Momentum Transfer and Mechanical Operations Lab

Multi-phase Gas Absorption

23rd September, 2024

Team: MTMO 2

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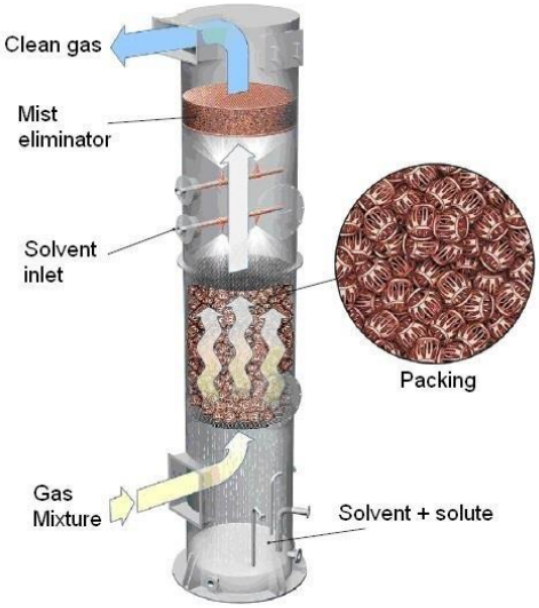
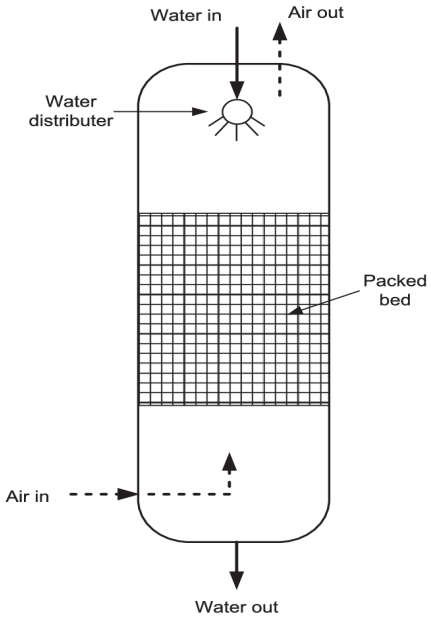
1 Abstract with Graphics

The experiment on multi-phase gas absorption (schematically shown in the following figures) aimed to study the absorption of carbon dioxide (CO_2) into an aqueous phase and calculate both the solubility and the bulk mass transfer coefficient of CO_2 in water. These values were then compared with theoretical calculations based on Henry's Law.

The experiment utilised two types of packing materials—Berl Saddles and Metal Pall Rings—in two separate columns to determine the effect of surface area on the efficiency of mass transfer. CO_2 was introduced from the bottom of the columns, while water flowed from the top. Various flow rates of CO_2 and water were used, and the absorbed CO_2 was measured through titration with sodium hydroxide (NaOH). The results showed that the column with Metal Pall Rings exhibited a higher mass transfer efficiency, as indicated by the larger volume of NaOH required for titration. The volumetric mass transfer coefficients were calculated, with the Metal Pall Rings showing a higher coefficient, confirming their superior performance in gas absorption.

The experiment highlighted the significance of efficient packing in improving the rate of gas absorption and provided insights into the mass transfer process under different con-





ditions and packing material efficiency in industrial applications such as air cleaning and chemical production, as well as in environmental efforts to capture and reduce greenhouse gases.

2 Aim & Objectives

- Validation of the mass transfer equations.
- By examining the absorption of CO_2 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 CO_2 with time (t).

3 Background and Motivation

Multi-phase gas absorption, particularly in packed beds, is an essential process in industries such as chemical manufacturing, gas purification, and environmental engineering. It involves the transfer of a gas phase into a liquid phase, often to facilitate mass and

heat transfer. The interactions that happen among the particles (figure 3) are commonly distributed in two different inter-phases, one is gas-liquid interface and the other being the liquid-solid interface (shown in figure 2). Among various gases, carbon dioxide (CO_2) absorption has garnered significant attention due to its environmental and industrial importance. CO_2 is a leading greenhouse gas, and its rising atmospheric levels are a major driver of global warming and climate change. Consequently, capturing CO_2 from industrial emissions has become a critical research area, with chemical absorption being one of the most widely explored methods.

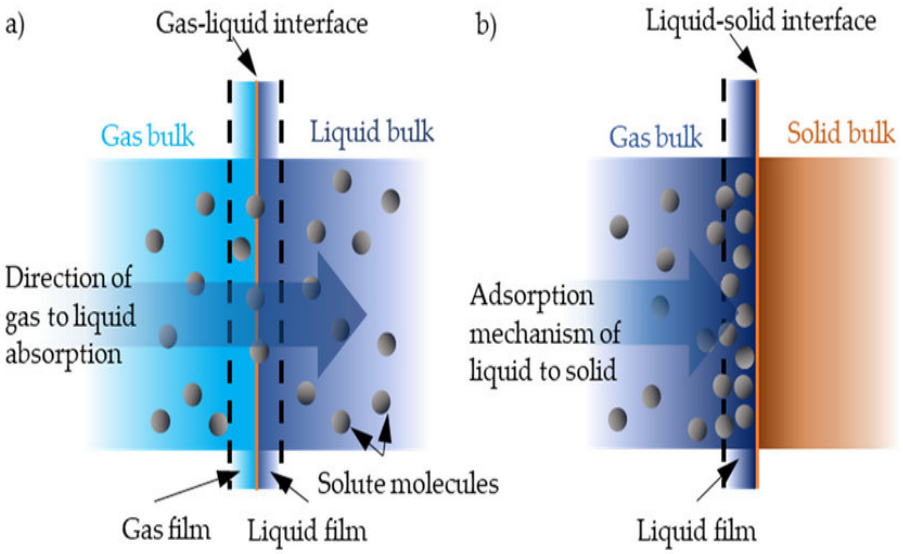


Figure 2: Interactions of particles in two different Interfaces

In the packed bed absorption system, a gas like CO_2 interacts with a liquid, typically water, in the presence of structured or random packing, which increases the contact area for mass transfer. This setup allows for highly efficient gas-liquid interactions, making it suitable for industrial-scale CO_2 capture. As industries worldwide seek solutions to reduce emissions and comply with environmental regulations, improving the efficiency of such systems has become a pressing need.

This experiment is motivated by the need to evaluate and optimize the CO_2 absorption process in an aqueous phase using a packed bed. Understanding how different packing materials, solvent compositions, and operating conditions affect absorption efficiency can provide valuable insights for enhancing the design and operation of CO_2 capture systems. By optimizing key parameters such as absorption rates and mass transfer coefficients, the findings from this study could contribute to the development of more effective CO_2 mitigation technologies, which are crucial for addressing the environmental challenges posed by industrial emissions.

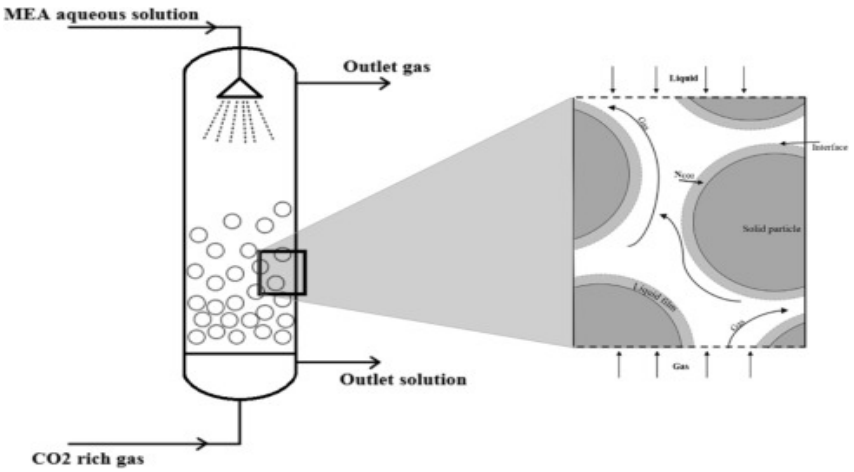


Figure 3: Interactions of liquid and gas particles inside the absorption column

4 Materials and Methods

4.1 Apparatus & Materials Required

Conical Flask, Phenolphthalein Indicator, 0.1 N NaOH Solution, Stopwatch, Water, Clamp Stand, Measuring Cylinder, Metal Pall Rings, Berl Saddle, CO_2 Cylinder.

4.2 Experimental Setup Description

The experimental setup is shown in figures 5a & 5b, which consists of two parts, that are performed separately.

Figure 4: Liquid and Gas flow rate control device

- **Glass Columns with Packing**: Two separate glass columns are prepared, one packed with **Berl Saddle** and the other with **Metal Pall Rings**. The packing is carefully placed to ensure uniform distribution and minimal void space.
- CO_2 **Gas Supply**: A CO_2 cylinder is connected to the bottom inlet of the gas absorption column, providing a controlled supply of CO_2 gas into the column.
- **Water Supply System**: Water is fed into the top of the column through a controlled inlet. Both the gas and water systems are equipped with flow controllers to regulate their respective rates.
- **Flow Control**: A **rotameter** (shown in figure 4) is used to monitor and adjust the flow rates of both CO_2 gas and water, with precise control to allow for varying the flow rates during the experiment.

(a) Set-up with Pall Rings (b) Set-up with Berl Saddle

Figure 5: Set-up with Pall Rings and
Berl Saddle as packing materials of the absorption column

- **Sampling System**: A setup is prepared for collecting 25 mL water samples from the column outlet at specific intervals, with collection containers positioned for easy access during sampling.

- **Titration Setup**: A conical flask, measuring cylinder, and phenolphthalein indicator are arranged for titration of the collected samples using 0.1 Normal NaOH solution to determine the amount of absorbed CO_2.

This experimental setup allows for the controlled introduction and regulation of CO_2 and water into packed columns, as well as the systematic collection and analysis of water samples after gas absorption.

4.3 Procedure

- Carefully fill the glass columns with Berl saddle packing material, ensuring proper and efficient packing.
- Connect the CO_2 gas source to the bottom inlet of the gas absorption column and the water source to the top inlet.
- Begin water flow through the top inlet and simultaneously initiate the CO_2 gas flow from the bottom, applying necessary pressure.
- Use the LPH rotameter to regulate the flow rates of both water and CO_2.
- Adjust the CO_2 flow rate while maintaining a constant water flow rate, collecting 25 mL water samples every 10 minutes. Repeat this procedure for three distinct CO_2 flow rates.
- Similarly, adjust the water flow rate while keeping the CO_2 flow constant, and collect samples using the same procedure.
- Take a total of 6 readings by varying the water flow rates to 10 LPH and 15 LPH, and the CO_2 flow rates to 5, 10, and 15 LPH.
- Perform titration on the collected samples using NaOH, given that the samples will be acidic.
- Repeat the entire procedure using Metal Pall Rings as the packing material, and obtain 6 readings with different water and CO_2 flow rate combinations.

5 Observation Tables

The tabulations include the observed data from each of the sub-experiments and are tabulated in the following tables (tables 1 & 2):

Note: Q_L = Volumetric flow rate of water, Q_G = Volumetric flow rate of CO_2, V_M = Volume of analyte (CO_2 + H_2O), V_1 = Volume in buret before titration, V_2 = Volume in buret after titration, V_T = Volume of titrant (N/10 $NaOH$)

Q_L (LPH)	Q_G (LPH)	V_M (mL)	V_1 (mL)	V_1 (mL)	V_T (mL)
10	5	25	14	15.7	1.7
10	10	25	15.7	17.2	1.5
10	15	25	17.2	18.5	1.3
15	5	25	18.5	20.2	1.7
15	10	25	20.2	21.6	1.4
15	15	25	21.6	22.9	1.3

Table 1: Tabulation for Berl Saddle as Packing Material

Q_L (LPH)	Q_G (LPH)	V_M (mL)	V_1 (mL)	V_1 (mL)	V_T (mL)
10	5	25	22.9	23.7	0.8
10	10	25	23.9	25.0	1.1
10	15	25	25.0	26.0	1.0
15	5	24	26.0	26.4	0.4
15	10	25	26.4	27.0	0.6
15	15	25	27.0	28.0	1.0

Table 2: Tabulation for Pall Ring as Packing Material

6 Results & Calculations

By applying CO_2 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 CO_2 in the liquid phase corresponding to the final pressure. This is calculated using Henry's law, which is as follows:

$$P = K_H \ \times \ C \tag{2}$$

here, P is the partial pressure of the gas in the liquid and K_H is the Henry's law constant which depends on temperature and C is the concentration of the gas dissolved in the liquid.

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

$$ln(\frac{C^* - C}{C^* - C_0}) = -k_l at \tag{3}$$

here, $a = \frac{A}{V}$, the surface area for mass transfer per unit volume of the liquid. And from literature data the values of a used in calculating the mass transfer coefficient values are:

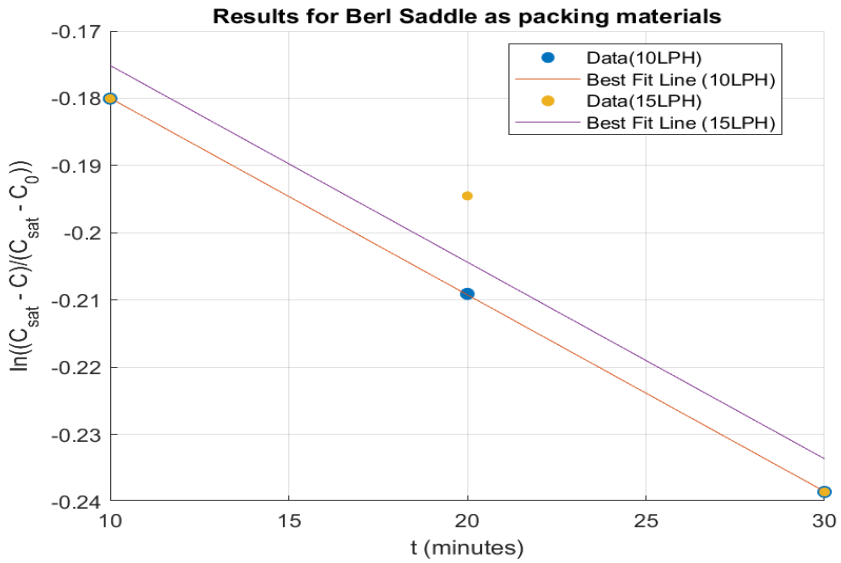


Figure 6: Best fit lines for columns with Berl Saddle

- For berl saddle, $a = 249.344832 m^2/m^3$.
- For metal pall rings, $a = 206.6929134 m^2/m^3$.

Then by plotting $ln(\frac{C^*-C}{C^*-C_0})$ vs t we get the following curves (shown in figures 6 & 7) and then by fitting them using ordinary least squared regression we get the best fit line. Then from the slope we get the value of $k_l a$ and diving the slope by the respective a values, we get the mass transfer coefficient. For each of the sub-parts, we take the average of the k_l values obtained and which are as follows:

- For berl saddle, $k_l = 1.173 \times 10^{-5}\ m/s$.
- For metal pall rings, $k_l = 1.319 \times 10^{-5}\ m/s$.

7 Conclusions and Remarks

- It is to be noted that the packing column with Ceramic Berl Saddle was operating in bubbly flow regime, whereas, the packed column with Metallic Pall Rings was operating in the tricking flow regime.
- The bubbly flow regime occurs when the liquid occupies most of the packing column and the gas is uniformly distributed throughout the column as minute gas bubbles.

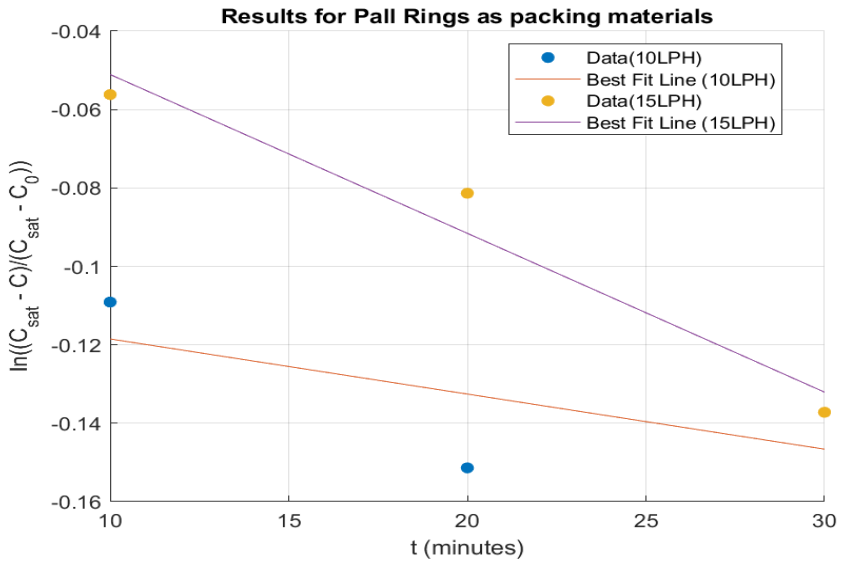


Figure 7: Best fit lines for columns with Metallic Pall Rings

- On the other hand, the tricking flow regime occurs when the gas occupies most of the packing column and the liquid slowly trickles down the packing, driven by force of gravity.
- From results, it's vivid that both towers show variations in mass transfer efficiency with changes in water flow rates. However, Berl Saddles consistently deliver better performance compared to Metal Pall Rings.
- In Metal Pall Rings, increasing the flow rate results in a moderate improvement in mass transfer. However, due to the occurrence of channelling, overall efficiency remains comparatively lower.
- Berl Saddles, starting with a high efficiency due to better flow distribution, are less impacted by changes in flow rate. This makes them more consistent in performance.

8 Error Analysis

Error in Volumetric Flow Rate $\equiv \Delta Q = 1$ LPH
Least Count of Stopwatch $\equiv \Delta t = 0.01$ sec
Least Count of Buret $\equiv \Delta V_T = 0.1$ mL
Least Count of Titration Flask $\equiv \Delta V_M = 1$ mL

Error in Concentration (C^*):

$$\frac{\Delta C^*}{C^*} = \frac{\Delta V_T}{V_T} + \frac{\Delta V_M}{V_M} \tag{4}$$

Error in Mass-Tranfer Coefficient (k_l):

$$\frac{\Delta k_l}{k_l} = \frac{\Delta t}{t} + \frac{(C - C_o)\ \Delta C^*}{(C^* - C)(C^* - C_o)\ ln\left(\frac{C^* - C}{C^* - C_o}\right)} \tag{5}$$

8.1 Sources of Error

- Possible leakage in the pipes connecting cylinder containing hot CO_2 gas and the packing column.
- Overflow of accumulated solution (CO_2 + H_2O) in the packing column, causing the solution to enter the gas inlet pipe, making the readings inaccurate.
- Human error in taking readings from different lab equipment.

9 Precautions

- The packing material (such as Berl saddles or Metal Pall Rings) in the absorption column must be carefully and uniformly arranged to prevent channeling and ensure consistent gas-liquid interaction.
- Ensure all connections in the experimental setup, particularly at the CO_2 gas and water inlets and outlets, are securely fastened to prevent any leakage of gas or liquid.
- Utilize calibrated rotameters to accurately monitor and adjust the flow rates of both CO_2 and water. Any abrupt changes in flow rates could compromise the precision of solubility and mass transfer measurements.
- Verify that the CO_2 source is of high purity to prevent any impurities from affecting the absorption process.
- Conduct titrations immediately after sample collection to minimize the risk of CO_2 degassing from the aqueous phase.
- The experiment should be carried out in a well-ventilated environment to safely manage the release of CO_2 gas.

10 Thought Question / Open-Ended

Q. Micro-gravity environments require rethinking your fluid mechanics equations. Many ISS systems for life support, carbon dioxide absorption and water treatment employ packed columns. How does gravity influence your equations that you used for calculations in your report so far? Can you reduce gravity to 1/10th and show how your final results change?

A. For the case of Bubbly Flow regime, the liquid phase occupies all of the voids in the packing columns and the gas phase is uniformly distributed as small bubbles. The equation which governs this regime is independent of external forces like gravity. As such there will be virtually no change in results after gravity is reduced to 1/10th.

On the other hand, the Trickling Flow regime occurs when the gas phase occupies all the voids in the packing column and the liquid phase is trickling down the packing due to the effect of gravity.

In reduced gravity (micro-gravity), the trickle flow regime becomes either pulse or bubbly flow and the pulse flow regime is observed over a much wider range of conditions. Rather than draining, the liquid tends to spread in a radial (as well as axial) direction until a sufficient amount of liquid has plugged a cross sectional area. Depending on the gas flow rate, the plug will either continue to fill the column until it is the continuous phase or at slightly higher gas flow rates, it will become the start of a liquid pulse. The other extreme of the gas continuous flow is spray or mist flow, which occurs at a very high gas-to-liquid ratio.
Provided below is a plot (figure 8) describing the effect of micro-gravity:

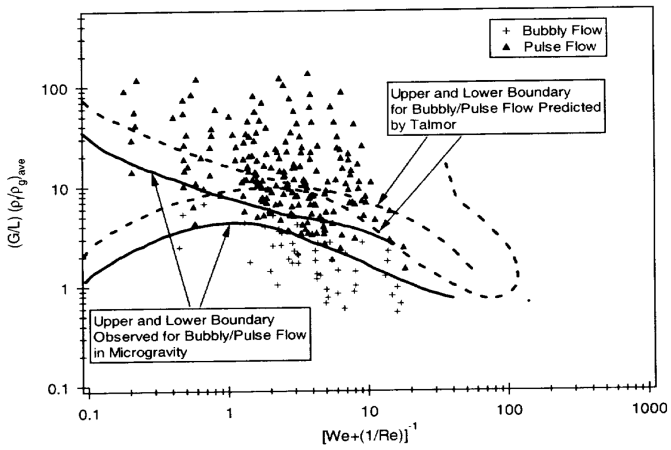


Figure 8: Flow map in microgravity | NASA/TM-2001-210705

Possible reasons behind such change :

- the wetting of the particles by the liquid in 0.1-g is different than in 1-g since the gravitational draining force is much less in 0.1-g.
- the enhanced role of the capillary forces in determining the amplitude of the waves that exist on the liquid film surrounding the packing materials.

11 Acknowledgements

We as a group contributed our respective parts into completing the above report on Multiphase Gas Adsorption.

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. 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 are 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 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.

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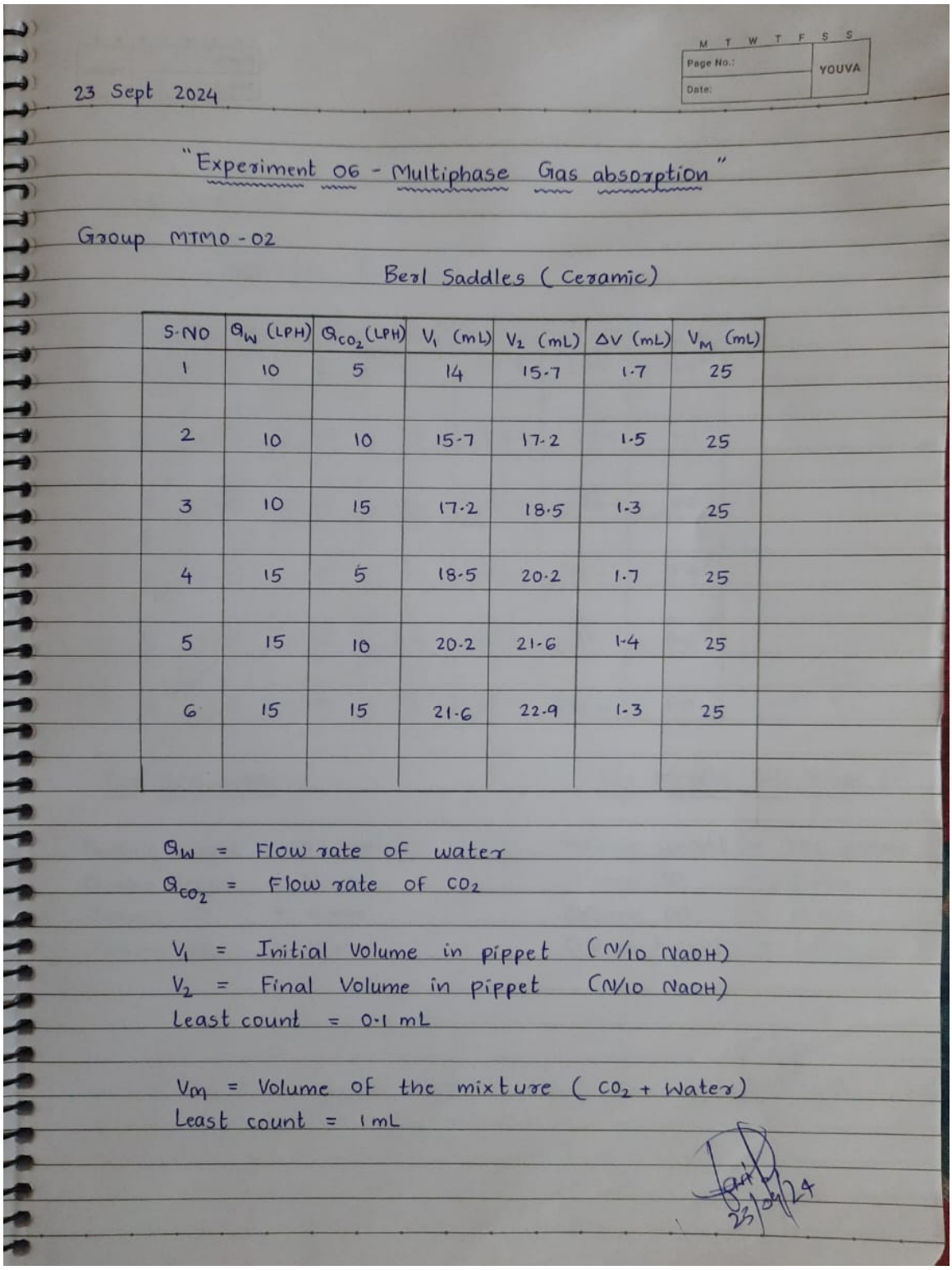
- A paper on gas absorption in packed bed (but with raschig ring) to get a genuine idea of the results, https://www.academia.edu/94548374/Gas_absorption_in_packed_tower_with_Raschig_rings_packings.
- Study of solubility of CO_2 in water at different temperatures, https://bionumbers.hms.harvard.edu/bionumber.aspx?s=n&v=4&id=106207.

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).

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_6. 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.



23 Sept 2024

"Experiment 06 - Multiphase Gas absorption"

Group MTM0-02

Berl Saddles (Ceramic)

S.NO	Q_W (LPH)	Q_{CO_2} (LPH)	V_1 (mL)	V_2 (mL)	ΔV (mL)	V_M (mL)
1	10	5	14	15.7	1.7	25
2	10	10	15.7	17.2	1.5	25
3	10	15	17.2	18.5	1.3	25
4	15	5	18.5	20.2	1.7	25
5	15	10	20.2	21.6	1.4	25
6	15	15	21.6	22.9	1.3	25

Q_W = Flow rate of water

Q_{CO_2} = Flow rate of CO_2

V_1 = Initial Volume in pippet (N/10 NaOH)

V_2 = Final Volume in pippet (N/10 NaOH)

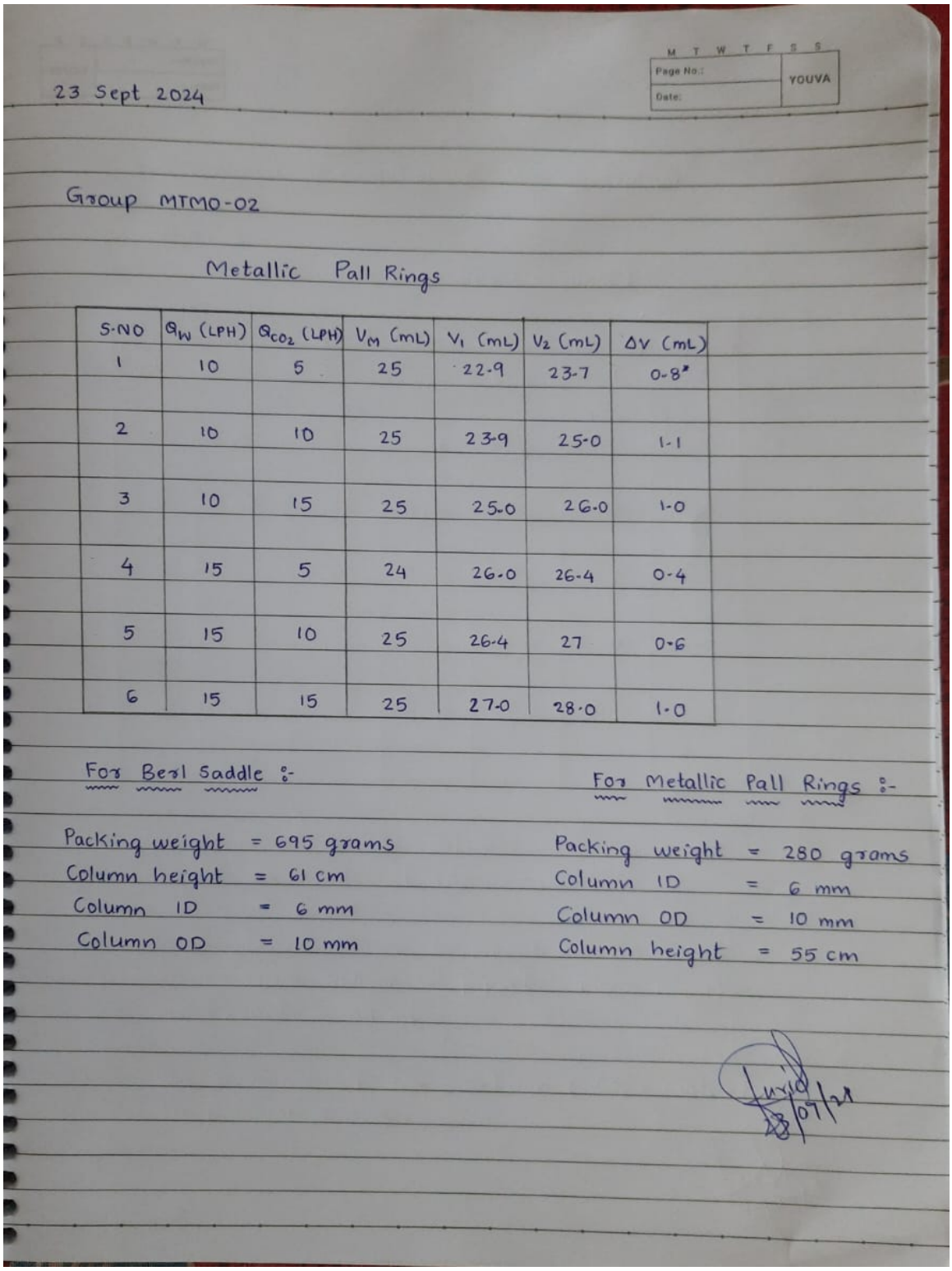
Least count = 0.1 mL

V_M = Volume of the mixture (CO_2 + Water)

Least count = 1 mL

23/09/24

Figure 9: Data for Berl Saddle as packing material



23 Sept 2024

Group MTMO-02

Metallic Pall Rings

S.NO	Q_W (LPH)	Q_{CO_2} (LPH)	V_M (mL)	V_1 (mL)	V_2 (mL)	ΔV (mL)
1	10	5	25	22.9	23.7	0.8
2	10	10	25	23.9	25.0	1.1
3	10	15	25	25.0	26.0	1.0
4	15	5	24	26.0	26.4	0.4
5	15	10	25	26.4	27	0.6
6	15	15	25	27.0	28.0	1.0

For Berl Saddle :-

Packing weight = 695 grams
Column height = 61 cm
Column ID = 6 mm
Column OD = 10 mm

For Metallic Pall Rings :-

Packing weight = 280 grams
Column ID = 6 mm
Column OD = 10 mm
Column height = 55 cm

Figure 10: Data for Metallic Pall ring as packing material