



CH 3522: Unit Operations Lab

Plate Column Distillation

Batch - R, Group - 05

Date of Experiment: 12th March 2025

Date of Submission: 18th March 2025

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1. Objective

- Number of theoretical plates using McCabe Thiele method
- Calculate the overall tower efficiency
- Compute the Murphree efficiency and number of transfer units for each tray for the given setup

2. Introduction

Distillation is a fundamental separation technique used for liquid mixtures, leveraging the differences in volatility among components to achieve separation. This process operates on the principle that each component in a mixture has a distinct boiling point. When the mixture is heated, the more volatile components vaporize first, enabling their separation from the less volatile ones. In a continuous distillation column, a high-purity product is obtained by integrating a rectifying section and a stripping section, with the feed typically introduced near the middle of the column. The feed, usually in liquid form, flows downward through the stripping section by gravity to the reboiler. The reboiler, heated by steam, vaporizes the liquid, and the resulting vapor ascends the column. The vapor is then condensed, collected, or returned as reflux to sustain the distillation process.

A plate column is a critical piece of equipment used in processes requiring mass transfer between liquid and gas phases. Distillation columns often employ trays or plates to enhance the interaction between vapor and liquid phases, thereby improving separation efficiency. Common tray designs include bubble cap trays, valve trays, and sieve trays, each contributing to the effective separation of components within the column.

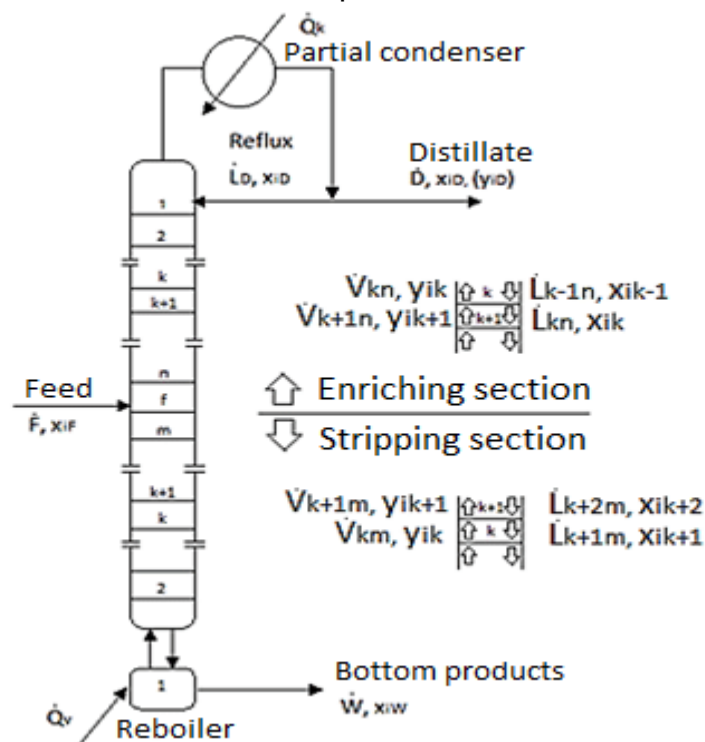


Figure 1: A Schematic of a Segmented Plate Distillation Column

3. Theory

This experiment involves a single feed introduction into the column, followed by sealing the system for continuous distillation. To analyze the process, we will utilize vapor-liquid equilibrium relationships, and apply the McCabe-Thiele method alongside the Murphree efficiency concept. These tools will help estimate the number of theoretical plates, overall column efficiency, and the number of transfer units required for each stage of the setup. Below is an explanation of the methods used in the calculations.

1. McCabe-Thiele Method

The McCabe-Thiele method is a graphical approach used to estimate the number of equilibrium stages required for binary distillation. Although it is commonly used for continuous distillation, it can be adapted for batch distillation by considering stepwise separation at different time intervals.

Application in Batch Distillation

Here are the steps for applying McCabe Thiele method in a batch distillation system:

A. Identify Initial Conditions:

- Determine the initial composition of the ethanol-water mixture in the still.



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- Set the target distillate composition (x_D).
- Define the reflux ratio (R) if a rectifying section is present.

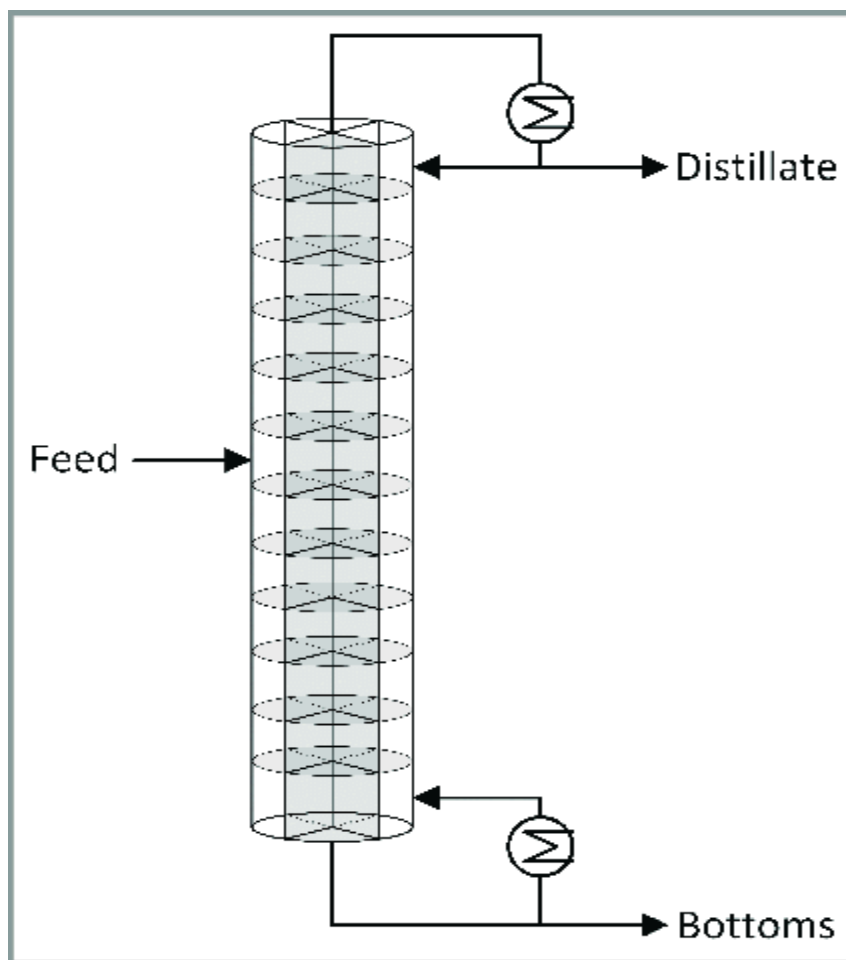


Figure 2: A simpler representation of a plate column distillation unit

B. Create the Equilibrium Diagram:

- Obtain or plot the equilibrium curve using vapor-liquid equilibrium (VLE) data.
- Draw a diagonal reference line ($y = x$).

C. Draw the Operating Line:

- For batch distillation, the rectifying operating line equation is:



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$$y_n = \frac{R}{R+1}x_n + \frac{x_D}{R+1} \quad (1)$$

Where:

- y_n = Vapor composition
- x_n = Liquid composition
- R = Reflux ratio
- x_D = Distillate composition

D. Graphical Stepwise Approach:

- Start at the desired distillate composition (x_D) on the x-axis.
- Move vertically to the equilibrium curve to determine the vapor composition (y_n).
- Move horizontally to the operating line to determine the liquid composition (x_n).
- Repeat the steps until reaching the initial still composition (x_w).
- The number of steps represents the required equilibrium stages (trays).

E. Account for Changing Still Composition:

- Since the still composition x_w changes over time, repeat the McCabe-Thiele analysis at different intervals.
- Adjust the still composition stepwise and update calculations as distillation progresses.

2. Murphree Tray Efficiency

Murphree Tray Efficiency assesses how effectively a tray in a distillation column brings the vapor and liquid into equilibrium. This efficiency helps estimate the number of theoretical stages needed for effective separation.

$$E_M = \frac{y_{actual} - y_{inlet}}{y_{equilibrium} - y_{inlet}} \quad (2)$$

Where:

- y_{actual} = Actual vapor composition leaving the tray
- y_{inlet} = Vapor composition entering the tray
- $y_{equilibrium}$ = Vapor composition at equilibrium with the exiting liquid

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Factors Influencing Tray Efficiency

- Flow rates of liquid and vapor
- Tray type (sieve, valve, bubble cap)
- Fluid properties (viscosity, surface tension)
- Degree of back-mixing and maldistribution

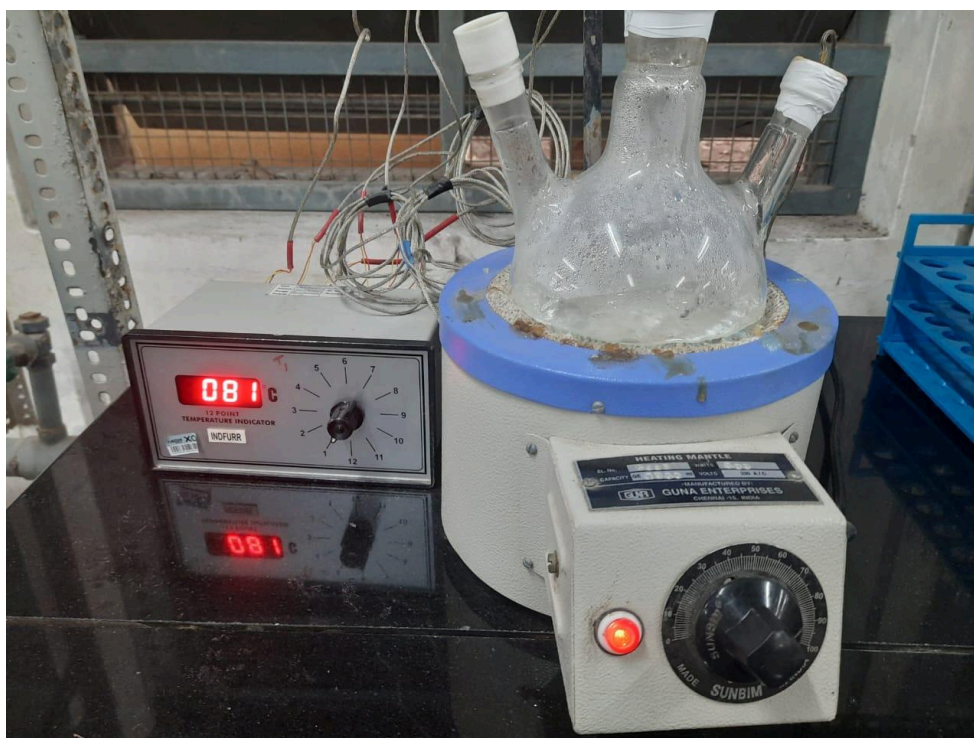


Figure 3: Steady State Heat Source

4. Apparatus Required

The apparatus and the chemicals used in this experiment are as follows,

- Chemicals required: Ethanol and water
- Triple-neck round bottom flask
- Plate column with 5 perforated 'trays' or 'stages'
- Collecting valves and inbuilt probes for temperature measurements
- Steady heat source and temperature display
- Glassware, TDS meter to measure density

5. Schematic of Experimental Setup

Experimental setup includes the following components and we have also provided the setup used during the experiment in figure 4.



Figure 4: Experimental setup of Plate Distillation Column

6. Procedure

- We start by preparing a solution of ethanol in water: Add 250 mL of Ethanol onto 500mL of water. This forms our feed which is then transferred to the triple neck round bottom flask. This feed is fed just once in the setup, thus making it a batch distillation process.
- Now, we turn on the condenser water supply and the pump, ensuring there is a continuous supply of coolant for condensing all vapours that would eventually go up through the column.
- Now switch on the power supply, setting to a heating mode below 80% of the maximum. Also switch on the temperature display and give enough time for steady state to reach.
- When the apparatus is all set and left undisturbed till the onset of steady state, we prepare a calibration plot connecting density with concentration of ethanol in water.
- Start the calibration table by adding 1mL of ethanol to 9 mL of water, subsequently measuring the density of the prepared solution with a TDS meter. Repeat the same for subsequent sets of 10 mL solutions with 2mL, 3mL ... up to 10 mL of ethanol itself.



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- Now we have 11 density readings corresponding to 11 ethanol-water concentration. This forms our calibration plot and can be used to identify concentration corresponding to some density value.
- While the calibration table is being prepared, make sure to take temperature readings from all 5 probes at regular intervals. This is done to track -state the onset of steady-state.
- Once the probe temperatures are fairly constant around a steady state value, ensure liquid levels at all 4 trays/stages. Now, we can collect the samples from the valves in the top part of the column to the bottom.
- Once we have all 6 samples collected, we measure the densities of each of these samples and tabulate the readings. Also measure the density of the residual liquid mixture left behind in the flask.

7. Experimental Observations

The following table (refer to table 1, 2) contains the data collected while performing the experiment, corresponding to which the datasheet is also provided at the end of this report.

The tabulated values are as follows,

Table 1: Calibration data of Composition and Density

Volume of Ethanol(ml)	Volume of Water(ml)	Density of mixture (g/cc)
10	0	0.785
9	1	0.817
8	2	0.829
7	3	0.867
6	4	0.894
5	5	0.915
4	6	0.936
3	7	0.953
2	8	0.972
1	9	0.983
0	10	0.993



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Table 2: Density of samples from different trays of the distillation column

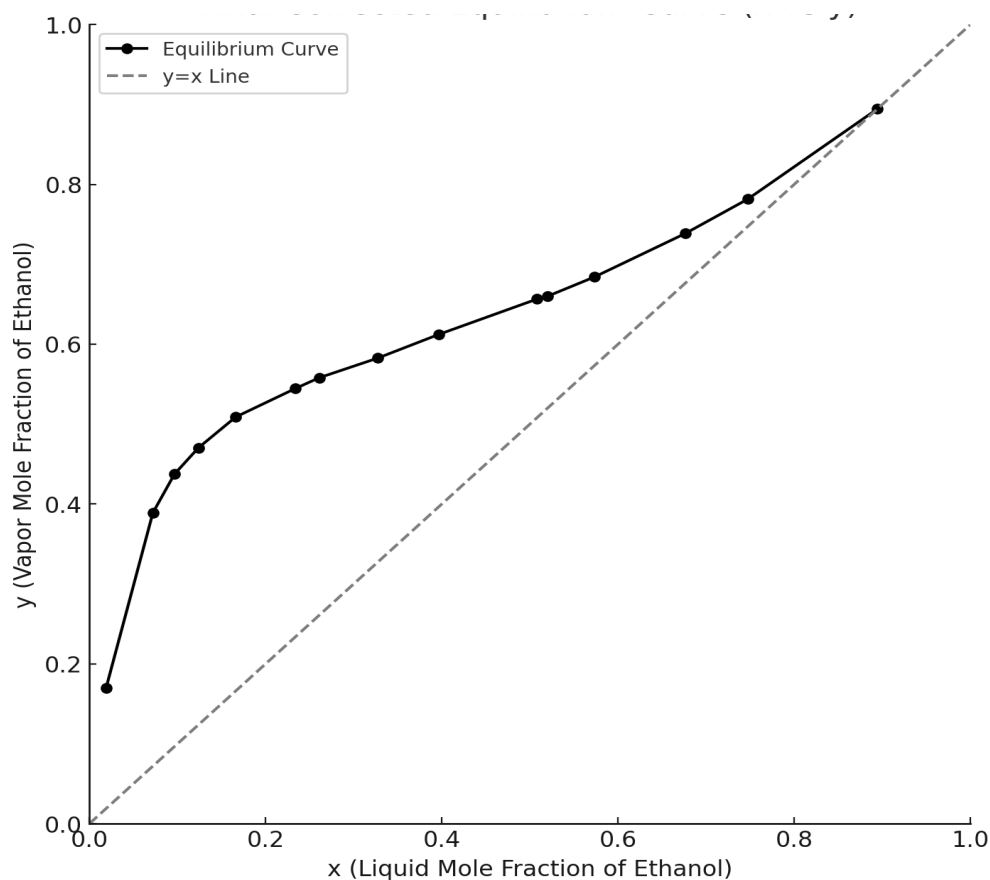
Thermocouple number	Temperature (°C) with offset	Density (g/cc)
6	74.8	0.815
5	77	0.822
4	75.5	0.837
3	77	0.860
2	77.5	0.896

Table 3: Liquid-vapour equilibrium data for ethanol-water at atmospheric pressure

T(°C)	x	y
95.5	0.0190	0.1700
89.0	0.0721	0.3891
86.7	0.0966	0.4375
85.3	0.1238	0.4704
84.1	0.1661	0.5089
82.7	0.2337	0.5445
82.3	0.2608	0.5580
81.5	0.3273	0.5826
80.7	0.3965	0.6122
79.8	0.5079	0.6564
79.7	0.5198	0.6599
79.3	0.5732	0.6841
78.74	0.6763	0.7385
78.41	0.7472	0.7815
78.15	0.8943	0.8943



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Equilibrium data for x vs y

Figure 5: Equilibrium Data

8. Sample Calculations & Results

Molecular weight of water = 18 g/mol

Molecular weight of ethanol = 46.07 g/mol

Density of water = 1 g/ml

Density of ethanol = 0.785 g/ml

Moles of ethanol :

$$n_{ethanol} = \frac{V_{ethanol} \times \rho_{ethanol}}{MW_{ethanol}} \quad (3)$$

Moles of water :

$$n_{water} = \frac{V_{water} \times \rho_{water}}{MW_{water}} \quad (4)$$

Mole fraction of ethanol:

$$x_{ethanol} = \frac{n_{ethanol}}{n_{ethanol} + n_{water}} \quad (5)$$



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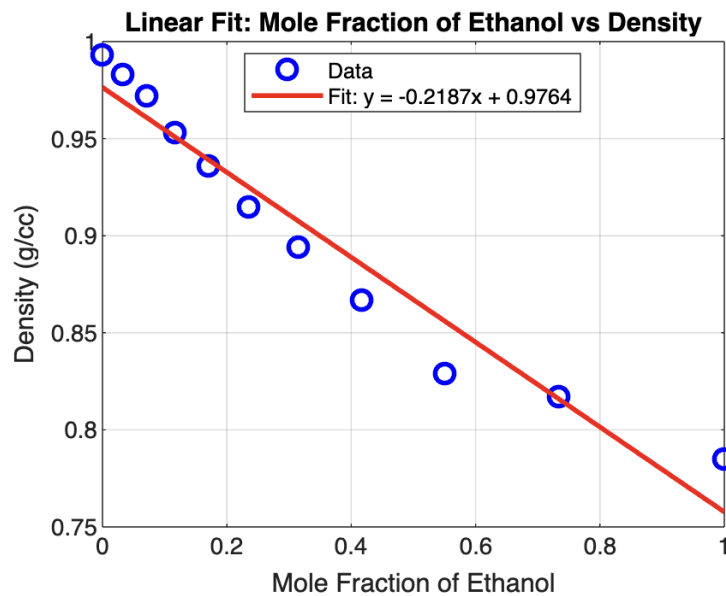


Figure 6: Fitted model for the calibration data

Equation of linear fit of Mole fraction of ethanol vs Density:

$$y = -0.2187x + 0.9764 \quad (6)$$

Sample calculation for Tray no. 1 (Thermocouple 6) : Density of liquid sample collected = 0.815 g/ml

$$y = -0.2187 \times x_1 + 0.9764$$

$$x_1 = \frac{y - 0.9764}{-0.2187} = 0.7379$$

Sample calculation of Murphree efficiency and NTU for Tray no. 1 (Thermocouple 6):

$$E_M = \frac{y_{actual} - y_{inlet}}{y_{equilibrium} - y_{inlet}} \quad (7)$$

$$NTU = \frac{y_{actual} - y_{inlet}}{y_{equilibrium} - y_{actual}} \quad (8)$$

$$y_{actual} = 0.7379$$

$$y_{inlet} = 0.7058 \text{ (from below tray)}$$

Finding $y_{equilibrium}$, using linear interpolation of VLE data.



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$$y = y_1 + \left(\frac{x - x_1}{x_2 - x_1}\right) \times (y_2 - y_1) \quad (9)$$

Where,

- x_1 and x_2 are the closest liquid compositions in the table
- y_1 and y_2 are the corresponding vapor compositions

$$y_{equilibrium} = 0.7759 \text{ (from VLE data)}$$

$$E_M = \frac{0.7379 - 0.7059}{0.7759 - 0.7059} = 0.457$$

$$NTU = \frac{0.7379 - 0.7059}{0.7759 - 0.7379} = 0.842$$

Thermocouple number	Temperature (°C) with offset	Density (g/cc)	Mole fraction of ethanol	$y_{equilibrium}$	E_M	NTU
6	74.8	0.815	0.7379	0.7759	0.457	0.842
5	77	0.822	0.7059	0.7562	0.577	1.362
4	75.5	0.837	0.6374	0.7156	0.574	1.345
3	77	0.860	0.5322	0.6481	0.587	1.420
2	77.5	0.896	0.3676	0.5402	-	-

Table 4: Murphree Tray Efficiency and NTU of corresponding stages

Calculation of ideal stages by McCabe-Thiele method:

Feed is 250 ml ethanol + 350 ml water, at room temperature. The quality factor (q) is calculated as:

$$q = 1 + \frac{C_p(T_{bp} - T_{feed})}{\lambda} \quad (10)$$

$$q = 1 + \frac{2.5 \times (78.37 - 25)}{840} = 1.159$$

Feed composition (x_F) = 0.200

Distillate composition (x_D) = 0.755 (from density vs mole fraction curve)



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Bottom composition (x_w) = 0.05 (assumed)

Assuming a Reflux Ratio (R) of 0.8, the intercept of the enriching line is 0.4.

The rectifying line is drawn from the intersection of the enriching line and q-line to (x_w, x_w)

Applying the McCabe-Thiele Method from the given data:

No. of stages as plotted by McCabe-Thiele method = 4.

Efficiency of the column = (Number of ideal stages/Number of real stages)*100 = 80%

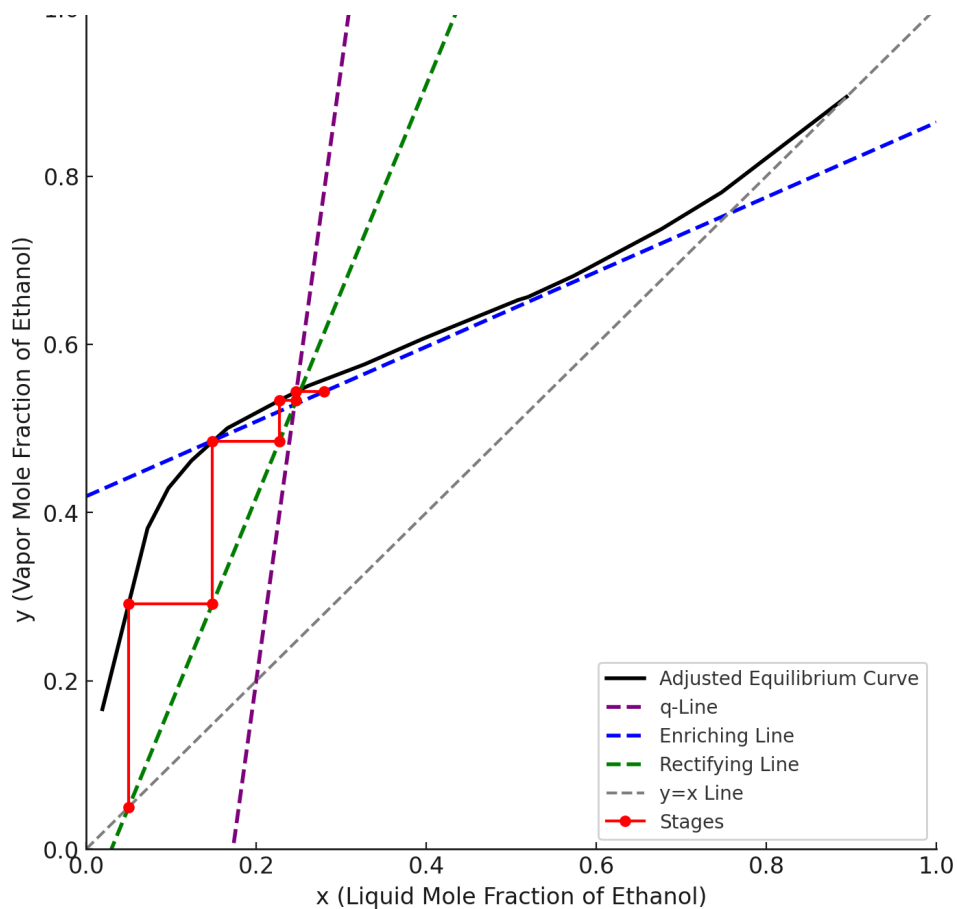


Figure 7: Calculation of ideal stages by McCabe Thiele Method



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9. Discussions & Conclusions

- The overall efficiency of the distillation column was calculated to be approximately 80%, indicating a relatively high performance for separating the ethanol-water mixture.
- Murphree tray efficiencies varied significantly across the column, with the highest efficiency observed in Tray 2 and lower efficiencies in other trays. This suggests non-uniform mass transfer performance throughout the column.
- The experiment demonstrated the practical application of concepts such as the Margules equation for modeling non-ideal vapor-liquid equilibrium and the McCabe-Thiele method for analyzing distillation column performance.
- The McCabe-Thiele method revealed that the column had 4 theoretical stages, which is close to the actual number of 5 physical trays in the experimental setup.
- Also it is to be noted that the thermocouples are not suitably calibrated and that's why the off-set corrected temperature values didn't follow a monotonic trend (table 3).
- Some vapours may escape from the top rather than completely collected and condensed by the condenser at the top. This can have an influence on the equilibrium compositions of collected samples.
- The TDS meter has an inbuilt instrumental error. Besides this, retention of small amounts of previously measured solution with a different density value may cause changes in the next measurement.

10. References

- I. Resource from Moodle.
- II. AMT Course Notes
- III. Chapter-7 Distillation of Binary Mixtures, Separation Process Principles, 3rd Edition, J. D. Seader, Ernest J. Henley, D. Keith Roper, John Wiley Incorporated, 2010
- IV. Equilibrium data: https://checalc.com/solved/binary_vle.html
- V. The [report](#) on McCabe Thiele method.
- VI. [Paper](#) on A general overview of distillation columns and column circuits.



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Data Sheet

Experiment: Plate column distillation

Date: 12/03/2025

Batch: R

Group No: 5

Roll No	Name
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TA Signature:

Anura Achary
12/03/25

CALIBRATION:

$T = 29.6^{\circ}\text{C}$

EtOH (ml)	H ₂ O (ml)	Density (g/cm^3)
10	0	0.785
9	1	0.817
8	2	0.829
7	3	0.867
6	4	0.894
5	5	0.915
4	6	0.936
3	7	0.953
2	8	0.972
1	9	0.984
0	10	0.993

Figure 8: Datasheet containing Lab-Data.



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Thermocouple reading:-						
	time(min)	T ₂	T ₃	T ₄	T ₅	T ₆
	0	29	29	29	29	29
	41	77	76	75	75	73
	43	77	76	75	75	73
	45	77	76	75	75	73
Values with offset		77.5°C	77°C	75.5°C	77°C	74.8°C
Density measures:-						
Column	Thermocouple no	density (g/cm ³)				
5	6	0.815				
4	5	0.822				
3	4	0.837				
2	3	0.860				
1	2	0.896				

Figure 9: Datasheet containing Lab-Data.