Momentum Transfer and Mechanical Operations Lab

Solid Dissolution

7th October, 2024

Team: MTMO 2

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1 Abstract with Graphics

The “Solid Dissolution" experiment (schematically shown in the following figure 1) explores the rate at which a solid substance dissolves in a solvent under different conditions. The primary objective of the experiment is to analyse the effects of various parameters, such as temperature, agitation speed, and particle size, on the dissolution rate of a solid material. By understanding these factors, the study aims to develop insights into optimising industrial processes, such as pharmaceutical tablet formulation or chemical reactions in solution.

In this experiment, a cylindrical solid of benzoic acid was submerged in a sodium hydroxide (NaOH) solution, and the dissolution was monitored over time. The initial and final heights of the benzoic acid cylinder were recorded, along with the volume of NaOH consumed at specific time intervals. The concentration of benzoic acid was calculated, and the mass transfer coefficient was derived from the experimental data. The results indicated that higher temperatures significantly enhance the dissolution rate, aligning with the theoretical understanding that increased temperature promotes solubility and mass transfer.

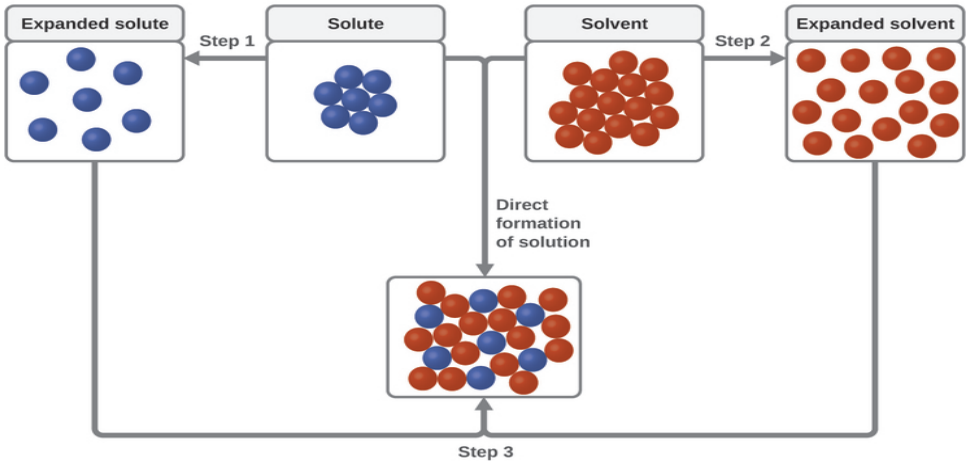

Figure 1

The experiment highlights the importance of factors such as increased temperature and agitation, which generally accelerate dissolution by enhancing molecular motion and reducing boundary layer resistance. Smaller particle sizes also lead to faster dissolution due to higher surface area-to-volume ratios. This knowledge is valuable for designing efficient systems where controlled dissolution is critical. Overall, the study successfully demonstrated the relationship between the physical parameters and the dissolution kinetics of benzoic acid, offering valuable data for future research in this field.

2 Aim & Objectives

- Validation of the Mass Transfer Equations.
- By examining the dissolution of Benzoic Acid in aqueous phase we aim to calculate the Mass Transfer Coefficients.
- To plot $ln(\frac{C^*-C}{C^*-C_0})$ vs. t and to find the mass transfer coefficient (k_l) by fitting the curve with a regression line.
- Validating the process of obtaining k_l by this plot using proper equations relating change in concentration of Benzoic Acid with time (t).

3 Background and Motivation

Solid dissolution (consisting general steps, shown in figure 2) is a fundamental process with significant applications across various fields, including pharmaceuticals, chemistry, environmental science, and material engineering. The rate at which a solid solute dissolves

in a solvent plays a critical role in determining the effectiveness of numerous chemical reactions, drug delivery mechanisms, and even environmental phenomena like pollutant dispersion in water bodies.

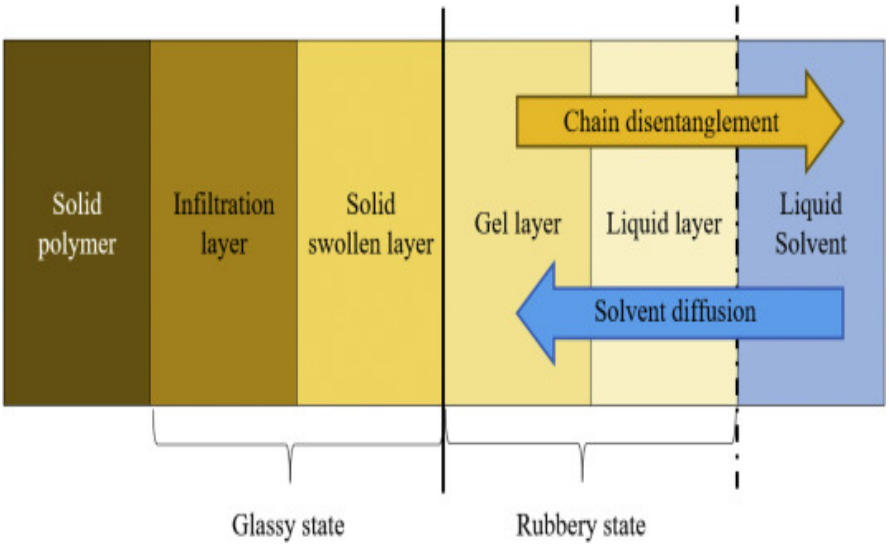

Figure 2: General Solid Dissolution Steps

Understanding the factors that influence the dissolution kinetics, such as solute properties (e.g., particle size, crystal structure), solvent characteristics (e.g., polarity, viscosity), and external conditions (e.g., temperature, agitation), is crucial for optimizing the dissolution process. For instance, in pharmaceuticals, controlling the dissolution rate is vital for drug bio-availability and efficacy, while in industrial processes, it is important for efficient material utilization and product consistency.

The motivation behind studying solid dissolution lies in its practical implications: improving the predictability and control of dissolution rates can lead to more efficient drug formulations, better environmental management, and optimized industrial processes. This experiment seeks to explore the interplay of various factors that affect the dissolution process, providing deeper insight into optimizing this essential chemical phenomenon.

4 Materials and Methods

4.1 Apparatus & Materials Required

- **Materials**: Benzoic Acid Cylinder (figure 3), 0.05N Sodium Hydroxide (NaOH) Solution, Phenolphthalein, Distilled Water.
- **Apparatus**: Batch Extractor, Motor-Controller Setup, Vernier Calipers, Burette, Pipette, Measuring Cylinder, Conical Flask, Beakers, Stopwatch.

Figure 3: Benzoic Acid Cylinder

4.2 Experimental Setup Description

- **Batch Extractor**: A batch extractor (figure 4) is used as the primary vessel for dissolving the benzoic acid in water, equipped with a motor-controller setup to rotate the benzoic acid cylinder at specified speeds.

Figure 4: Batch Extractor

- **Motor-Controller Setup**: The motor-controller is used to rotate the benzoic acid cylinder at different speeds (400 rpm and 900 rpm), allowing for control of the dissolution process under various conditions.
- **Vernier Callipers**: Vernier callipers are employed to accurately measure the initial and final dimensions (diameter and height) of the benzoic acid cylinder before and after the experiment.
- **Heating System**: The water in the cavity is heated to specific temperatures (50^oC and 60^oC) to observe the effect of temperature on the dissolution rate. The setup includes a temperature controller to maintain the required temperature.
- **Titration Setup**: A burette, pipette, and conical flask are set up for titration (figure 5). The benzoic acid solution is sampled at intervals and titrated with NaOH solution using phenolphthalein indicator to measure the concentration of benzoic acid dissolved in the water.

Figure 5: Titration Setup

- **Sampling and Measurement**: A stopwatch is used to time the rotation intervals.
- **Beakers and Measuring Cylinder**: Beakers and a measuring cylinder are used for handling and preparing solutions, such as the NaOH titrant and the benzoic acid solutions.

This setup enables the precise control and measurement of benzoic acid dissolution under varying temperatures and rotational speeds, facilitating a comprehensive study of the dissolution process.

4.3 Procedure

- Prepare a 0.05 N NaOH solution by diluting 0.1 N NaOH with an equal volume of distilled water. Fill the burette with the prepared NaOH solution.
- Thoroughly rinse the motor housing, ensuring that all equipment is clean and completely dry before proceeding.
- Use vernier calipers to accurately measure the benzoic acid cylinder, then securely attach it to the motor and immerse it in pure water.
- Set the experimental conditions to 50^{o}C and 900 RPM stirring speed, and initiate the timer.
- Collect samples at consistent 5-minute intervals and perform titrations using the prepared NaOH solution with phenolphthalein as the indicator to identify the titration endpoints.
- After completing the final sample extraction, remeasure the acid cylinder with vernier calipers to assess material loss.
- Repeat the procedure under the following conditions: 60^{o}C at 900 RPM and 60^{o}C at 400 RPM, to examine the effects on dissolution rate and mass transfer.

5 Observation Tables

The tabulations include the observed data from each of the sub-parts of the overall experiment and are tabulated in the following tables (tables 1, 2, 3 & 4):

$Case$	$T\ (^oC)$	$w\ (RPM)$	$D\ (mm)$	$H\ (mm)$
1	50	905	30.15	60.54
2	60	900	21.70	52.05
3	60	400	22.20	55.34

Table 1: Diameter and Height of Solid Benzoic Acid cylinder

$t\ (min)$	$T\ (^oC)$	$V_M\ (mL)$	$V_1\ (mL)$	$V_2\ (mL)$	$V_{NaOH}\ (mL)$
5	49.9	5.8	5.0	6.7	1.7
11	49.7	4.8	6.8	9.2	2.4
15	49.1	5.0	9.3	12.1	2.8
20	48.3	5.0	12.2	15.9	3.7
25	47.7	5.0	15.9	19.9	4.0

Table 2: [Case 1] 50^oC and 905 RPM

$t\ (min)$	$T\ (^oC)$	$V_M\ (mL)$	$V_1\ (mL)$	$V_2\ (mL)$	$V_{NaOH}\ (mL)$
5	61.9	5.0	20.4	22.8	2.4
10	60.5	5.0	23.0	27.4	4.4
15	59.2	5.0	27.5	32.8	5.3
20	58.2	5.0	32.9	39.2	6.3
25	57.3	5.0	39.3	46.4	7.1

Table 3: [Case 2] 60^oC and 900 RPM

$t\ (min)$	$T\ (^oC)$	$V_M\ (mL)$	$V_1\ (mL)$	$V_2\ (mL)$	$V_{NaOH}\ (mL)$
5	61.9	5.0	5.8	6.6	0.8
10	60.9	5.0	6.6	8.8	2.2
15	59.4	5.2	8.8	11.2	2.4
20	58.4	5.0	11.2	13.9	2.7
25	57.7	5.2	13.9	16.8	2.9

Table 4: [Case 3] 60^oC and 400 RPM

Notations used: t = Time, T = Temperature, w = Rotation Speed, D = Diameter of solid Benzoic Acid cylinder, H = Height of solid Benzoic Acid cylinder, V_M = Volume of mixture (Benzoic Acid + Water), V_1 = Volume in buret before titration, V_2 = Volume in burret after titration, V_{NaOH} = Volume of titrant (N/20 $NaOH$).

6 Results & Calculations

By applying Benzoic Acid balance over the liquid phase, we get

$$V\frac{dC}{dt} = k_l A(C^* - C) \tag{1}$$

where C^* is the saturation concentration or the solubility, which represents the maximum concentration of Benzoic Acid in the liquid phase. This value is dependent on the temperature and is found from theoretical data mentioned in the **Appendix** section. Which are as follows:

- At 50^{o}C, solubility of benzoic acid in water (density = 0.98802 kg/L) is 0.07705 mol/kg of Solvent.
- At 60^{o}C, solubility of benzoic acid in water (density = 0.98313 kg/L) is 0.0976 mol/kg of Solvent.

To calculate the concentration of Benzoic Acid in liquid at time t, we can use the following relation,

$$V_{NaOH} N_{NaOH} = V_{\text{Benzoic Acid}} N_{\text{Benzoic Acid}} \tag{2}$$

The required data for the computations are used from the above tabulations. since benzoic acid has very low dissolution in water its normality value is not equalt to its molarity value. From theoretical data, the n-factor for this solid is found to be 0.5. So by following the below relation we obtain the molarity or the concentration value for benzoic acid:

$$M = N/n = 2N \tag{3}$$

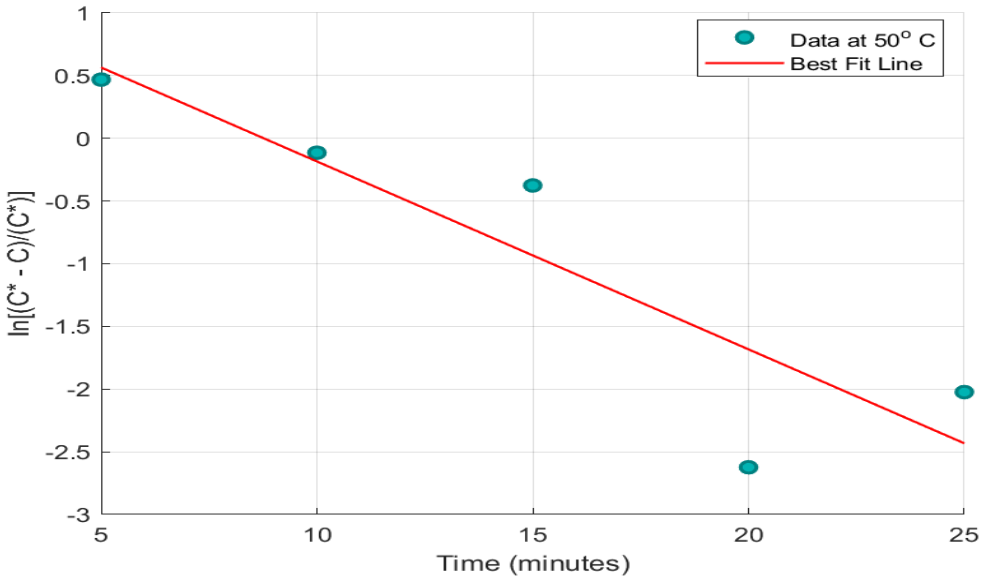

Figure 6: Case 1 | 50^{o}C and 905 RPM

Then integrating the above differential equation by assuming C^* to be constant,

$$ln(\frac{C^* - C}{C^* - C_0}) = -k_l a t \tag{4}$$

where C_0 is the concentration from where we started the dissolution experiment which in each of the specific cases is 0 initially since we changed the water in the batch extractor for each specific cases in order to take fresh observations without any previous temperatures and rotational effects to find the their accumulative effects on the mass transfer coefficient.

Also here, $a = \frac{A}{V}$, the surface area for mass transfer per unit volume of the liquid. And using table 1 we can calculate the a values for all the three cases while considering the total volume of liquid water in the batch extractor to be constant and is equal to 2400 mL.

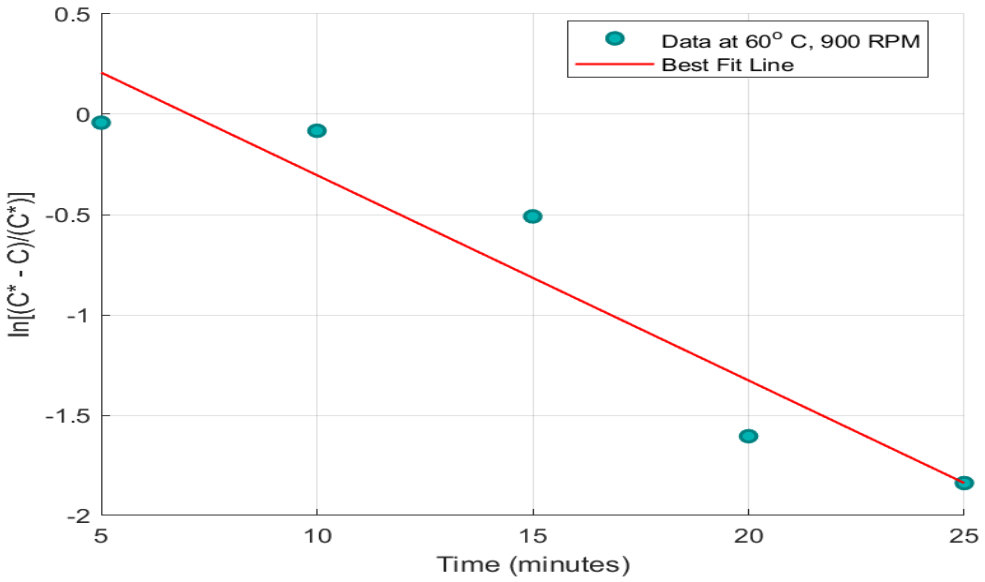

Figure 7: Case 2 | 60^{o}C and 900 RPM

Respective a values obtained from calculation are:
Case 1, a = 2.389 m^{-1}; **Case 2**, a = 1.478 m^{-1}; **Case 3**, a = 1.608 m^{-1}.

Then by plotting $ln(\frac{C^*-C}{C^*-C_0})$ vs t we get the following curves (shown in figures 6, 7 & 8) and then by fitting them using ordinary least squared regression method we get the best fit line.

From the slope we get the value of $k_l a$ and dividing the slope by the respective a values, we get the mass transfer coefficient. For each of the sub-parts, which are as follows;

- **Case 1**, $k_l = 0.0511\ m/s$
- **Case 2**, $k_l = 0.3581\ m/s$
- **Case 3**, $k_l = 0.1513\ m/s$

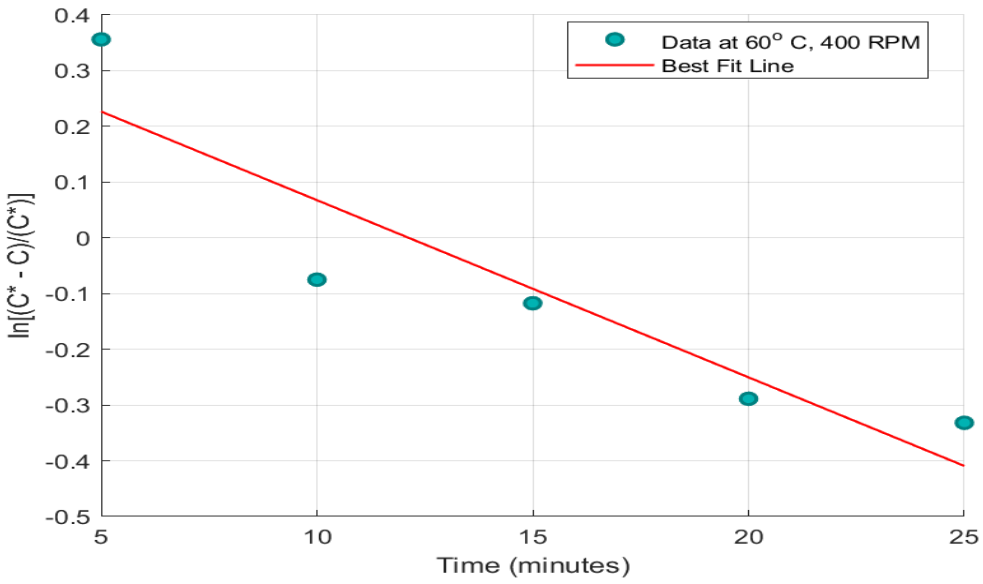

Figure 8: Case 3 | 60^{o}C and 400 RPM

7 Conclusions and Remarks

- From the obtained results we can see there is a high effect of temperature on the mass transfer coefficient.
- With the increase in temperature, the value of the mass transfer coefficient increases.
- This also satisfies the theory which says because of increased diffusion rate, reduction in viscosity in liquid and increased solubility of the solid in the liquid the mass transfer coefficient increases with the increase in temperature and this differentiates the case 1 from cases 2 and 3.
- Also the effect of rotation in the batch extractor is vivid from the results that with a larger RPM value the dissolution of the solid in the liquid increases which differentiates the case 2 and 3.

8 Error Analysis

Error in Timer $\equiv \Delta t = 5$ sec $= 0.0833$ min
Least Count of Vernier Calliper $\equiv \Delta D = \Delta H = 0.02$ mm
Least Count of Buret $\equiv \Delta V_T = 0.1$ mL
Least Count of Titration Flask $\equiv \Delta V_M = 0.2$ mL

Error in Surface Area to Volume Ratio (a):

$$\frac{\Delta a}{a} = \frac{\Delta D}{D} + \frac{\Delta H}{H} \tag{5}$$

Error in Concentration (C^*):

$$\frac{\Delta C^*}{C^*} = \frac{\Delta V_T}{V_T} + \frac{\Delta V_M}{V_M} \tag{6}$$

Error in Mass-Tranfer Coefficient (k_l):

$$\frac{\Delta k_l}{k_l} = \frac{\Delta t}{t} + \frac{(C - C_o)\ \Delta C^*}{(C^* - C)(C^* - C_o)\ ln\left(\frac{C^* - C}{C^* - C_o}\right)} \tag{7}$$

8.1 Sources of Error

- The measurement of diameter D and height H in Case 3 have significant error in them. This is because the Benzoic Acid cylinder had mostly dissolved into the water during Case 1 and Case 2, causing many fractures and breakages to appear on the cylinder.
- The error in the value of a (Surface Area to Volume) is also very high in Case 3 as the Benzoic Acid cylinder had mostly dissolved by that time, leaving behind a much more rough and complex shape instead of a simple cylinder.
- The time taken for the (Benzoic Acid cylinder + Water) system to reach the required temperature is not taken into account during our calculations. This can cause some error in the final results.
- Human error (spilling the solution over the table, not cleaning the cylinders and flasks properly, effects of parallax, etc.)

9 Precautions

- Ensure all measuring instruments, such as vernier calipers, burettes, and temperature sensors, are properly calibrated before starting the experiment to avoid measurement errors.
- Maintain the desired temperature precisely during each trial. Use a reliable heating system and thermometer to monitor and control temperature fluctuations, which can affect the rate of mass transfer.

- Ensure the motor operates at a consistent and accurate speed throughout the experiment. Verify the rotational speed with a tachometer if needed.
- Ensure the entire benzoic acid cylinder is fully immersed in water during each trial to facilitate uniform dissolution and accurate measurement.
- When filling the burette with NaOH, ensure there are no air bubbles that could affect the accuracy of titration results.
- Maintain consistent placement of the equipment and cylinder in the water bath to avoid any positional variation that could influence the hydrodynamics and, consequently, the mass transfer coefficient.

10 Thought Question / Open-Ended

Q. How will the dissolution equation change in case of a complex solvent? How can this be exploited in case of drug delivery in pharmaceuticals?

A. A complex solvent is a mixture of multiple solvents or one with specific properties such as pH buffers or co-solvents. The dissolution process can be effected by complex solvents due to new solvent-solute interactions in the system. Our objective is to look at different effects of complex solvents on solid dissolution and the use of such in drug delivery (pharmaceuticals).

Given below are some approaches to enhance solubility of drugs with respect to complex solvents :

- **pH Control**
  Drugs that are either weak acids (ibuprofen, aceclofenac, glipizide, etc.) or weak bases (olanzapine, telmisartan, sildenafil citrate, etc.), show improved solubility when the pH is adjusted properly.
  For example - ibuprofen shows increase in solubility at high pH values, whereas, olanzapine shows a jump in solubility at very low pH values.
- **Co-Solvency**
  The solubility of a poorly water soluble drugs, such as steroids and antifungal agents, can be increased by the addition of a water miscible solvent in which the drug has good solubility (ethanol, propylene glycol, etc).
  Examples of co-solvent formulations - Nimodipine Intravenous Injection and Digoxin Elixir Pediatric.

Drug	pH	Solubility (mg/mL)	Kp (cm/h)
Ibuprofen	1.2	0.14	0.3079
Ibuprofen	5.0	0.28	0.1354
Ibuprofen	8.0	3.51	0.0397
Olanzapine	1.2	16.91	0.0004
Olanzapine	5.0	5.55	0.0050
Olanzapine	8.0	0.38	0.0335

Table 5: Solubility and Kp values of different drugs

- **Hydrotrophy**
  Hydrotrophy is a solubilization process whereby addition of a large amount of second solute results in an increase in the aqueous solubility of another solute.
  Solute consists of alkali metal salts of various organic acids. Hydrotropic agents are ionic organic salts. Additives or salts that increase solubility in given solvent are said to “salt in” the solute and those salts that decrease solubility “salt out” the solute.
  Several salts with large anions or cations that are themselves very soluble in water result in “salting in” of non electrolytes called “hydrotropic salts” a phenomenon known as “hydrotropism”.

11 Acknowledgements

We as a group contributed our respective parts into completing the above report on Solid Dissolution.

In terms of specifications, Rapolu Paranay Reddy helped with “Apparatus & Materials" and “Experimental Setup Description" part of the report. Atharva Sunilkumar Ghodke contributed in “Procedure" & “Precaution" parts. Anmol Upadhyay delivered the content for “Background & Motivation" along with Lakkireddy Vishnu Vardhan Reddy helping in “Abstract” part of the report and rest of all the parts 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 Sparsh bhaiya for this experiment for his 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 CH2014 : Heat and Mass Transfer by Prof Renganathan.
- Notes from the course CH3030 : Applied Mass Transfer by Prof Ethayaraja.
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- "Solubility of Benzoic Acid in Mixed Solvents" | January 2007 Journal of Chemical & Engineering Data 52(1):298-300 DOI: 10.1021/je060408x https://www.researchgate.net/publication/277518455_Solubility_of_Benzoic_Acid_in_Mixed_Solvents
- "The effect of temperature on the solubility of benzoic acid derivatives in water" | Fluid Phase Equilibria to obtain the solubility values for benzoic acid in water https://www.sciencedirect.com/science/article/pii/S0378381206004420
- To obtain the density of water at the specified temperatures. https://www.vip-ltd.co.uk/Expansion/Density_Of_Water_Tables.pdf

Appendix

Lab Data: All the experimental observations with each of the sub-parts of the main experiment that was performed and tabulated during the laboratory session are included in order in the following (in figures 9, 10, 11 & 12).

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_8. 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.

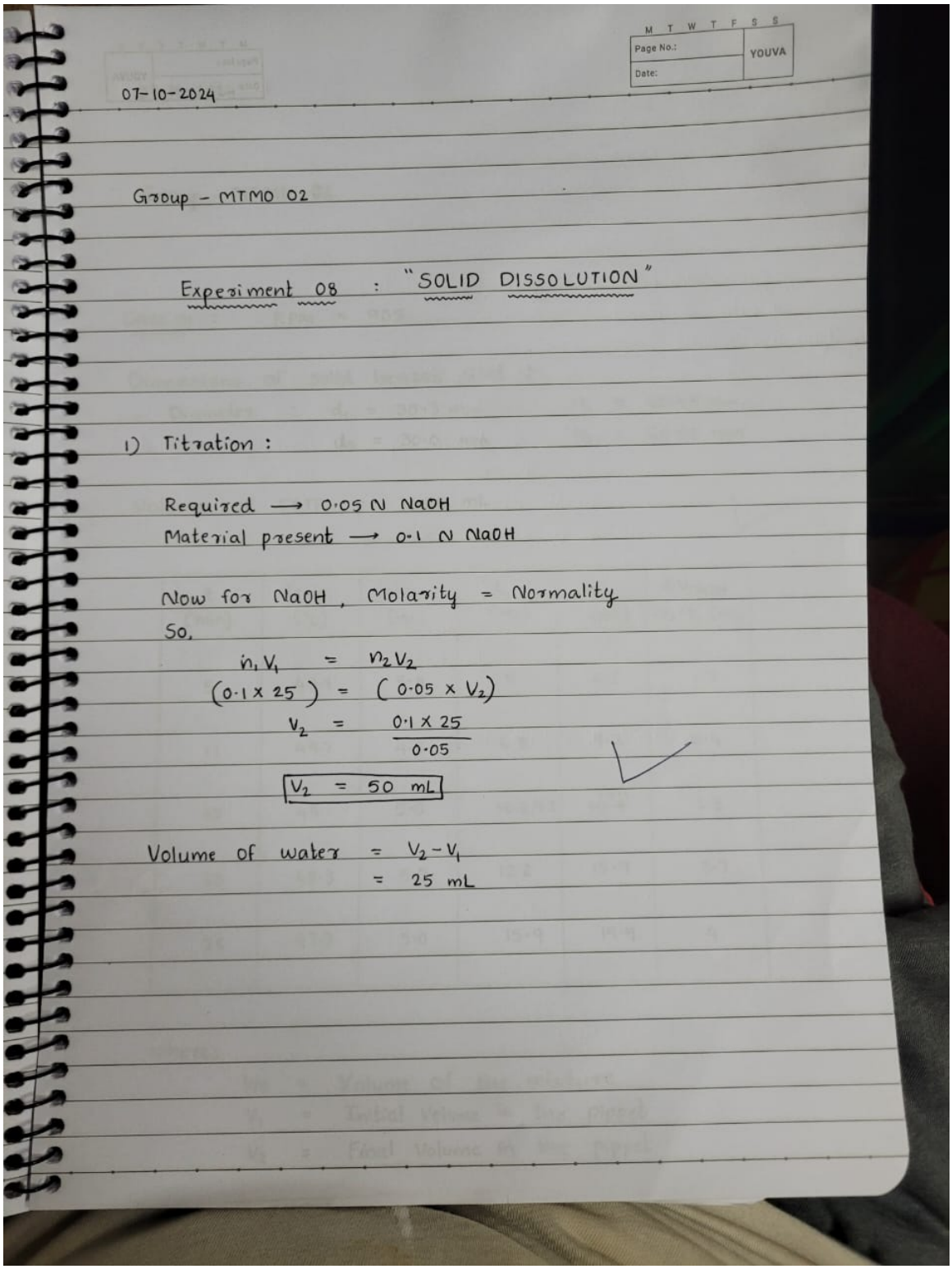
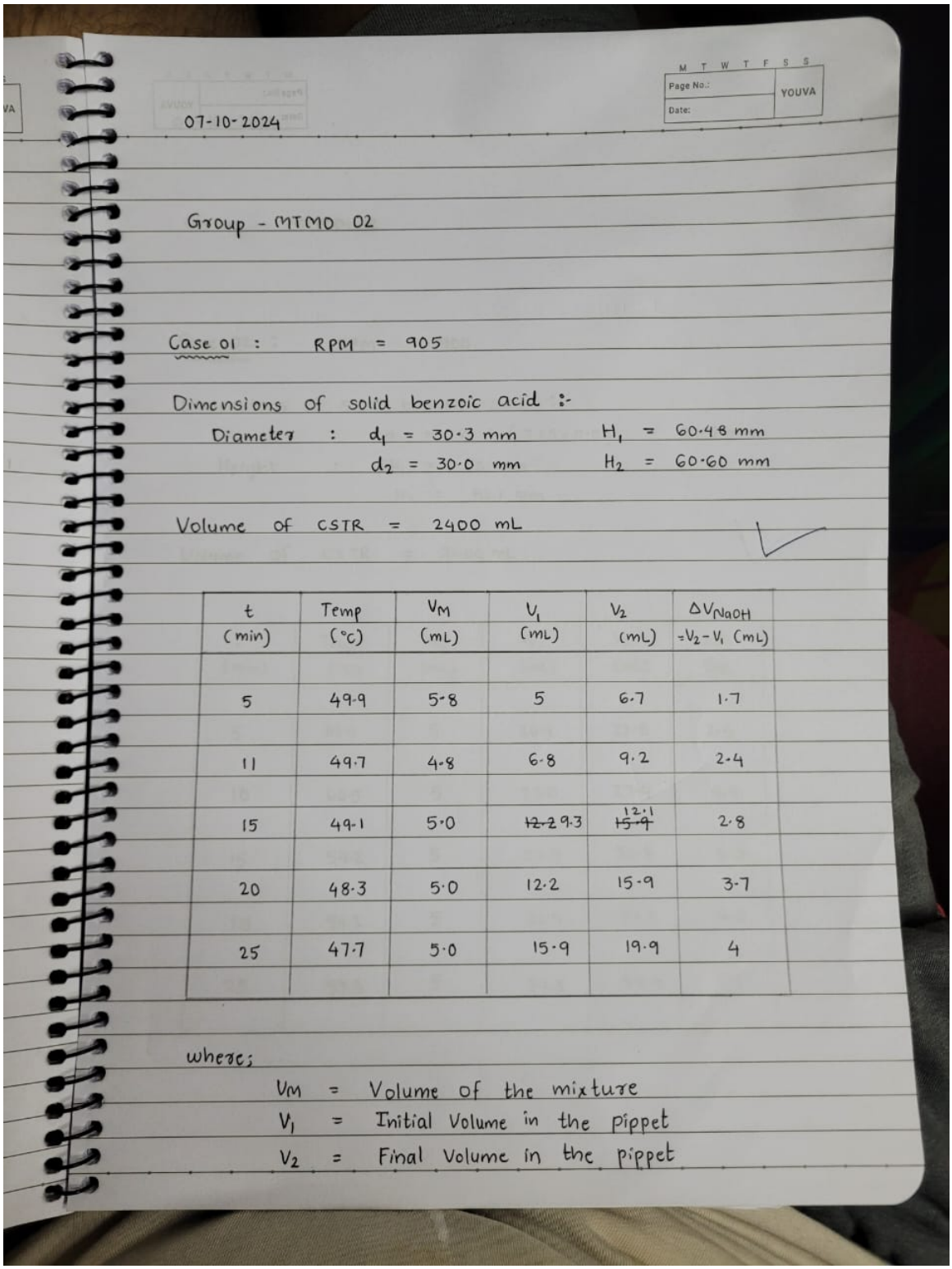


Figure 9: Titration Data



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Group - MTMD 02

Case 01 : RPM = 905

Dimensions of solid benzoic acid :-

Diameter : d_1 = 30.3 mm H_1 = 60.48 mm

d_2 = 30.0 mm H_2 = 60.60 mm

Volume of CSTR = 2400 mL

t (min)	Temp (°C)	V_M (mL)	V_1 (mL)	V_2 (mL)	$\Delta V_{NaOH} = V_2 - V_1$ (mL)
5	49.9	5.8	5	6.7	1.7
11	49.7	4.8	6.8	9.2	2.4
15	49.1	5.0	~~12.2~~ 9.3	~~15.9~~ 12.1	2.8
20	48.3	5.0	12.2	15.9	3.7
25	47.7	5.0	15.9	19.9	4

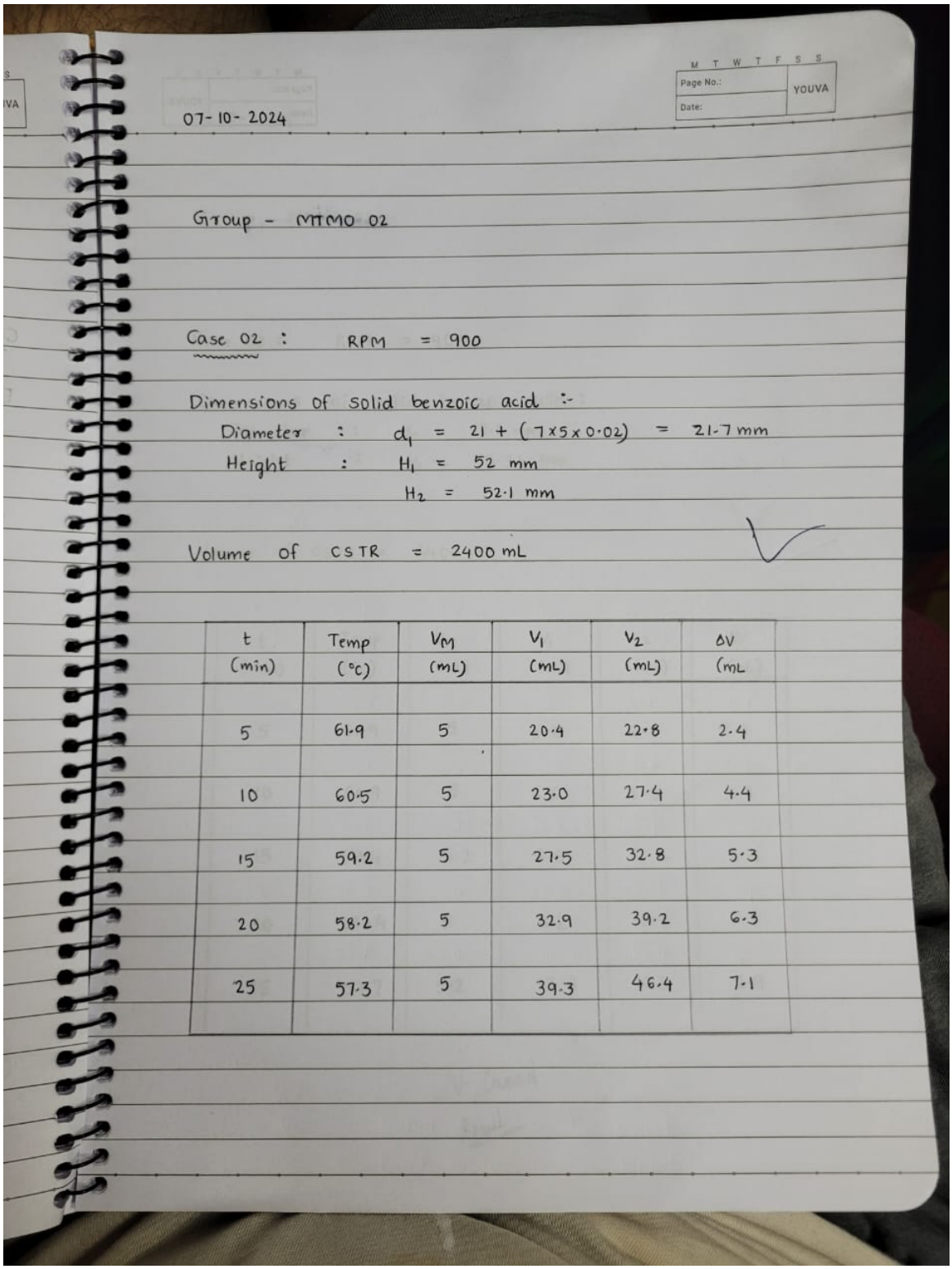
where;

V_M = Volume of the mixture

V_1 = Initial Volume in the pippet

V_2 = Final Volume in the pippet

Figure 10: Case 1 of Observation



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Group - MTMO 02

Case 02 : RPM = 900

Dimensions of solid benzoic acid :-

Diameter : $d_1 = 21 + (7 \times 5 \times 0.02) = 21.7$ mm

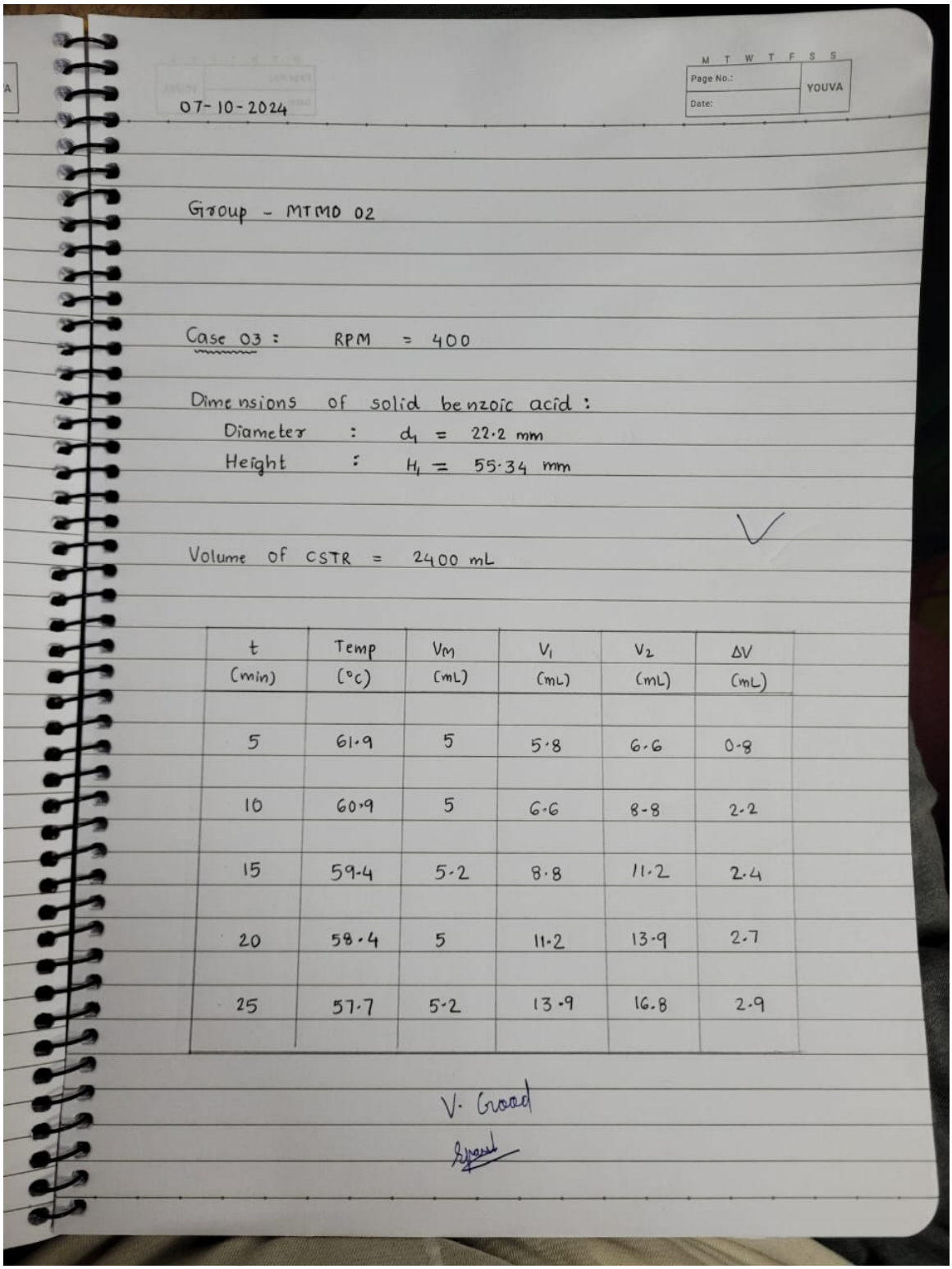
Height : $H_1 = 52$ mm

$H_2 = 52.1$ mm

Volume of CSTR = 2400 mL

t (min)	Temp (°C)	V_M (mL)	V_1 (mL)	V_2 (mL)	ΔV (mL
5	61.9	5	20.4	22.8	2.4
10	60.5	5	23.0	27.4	4.4
15	59.2	5	27.5	32.8	5.3
20	58.2	5	32.9	39.2	6.3
25	57.3	5	39.3	46.4	7.1

Figure 11: Case 2 of Observation



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Group - MTMD 02

Case 03 : RPM = 400

Dimensions of solid benzoic acid :
Diameter : d_1 = 22.2 mm
Height : H_1 = 55.34 mm

Volume of CSTR = 2400 mL

t (min)	Temp (°C)	V_M (mL)	V_1 (mL)	V_2 (mL)	ΔV (mL)
5	61.9	5	5.8	6.6	0.8
10	60.9	5	6.6	8.8	2.2
15	59.4	5.2	8.8	11.2	2.4
20	58.4	5	11.2	13.9	2.7
25	57.7	5.2	13.9	16.8	2.9

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Figure 12: Case 3 of Observation