

# Momentum Transfer and Mechanical Operations Lab

## Batch Distillation

30th September, 2024

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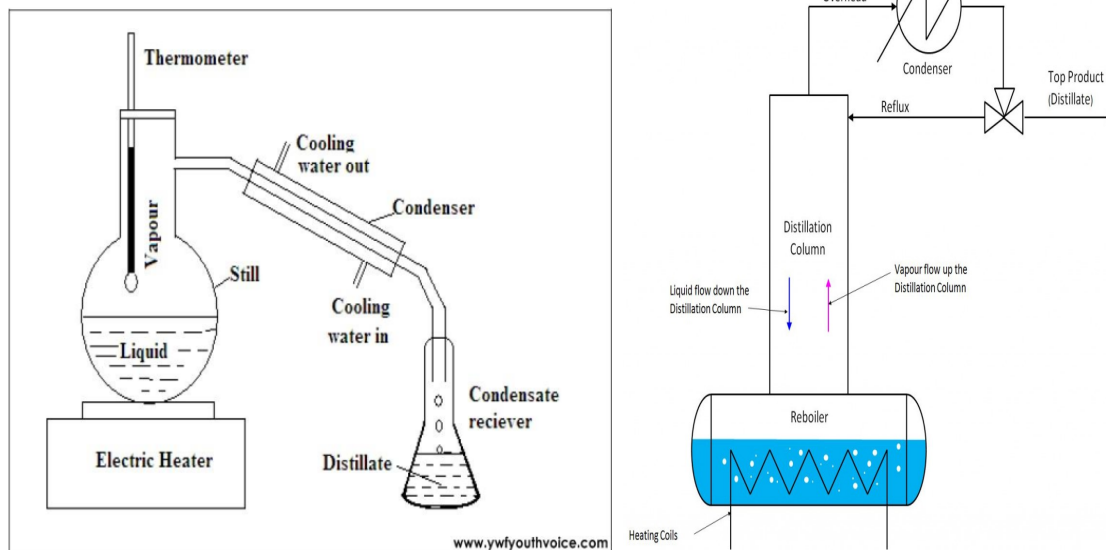
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## 1 Abstract with Graphics

Batch distillation (schematically shown in the following figures) is a common method used in chemical engineering to separate liquid mixtures based on the differing volatilities of their components. Unlike continuous distillation, batch distillation operates in discrete cycles, with mixtures placed in a reboiler and heated until the most volatile components vaporise. As the vapour rises through the distillation column, it is partially condensed and collected as distillate.

In this batch distillation experiment, the objective was to validate Rayleigh's Law and determine the equilibrium line for a binary mixture of ethanol and water by analysing data obtained during the distillation process. The experiment aimed to collect the distillate and analyse its composition, comparing it with the residue left in the reboiler after a significant portion of the mixture had been distilled.

The experiment involved charging a two-necked round bottom flask with a mixture of 200ml of water and 400ml of ethanol, gradually heating it, and collecting the distillate. The temperature at which the first drop of distillate appeared was recorded as the dew point, and the distillation continued until approximately three-fourths of the initial mixture volume had been distilled. The refractive indices of the distillate and residue were



measured to determine their ethanol concentrations. A calibration curve was created by preparing mixtures of water and ethanol with varying volume fractions and measuring their refractive indices. The experimental data was used to plot the equilibrium line and verify Rayleigh's equation, which describes the relationship between the amount of liquid remaining in the reboiler and the composition of the distillate over time.

The results showed that the distillation process efficiently separated the components, and the refractive index measurements accurately reflected the ethanol concentration in the mixture. However, potential sources of error included parallax error in volume measurements, leakage during sample collection, and thermo-couple calibration drift.

## 2 Aim & Objectives

- To obtain equilibrium data for ethanol-water from literature.
- To plot the volume calibration data and to use it for calculating volume fraction.
- To validate Rayleigh equation,  $\ln\left(\frac{L_1}{L_2}\right) = \int_{x_2}^{x_1} \frac{dx}{(y^* - x)}$ .

## 3 Background and Motivation

Batch distillation (figure 2) is a crucial separation process widely used in industries such as pharmaceuticals, petrochemicals, and fine chemicals, particularly for small-scale or

flexible production. Unlike continuous distillation, batch distillation operates in discrete cycles, making it ideal for processes where feed composition changes between batches or where high-purity products are required. The process separates components of a liquid mixture based on differences in their boiling points, with the more volatile components vaporizing first and being collected as distillate, while the less volatile components remain in the liquid phase.

The Rayleigh equation, which describes the relationship between the vapor and liquid compositions in ideal binary mixtures, plays a vital role in understanding and controlling the batch distillation process. It helps predict how the composition of the liquid and vapor phases evolve during distillation, providing a theoretical basis for optimizing the separation.

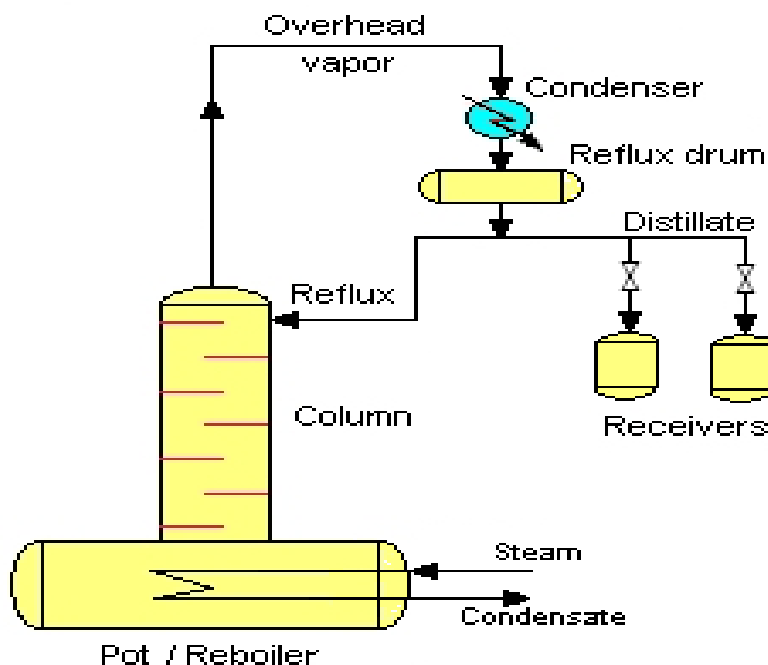


Figure 2: Overall Representation of the workflow of Batch Distillation

The motivation for studying batch distillation lies in its broad industrial applications and its potential for improving product purity, reducing energy consumption, and minimizing waste. As demand grows for more efficient and sustainable separation techniques, understanding the fundamental principles of batch distillation becomes increasingly important. This experiment provides an opportunity to explore these principles in practice, offering insights into the design, operation, and optimization of batch distillation systems for real-world applications. By mastering this process, one can make informed decisions about the best separation method for specific industrial needs, enhancing both economic and environmental sustainability.

## 4 Materials and Methods

### 4.1 Apparatus & Materials Required

- **Materials:** Water (with ethanol as a mixture and also used as cooling Water Inlet and Outlet), Ethanol.
- **Apparatus:** Still (Distillation Flask), Electric Heater, Condenser, Thermometer, Condenser Receiver, Rubber Tubing, Funnel, stand with Clamps.

### 4.2 Experimental Setup Description

The experimental setup is shown in the figure 3.

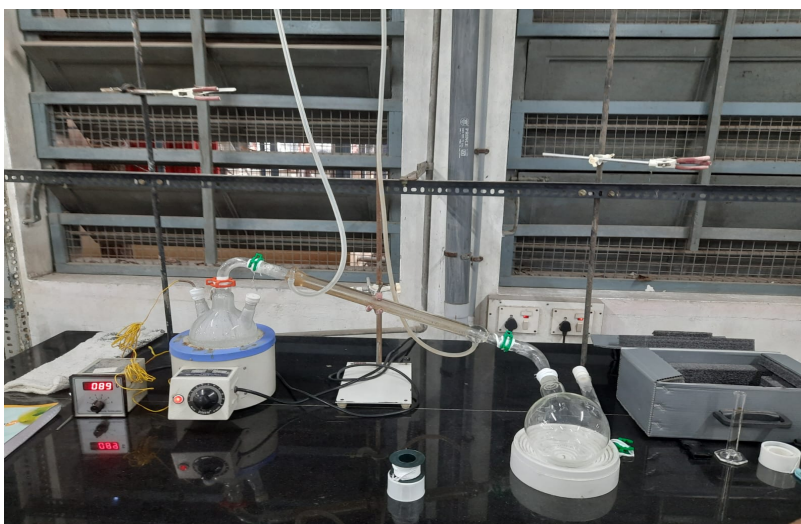


Figure 3: Experimental Set-up of the Batch Distillation Experiment

- **Distillation Flask Setup:** The still flask containing the liquid mixture to be distilled is placed on the electric heater.
- **Thermometer Placement:** The thermometer is placed at the neck of the still flask to measure the vapor temperature.
- **Condenser Attachment:** The condenser is connected to the vapor outlet of the still flask. Coolant water flows through the condenser's jacket from the inlet to the outlet.
- **Distillate Collection:** The condensed vapor (now liquid) flows into the condenser receiver flask.

- **Heating:** The electric heater (shown in figure 4) is turned on to gradually heat the liquid mixture in the still flask until it starts boiling and producing vapor.
- **Cooling Water Flow:** Water is circulated through the condenser to cool and condense the vapor into liquid distillate.
- **Collection of Distillate:** The distilled liquid is collected in the receiver flask, and the process is monitored and controlled using the thermometer readings.

This setup ensures that the vapor is condensed back into a liquid efficiently and that the distillation process is conducted safely.

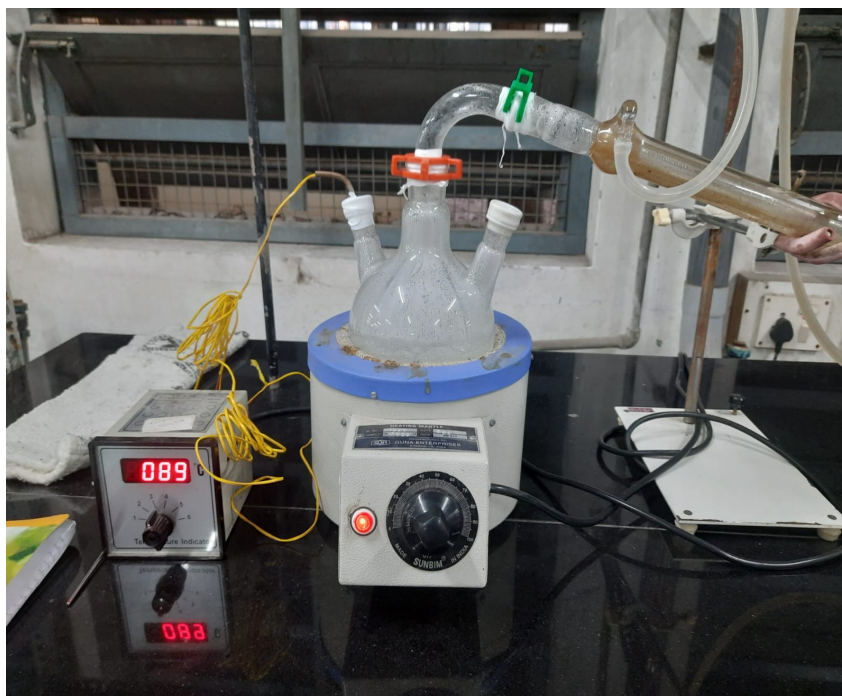


Figure 4: Heater and Thermometer in the Experimental Set-up

### 4.3 Procedure

- Pour 300 mL of water and 300 mL of ethanol into a beaker and mix thoroughly using a glass rod. Transfer the ethanol-water solution into a three-necked round bottom flask. Ensure the condenser is set up with running water flowing through it.
- Gradually increase the temperature of the heating mantle, monitoring the mixture's temperature using a thermo-couple.

- As the temperature rises, the mixture will begin to vaporize. The first drop of distillate will appear, with the temperature recorded at the dew point.
- Continue the distillation process, collecting the distillate in the receiving flask until approximately three-quarters of the original solution volume has been distilled.
- Allow the distillate and the remaining residue to cool to room temperature. Once cooled, measure the density and volume of each.
- Use a densitometer to accurately determine the density of the collected distillate.
- Prepare mixtures of water and ethanol with ethanol volume fractions of 0, 0.2, 0.4, 0.6, 0.8, and 1, each having a total volume of 10 mL.
- Measure the density of each prepared mixture to create a calibration curve, which will be used to analyze the composition of both the distillate and the residue.

## 5 Observation Tables

The tabulations include the observed data of the experiments and are tabulated in the following table 1;

Volume of Ethanol (mL)	Volume of Water (mL)	Volume Fraction of Ethanol	Density of mixture ( $g/cm^3$ )
10	0	1.0	0.784
8	2	0.8	0.844
6	4	0.6	0.900
4	6	0.4	0.936
2	8	0.2	0.995
0	10	0.0	0.997

Table 1: Volume Calibration data for Ethanol-Water mixture

Rest of the important data observed from the onset experiment are in the **Appendix** section uploaded as **Lab Data**.

## 6 Results & Conclusions

This section includes all the results and conclusions obtained using the Lab Data and using some of the theoretical equations to meet all the aim and objectives of this report.

## 6.1 Calculating Van-Laar paramters

To obtain the equilibrium curve of the Ethanol-Water solution, we fit the data obtained from one paper into the Van-Laar equation to calculate the model parameters  $A_{12}$  and  $A_{21}$ . Then we check the model accuracy by using it on the data of another paper.

So, the Van-Laar equation can be written as :

$$\ln(\gamma_1) = A_{12} \left( 1 + \frac{A_{12}x_1}{A_{21}x_2} \right)^{-2} \quad (1)$$

$$\ln(\gamma_2) = A_{21} \left( 1 + \frac{A_{21}x_2}{A_{12}x_1} \right)^{-2} \quad (2)$$

We used optimization functions in MATLAB to calculate the parameters by minimizing the following equations :

$$F_1(A_{12}, A_{21}) = \text{norm} \left( \gamma_1 - \frac{y_1 P}{x_1 P_1^{sat}} \right) \quad (3)$$

$$F_2(A_{12}, A_{21}) = \text{norm} \left( \gamma_2 - \frac{y_2 P}{x_2 P_2^{sat}} \right) \quad (4)$$

Here, we  $x_1$  and  $y_1$  are the mole fractions of Ethanol in Liquid and Vapor phase respectively. The T-x-y values were taken from the paper (Hadrich, 2009).

The values of  $P^{sat}$  were calculated using the Antoine equation :

$$\log_{10} P^{sat} = A - \frac{B}{T + C} \quad (5)$$

The values of A, B and C were taken as :

Compound	A	B	C
Ethanol	4.92531	1432.526	-61.819
Water	4.6543	1435.264	-64.848

Table 2: Antoine Equation parameter values

After running the MATLAB scripts, we obtain the following values :

$$A_{12} = 1.730296 \quad (6)$$

$$A_{21} = 0.9939354 \quad (7)$$

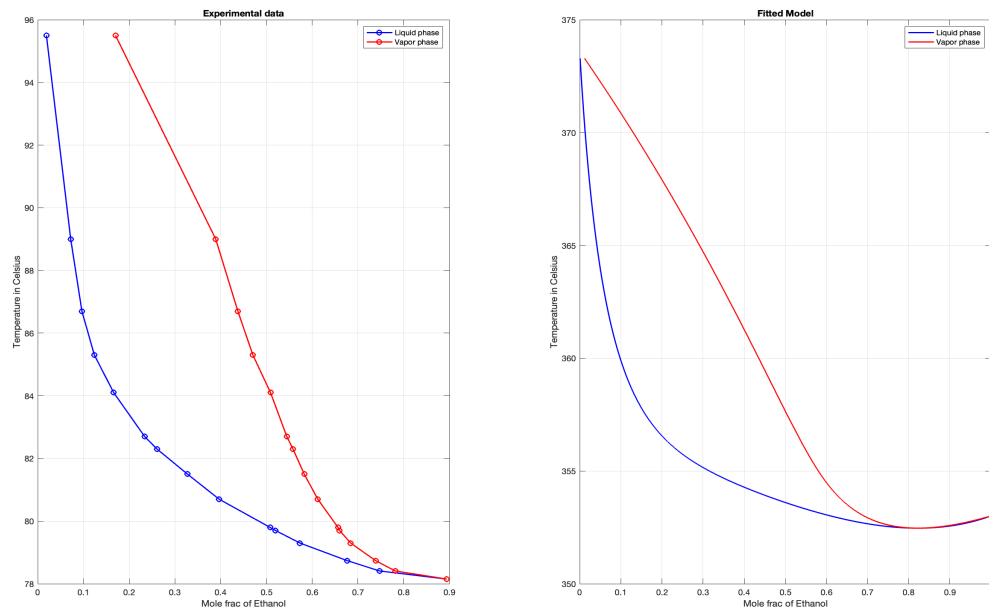


Figure 5: T-x-y plot of Ethanol-Water Solution

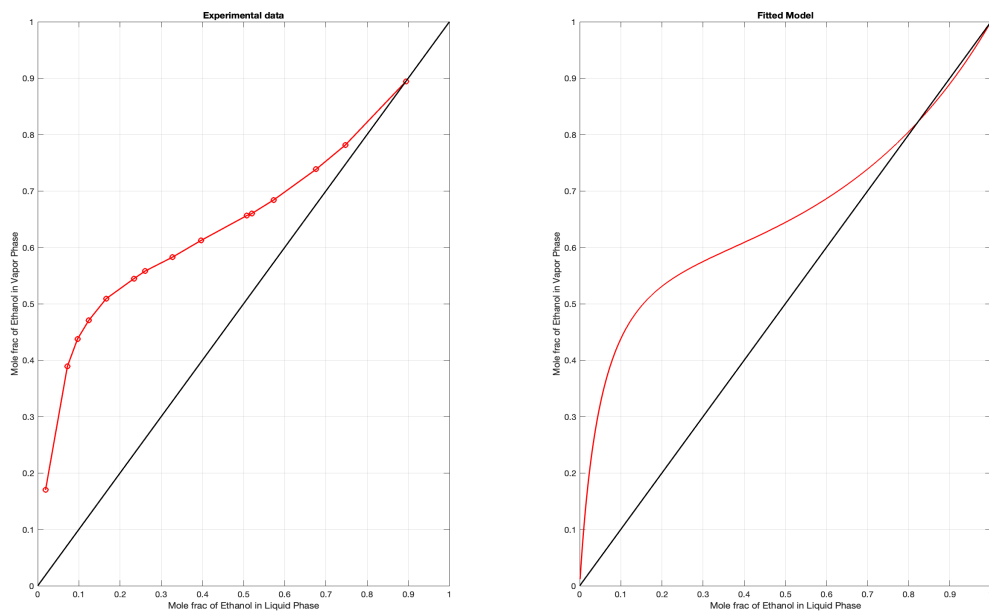


Figure 6: x-y plot of Ethanol-Water Solution



## 6.2 Checking Model Accuracy

Now, we check the accuracy of our fitted Van-Laar parameters by testing them on the VLE data obtained from another paper (Cornell, 1933). We obtain the following results :

$$SS_{res} \equiv \sum (y_i - \hat{y}_i)^2 = 0.004348 \quad (8)$$

$$R^2 = 0.997784 \quad (9)$$

We observe that the  $R^2$  value is larger than 0.995, meaning that our model is very accurate and can be used in further calculations.

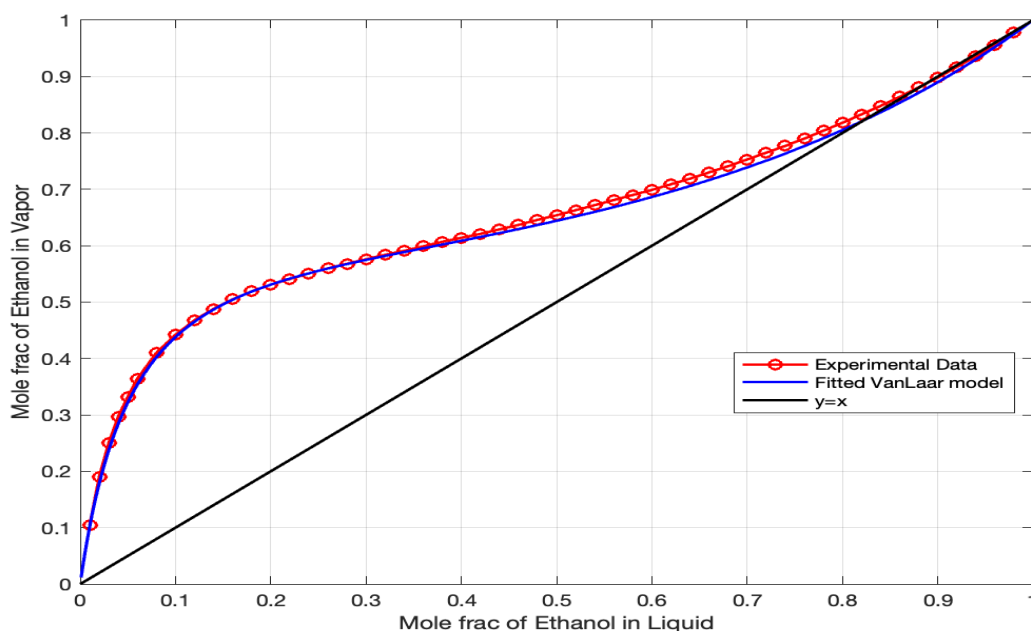


Figure 7: Plot of Experimental Data from Cornell, 1933 and Fitted Van-Laar Model

## 6.3 Density Calibration

We plot the density of Ethanol-Water solution at different mole fractions obtained from experiment, and obtained from literature data.

As observed in this graph, there is one outlier ( $x = 0.2$ ,  $\rho = 0.995$ ) in the experimental values. After removing it, we get the following fitted equation of Mixture Density ( $\rho_{mix}$ ) in terms of Ethanol mole fraction ( $x$ ):

$$\rho_{mix} = 0.1343x^2 - 0.3454x + 0.9939 \quad (10)$$

$$R^2 = 0.999878 \quad (11)$$

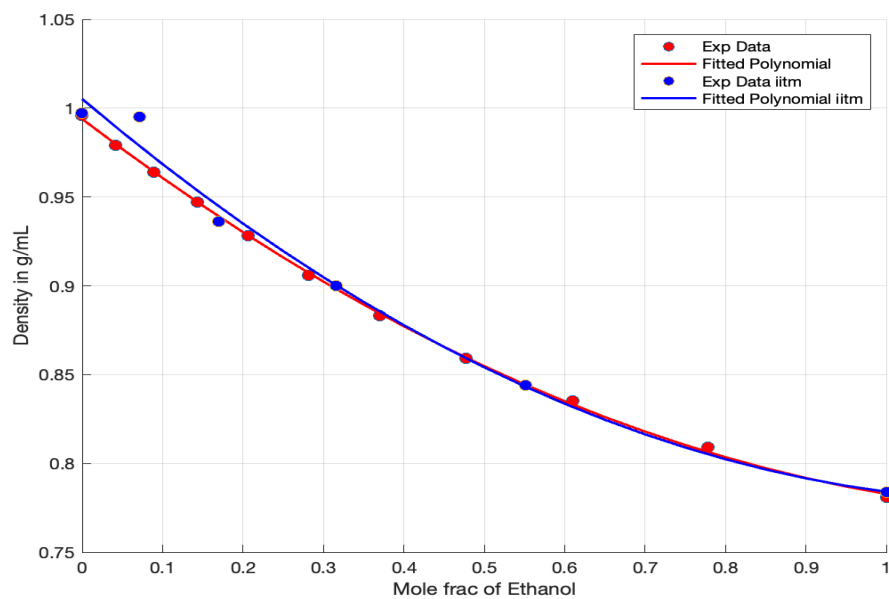


Figure 8: Plot of Density values (one outlier)

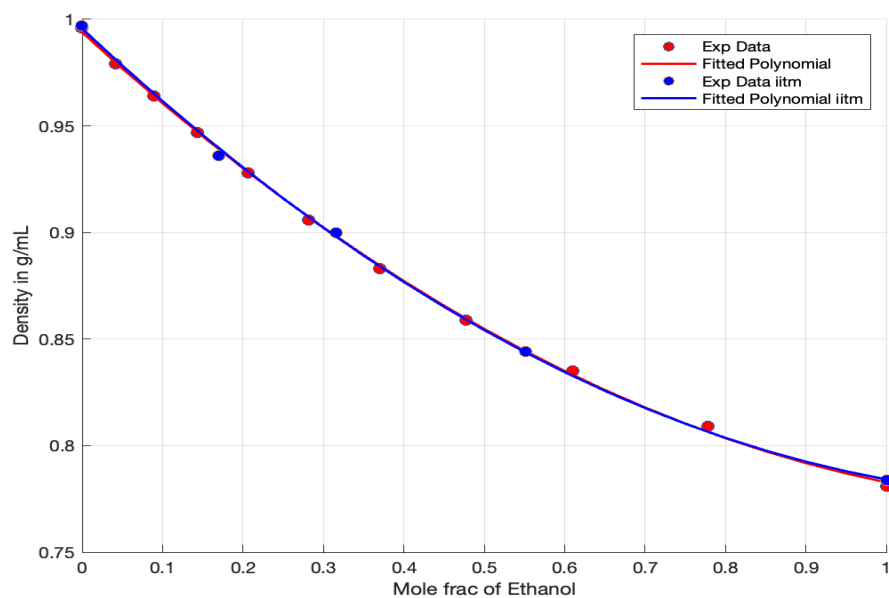


Figure 9: Plot of Density values (zero outliers)

## 6.4 Initial and Final Values

The following are the values of variables corresponding to Still Volume in the flask at the start of the experiment :

Compound	Mass (g)	Volume (mL)	Density (g/mL)	Moles (mol)	Mole Fraction
Ethanol	156.56	200	0.7828	3.3985	0.1334
Water	397.55	400	0.9939	22.0679	0.8666
Total	554.11	583.16	0.9502	25.4664	-

Table 3: Initial Values of Still Volume

The following are the values of variables corresponding to Residual Volume in the flask at the end of the experiment :

Compound	Mass (g)	Volume (mL)	Density (g/mL)	Moles (mol)	Mole Fraction
Ethanol	15.68	20.03	0.7828	0.3404	0.0180
Water	333.96	336.02	0.9939	18.5381	0.9820
Total	349.64	354	0.9877	18.8785	-

Table 4: Final Values of Residual Volume

Finally, the given below values are of variables corresponding to the Distillate condensed into another beaker :

Compound	Mass (g)	Volume (mL)	Density (g/mL)	Moles (mol)	Mole Fraction
Ethanol	117.03	149.50	0.7828	2.5404	0.3988
Water	69.00	69.42	0.9939	3.8300	0.6012
Total	186.03	212	0.8775	6.3704	-

Table 5: Final Values of Distillate

## 6.5 Checking Mass Conservation

$$Initial : M_i = 554.11 \text{ grams} \quad (12)$$

$$Final : M_f = 349.64 + 186.03 = 535.67 \text{ grams} \quad (13)$$

$$Error : \left| \frac{M_f - M_i}{M_i} \right| \cdot 100\% = 3.33\% \quad (14)$$

## 6.6 Checking Rayleigh's equation

$$LHS : \ln \left( \frac{N_i}{N_f} \right) = 0.2993 \quad (15)$$

$$RHS : \int_{x_f}^{x_i} \frac{1}{y^* - x} dx = 0.4082 \quad (16)$$

$$Theoretical \hat{N}_f = \frac{N_{initial}}{\exp(RHS)} = 16.9308 \quad (17)$$

$$Error : \left| \frac{N_f - \hat{N}_f}{\hat{N}_f} \right| \cdot 100\% = 11.50\% \quad (18)$$

## 6.7 Remarks

- It is seen that the batch distillation on Ethanol-Water solution gives good results due to the large difference between their boiling points;  $T_{ethanol} = 78^\circ C$  and  $T_{water} = 100^\circ C$ .
- It is observed that the temperature at which saturated vapor starts to condense (Dew Point) is at about  $87^\circ C$ .
- The temperature at which liquid starts to vaporize in the flask (Bubble Point) is about  $79^\circ C$ .
- Considering that the error calculated while checking Rayleigh equation is quite low, we can conclude that the equation does hold true for batch distillation.

## 7 Error Analysis

Least Count of (large) measuring cylinder  $\equiv \Delta V_L = 2 \text{ mL}$

Least Count of (small) measuring cylinder  $\equiv \Delta V_S = 0.1 \text{ mL}$

Least Count of Stopwatch  $\equiv \Delta t = 0.01 \text{ sec}$

Least Count of Densitometer  $\equiv \Delta \rho = 0.0001 \text{ g/mL}$

Error in Temperature  $\equiv \Delta T = 1 \text{ K}$

Error in Generated Heat  $\equiv \Delta V_M = 1 \text{ Watt}$

### 7.1 Sources of Error

- Leakage of saturated vapor through gaps or holes.
- Reading density at a higher temperature than the temperature at which density calibration was performed.

- Presence of Outliers in data collected from experiment.
- Human error (spilling the solution over the table, misreading the value shown in densitometer, ignoring effects of parallax, etc.)

## 8 Precautions

- Verify that all components of the distillation apparatus, including the three-necked flask, condenser, and receiving flask, are properly assembled and tightly sealed to prevent leaks or loss of vapor.
- Ensure the thermocouple is correctly positioned to accurately measure the temperature of the vapor phase, as this is critical for validating Rayleigh's equation.
- Maintain a consistent flow of cooling water through the condenser to efficiently condense the vapor, preventing the loss of ethanol vapor into the surroundings.
- Conduct the experiment in a well-ventilated area or under a fume hood to avoid the buildup of ethanol vapor, which is flammable and can be hazardous when inhaled in high concentrations.
- Regularly check for pressure buildup in the system, especially in the round-bottom flask and condenser, to ensure safe operation during the distillation process.
- Do not fill the distillation flask above two-thirds of its capacity to prevent splashing of the liquid into the condenser or uneven boiling.
- Ensure that all measuring instruments, such as thermocouples and density meters, are properly calibrated before starting the experiment for precise measurements.

## 9 Thought Question / Open-Ended

**Q.** Can you distill crude oil into petroleum products using batch distillation? If yes, how? If no, then what are the limitations? How will the Rayleigh Equation change in case of multi-component distillation?

**A.** (**Note.** Two papers were taken as a reference before answering the open-ended question and they are mentioned in the answer as per their usage arrives and also referred in **Reference** section)

Batch distillation can technically be used to separate crude oil into different petroleum fractions, but it is not the preferred method for crude oil processing in industrial settings. Instead, continuous distillation is used in refineries for this purpose. Below are the reasons why batch distillation is not ideal for distilling crude oil and what limitations arise:

### 1. Limitations of Batch Distillation for Crude Oil Processing:

- Crude oil contains a very wide range of hydrocarbons with varying boiling points, from gases to heavy residues. This makes it impractical to separate all components efficiently in a batch distillation setup, which works better for simpler mixtures.
- Crude oil components require precise temperature control across a broad range of boiling points. Heating crude oil in a batch distillation would demand frequent adjustments in temperature, making the process inefficient.
- In batch distillation, the oil is heated and kept at high temperatures for extended periods. This prolonged heating can cause thermal cracking or decomposition of heavier components, leading to undesirable products.
- Large-scale processing of crude oil requires continuous operation to handle the high volume of crude. Batch distillation is typically done in smaller quantities, making it impractical for industrial-scale separation.

### 2. Continuous Fractional Distillation is Used:

- They operate without interruption, allowing for the processing of large quantities of crude oil.
- They enable the separation of various fractions (e.g., gasoline, kerosene, diesel) based on their boiling points through multiple stages of condensation and vaporization within the column.
- The heat used in continuous distillation is better managed and recycled within the system, reducing overall energy consumption and continuous fractional distillation produces petroleum products of consistent quality because of the better control over the separation process.

“*Simulation and Optimization of Crude Oil Distillation Unit*” (Nasiri et al., 2008) explain how continuous fractionating columns are optimized for energy efficiency and yield, showing the advantages over batch distillation.

### Changes in the Rayleigh Equation in case of multi-component distillation:

The derivation of Rayleigh Equation says that the equations are valid for multi-component mixtures (discussed in section **9.7 Multicomponent Simple Batch Distillation** in the book “*Separation Process Engineering: Includes Mass Transfer Analysis, 4th Edition*”). However, since the general equilibrium expression

$$y_i = f(x_1, x_2, \dots, x_C, T, p) \quad (19)$$

depends on the concentration of all components. And for batch distillation the equilibrium expression,

$$y_j = \frac{x_j \alpha_{j-ref}}{\sum_{i=1}^C (x_i \alpha_{i-ref})} \quad (20)$$

The Rayleigh equation for the component A becomes,

$$\ln\left(\frac{L_1}{L_2}\right) = - \int_{x_{final}}^{x_0} \frac{dx}{y_A - x_A} = - \int_{x_{final}}^{x_0} \frac{dx}{\frac{x_A \alpha_{A-ref}}{\sum_{i=1}^C (x_i \alpha_{i-ref})} - x_A} \quad (21)$$

## 10 Acknowledgements

We as a group contributed our respective parts into completing the above report on Batch Distillation.

In terms of specifications, Atharva Sunilkumar Ghodke contributed in "Procedure" & "Precaution" parts. Anomol Upadhyay delivered the content for "Aim (Objective)"; "Background & Motivation" along with Lakkireddy Vishnu Vardhan Reddy helping in "Abstract" part of the report and rest of all the parts including calculations are collaboratively done & organized by Deepanjhan Das (general editor) & Aayush Bhakna (proof reader).

Regarding AI transcript for the open-ended thought question asked, we didn't use ChatGpt for our thought question. It was more confusing and so we, after discussing the scenario and after reading some related papers, we wrote as per our understanding. Therefore no such transcript is provided in the **Appendix** section.

And at last but not the least, we specially thank the respective TA for this experiment for her kind help and to let us have a thorough understanding of the whole process and the concept. We thank all the course instructors for their effective control and high co-operation as per the need.

## References

- Notes from the course CH5140 : Process Modelling Simulation and Analysis by Prof Renganathan
- Notes from the course CH3030 : Applied Mass Transfer by Prof Ethayaraja
- Cornell, 1933 | Liquid-Vapor Equilibria in systems Ethanol-Water, Methanol-Water and Acetic Acid-Water | University of Minnesota  
[https://drive.google.com/file/d/1lf5WHVYy38Lte2laCpVks43LZcnWicT5/view?usp=share\\_link](https://drive.google.com/file/d/1lf5WHVYy38Lte2laCpVks43LZcnWicT5/view?usp=share_link)

- Hadrich, 2009 | Identification of the Best Model and Parameters for T-X-Y equilibrium data of Ethanol-Water Mixture | Imam Muhammad bin Saud Islamic University  
[https://drive.google.com/file/d/1wr89ulSt0FU0xOwHhRJIU0PxRilWxd9A/view?usp=share\\_link](https://drive.google.com/file/d/1wr89ulSt0FU0xOwHhRJIU0PxRilWxd9A/view?usp=share_link)
- Properties of Water (Antoine parameters, density, molecular weight, etc)  
<https://webbook.nist.gov/cgi/cbook.cgi?ID=C7732185&Mask=4&Type=ANTOINE&Plot=on>
- Properties of Ethanol (Antoine parameters, density, molecular weight, etc)  
<https://webbook.nist.gov/cgi/cbook.cgi?ID=C64175&Mask=4#>
- Density of Ethanol-Water Solution at different weight% at 30°C  
[https://www.engineeringtoolbox.com/ethanol-water-mixture-density-d\\_2162.html](https://www.engineeringtoolbox.com/ethanol-water-mixture-density-d_2162.html)
- “Simulation and Optimization of Crude Oil Distillation Unit” (Nasiri et al., 2008)  
<https://dergipark.org.tr/en/download/article-file/721742>
- “Separation Process Engineering: Includes Mass Transfer Analysis, 4th Edition”  
[https://www.informit.com/articles/article.aspx?p=2738308&seqNum=8#:~:text=The%20profiles%20of%20xi,numerically%20integrate%20the%20Rayleigh%20equation.&text=where%20%CE%94xA%20%3D%20x,%E2%80%93%20xA%5D\)k%5D](https://www.informit.com/articles/article.aspx?p=2738308&seqNum=8#:~:text=The%20profiles%20of%20xi,numerically%20integrate%20the%20Rayleigh%20equation.&text=where%20%CE%94xA%20%3D%20x,%E2%80%93%20xA%5D)k%5D)

## Appendix

**Lab Data:** All the experimental observations of the main experiment that was performed and tabulated during the laboratory session are included in order in the following (in figures 10, 11, 12 & 13).

**Reference to all the contents:** The official GitHub repository which contains all the related data and coded scripts for calculations is also provided below: [https://github.com/deep183Das/CH3510\\_MTMO\\_Lab\\_Group\\_2/tree/main/Experiment\\_7](https://github.com/deep183Das/CH3510_MTMO_Lab_Group_2/tree/main/Experiment_7). One can easily refer to all the related lab resources from this GitHub repository from where screenshots of few instances are shown in the above figures, in in this report.



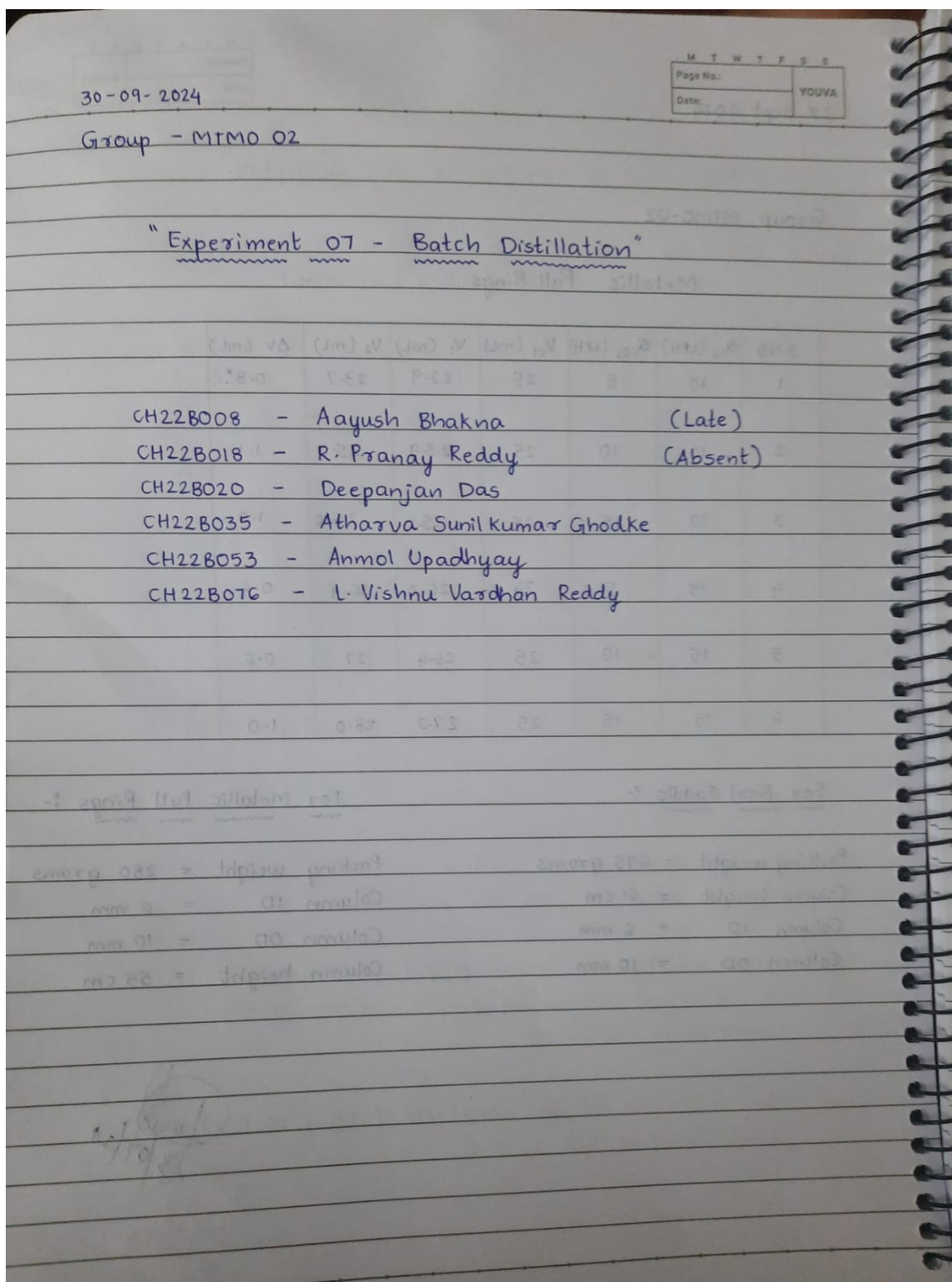


Figure 10: Group Members

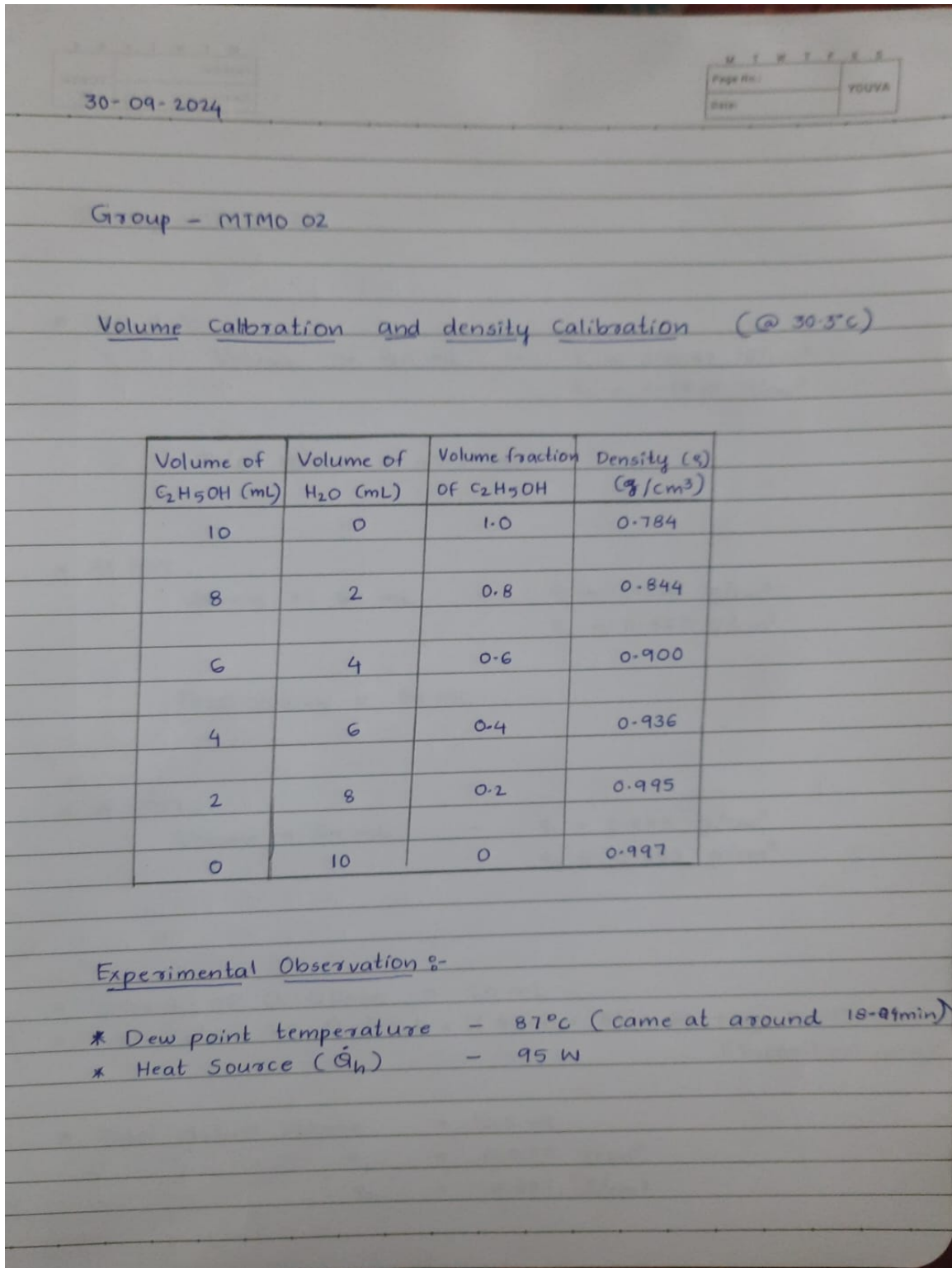


Figure 11: Volume Calibration Data

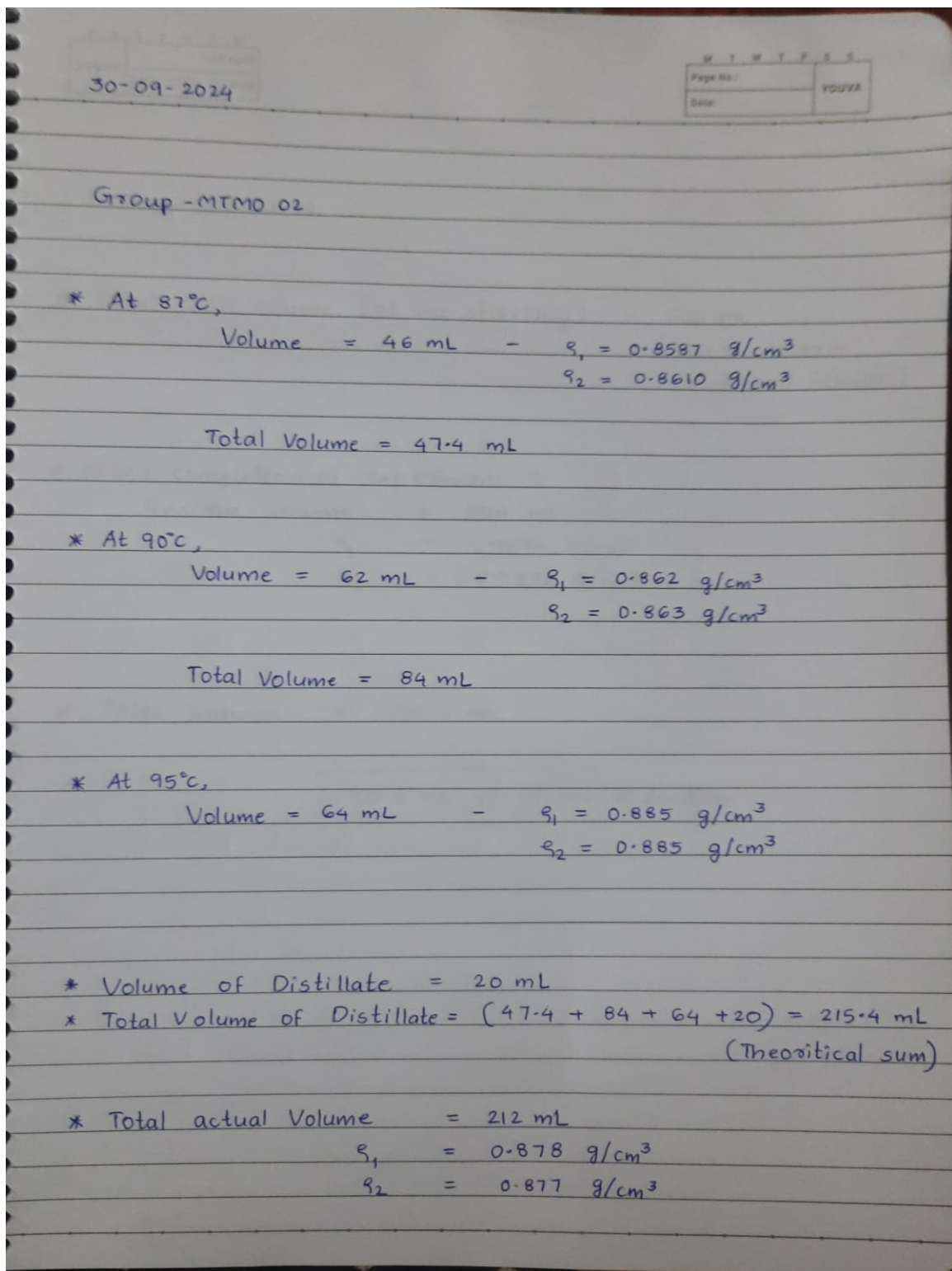


Figure 12: Batch Distillation Data - 1

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\* Total Still Volume (at the starting) = 600 mL  
(400 mL water +  
200 mL  $C_2H_5OH$ )

\* After completion of experiment :-  
Residue Volume = 354 mL  
 $\rho_1 = 0.9878 \text{ g/cm}^3$   
 $\rho_2 = 0.9876 \text{ g/cm}^3$

\* Total Volume = 566.4 mL

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33.6 mL of Volume reduction

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Figure 13: Batch Distillation Data - 2