Continuous Distillation

Experiment IV

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Objective

The objective is to study the process of continuous distillation in a sieve plate column, for the separation of a water-ethanol mixture. The tray efficiency for the column, mass and energy balances, and heat loss should also be determined.

Apparatus Required

Equipment required: Distillation column made of glass with perforated plates, specific gravity bottle, specific gravity balance, Abbe refractometer, laboratory droppers, laboratory glassware.

The mixture to be separated is ethanol-water. The actual number of plates in the distillation column is 12. Metering pumps have been used for the feed and the reflux.

Theory
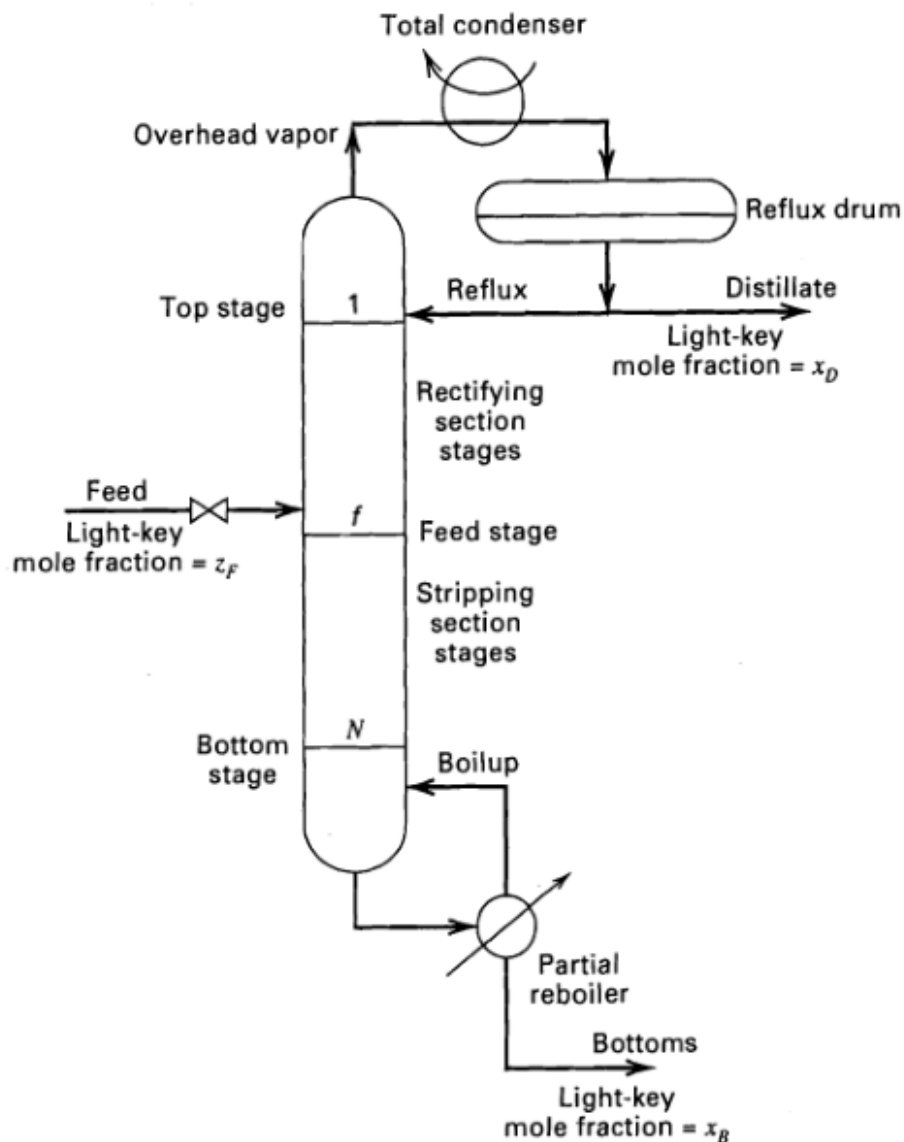
Figure 0.1: *Distillation Operation Using a Total Condenser and Partial Reboiler*

The general countercurrent-flow, multistage, binary-distillation operation consists of a column containing the equivalent of N theoretical stages; a total condenser in which the overhead vapor leaving the top stage is totally condensed to give a liquid distillate product and liquid reflux that is returned to the top stage; a partial reboiler in which liquid from the bottom stage is partially vaporized to give a liquid bottoms product and vapor boil-up that is returned to

the bottom stage, and an intermediate feed stage Seader, Henley, and Roper (2016). By means of multiple, countercurrent contacting stages arranged in a two-section cascade with reflux and boil-up, it is possible to achieve a sharp separation between the two components in the feed unless an azeotrope is formed, in which case one of the two products will approach the azeotropic composition. For the ethanol-water system, separation can only be achieved up to 95.5% ethanol.

Assumptions

In order to plot the locus of all passing streams in the rectifying section as a straight line of the form $y = mx + c$, the total molar flow rates L and V cannot vary from stage to stage. The following are the *McCabe-Thiele assumptions*[1].

1. The mixture is binary.
2. The column operates in the steady-state condition.
3. The feed stream is mixed with the feed stage fluids prior to separation.
4. Each stage reaches equilibrium.
5. The two components have equal and constant molar enthalpies of vaporization (latent heats).
6. Component sensible-enthalpy changes ($C_p \Delta T$) and heat of mixing are negligible compared to latent heat changes.
7. The column is well insulated so that heat loss is negligible.
8. The pressure is uniform throughout the column (no pressure drop).

The assumptions above lead to the condition of *constant molar overflow* in the rectifying section. Constant molar overflow refers to a molar liquid flow rate that remains constant as the liquid flows over each weir, from one stage to the next.

External Reflux Ratio

The liquid entering the top stage is the external reflux rate, L_0, and its ratio to the distillate rate, L_0/D, is the reflux ratio, R. Because of the assumption of constant molar overflow, R is a constant in the rectifying section Seader, Henley, and Roper (2016), Smith (2012):

$$R = \frac{L}{D} \tag{0.1}$$

[1]Seader, Henley, and Roper (2016),Gorak and Sorensen (2014)
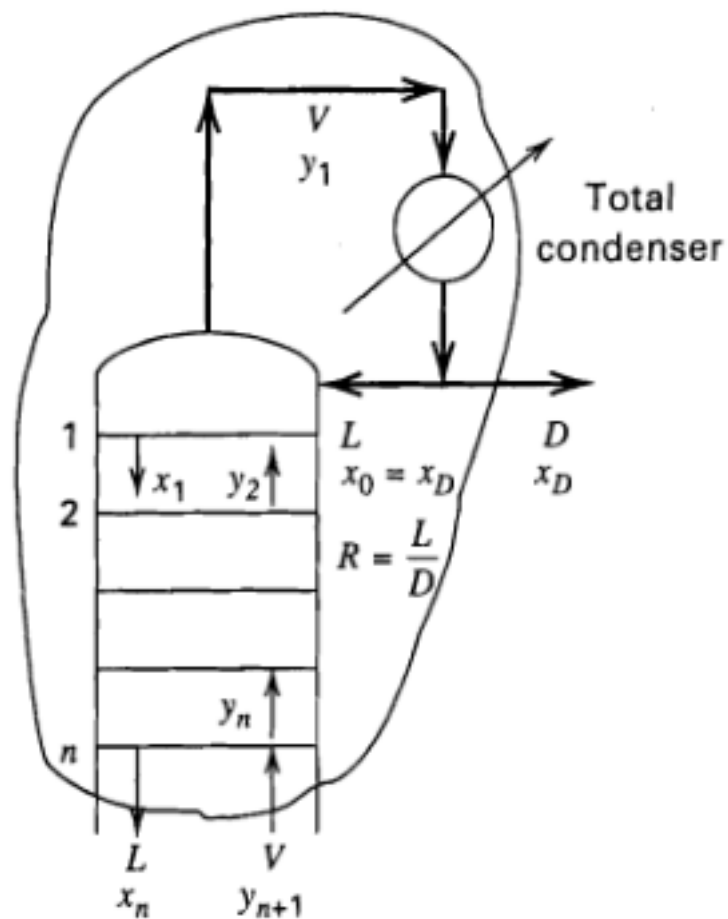
Rectifying Section

Figure 0.2: *Rectifying Section*

A material balance for the light key over the envelope shown in figure 0.2 for the total condenser and stages 1 to n is as follows, where y and x refer to vapor and liquid mole fractions, respectively, for the light key:

$$V_{n+l}y_{n+l} = L_n x_n + D x_D \tag{0.2}$$

Solving for y_{n+1}, we get:

$$y_{n+1} = \frac{L_n}{V_{n+1}} x_n + \frac{D}{V_{n+1}} x_D \tag{0.3}$$

Where we note that:

- L_0 is the external reflux rate
- D is the distillate rate
- $\frac{L_0}{D}$ is the reflux ratio, R

Additionally we know from the overall mass balance around the equipment envelope that,

$$V = L + D \tag{0.4}$$

From this, we may now note the relation between the operating line and the reflux ratio, ie. 0.5

$$\frac{L}{V} = \frac{L}{L+D} = \frac{L/D}{L/D + D/D} = \frac{R}{R+1} \tag{0.5}$$

Similarly,

$$\frac{D}{V} = \frac{D}{L+D} = \frac{1}{R+1} \tag{0.6}$$

From equations 0.4,0.5 and 0.6 we now have the most used form, that of 0.7

$$y = \left(\frac{R}{R+1} \right) x + \left(\frac{1}{R+1} \right) x_D \tag{0.7}$$

Stripping Section
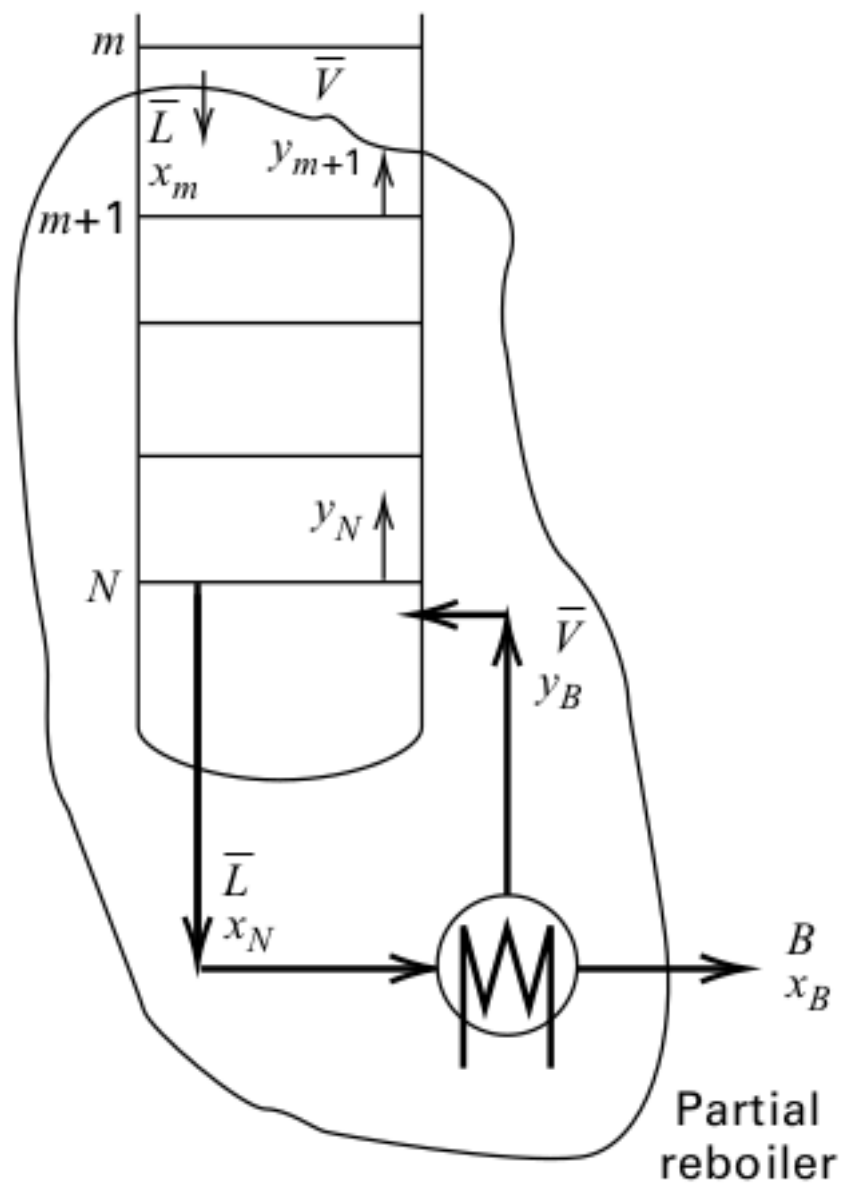
Figure 0.3: *Stripping Section*

Analogous to the develop above, we shall step through the equations of note in the stripping section, marked in 0.3. Vapor leaving the partial reboiler is assumed to be in equi- librium with the liquid bottoms product, B, making the partial reboiler an equilibrium stage. The vapor

rate leaving it is the boilup, $\bar{V}_{N+1}$, and its ratio to the bottoms product rate, $V_B = \bar{V}_{N+1}/B$ is the *boilup ratio*. With the constant-molar overflow assumption, V B is constant in the stripping section.

Since $\bar{L} = \bar{V} + B$, we have

$$\frac{\bar{L}}{\bar{V}} = \frac{\bar{V} + B}{\bar{V}} = \frac{V_B + 1}{V_B} \tag{0.8}$$

$$\frac{B}{\bar{V}} = \frac{1}{V_B} \tag{0.9}$$

Similar to the section above we now have the final equation in 0.10.

$$y = \left(\frac{V_B + 1}{V_B}\right) x - \left(\frac{1}{V_B}\right) x_B \tag{0.10}$$

Feed Conditions

The feed line equation is given by:

$$y = \frac{q}{q - 1} x - \frac{z_F}{q - 1} \tag{0.11}$$

Table 0.1: *Possible Feed Conditions and Corresponding q Values[2].*

Feed condition	f	q	q Calculation
Subcooled liquid	$f < 0$	$q > 1$	$q = [c_{P,L}(T_{bp} - T_F) + \Delta H_{vap}]/\Delta H_{vap}$
Liquid at bubble point	0	1	$q = \Delta H_{vap}/\Delta H_{vap} = 1$
Partially vaporized	$0 < f < 1$	$1 < q < 0$	$q = [(1 - f)\Delta H_{vap}]/\Delta H_{vap}$
Vapor at dew point	1	0	$q = 0$

Feed condition	f	q	q Calculation
Superheated vapor	$f > 1$	$q < 0$	$q = -[c_{P,V}(T_F - T_{dp})]/\Delta H_{vap}$

Procedure

1. The flow rate through the condenser was set at $2.5 L/min$.

2. The calibration curve for refractive index with alcohol percent was obtained. The calibration curve for specific gravity was also calculated.

3. The feed composition was determined and the reboiler was filled with it.

4. The reboiler was started and cooling water began circulation in the condenser.

5. For operating the column using total reflux, the entire distillate was returned to the column, and no secondary feed was provided. Secondary feed was provided only for the partial reflux cases.

6. The minimum reflux ratio for the feed conditions was determined, assuming 85 mole purity of the distillate.

7. The column was operated under a reflux ratio higher than minimum reflux ratio, and samples were obtained at the steady states.

8. A sample from the bottoms was collected every 10 minutes. The refractive index was measured using an Abbe refractometer. The specific gravity of the distillate was measured.

9. All the flow rates (distillate, reflux, feed, cooling water) and temperature (bottom, distillate, condenser inlet and outlet) were measured. The power

10. After the total reflux condition was achieved, a secondary feed was used to operate the column using a reflux ratio between 3 to 4.

11. The flow rates of the distillate and bottoms were adjusted so that the overall mass balance adjusted for the desired reflux ratio.

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[2]Gorak and Sorensen (2014)

12. The total distillate and bottoms were collected and measured to verify the overall mass balance.

13. The refractive index of the total bottoms and the specific gravity of the distillate was measured to get an indication of the degree of purification for a fixed reflux ratio.

    The overall electricity consumption was also measured for the entire process.

Line Diagram

Figure 0.4: *Lab Setup*

The actual lab equipment is represented in Figure 0.4.

Tables and Graphs

The observation table is depicted in image form. This has been done to preserve the authenticity of the data and the format of the report. The relevant figure is Figure 0.5.

Continuous Distillation

DATE - 30/07/2018

	FLOW RATE OF COLD WATER = 2.25 lit/min	NAME OF STUDENT
Reboiler feed volume= 1500 ml	COLD WATER IN CONDENSOR	1.
Secondary feed volume= 500 ml	INLET TEMP =	2.
Reboiler feed RI= 1.3555 and/or X_f	OUTLET TEMP =	
SG=		3.
		4.

	Initial Energy meter reading =	Kwh
	final energy meter reading =	
	Heating time start = 5301	
	Heating time end = 5909	

Secondary feed RI= 1.3535 and/or
SG =

Empty weight of SG bottle = 18·911 g
weight of SG bottle(water) = 28·341 gm

Part I - Total reflux condition:

Sr. No.	1	2	3
Time start	4:41 pm	5:11	5:21
Time end	5:11 PM	5:21	5:31
Temp of vapor=	75 °C	74	74
Cold water inlet temp =	29°C	28	28
Cold water outlet temp=	29°C	29	29
Reboiler temp =	91	91	91
Secondary feed flow rate	ON/ min.	0	0
Flow rate at the TOP section	8·5 ml/min	8·5	8·5
Distillate flow Rate =			
L(reflux)	—		
D(distillate out)			
SG of distillate	0·82	0·82	0·82
RI at the bottom	1·344	1·342	1·340

RI of total distillate = 1·36

Total volume of distillate	160 ml
SG of total vol of distillate	

Part II - Partial reflux condition:

Sr. No.	1	2	3	4
Time start	5:50 pm	6:10 pm		
Time end	6:10 pm	6:10 pm		
Temp of vapor=	74	74		
Cold water inlet temp =	28	28		
Cold water outlet temp=	29	29		
Reboiler temp=	98	114		
Secondary feed flow rate (F)	8·57 m/n.	8·57		
Distillate flow Rate = (D)				
Flow rate at the TOP	7:32 ml/min	7·32		
L(reflux)				
D(distillate out)	= 2·47 ml/m.	2·47		
SG of distillate	0·82	0·82		
RI at the bottom	1·341	1·341		
Partial ratio L/D	2·96	2·96		

reflux ratio
bottom collected after 20 min = 60+60 = 120 ml (60 ml/min)

Total volume of bottom collected = 621 ml	Total consumption of secondary feed = ml (500-330)	NAME OF TA
RI of bottom collected = 1·345		SING.OF TA

RI of total distillate = 1·36

x_D

Figure 0.5: *Observation Table*

The calibration curves from both the refractive index observations as well as the specific

Table 0.2: *Calibration Curve Data*

Volume (ml)		Moles		Mole Fraction		Refraction Index	Mass of Bottle (+Sample) (mg)	Specific Gravity
Ethanol	Water	Ethanol	Water	Ethanol	Water			
0	18	0	1	0	1	1.333	28.341	1
15	14	0.026	0.780	0.032	0.968	1.353	27.619	0.920
23	13	0.4	0.730	0.354	0.646	1.361	27.207	0.880
31	11	0.53	0.610	0.465	0.535	1.363	27.126	0.870
38	9	0.65	0.500	0.565	0.435	1.363	26.977	0.860
46	7	0.79	0.390	0.669	0.331	1.364	26.808	0.840
53	5	0.91	0.280	0.765	0.235	1.363	26.795	0.840
61	4	1.05	0.230	0.820	0.180	1.362	26.618	0.820
68	2	1.17	0.120	0.907	0.093	1.361	26.468	0.800
76	0	1.3	0	1	0	1.361	26.434	0.800

gravity ones are plotted in Figures 0.6 and 0.7. The data for both curves has been tabulated in Table 0.2.
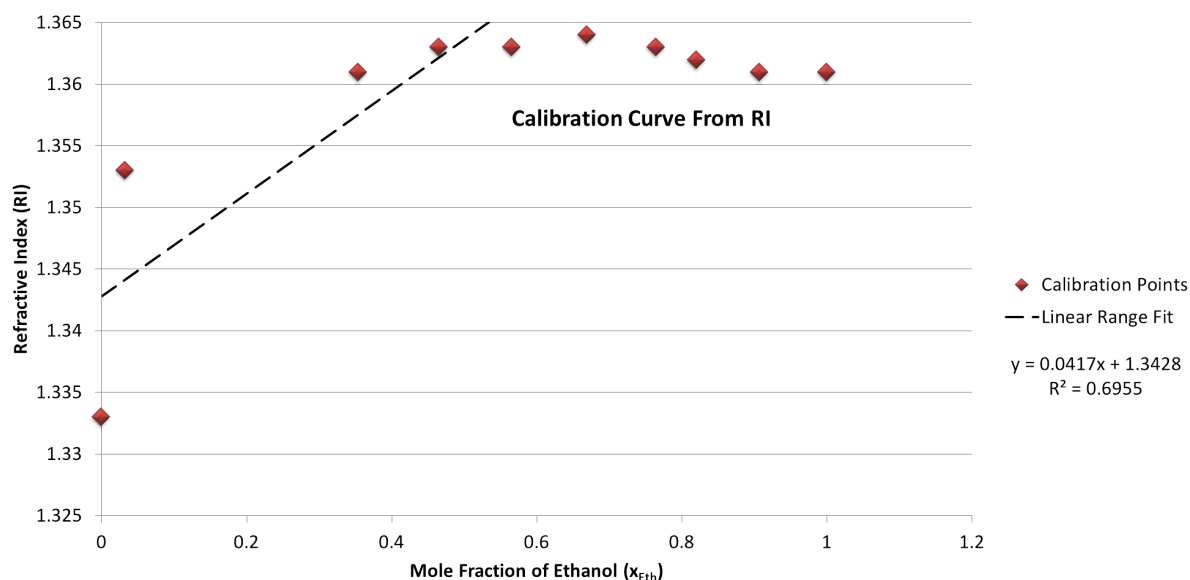

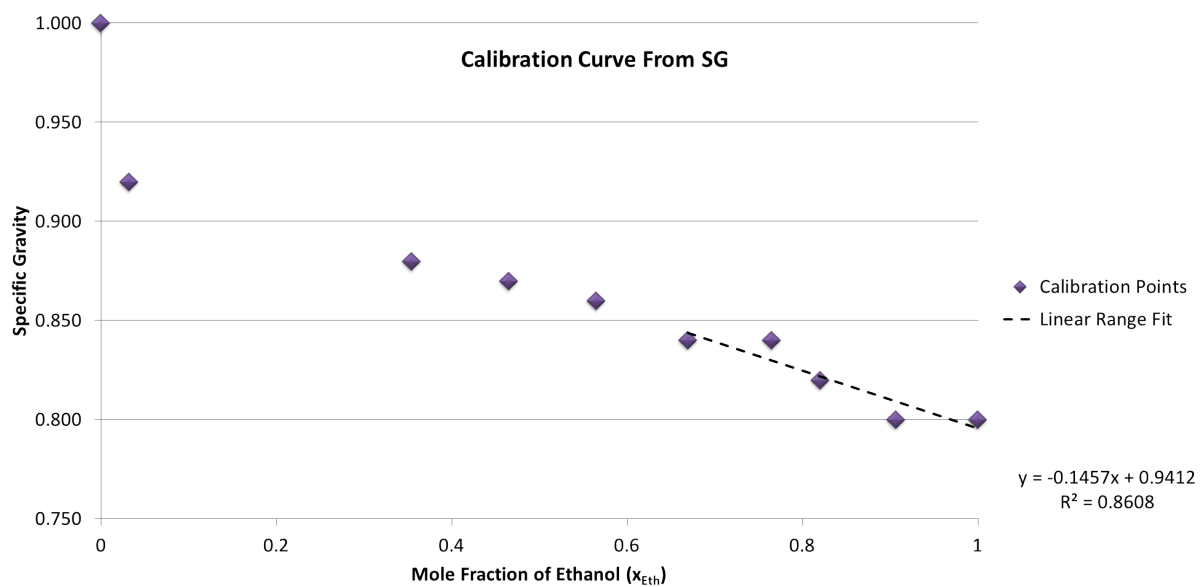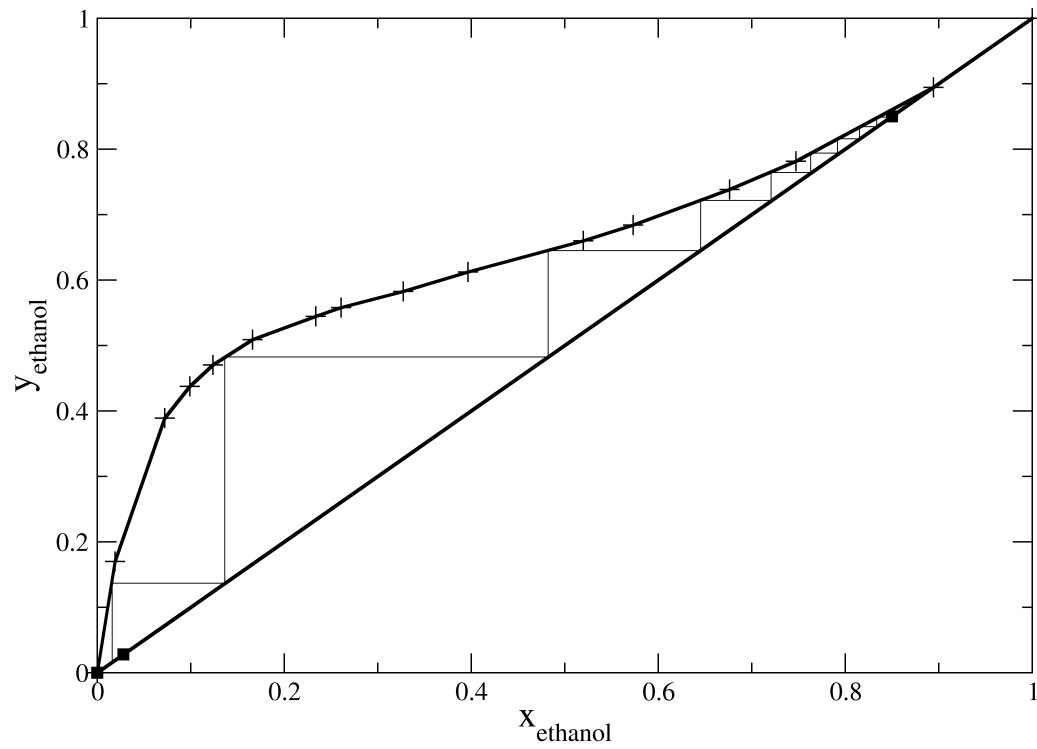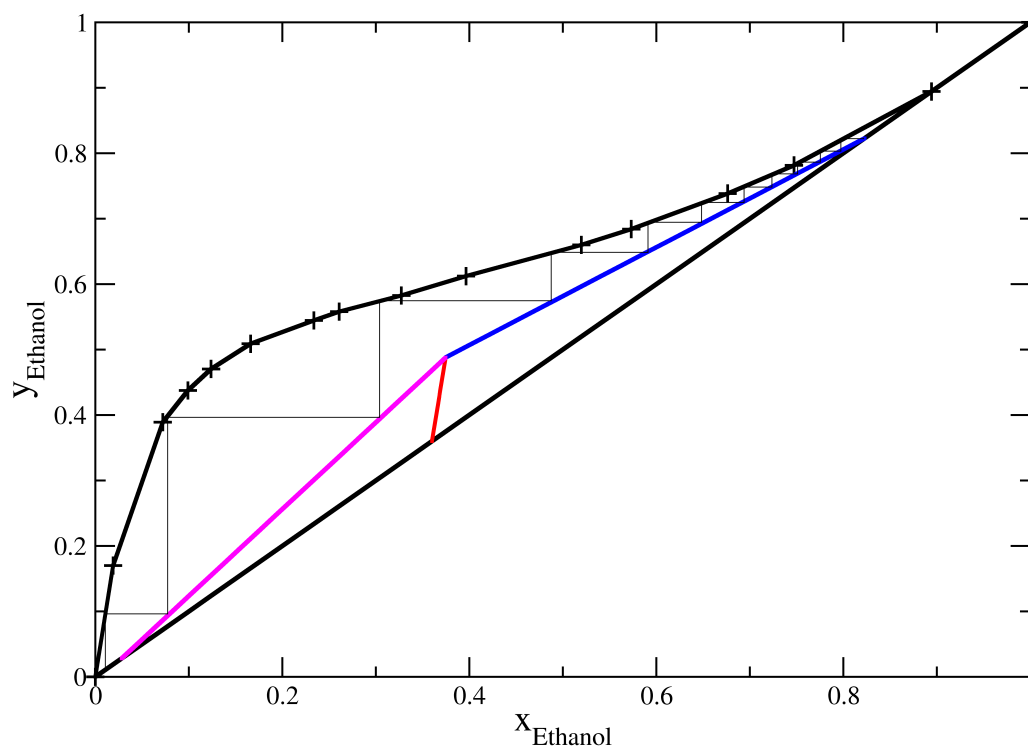Figure 0.6: *Calibration Curve from Refractive Index Data*

Figure 0.7: *Calibration Curve from Specific Gravity Data*

Using the McCabe Thiele method, the number of stages were plotted in 0.8 and 0.9.

Table 0.3: *Vapor Liquid Equilibrium Data*

xEtoh	xw	yEtoh	yw	T, oC
0	1	0	1	100
0.019	0.981	0.17	0.83	95.5
0.0721	0.9279	0.3891	0.6109	89
0.099	0.9034	0.4375	0.5625	86.7
0.1238	0.8762	0.4704	0.5296	85.3
0.1661	0.8339	0.5089	0.4911	84.1
0.2337	0.7663	0.5445	0.4555	82.7
0.2608	0.7392	0.558	0.442	82.3
0.3273	0.6727	0.5826	0.4174	81.5
0.3965	0.6035	0.6122	0.3878	80.7
0.5198	0.4802	0.6599	0.3401	79.7
0.5732	0.4268	0.6841	0.3159	79.3
0.6763	0.3237	0.7385	0.2615	78.74
0.7472	0.2528	0.7815	0.2185	78.41
0.8943	0.1057	0.8943	0.1057	78.15
1	0	1	0	78.3

Figure 0.8: *Total Reflux*

Figure 0.9: *Partial Reflux*

Calculations

Specific Gravity Calculations

Weight of the empty bottle $= 18.911g$

The weight of $10g$ liquid in the bottle is the sum of the weight of the liquid and the weight of the empty bottle. To get the weight of only the liquid, the weight of the empty bottle must be subtracted from the reading obtained from the specific gravity balance.

Weight of liquid water $= 28.341 - 18.911 = 9.43g$

Specific gravity of the liquid $= \frac{9.43}{10} = 0.943g/mL$

Calibration Curve

From the calibration curves Figures 0.6 and 0.7, relations for the mole fractions of ethanol can be determined:

$$x_{Eth} = \frac{(RI - 1.3428)}{0.04168} \tag{0.12}$$

$$x_{Eth} = \frac{(SG - 0.94122)}{0.14565} \tag{0.13}$$

0.12 is used for lower mole fractions of ethanol, while 0.13 is used for higher mole fractions.

Feed Line Equation

Refractive Index of the feed mixture $= 1.357 = 0.36$ mole fraction

The bubble point of feed mixture must be determined. The temperature of the feed is room temperature; i.e. $30°C$.

The Antoine equations for ethanol[3] and water[4] are :

$$log_{10}(P^*) = 4.92531 - \frac{1432.526}{(T - 61.819)} \tag{0.14}$$

$$log_{10}(P^*) = 3.55959 - \frac{643.748}{(T - 198.043)} \tag{0.15}$$

where,

- P is the vapour pressure in bar
- T is the temperature in Kelvin

Rault's law is given by:

$$P = P_A^* x_A + P_B^* x_B \tag{0.16}$$

Using 0.14 , 0.15, 0.16 we obtain, at atmospheric pressure:

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[3]Ambrose, Lawrenson, and Sprake (1975)
[4]Liu and Lindsay Jr (1970)

$$760 = x_{Eth}[10^{4.92531-1432.526/(T-61.819)}] + (1 - x_{Eth})[10^{3.55959-643.748/(T-198.043)}] \quad (0.17)$$

Using 0.17, we calculate the bubble point of the feed to be $363.2K = 90.2°C$.

Specific heat capacity of the mixture is given by:

$$c_{p,mix} = x_{Eth}c_{p,Eth} + (1 - x_{Eth})c_{p,wat} \quad (0.18)$$

ΔH_{vap} of the mixture is given by:

$$\Delta H_{vap,mix} = x_{Eth}\Delta H_{vap,Eth} + (1 - x_{Eth})\Delta H_{vap,wat} \quad (0.19)$$

Using the equations from Table 0.1, we calculate:

$$q = 1.133 \quad (0.20)$$

From @#eq:subcool and Table 0.1, it can be concluded that the feed is *subcooled*.

Using 0.11, the equation for the feed line can be obtained from the calculated value of q:

$$y = 8.52x - 2.707 \quad (0.21)$$

Total Reflux Condition

Specific gravity of distillate $= 0.82$

Refractive index of bottoms $= 1.344$

x_{Eth} for distillate $= 0.823$

x_{Eth} for the bottoms $= 0.028$

Rectifying Line

For the total reflux condition, $R \rightarrow \infty$, and thus, on substituting into 0.7, we obtain:

$$y_{n+1} = x_n \tag{0.22}$$

Stripping Line

$$V = L + D \tag{0.23}$$

where:

- V and L are the constant molar overflows
- D is molar flow rate of the distillate

Since $D = 0, V = L$.

$$y_{m+1} = x_m \tag{0.24}$$

Tray Efficiency

Number of trays from the plot $= 9$

Actual number of trays $= 12$

The tray efficiency is calculated as:

$$Efficiency = \frac{9}{12} \times 100 = 66.67\%$$

Partial Reflux Condition

Specific gravity of distillate $= 0.82$

Refractive index of bottoms $= 1.343$

x_{Eth} for distillate $= 0.823$

x_{Eth} for the bottoms $= 0.02$

Reflux ratio $R = 2.96$

Material Balance

Overall Material Balance

$$F = D + B \tag{0.25}$$

From the observation table, $F = 8.57 mL/min$, $B = 6 mL/min$, $D = 2.47 mL/min$

The total feed supplied is the sum of the primary feed and the secondary feed.

Secondary feed $= 500 - 330 = 170 mL$

Primary feed $= 1500 mL$

Total feed $= 1500 + 170 = 1670 mL$

Distillate volume $= 160 mL$

Total volume of bottoms collected $= 621 mL$

According to the ideal material balance equation in 0.25, the L.H.S and R.H.S. are calculated as:

$$L.H.S. = F = 1670 mL \tag{0.26}$$

$$R.H.S. = B + D = 160 + 621 = 781 mL \tag{0.27}$$

Density of ethanol $= 0.789 g/mol$

Density of water $= 1.0 g/mol$

Density of the feed, with z_F of $0.36 = z_F \rho_{Eth} + (1 - z_F)\rho_{wat} = 0.924 g/mol$

Density of distillate $= x_D \rho_{Eth} + (1 - z_F)\rho_{wat} = 0.826 g/mol$

Density of the bottom $= x_w \rho_{Eth} + (1 - z_F)\rho_{wat} = 0.996 g/mol$

Molecular weight of the feed $= 0.36 \times 46 + (1 - 0.36) \times 18 = 28.08 g/mol$

Molecular weight of the distillate $= 0.823 \times 46 + (1 - 0.823) \times 18 = 41.04 g/mol$

Molecular weight of the bottom $= 0.02 \times 46 + (1 - 0.02) \times 18 = 18.56 g/mol$

Moles in the feed $= 53.97 mol$

Moles in the distillate $= 3.22 mol$

Moles in the bottom $= 33.32 mol$

Component Material Balance

The component material balance is given by:

$$Fz_F = Dx_D + Bx_w \tag{0.28}$$

On solving 0.28, we get:

$$L.H.S. = 53.97 \times 0.36 = 19.43 mol \tag{0.29}$$

$$R.H.S. = 3.22 \times 0.823 + 33.32 \times 0.02 = 3.31 mol \tag{0.30}$$

Energy Balance

Heat capacity of water $= 75.6 J/mol K$

Heat capacity of the ethanol $= 111.46 J/mol K$

Heat capacity of the distillate $= x_D c_{p,Eth} + (1 - x_D)c_{p,wat} = 105.11 J/mol K$

Heat capacity of the feed $= z_F c_{p,Eth} + (1 - z_F)c_{p,wat} = 88.51 J/mol K$

Heat capacity of the bottom $= x_w c_{p,Eth} + (1 - x_w)c_{p,wat} = 76.31 J/mol K$

Energy Input

Energy given to the reboiler $= 590.9 - 590.1 = 0.8 kWh = 0.8 \times 3600 kJ = 2889 kJ$

Energy Output

The feed is at room temperature; i.e. at $30°C$.

Temperature of the distillate $= 74°C$

Average temperature of the bottom $= 0.5 \times (98 + 114) = 106°C$

Energy of the distillate $= mc_p \Delta T = 14.89 kJ$

Energy of the bottom $= mc_p \Delta T = 33.32 \times 76.31 \times (106 - 30) = 193.24 kJ$

Energy lost in the condenser $= \rho V_{flow} c_p \Delta T \times time = 2196.6 kJ$

Total Heat Lost

Total heat loss $= 2889 - (14.89 + 193.24 + 2196.6) = 484.3kJ$

Rectifying Line

Using 0.7, the rectifying line obtained is:

$$y_{n+1} = 0.747x_n + 0.25 \qquad (0.31)$$

Stripping Line

From 0.23, 0.10, we obtain:

$$y_{m+1} = \frac{L}{(L+D)}x_m - \frac{B}{(L+D)} \qquad (0.32)$$

On solving 0.32, the stripping line can be obtained:

$$y_{m+1} = 0.748x_m - 0.613 \qquad (0.33)$$

Tray Efficiency

Number of trays from the plot $= 11$

Actual number of trays $= 12$

The tray efficiency is calculated as:

$$Efficiency = \frac{11}{12} \times 100 = 91.67\%$$

Results and Discussion

1. The overall material balance indicated a loss in mass. This can be explained by a mass accumulation inside the column, which may be a result of non-ideal tray hold-up, which does not remain constant throughout the operation.

2. When the column reflux ratio was changed from total reflux to partial reflux, a rise in the reboiler temperature, from an average of $91°C$ to $106°C$.

3. A higher number of trays was required for the partial reflux case, consistent with the decline of driving force for the separation.

4. The heat loss during the separation was about 16.7%. This may be attributed to insufficient insulation.

5. Ethanol and water form a minimum boiling azeotrope at a composition of 95.5% by weight of ethanol,[5] (Lide 2003). Once this composition has been achieved, the liquid and vapour have the same composition, and no further separation is possible without using an entrainer. Absolute alcohol can be obtained using the process of heterogenous azeotropic distillation Seader, Henley, and Roper (2016). The addition of benzene to an ethanol-water mixture results in the formation of a minimum-boiling, heterogeneous ternary, azeotrope containing, by weight, 18.5% alcohol, 74.1% benzene, and 7.4% water, boiling at $64.85°$C. Upon condensation, the ternary azeotrope separates into two liquid layers: a top layer containing 14.5% alcohol, 84.5% benzene, and 1% water, and a bottoms layer of 53% alcohol, 11% benzene, and 36% water, by weight. The benzene-rich layer is returned as reflux. The other layer is sent to a second distillation column for recovery and recycling of alcohol and benzene. Absolute alcohol, which has a boiling point above that of the ternary azeotrope, is removed at the bottom of the column.

Conclusions

1. The value of q obtained for the feed is 1.133, which indicates that the feed is in the subcooled condition. This is in accordance with the feed actually used in the experiment, which was a liquid at room temperature.

2. In the case of partial reflux, the tray efficiency is 91.67%, which is higher than the tray efficiency 66.67% for the total reflux condition. This may be attributed to the ease of separation for the two cases.

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[5]Speight (2005)

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