Exercise 5B: Batch Distillation

Course 28121: Chemical Unit Operations Laboratory

Team B

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1. xb0 = initial mole fraction of ethanol in tank 1
2. xb1 = final mole fraction of ethanol in tank 1
3. xd_{theo} = average mole fraction of ethanol in tank 2
4. n0 (moles) = total initial number of moles in tank 1 (water and ethanol)
5. n1 (moles) = total final number of moles in tank 1 (water and ethanol)
6. x_1 = final mol fraction of ethanol in still
7. x_0= initial mol fraction of ethanol in still
8. nA (moles) = number of moles of ethanol in still at time t
9. nB (moles) = number of moles of water in still at time t
10. n0A (moles) = number of moles of ethanol in still at time t = 0
11. n0B (moles) =number of moles of water in still at time t = 0
12. α AB = relative volatility for a binary mixture

Abstract

Distillation is a separation process where liquid or vapor mixture of two or more substance is separated into its desired component fractions through boiling and condensation. In this experiment, mixture of ethanol and water were used. Rectification using a constant reflux ratio, which results in a varying distillate composition. In this experiment we have used constant reflux ratio as it has certain benifits such as no need of automatic equipment. Constant reflux ratio is maintained manually as 1.5 throughout the process. Number of plates are estimated by using MaCabe-Thiele Diagram, moreover this method is also used in calculation of reflux ratio. The general mass balances of the ethanol in the the still and the tank is disccused and two methods rayleigh's modified equation and simplified relative volatility procedure are compared in accuracy. The theoritical value of column pressure and the experimental value are compared. The Heat flow and balance in the system is studied. And at last the various components of the steam-condensate system are discussed in their utility and functions.

Introduction

Many industrial distillations are performed batch-wise. In the experiment the batch distillation is carried out in order to separate water and ethanol from mixtures with quantities (weight or volume basis) in the same magnitude of order of the two components. The process is applied under constant reflux ratio, so the distillate and still compositions will continuously vary with time. The initial distillate contains the highest concentration of the light component (ethanol), but distillate composition continuously gets more and more concentrated with the heavier component as the distillation proceeds. The progress of the separation process is followed by determining the concentration of samples from various places in the plant and at various times by measuring the density. The plant is designed to separate a batch of maximum 100 liters using a column with a structured packing. The distillation capacity is 30-40 kg/h. Based on a simple alcohol-water separation it is possible to determine the number of stages, mass balance, minimum reflux ratio, heat efficiency, pressure drop etc. and compare with theory.

Design and construction

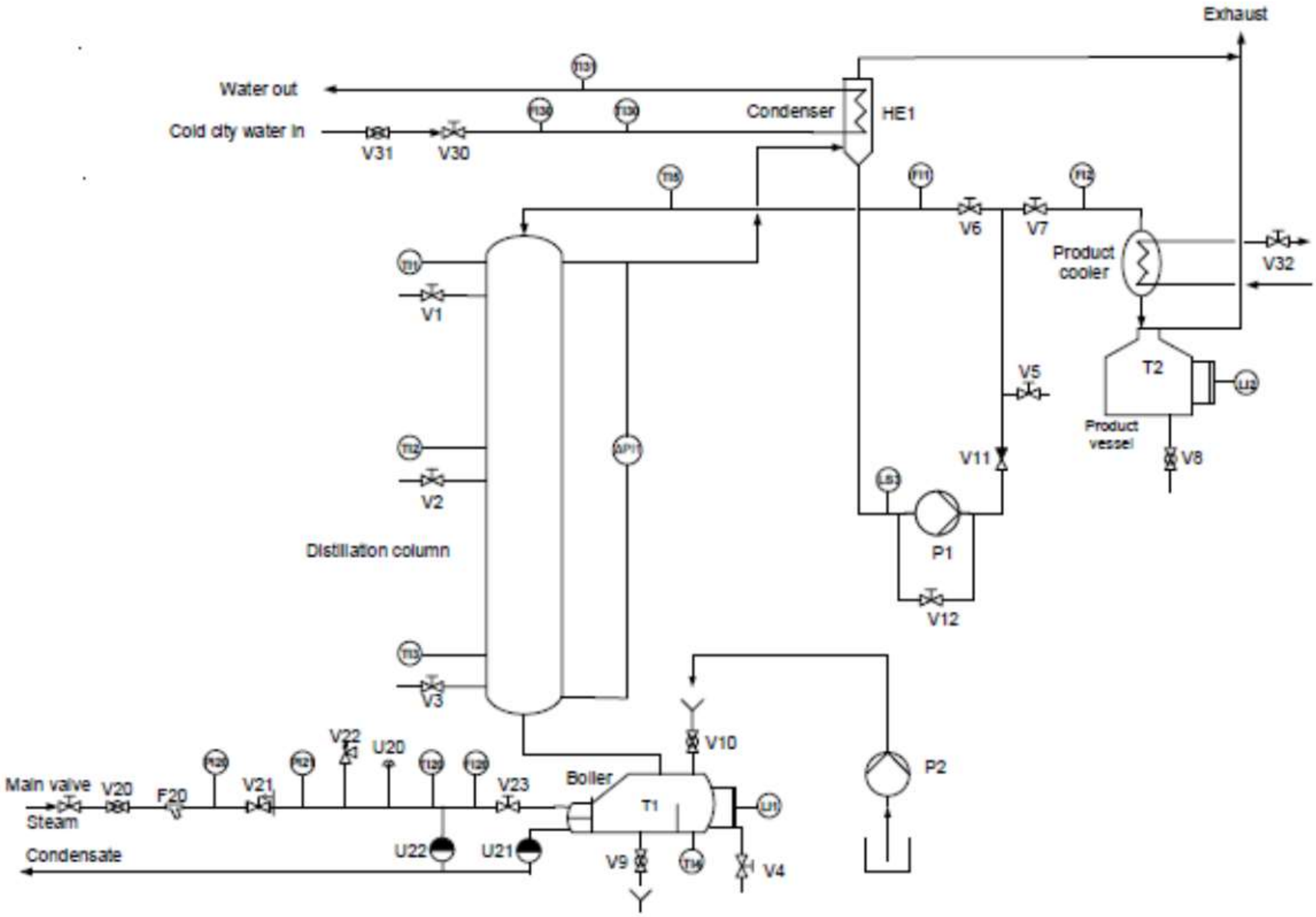


Figure 1. P&I Diagram of Batch Distillation Plant

The experimental setup is shown on the P&I diagram in *Figure 1*. It includes a steam-heated boiler T1 (100 liter) which contains the initial water/alcohol mixture. On top of the boiler (=still) is the column, made in glass tubes of diameter 10 cm. The column is filled with a structured packing called Mellapak 250Y. The height of the packing is 1.68 m. Sample valves and temperature elements are placed at the top, at the middle and at the bottom of the column. The vapors leaving the column are condensed in the glass heat exchanger HE1 using cold city water, and all the condensate is transferred to the pump P1. From here a part of the condensate is taken to the condensate vessel T2 through valve V7, while the rest of the condensate is returned to the top of the column as reflux. The reflux ratio is determined using the flow meters FI1 and FI2. To protect the pump P1 against running dry a flow switch LS3 is installed upstream of the pump. A bypass with a valve V12 around P1 is used to adjust the pump capacity to the achieved condensate flow. The liquid level in the boiler can be controlled by the transparent liquid glass LI1. The level in T2 is controlled by a similar device LI2. Condensate and still samples are taken from V5 resp. V4. The steam flow is controlled by the reducing valve V21 and the manual valve V23 and recorded by the flow meter FI20. The flow of steam through the coils in the boiler is determined by the condensing quantity of steam, since only condensate can pass the steam traps U21 and U22.

Methods, results and analysis

Plot of temperatures versus time

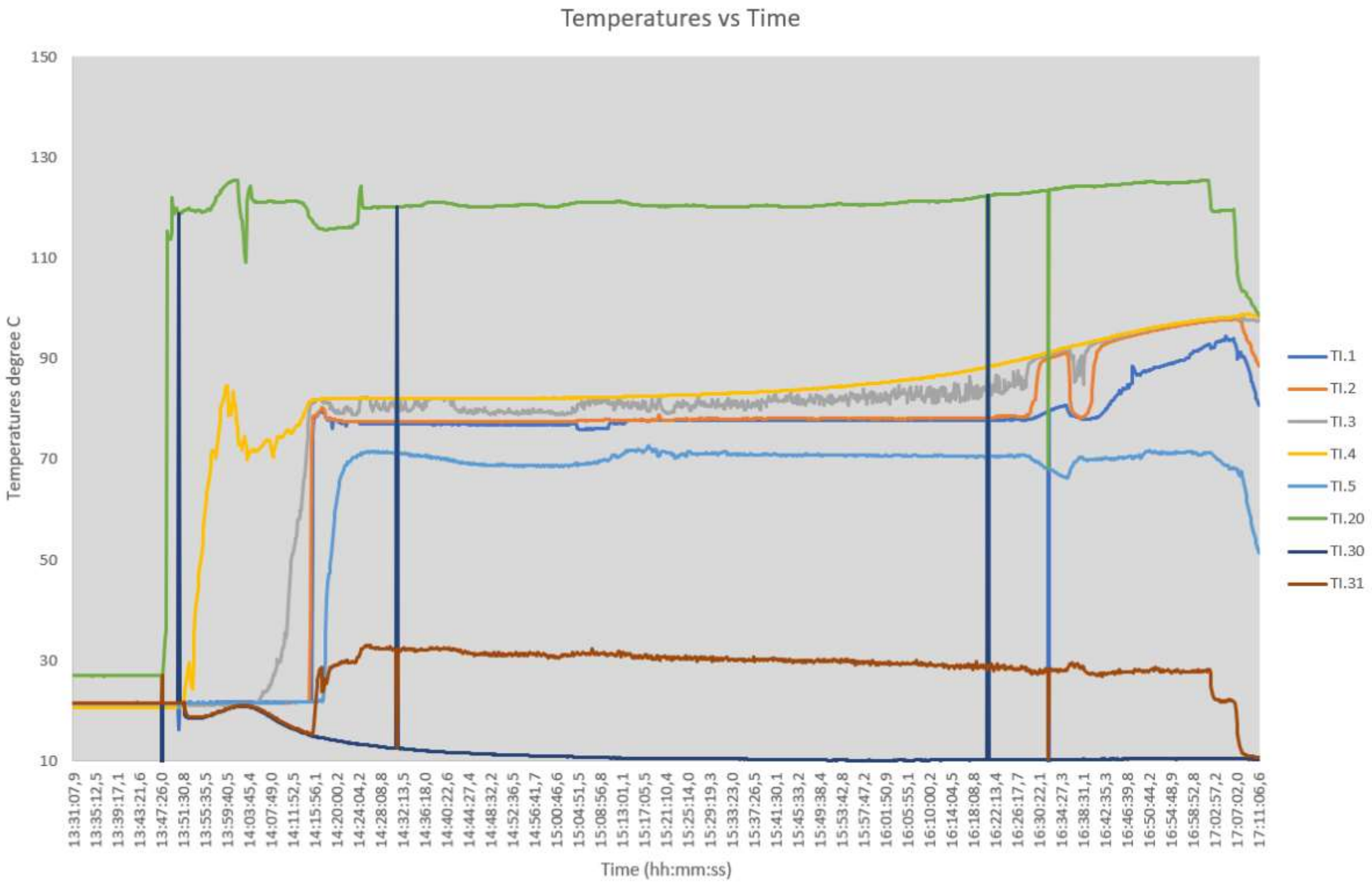


Figure 2 Temperature Vs Time

Calculation of Mole Fraction

The raw data Collected using the Densitometer DMA38 for different samples in V2,V3,V4,V5 is shown in the *Table 1*.

Measurement Time	V2	Alcohol	Water	V3	Alcohol	Water	V4	Alcohol	Water	V5	Alcohol	Water
	Temp	V/V	V/V	Temp	V/V	V/V	Temp	V/V	V/V	Temp	V/V	V/V
14:35:27	22	80.1	19.9	22.5	69.5	30.5	22	29.1	70.9	22.4	89.1	10.9
15:22:21	23.4	87.2	12.8	22.8	63	37	23.6	40.6	59.4	23.5	93.1	6.9
15:40:59	22.4	85	15	21.3	52.8	47.2	23.2	38	62	22	91.7	8.3
16:07:26	22	84.2	15.8	22.3	44.4	55.6	23.1	27.4	72.6	22.6	91.2	8.8
16:32:14	21.6	37.9	62.1	21.7	32.2	67.8	22.4	15.5	84.5	22.9	86.2	13.8
16:58:42	22	5.7	94.3	21	5.6	94.4	21.8	4.4	95.6	22.3	88.4	11.6

Table 1. Raw Data

Here, Temp stands for Temperature and V/V stands for % Volume by Volume

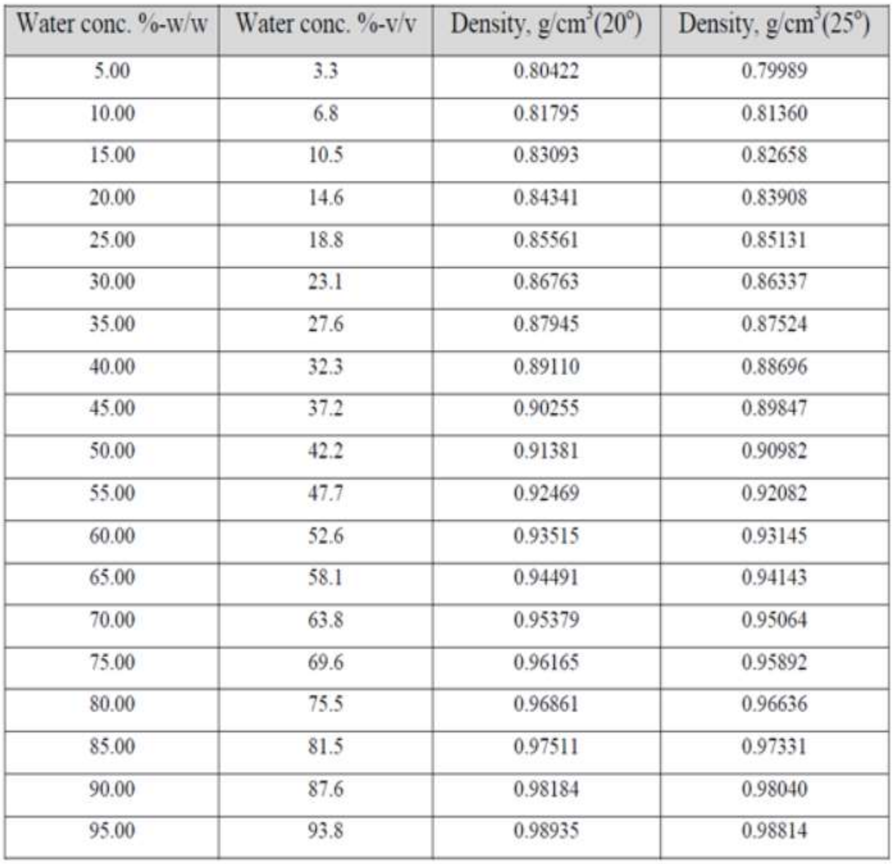
Using the Table 1. we can interpolate the values of Water conc. %-w/w at the specific Water conc. %-v/v. Linear Interpolation Formula is given as

$$y = y_1 + \frac{(x - x_1)\ (y_2 - y_1)}{x_2 - x_1} \tag{1}$$

For $(x_1, y_1) = (6.8,\ 10)$, $(x_2, y_2) = (10.5,\ 15) \cdot (\textit{values taken from Table 2})$ and for $x = 6.9$ in *equation 1* we get,

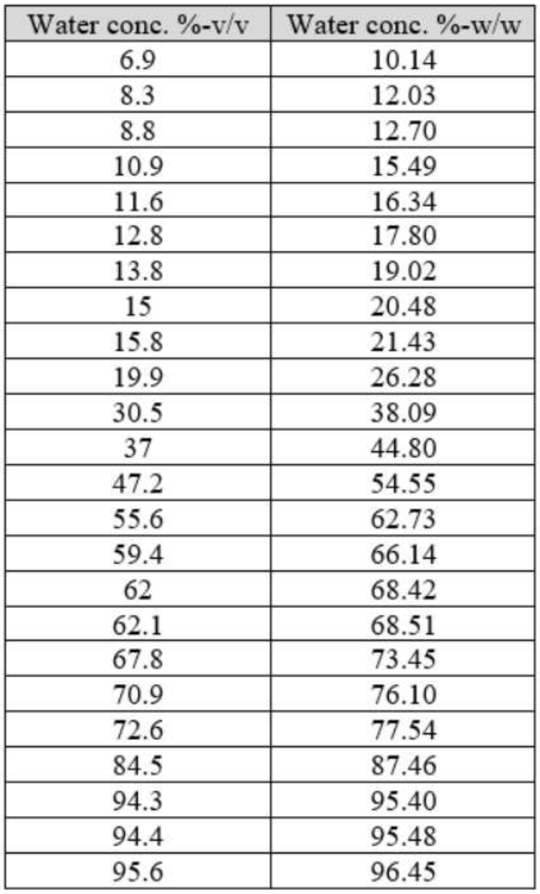
$$y = 10 + \frac{(6.9 - 6.8) \cdot (10.5 - 6.8)}{(15 - 10)} = 10.14$$

Similarly all the values of Water conc. %-w/w are calculated and are given in *Table 3*



Water conc. %-w/w	Water conc. %-v/v	Density, g/cm^3(20°)	Density, g/cm^3(25°)
5.00	3.3	0.80422	0.79989
10.00	6.8	0.81795	0.81360
15.00	10.5	0.83093	0.82658
20.00	14.6	0.84341	0.83908
25.00	18.8	0.85561	0.85131
30.00	23.1	0.86763	0.86337
35.00	27.6	0.87945	0.87524
40.00	32.3	0.89110	0.88696
45.00	37.2	0.90255	0.89847
50.00	42.2	0.91381	0.90982
55.00	47.7	0.92469	0.92082
60.00	52.6	0.93515	0.93145
65.00	58.1	0.94491	0.94143
70.00	63.8	0.95379	0.95064
75.00	69.6	0.96165	0.95892
80.00	75.5	0.96861	0.96636
85.00	81.5	0.97511	0.97331
90.00	87.6	0.98184	0.98040
95.00	93.8	0.98935	0.98814

Table 2. Densities of water-ethanol mixtures at 20°C and 25°C (ref. 2)



Water conc. %-v/v	Water conc. %-w/w
6.9	10.14
8.3	12.03
8.8	12.70
10.9	15.49
11.6	16.34
12.8	17.80
13.8	19.02
15	20.48
15.8	21.43
19.9	26.28
30.5	38.09
37	44.80
47.2	54.55
55.6	62.73
59.4	66.14
62	68.42
62.1	68.51
67.8	73.45
70.9	76.10
72.6	77.54
84.5	87.46
94.3	95.40
94.4	95.48
95.6	96.45

Table 3. Water conc. %-w/w in water-ethanol mixtures

Molar Mass of ethanol is 46.07 g/mol and that of water is 18.01528 g/mol

$$\textit{Water conc. \%} - w/w = \frac{(\textit{weight of Water})}{(\textit{weight of ethanol})}$$

$$\frac{(\textit{molar mass of ethanol})}{\textit{molar mass of water}} = \frac{46.07}{18.01528} = 2.557$$

$$\frac{\textit{moles of water}}{\textit{moles of ethanol}} = \frac{(\textit{weight of Water})}{(\textit{weight of ethanol})} \cdot \frac{(\textit{molar mass of ethanol})}{\textit{molar mass of water}}$$

$$\textit{mole fraction of ethanol} = \frac{1}{\left(\frac{\textit{moles of water}}{\textit{moles of ethanol}} + 1\right)}$$

$$For\ Water\ conc.\ \%-w/w = 10.14\ \text{(From Table 3 row 1)}$$

$$\frac{(weight\ of\ Water)}{(weight\ of\ ethanol)} = \frac{10.14}{100}$$

$$mole\ fraction\ of\ ethanol = \frac{1}{(.1014\cdot 2.557+1)}$$

$$mole\ fraction\ of\ ethanol = 0.7941046938 \qquad \textbf{(2)}$$

Similarly, All the values of mole fraction are calculated and is reported in *Table 4.* and the variation of mole fraction in V2,V3,V4,V5 with respect to time is shown in *Figure 3.*

Measurement Time hh:mm:ss	V2 Water V/V	V2 Mole fraction	V3 Water V/V	V3 Mole fraction	V4 Water V/V	V4 Mole fraction	V5 Water V/V	V5 Mole fraction
14:35:27	19.9	0.598102	30.5	0.506628	70.9	0.339452	10.9	0.71632
15:22:21	12.8	0.687157	37	0.466107	59.4	0.37158	6.9	0.794183
15:40:59	15	0.656351	47.2	0.417584	62	0.363699	8.3	0.7648
16:07:26	15.8	0.646025	55.6	0.384034	72.6	0.33526	8.8	0.754826
16:32:14	62.1	0.363403	67.8	0.347455	84.5	0.308992	13.8	0.672742
16:58:42	94.3	0.290743	94.4	0.290569	95.6	0.288495	11.6	0.705293

Table 4 Mole fraction for corresponding Water Conc. w/w

Plot of measured concentrations (mole fractions of Ethanol) versus time

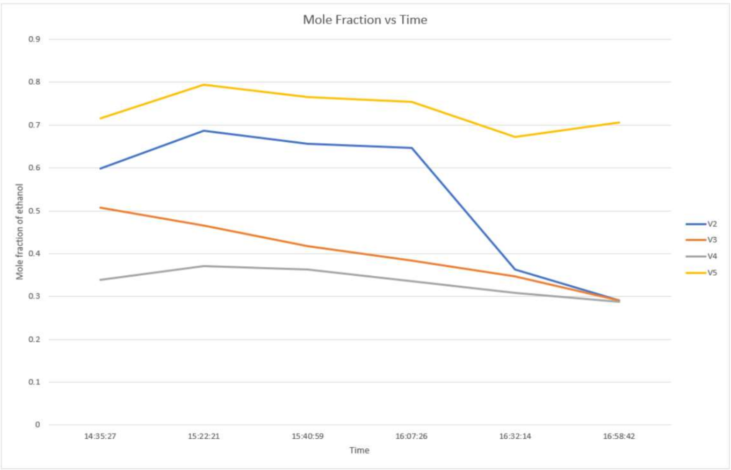


Figure 3. Concentration (Mole Fraction vs Time)

McCabe-Thiele method

The steps between operating lines and the equilibrium line and then count them. Those steps represent the theoretical plates (or equilibrium stages). The required number of theoretical plates is 5 for the binary distillation depicted in Figure 4.

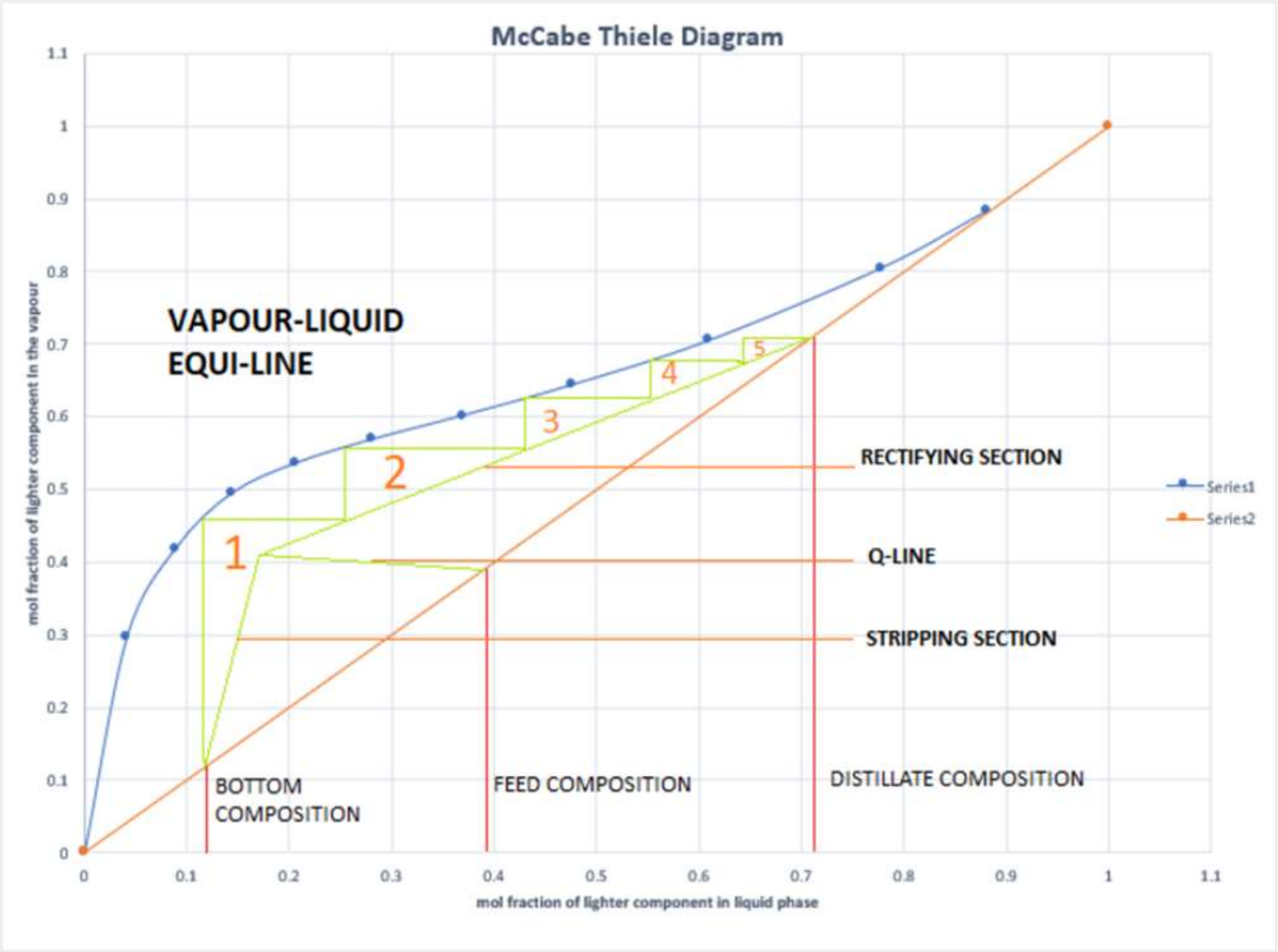


Figure 4:- Typical McCabe–Thiele diagram for distillation of a binary feed

Steps used to get this diagram is given below-
The first step is to draw equal sized vertical and horizontal axes of a graph. The horizontal axis will be for the mole fraction (denoted by x) of the lower-boiling feed component in the liquid phase. The vertical axis will be for the mole fraction (denoted by y) of the lower-boiling feed component in the vapor phase. this x = y line in Figure 1 this 45-degree line is used simply as a graphical aid for drawing the remaining lines. Then the equilibrium line using the VLE data points of the lower boiling component, representing the equilibrium vapor phase compositions for each value of liquid phase composition. Also draw vertical lines from the horizontal axis up to the x = y line for the feed and for the desired compositions of the top distillate product and the corresponding bottoms product. Starting at the intersection of the distillate composition line and the x = y line, draw the rectifying operating line at a downward slope

$$(\Delta y/\Delta x) \text{ of } L / (D + L)$$

where L is the molar flow rate of reflux and D is the molar flow rate of the distillate product. The number of steps used will give us number of plates.

So the calculated number of plates is 5 as per applies McCabe-Thiele Diagram. The number of theoretical plates calculated by the McCabe-Thiele method are more than estimated by the packing supplier of Mellapak 250Y. There exist error in estimation due to method of calculation and error in experimental observation.

Composition Calculations

Initial number of moles in the Still

Initial moles of water in tank = (Initial volume of tank * initial water conc. in sample (V4)) / (molar volume of water)
Initial moles of water in tank = ((80 L*1000 cm3/L) * 0.61(v/v)%)/ 18 cm3/mol
= 2711.11 mol (n_{0B})
Similarly, for ethanol we have-

Initial moles of ethanol in tank = (Initial volume of tank * initial ethanol conc. in sample (V4)) / (molar volume of ethanol)
Initial moles of ethanol in tank = ((80 L*1000 cm3/L) * 0.39 (v/v)%)/ 58 cm3/mol

= 537.93 mol (n_{0A})

Hence the total number of moles in the Still initially = 2711.11+ 537.93 = 3249 mol (n_0)
Initial Mole fraction of ethanol in still = 0.1655 (x_0)

Final number of moles in the Still

Final moles of water in tank = (final volume of tank * final water conc. in sample (V4)) / (molar volume of water)
Final moles of water in tank = ((50 L*1000 cm3/L) * 0.94(v/v)%)/ 18 cm3/mol
= 2611.11 mol (n_B)
Similarly, for ethanol we have-

Final moles of ethanol in tank = (final volume of tank * final ethanol conc. in sample (V4)) / (molar volume of ethanol)
Final moles of ethanol in tank = ((50 L*1000 cm3/L) * 0.06(v/v)%)/ 58 cm3/mol
= 51.72 mol ($n_{A(Calc)}$)

Hence the total number of moles in the Still finally at the end = 2611.11+51.72 = 2662.83 mol (n_1)
Final Mole fraction of ethanol in still = 0.01942 (x_1)

Average distillate composition

We can calculate total moles of water and ethanol in distillate by subtracting initial moles from final moles of still.
Moles of water in distillate = 2711.11 mol -2611.11 mol = 100 mol
Moles of ethanol in distillate = 537.93 mol - 51.72 mol = 486.21 mol

So average distillate composition will be $x_{d(theo)} = 0.829$

Measured distillate composition

Calculations:

Total number of distillate Barrel : 6

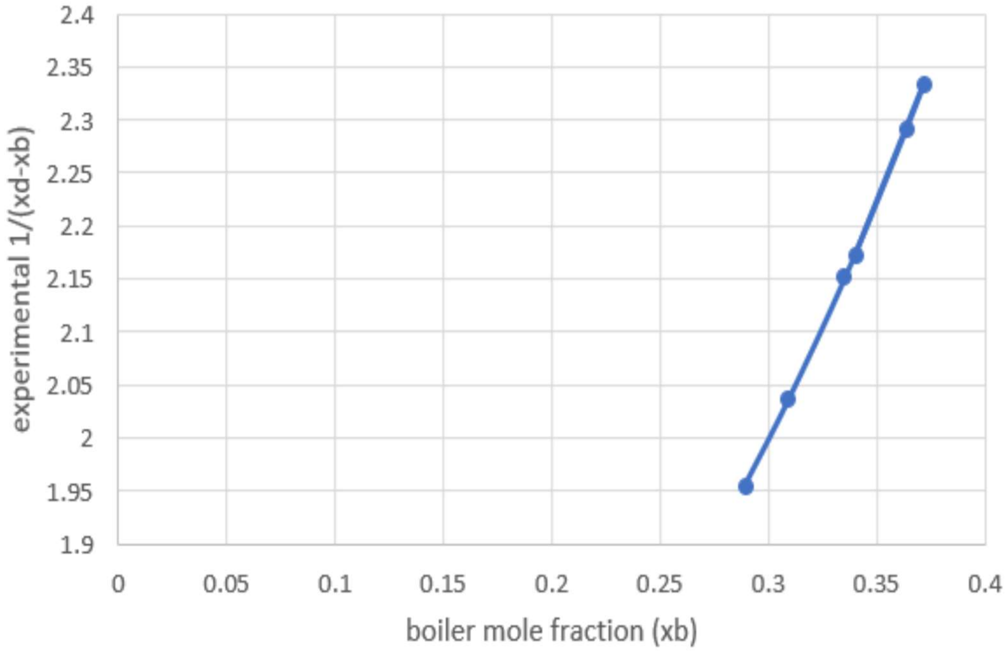
Barrel No.	*Empty*	1	2	3	4	5	6
Composition %V/V	–	95.6	92.1	87.3	85.6	84.2	82.5
Composition %w/w	–	96.45	93.63	89.75	88.36	87.21	85.82
Weight of Barrel·(kg)	0.32	4.77	4.58	4.86	4.52	3.92	1.43
Weight of solution·(kg)	–	4.45	4.26	4.54	4.2	3.6	1.11
Weight of water	–	0.157975	0.271362	0.46535	0.48888	0.46044	0.157398
Weight of ethanol	–	4.292025	3.988638	4.07465	3.71112	3.13956	0.952602
Moles of water	–	8.77	15.07	25.85	27.16	25.58	8.74
Moles of ethanol	–	93.30	86.70	88.58	80.68	68.25	20.71

Distillate Measured Composition

Total Number of Moles of ethanol = 438.22 , Total Number of Moles of water = 111.17

Measured distillate composition (molefraction of ethanol) = 0.79

The Measured distillate composition and Calculated average distillate compositions are almost same and hence x



Fiqure 5. Plot of experimental 1/(xd-xb) as function of the measured boiler mole fraction xb

Mass Balance

The total initial mass is found by addition of masses of water and ethanol in the tank

$$\textit{moles of ethanol}\cdot\textit{molecular mass of ethanol} + \textit{moles of water}\cdot\textit{molecular mass of water}$$

$$\frac{(537.93\cdot 46 + 2711.11\cdot 18)}{1000}$$

$$73.54 \text{ kg}$$

The total End mass is found by addition of masses of water and ethanol in the tank and distillate at end of the process.

Mass in Tank at the end

$$\textit{moles of ethanol}\cdot\textit{molecular mass of ethanol} + \textit{moles of water}\cdot\textit{molecular mass of water}$$

$$\frac{(51.72\cdot 46 + 2611.11\cdot 18)}{1000}$$

$$49.37 \text{ kg}$$

Mass in Distillate at the end

$$\textit{moles of ethanol}\cdot\textit{molecular mass of ethanol} + \textit{moles of water}\cdot\textit{molecular mass of water}$$

$$\frac{(438.22\cdot 46 + 111.17\cdot 18)}{1000}$$

$$22.159 \text{ kg}$$

Thus total end mass is 49.37 + 22.159 = 71.53 kg

The total mass found in the end is slightly less than what was the input initially this error could be due to the measurement error in volume % of the solutions during the total moles calculations.

For batch distillation of binary systems under constant reflux the mass balance for the process can be described by the Rayleigh equation

$$\ln\left(\frac{n1}{n0}\right) = \int_{x_{b0}}^{x_{b1}} \frac{dx_b}{x_d - x_b}$$

where n0 and n1 are the total initial number resp. the final number of moles in the still T1 (water and alcohol), xb and xd are the actual mole fraction of alcohol in the still (boiler) resp. in the condensate (distillate), and xb0 and xb1 denotes the initial and final mole fraction in the still. This expression can be integrated graphically using a diagram where $\frac{1}{(x_d - x_b)}$ is plotted against x_b. The area under the curve represents the solution to the integral.

Thus Using rayleigh's modified equation we have,

$$\frac{n_1}{n_0} = \frac{x_{d(theo)} - x_0}{x_{d(theo)} - x_1}$$

$$n_{1(theo)} = n_0 \cdot \left(\frac{x_{d(theo)} - x_0}{x_{d(theo)} - x_1} \right)$$

$$n_{1(theo)} = \frac{3249 \cdot (0.829 - 0.1655)}{(0.829 - 0.01942)}$$

$$n_{1(theo)} = 2662.75\ mol$$

The calculated value for $n_{1(calc)}$ is 2662.83 mol and the theoritical value found using rayleigh's modified equation is $n_{1(theo)}$ is 2662.75 mol. The error is 0.003 % which is very low and thus we can conclude that the rayleigh's modified equation is a highly accurate in terms of mass balance or mole balance

Now Using the simplified relative volatility procedure

$$\frac{n_B}{n_{0B}} = \left(\frac{n_A}{n_{0A}} \right)^{\frac{1}{\alpha_{AB}}}$$

Rearranging the equation to find the value of n_A

$$n_{A(theo)} = n_{0A} \cdot \left(\frac{n_B}{n_{0B}} \right)^{\alpha_{AB}}$$

where A is ethanol and B is water , n_{0A} and n_{0B} denotes the number of moles of A and B in the still at time = 0 and n_A and n_B is the number of moles in the still at a later time that is of intrest.

n_B = 2611.11 mol

n_{0B} = 2711.11 mol

n_{0A} = 537.93 mol

$n_{A(Theo)}$ = 51.72 mol

$$\alpha_{AB} = \frac{y_A \cdot x_B}{y_B \cdot x_A} = \frac{(0.672) \cdot (0.971)}{(0.028) \cdot (0.327)} = 65$$

$$n_{A(calc)} = 537.93 \left(\frac{2611.11}{2711.11} \right)^{65}$$

$$n_{A(calc)} = 46.75\ \text{mol}$$

From the relative volatility procedure the moles of ethanol found are less and the error is 9 % which is larger than the error from rayleigh's modified equation. Thus it can be deduced from these calculations that rayleigh's modified equation approach is better and more accurate than the relative volatility procedure.

Column Pressure

The distillation column is filled with 1.68m of Mellapak 250Y, with the tube diameter being 0.1m and has a specific surface of 250 $\left(\frac{m^2}{m^3}\right)$ volume. The liquid phase flowing down the column impends the upward flow of the vapour and thus the pressure at the top of the distillation column is lower than that of the bottom. Thus, the experimentally determined pressure drop of 120 Pa is in like with the theoretical expectation. The manufacturer of the Mellapak 250Y structured packing provides theoretical pressure drop data as a function of F-factor. The F-factor is obtained from the following equation :

$$F-factor = w_G\sqrt{\rho_G} = 0.002533\ g\sqrt{\frac{T}{M \cdot p}}$$

where,

Gas velocity referring to empty column : w_G (m/s),

Gas density : ρ_G (kg/m3),

Gas load : $g\left(\frac{kg}{m^2 h}\right)$
Operating temperature : T (K)
Operating pressure : p (mbar)
Molar mass : M ($\frac{kg}{kmol}$)

To calculate the above $F-factor$ the Operating Pressure is taken to be 100 mbar

The gas load $g = \frac{vap_{rate}}{Area_{column}}$

$$Area_{column} = 2 \cdot \pi \cdot h \cdot r = \frac{2 \cdot \pi \cdot 1.68 \cdot 0.1}{2} = 0.5277\ m^2$$

Now to find the rate of formation of vapour of ethanol in the distillation column the relation below is used :

$$vap_{rate} = \frac{\Delta m}{\Delta t}$$

where, Δm is the total change in mass of ethanol and Δt is the total time of experiment i.e 3 hours.

From the mass balance calculations the mass of ethanol before the experiment started can be stated as

$$Mass\ of\ ethanol = moles\ of\ ethanol\ at\ the\ start \cdot molecular\ mass\ of\ ethanol$$

$$537.93\ \text{x}\ \frac{46}{1000} = 24.75\ kg$$

and the mass of ethanol after the experiment ended can again be stated as

$$Mass\ of\ ethanol = moles\ of\ ethanol\ at\ the\ end \cdot molecular\ mass\ of\ ethanol$$

$$51.72\ \text{x}\ \frac{46}{1000} = 2.38\ kg$$

thus,

$$vap_{rate} = \frac{24.75 - 2.38}{3}$$

$$vap_{rate} = 7.45\ \frac{kg}{hr}$$

$$g = \frac{7.45}{0.5277}\left(\frac{kg}{m^2 h}\right)$$

$$g = 14.11\left(\frac{kg}{m^2 h}\right)$$

Operating Temperature used in the equation can be calculated as the average of the temperatre reading taken all through the experiment for the distillation column i.e.

$$T_{avg} = \frac{\left(\frac{\left(\sum TI.1 + \sum TI.2 + \sum TI.3\right)}{3}\right)}{Total\ number\ of\ readings}$$

this calculation gives the value of temperature to be 67.4 °C i.e 340.55 Kelvin

$$F\text{-}factor = 0.002533\ g\sqrt{\frac{T}{M \cdot p}}$$

$$F\text{-}factor = 0.002533 \cdot 14.11\sqrt{\frac{340.55}{46 \cdot \frac{100}{10000}}}$$

$$F - factor = 0.972$$

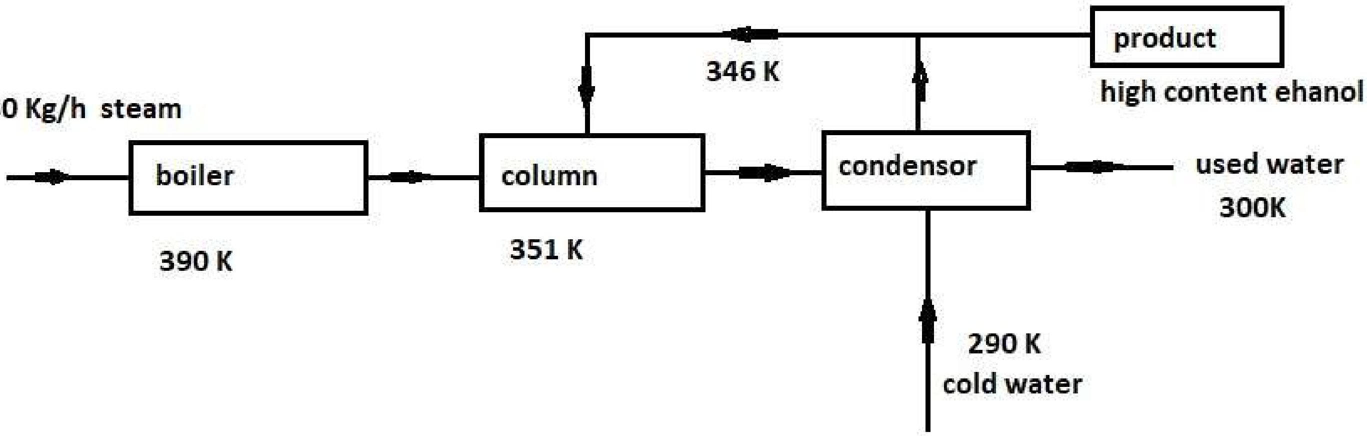
Using the graph provided in manufacturer sheets this corresponds to a theoretical pressure drop of 35 Pa, the following deviation from the experimental pressure drop is observed :

$$percentage\ error = \frac{(experimental\ value\ -\ theoritical\ value)}{theoritical\ value} = \frac{(120 - 35)}{120} \cdot 100 = 70.83\ \%$$

Heat Flow and Balance in the System

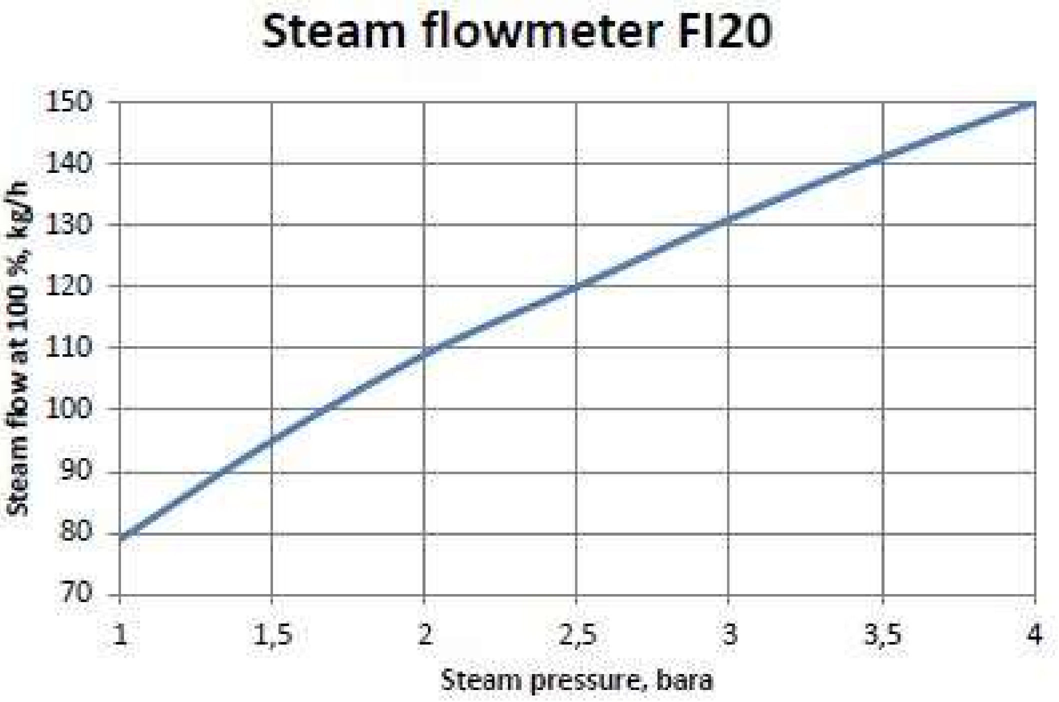
General Heat Balance in any system is
Heat in + Heat Generated = Heat out + Heat Accumulated

But their is no Generation or Accumulation in the system hence these terms are zero. Thus theorotically speaking the energy transfer of the boiler and condenser should be identical, but in practice these results vary largely due to energy losses in friction, heat transfer to the surrounding and so much more. The diagram below provides a basic outline of the main process components involved in the batch distillation investigation



Fiqure 6. Feed flow direction in a system

The reading of the steam flowmeter FI20 is recorded in % full scale on the data logging system and to convert to actual flow the below diagram is used



Fiqure 7. Plot of steam flow VS steam pressure

From the above figure we can conclude that the rate of heat stream entering the system is 80kg/h. The enthalpy of vaporization for ethanol at 120 degrees C is listed to be 764.05 kJ/kg from the nist website[9] and The constant pressure specific heat capacity of water was found to be : $C_{p,\,w}$ = 2.187 kJ/kgK refrence [4] is udes here.

The rate of heat transfer for the boiler is determined using the following equation:

$$\frac{Q}{t} = \hat{m} \cdot \Delta H_{vap}$$

$$\frac{Q}{t}\left(\frac{kJ}{s}\right) = 0.02222\left(\frac{kg}{s}\right)\cdot 764.05\left(\frac{kJ}{kg}\right) = 16.98\left(\frac{kJ}{s}\right)$$

In order to determine heat transfer rate for the condenser, a different equation is used

$$\frac{Q}{t} = \hat{m} \cdot C_{p,\,w} \cdot \Delta T$$

As given in the manual, rotameter FI30 for cooling water has a full scale (100 %) flow of 737 kg/h. And thus the cold water flow is taken to be a constant 737kg/h = 0.2047kg/s.

$$\frac{Q}{t}\left(\frac{kJ}{s}\right) = 0.2047\left(\frac{kg}{s}\right)\cdot 2.187\left(\frac{kJ}{kg\cdot K}\right)\times(306 - 286)\,(K) = 8.95\left(\frac{kJ}{s}\right)$$

Overall, the calculated energy transfer rate through the condenser is significantly lower than that of the boiler. The percentage difference between the two values can be quantified as the difference in the vaue divided by the average multiplied by hundred .

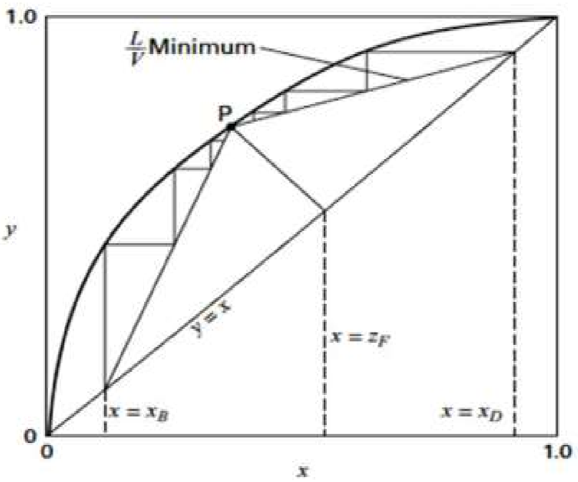
Thus Diff = $\frac{(16.98-8.95)\cdot 2}{(16.98+8.95)}\cdot 100 = 61\ \%$

A percent difference of 61% is huge and can be understood due to a noticeably lower energy transfer rate in a condenser compared to a boiler. Evaporation losses can be said to have contributed a lot.

Minimum Reflux Ratio

At total reflux, the number of theoretical plates required is a minimum. As the reflux ratio is reduced (by taking off product), the number of plates required increases. The Minimum Reflux Ratio (R min) is the lowest value of reflux at which separation can be achieved even with an infinite number of plates.

MINIMUM REFLUX RATIO DETERMINATION



Fiqure 8. Minimum reflux ratio analysis.

At the minimum reflux ratio condition there are invariant zones that occur above and below the feed plate, where

the number of plates is infinte and the liquid and vapour compositions do not change from plate to plate.
Unlike in binary distillation, in multicomponent mixture these zones are not necessary adjacent to the feed plate location.

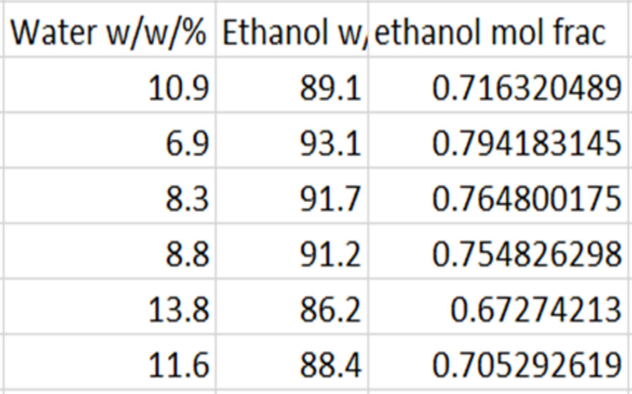
The McCabe-Thiele plot Diagram is used in the minimum reflux ratio determination. In order to accomplish this, theoretical mole fraction of ethanol in vapour (y)is plotted against the mole fraction of ethanol in liquid (x) are plotted.
For determination of minimum reflux ratio for initial and final condition reference lines are plotted (mol fractions of ethanol in liquid sample xB and for condenser xP.
Y intercept of the line is defined by-
$Y= xP/(R+1)$
R representing the minimum theoretical reflux rate for the process.
Mathematically R can be defined as-

$RMIN = L/VMIN/(1- L/VMIN)$

whereas L/VMIN is given by-

$L/VMIN = (yD – yP)/(xD – xP)$

By using linear interpolation for the given data, we will have-



Water w/w/%	Ethanol w,	ethanol mol frac
10.9	89.1	0.716320489
6.9	93.1	0.794183145
8.3	91.7	0.764800175
8.8	91.2	0.754826298
13.8	86.2	0.67274213
11.6	88.4	0.705292619

By using initial and final data we can calculate required reflux ratio,

$Y= xp/R+1$
$Y= xp/y – 1$
For initial conditions we have-
$R = 0.598/0.295 - 1 = 1.02$

For initial conditions we have-
$R= 0.210/0.0547 – 1 = 2.8$

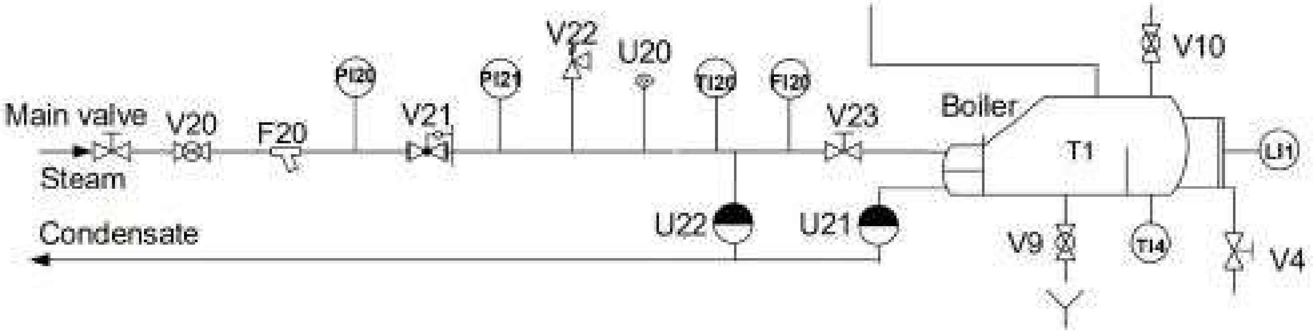
The process is applied under constant reflux ratio, so the distillate and still compositions will continuously vary with time. We have used constant reflux ratio which was set to 1.5 manually.

There are two options for reflux in batch distillation.
1. Increase the reflux ratio with time to keep the product concentration constant. Low reflux initially; high reflux towards the end.
2. Use a fixed reflux ratio. Operate the still until the top concentration falls below a setpoint.

But we have some benefits of using constant reflux ratio is that it does not require expensive, and occasionally unreliable, automatic equipment. But we get little bit different values for the initial and final conditions, we have smaller value for initial condition and greater value for final condition. This can be due to the observed boiler liquid mole fraction xB being a very small value, causing a larger slope for final conditions.

Various components in the process steam/steam-condensate system

The part of PI diagram which has the steam-condensate system is shown below :



Fiqure 9. Steam-Condensate System

Steam Generator

Steam is used to heat the mixture of water and ethanol in a boiler, which can be controlled by valves and controller (main valve) as per the demand.

Boiler

Boiler is a heart of Batch Distillation process a container of 100 L capacity, which is used to heat the mixture of water and ethanol using steam.

Batch Columns

In batch operation, the feed to the column is introduced batch-wise. That is, the column is charged with a 'batch' and then the distillation process is carried out. When the desired task is achieved, a next batch of feed is introduced.

Main Components of Distillation Columns
Distillation columns are made up of several components, each of which is used either to transfer heat energy or enhance material transfer.
A typical distillation contains several major components:

- A vertical shell where the separation of liquid components is carried out
- Column internal such as trays/plates and/or packing which are used to enhance component separations
- A reboiler to provide the necessary vaporizations for the distillation process
- A condenser to cool and condense the vapour leaving the top of the column
- A reflux drum to hold the condensed vapour from the top of the column so that liquid (reflux) can be recycled back to the column

Condensor

The condenser is located at the top of the distillation column and removes energy from the distillation column. The purpose of the condenser is to condense the vapor leaving the top tray of the column.
Reflux percent is the percent of liquid condensed in the condenser that is allowed to be collected as “distillate”. The distillate is the product stream from the top of the distillation column, which is rich in component A of the binary mixture. The remaining liquid that is not collected as distillate, termed “reflux,” is returned to the distillation column for further separation.

Discussion and conclusions

During experimentation of the distillation column the group noticed distillate being formed even during total reflux. This could indicate a problem with the reflux valve. This could also explain the problem with the reboiler volume during batch distillation experiments. The behavior of the reflux valve should be monitored to determine whether it is functioning correctly. The plots of temperature vs time and mole fraction vs time reveal that the mole fraction of ethanol decreases continuosly when the sysytem is started and which is expected as the ethanol molefraction should increase in the distillate and decrease in the still. MaCabe-Thiele method is used for calculation of number of plates, by estimation we get the number of theoretical plates calculated by the McCabe-Thiele method are more than estimated by the packing supplier of Mellapak 250Y. In the whole process we used constant reflux ratio, which was already set to 1.5 but the calculated value is less for initial condition and greater for final condition. Mostly we are constrained to maintain it to constant but values calculated are different due to high difference in flow rate of steam into the boiler. Which would have led to requirement of high reflux ratio due to high flow rate. Similarly, for less flow rate we have less reflux ratio. ∇In the mass balance theory we found that the rayleigh's modified equation is much more accurate than the simplified relative volatility procedure. The error value for the fisrt is 0.003% while for the latter is 9% which very huge compared to the first one. In finding the column pressure difference the error in experimental value and theoritical value is as large as 70 %. Heat balance in the system gives out the fact that their are many losses in heat due to frictioin, heat release in the environment and the percentage difference due to such losses is 61%. Being able to perform experiments on the distillation column was very useful task as it provides practical experience in using the theoretical knowledge acquired in the Unit Operations class throughout the semester.

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