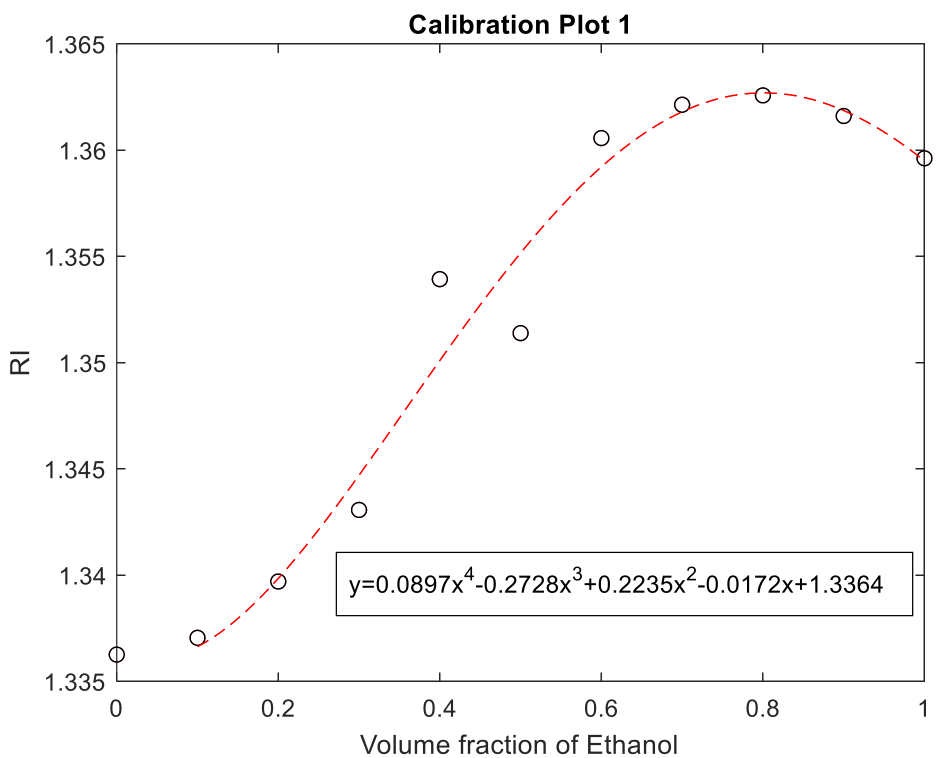
8. Record the temperature of bottoms and distillate for heat balance. .

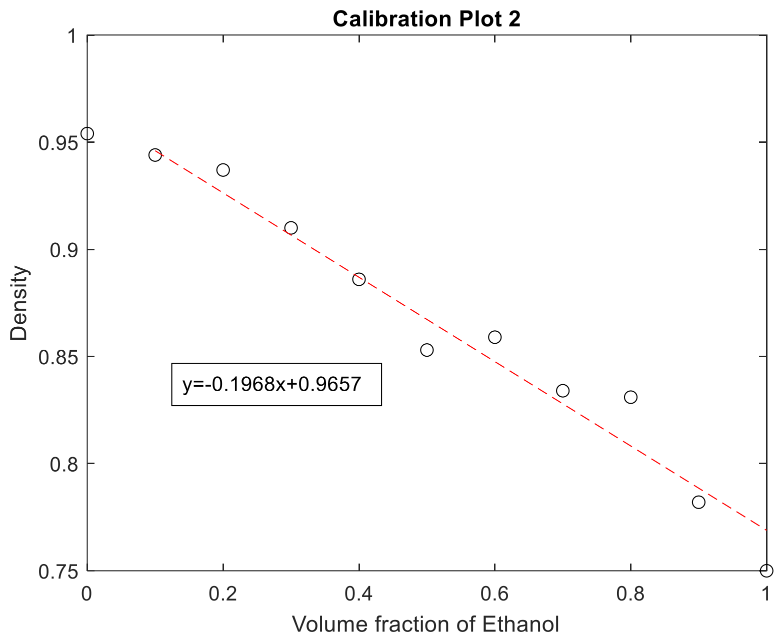
Observations and Calculations:

Calibration Chart:

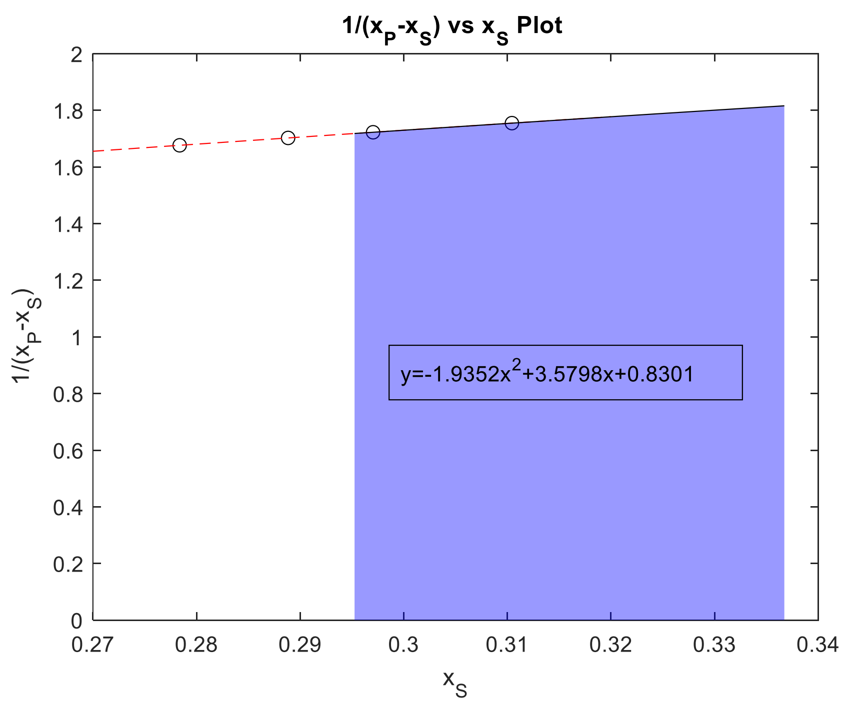
%V/V of Ethanol	RI	Density
0	1.33626	0.954
10	1.33705	0.944
20	1.3397	0.937
30	1.34307	0.91
40	1.35393	0.886
50	1.35139	0.853
60	1.36057	0.859
70	1.36214	0.834
80	1.36258	0.831
90	1.36161	0.782
100	1.35962	0.75

	Sr.NO.	Time(min)	Bot_Temp	Top_temp	RI_bot	Density_top	vf E top	vf E bot	xp	xs	1/(xp-xs)
Total	1	30	82	77.2	1.35910	0.7733					
Reflux	2	40	82	76.6	1.35900	0.7723					
	3	50	82	77.6	1.35893	0.7769	0.9593	0.591	0.8803	0.31044	
Partial	1	0	82	77.6	1.35893	0.7769	0.9593	0.591	0.8803	0.31044	1.7549
Reflux	2	10	83	78.9	1.35837	0.7771	0.9583	0.5756	0.8775	0.29704	1.7227
	3	20	83	79	1.358	0.7772	0.9578	0.5659	0.8762	0.28884	1.7026
	4	30	83	79.1	1.3575	0.7773	0.9573	0.5532	0.8748	0.27837	1.6766





Calibration Plot 2
Density
Volume fraction of Ethanol
y=-0.1968x+0.9657
1
0.95
0.9
0.85
0.8
0.75
0
0.2
0.4
0.6
0.8
1



1/(x_P-x_S) vs x_S Plot
1/(x_P-x_S)
x_S
y=-1.9352x^2+3.5798x+0.8301
2
1.8
1.6
1.4
1.2
1
0.8
0.6
0.4
0.2
0
0.27
0.28
0.29
0.3
0.31
0.32
0.33
0.34

→ **Total Reflux :-**

at 50 min : Top Temp. = 77.6°C , Bottom Temp. = 82°C

$RI_{bot} = 1.35893$, $\rho_{top} = 0.7769$ g/ml

$\Downarrow$ $\Downarrow$

From calibration plot : volume fraction of E at Bottom $= 0.591$, volume fraction of E at Top $= 0.9593$

From (Density vs volume fraction of ethanol curve) $\Rightarrow \rho_E = 0.7689$ g/ml, $\rho_W = 0.9657$ g/ml

- Antoine Eqn :- $A - \left(\frac{B}{C+T}\right)$

$$\log P^{sat} = A - \frac{B}{C+T}$$

P unit = mmHg

T unit : K

Antoine Constant

	Ethanol	Water
A	5.25	5.08
B	1598.673	1659.793
C	-46.424	-45.854

- Top Temp = 77.6°C

$$P_E^{sat} = 10^{\left(A - \frac{B}{C+T}\right)} = 0.9858 \text{ mmHg}$$

$$P_W^{sat} = 0.4295 \text{ mmHg}$$

$$\alpha_{Top} = \frac{P_E^{sat}}{P_W^{sat}} = 2.2941$$

Bottom Temp. = 82°C

$$P_E^{sat} = 1.1708$$

$$P_W^{sat} = 0.5134$$

$$\alpha_{Bottom} = \frac{P_E^{sat}}{P_W^{sat}} = 2.2805$$

$$\alpha_{avg} = \frac{\alpha_{top} + \alpha_{bot}}{2} = 2.29$$

- m.w of E = 46 g/mol

m.w. of W = 18 g/mol

Concⁿ of E at Top $= \dfrac{\rho_E \times \text{Vol. fraction top}}{MW_E}$

Concⁿ of W at Top $= \dfrac{\rho_W \times (1 - \text{vol. fraction top})}{MW_W}$

$\rightarrow x_p = \dfrac{C_E}{C_E + C_W} = 0.8803$

Similarly : concⁿ of E at bot. $= \dfrac{\rho_E \times \text{vol. fra. Bot}}{MW_E}$

concⁿ of W ,, ,, $= \dfrac{\rho_W \times (1 - \text{vol. fract. Bot})}{MW_W}$

$\rightarrow x_s = \dfrac{C_E}{C_E + C_W} = 0.31044$

- No. of Stages:

$$N_{th} + 1 = \frac{\log\left(\frac{x_p(1-x_s)}{x_s(1-x_p)}\right)}{\log \alpha_{avg.}} = 3.37$$

$N_{th} = 2.37 \approx 2$

$N_{act} = 21$

efficiency $= \eta =$ $\cancel{\frac{N_{th}}{N_{act}} \times 100 = \frac{2}{21} \times 100}$ $\Rightarrow \eta = \dfrac{N_{th}}{N_{act}} \times 100 = \dfrac{2}{21} \times 100 = 9.52\%$

⇒ Partial Reflux :

Reflux Ratio if fixed at 2.6

Experimental Residue : volume = 1340 ml , RI = 1.35829 , Density = 0.8928

F_{th} (moles) = moles of E + moles of W ∵ (Initial RI = 1.35988, volume = 1800 ml)

$= 18.65 + 36.73$

$= 55.37$ moles

$x_F = 0.3367$

$x_W = 0.2952$

• Area Under the curve of $\frac{1}{x_p - x_s}$ vs x_s

Area(A) = 0.0551

Rayleigh eqn $\ln\left(\frac{F_{th}}{W_{th}}\right) = \int_{x_W}^{x_F} \frac{dx_s}{x_p - x_s} = 0.0712$

$W_{th} = \frac{F_{th}}{e^A} = 51.57$ mol

$\%\ \text{Error} = \frac{|W_{th} - W_{exp}|}{W_{exp}} \times 100$

$= 15.63\%$

W_{exp} = moles of E + moles of W

$= 12.85 + 30.66 = 43.51$ mol

~~$F_{exp} = D + B$~~

F_{exp} (ml) = Total Distillate vol. (ml) + Total Bottom vol. (ml) + Residue vol. (ml)

$= \Sigma V + \Sigma V + 1340$

$= 284 + 85 + 1340$

$= 1709$ ml

F_{th} (ml) = 1800 ml

$\%\ \text{Error} = \frac{|F_{th} - F_{exp}|}{F_{th}} \times 100 = 5.05\%$

Results:

- The theoretical number of minimum stages (2) is significantly less than the actual number of stages (21).
- We conclude that the efficiency of the column in the case of total reflux is 9.52%.
- After the process, Wexp = 43.51 and Wtheory = 51.57moles. This leaves an error in W of = 15.63%
- Vapor loss obtained from mass balance = 5.05 %

Conclusion:

- The mixture shows an ideal behavior as its density varies linearly with volume fraction.
- Under **total reflux** conditions, the system reaches an equilibrium where both distillate and residue maintain constant composition due to the complete condensation of all vapor, which is then returned to the still. Total reflux operation is advantageous for establishing feed composition and determining the minimum theoretical plates needed for effective separation.
- In **partial reflux** operations, a portion of the vapor condenses and returns to the still, while the rest is collected as distillate. Over time, the distillate composition evolves, eventually reaching equilibrium within the system.