CH 3522: Unit Operations Lab

Adsorption Breakthrough

Batch - R, Group - 05
Date of Experiment: 5th February, 2025
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1. Objective

- Plotting of the breakthrough curve.
- Estimation of the equilibrium capacity of the silica gel bed with water.
- Estimation of the mass transfer coefficient using the breakthrough curve.

2. Introduction

Adsorption is a surface phenomenon in which molecules from a gas or liquid adhere to the surface of a solid material, known as the adsorbent. In this experiment, silica gel serves as the adsorbent, while water vapor in humid air acts as the adsorbate. Adsorption occurs due to a difference in chemical potential between the fluid phase and the solid surface, leading to the accumulation of molecules on the adsorbent. This process is primarily governed by weak intermolecular forces, such as van der Waals forces and hydrophobic interactions, making adsorption a reversible process where the adsorbent can be regenerated after reaching its capacity.

Over time, as silica gel continues to capture water vapor, it eventually reaches saturation, resulting in breakthrough, the point at which adsorbate starts escaping with the effluent air. Understanding breakthrough behavior is essential for optimizing the performance of silica gel-based adsorption systems, which are widely used in applications such as air dehumidification, gas purification, and environmental remediation. By experimentally analyzing breakthrough curves, valuable insights can be gained into improving adsorption efficiency, system design, and operational parameters for industrial-scale packed bed adsorption processes.

3. Theory

Few of the important theoretical aspects behind the current experiments are explained below.

Adsorption Breakthrough in Silica Gel

Silica gel is a highly porous material with a large surface area (typically between 100 and 1200 m^2/g), making it an effective adsorbent for various applications such as water purification, air filtration, and chromatography. However, its adsorption capacity is finite, and it eventually reaches a **breakthrough point**, where it can no longer retain contaminants efficiently. Understanding this phenomenon is crucial for optimizing adsorption-based separation and purification systems.

Breakthrough Phenomenon and the Mass Transfer Zone (MTZ)

In a packed bed adsorption column, the **mass transfer zone (MTZ)** is the active region where adsorption occurs. As the contaminated air or liquid flows through the silica gel bed, the MTZ moves along the column length. The efficiency of the adsorption process depends on how well this zone is managed. A shorter MTZ indicates better utilization of the adsorbent, while a longer MTZ may result in inefficient adsorption. The breakthrough curve, which plots the ratio of outlet to inlet adsorbate concentration C/C_0 against time, helps analyze this behavior.

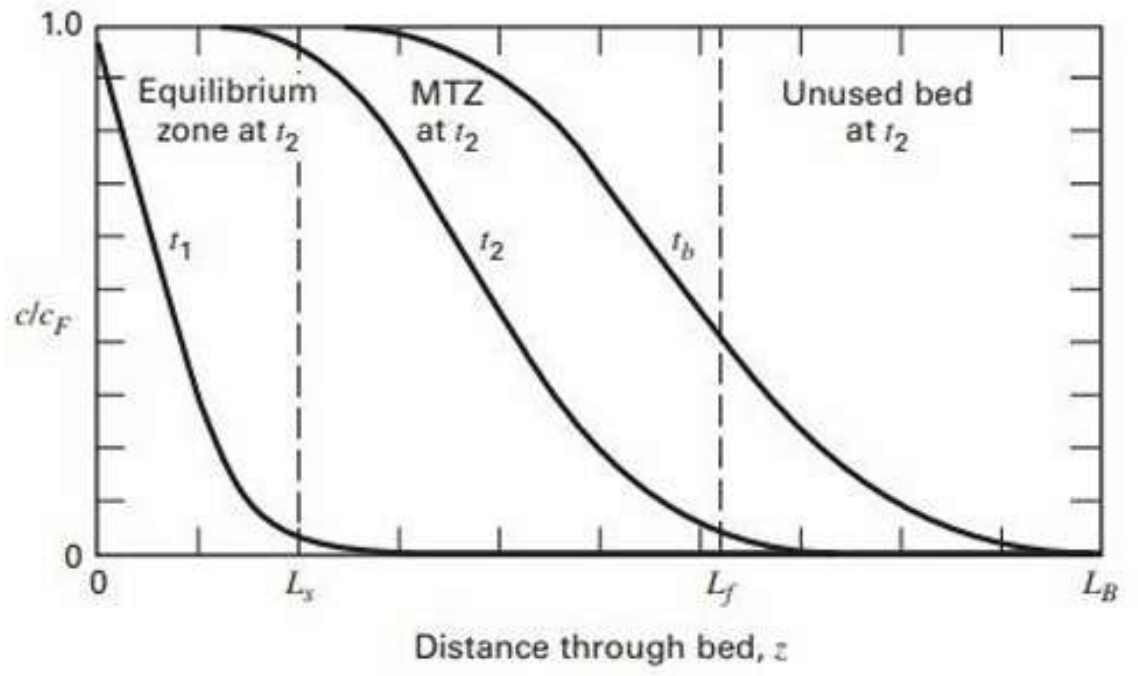


Figure 1: MTZ along the length of bed

Characteristics of the Breakthrough Curve

1. **Initial Plateau:** At the start, fresh silica gel effectively adsorbs all contaminants, keeping the effluent concentration near zero.
2. **Breakthrough Point:** As adsorption sites near the inlet become saturated, some pollutants escape, causing the curve to rise.
3. **Mass Transfer Zone:** The gradual rise in the curve indicates the movement of remaining pollutants deeper into the bed, occupying more adsorption sites.
4. **Saturation Plateau:** Eventually, the effluent concentration equals the inlet concentration $C/C_0 = 1$, indicating complete saturation and the end of the adsorption cycle.

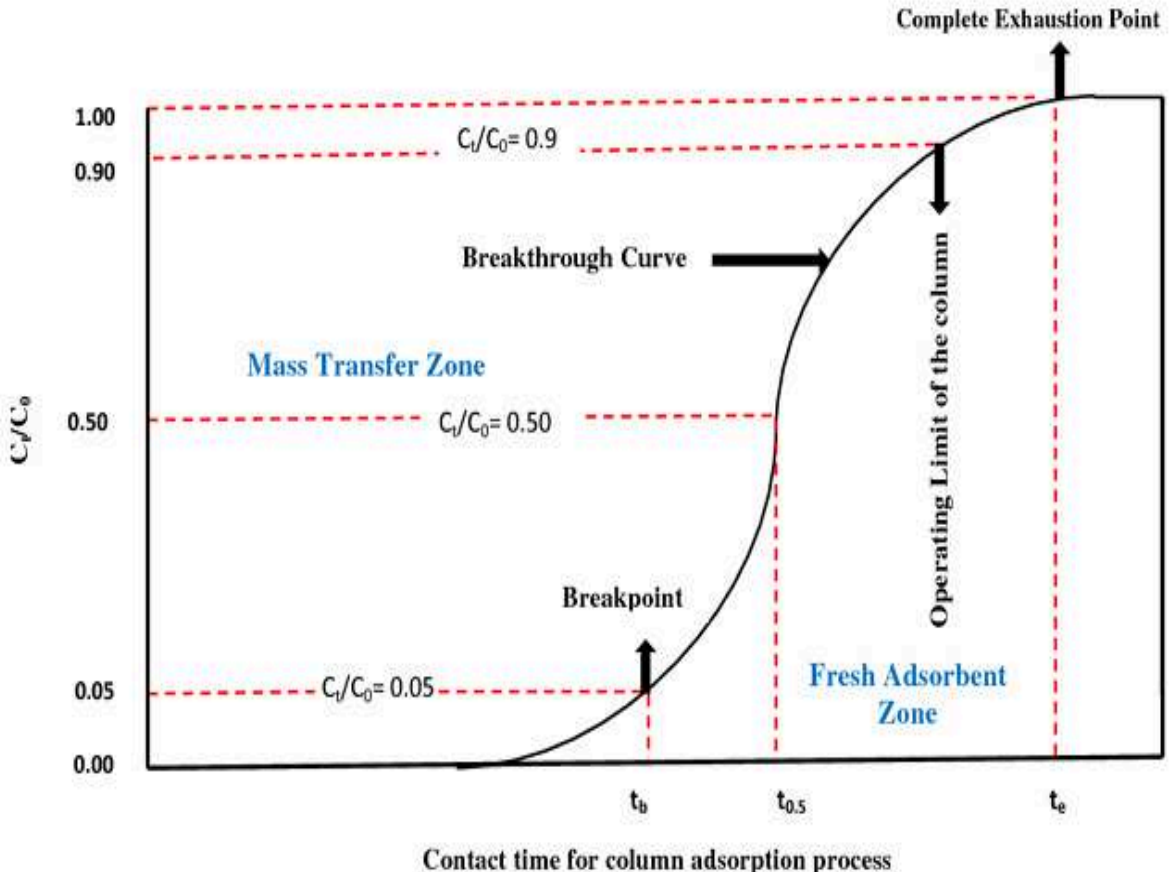


Figure 2: Breakthrough curve characteristics

Factors Influencing Adsorption Performance

Several factors affect the efficiency of silica gel adsorption:

- **Silica Gel Characteristics:** Surface area, pore size, and chemical composition determine the adsorption capacity.
- **Operating Conditions:** Temperature, flow rate, and contaminant concentration influence how quickly breakthrough occurs.
- **Adsorbate Properties:** Molecular size, polarity, and humidity levels impact adsorption efficiency.
- **Particle Size:** Smaller silica gel particles improve mass transfer rates but may increase pressure drop.

Optimizing Adsorption Systems

To maximize adsorption efficiency, factors such as **column design, adsorbent selection, and operational parameters** must be carefully optimized. Monitoring the breakthrough curve provides valuable insights for improving system performance and ensuring the best utilization of silica gel in real-world applications.

4. Apparatus Required

The apparatus required for this experiment are listed below;

- Activated Silica gel
- RH Meter
- Glass Column
- Connecting Pipes
- Bubble Flow Meter
- Cotton Plug
- Stop watch
- Retort Stand
- Squeeze bulb

5. Schematic of Experimental Setup

Experimental setup includes the following components:

- Glass column with Activated Silica gel.
- Bubble flow meter connected to one end of a column.
- Relative humidity (RH) meter connected to the other end of a column.

We show the experimental setup used in the laboratory in the following figures (figure 3) along with the schematic (figure 4) of the adsorption breakthrough experiment.

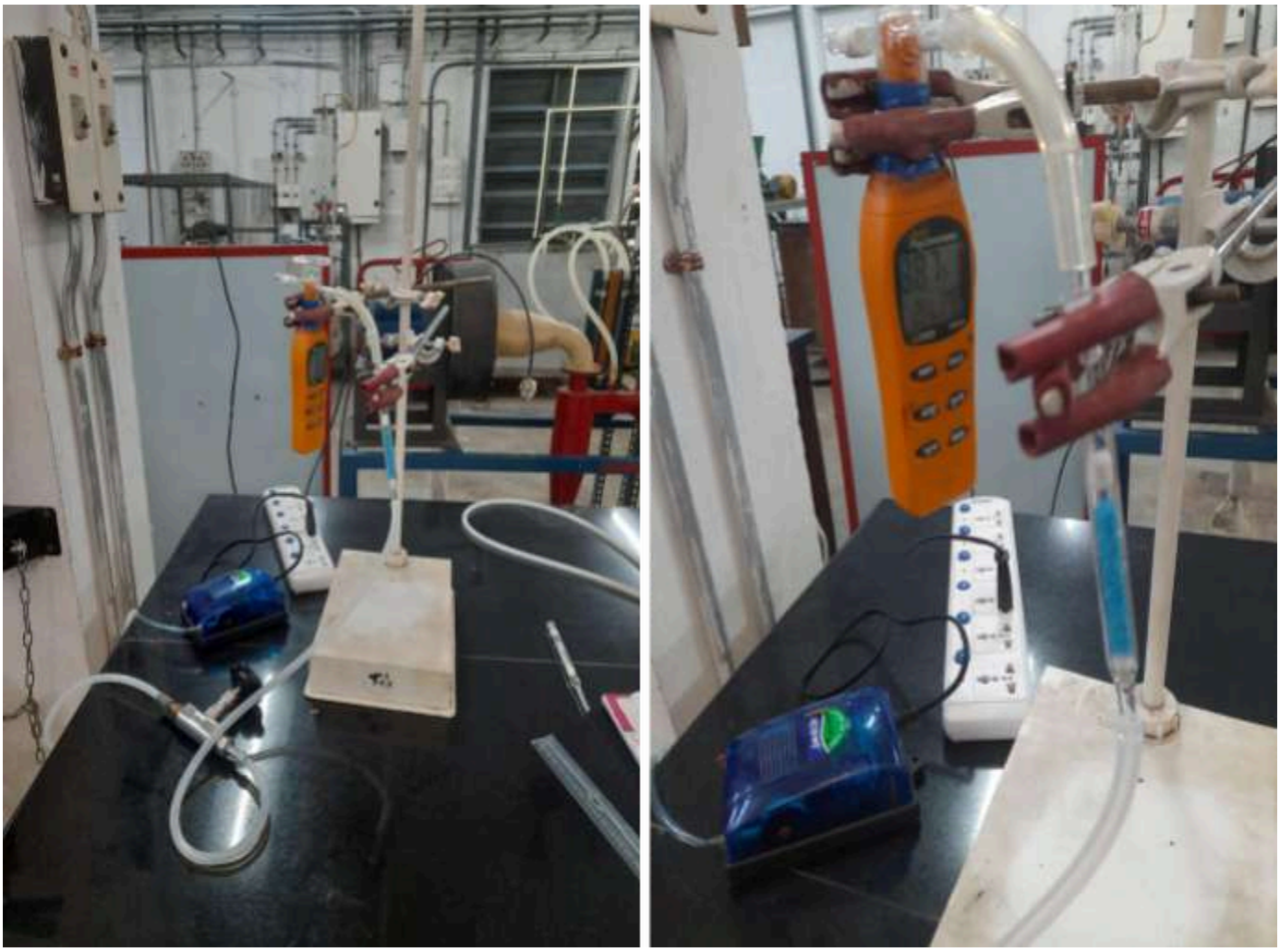


Figure 3: The Experimental Set-up used in the experiment

6. Procedure

● Fill a beaker with soapy water and generate a soap bubble at the tip of the bubble flow meter by allowing air to pass through it.

● Mark two fixed points along the tube and measure the time taken for the bubble to travel between these points using a stopwatch.

● Using the known volume between these points we can calculate the volumetric flow rate.

● We use a 3 cm column of activated silica in a glass column which is sealed at both ends with cotton plugs to prevent external airflow.

● Then secure the column to a retort stand and connect one end to a bubble flow meter and the other to a relative humidity (RH) meter using connecting pipes.

● Maintain a constant flow rate and start the bubble flow meter.

● Record RH readings at one-minute intervals.

● Determine the volumetric flow rate of the gas using soapy water and a squeeze bulb.
● Capture snapshots of the glass column every minute to monitor colour changes in the silica over time.

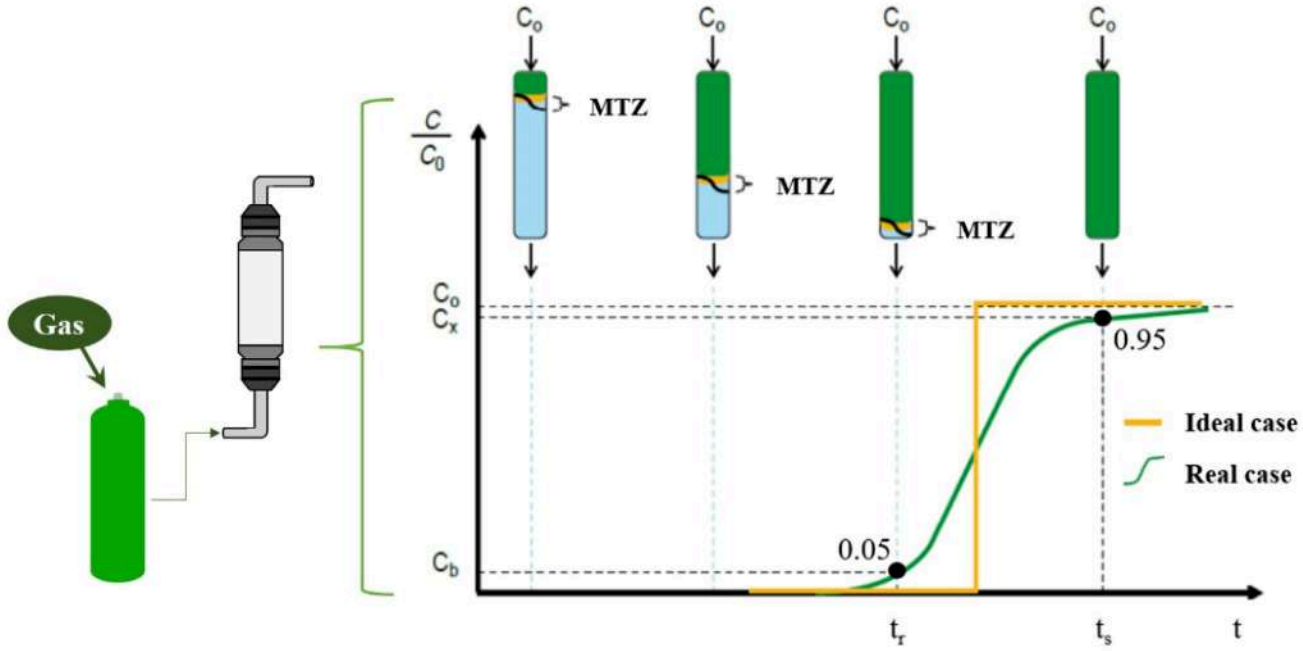


Figure 4: Schematic representation of Adsorption Breakthrough

7. Experimental Observations

In the following two tables (table 1 & 2), we include the experimentally observed data along with the values of several parameters related to the experimental equipment and the materials used.

Parameters	
D - Column Diameter	10.2 mm
Air volume flow rate	3.37078 mL/s
Packing height	5.7 cm
Inlet humidity	74.1 %
Weight of packing	1.46 g

Table 1: All the required parameter values

The following data contains the relative humidity values with time.

Time (min)	RH (%)	Time (min)	RH (%)	Time (min)	RH (%)
0	74.1	21	71.9	42	72.9
1	59.3	22	72.1	42	72.9
2	56.7	23	71.9	43	72.9
3	58.4	24	72.1	44	72.9
4	60.5	25	72.1	45	72.8
5	62.2	26	72.3	46	72.7
6	64.5	27	72.4	47	72.7
7	66.1	28	72.6	48	72.6
8	66.1	29	72.8	49	72.6
9	66.8	30	72.8	50	72.4
10	67.4	31	72.9	51	72.2
11	67.9	32	72.8	52	71.7
12	68.8	33	72.9	53	71.4
13	69.6	34	73	54	69.5
14	69.7	35	73.2		
15	70.1	36	73.2		
16	70.3	37	73.3		
17	70.7	38	73.3		
18	70.9	39	73.2		
19	71.4	40	73.2		
20	71.8	41	73		

Table 2: Relative Humidity values of the outlet

8. Sample Calculations

The relative humidity is the ratio of how much water vapor is in the air to how much water vapor the air could potentially contain at a given temperature. Using this definition we can approximate the trend of $C(t)/C_{initial}$ using the RH (%) trend with time which is shown in figure 6 in the "*Results & Discussion*" section.

We have observed the fraction of white part, which symbolises the amount of mass of silica gel that has been hydrated. We have observed those values for 11, 20, 28th minutes which are shown in the following figures (5).

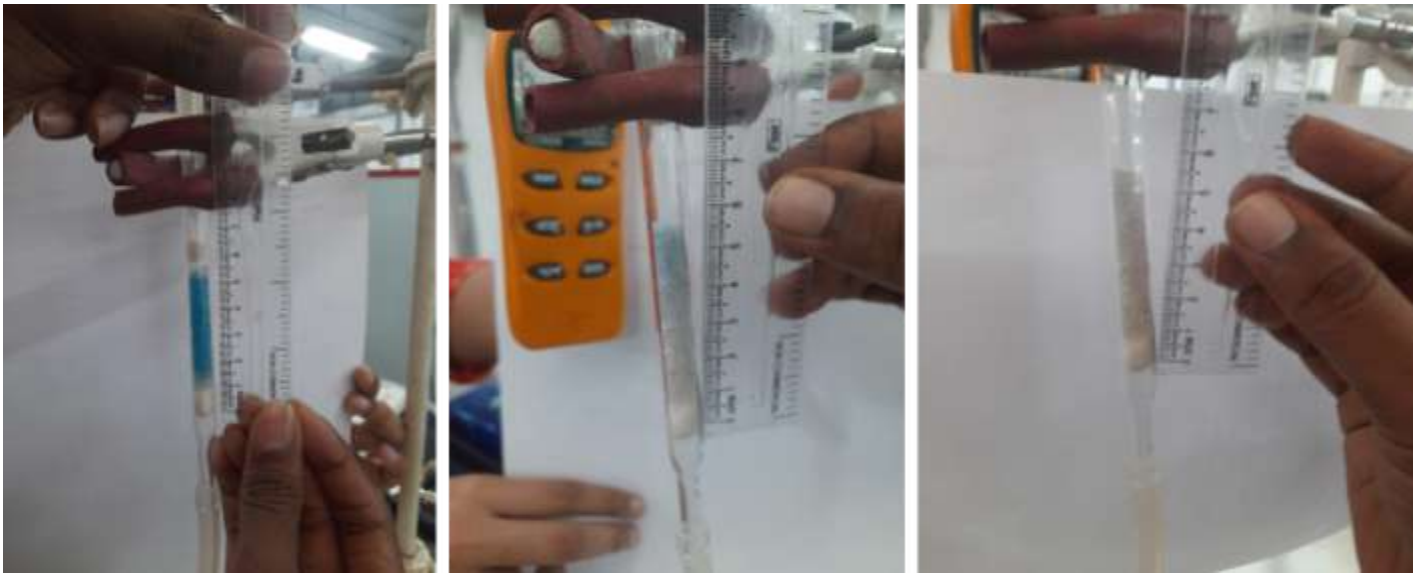


Figure 5: The fraction of white section packing at the time 11, 20 & 28th minutes

Then with the available RH (%) and time data we have used **Rosen model** to fit them with in order to obtain an estimate of the overall mass transfer coefficient (K_{Ai}). The Rosen model combines internal & external mass transfer resistances and is given by the following expression,

$$\rho_A / \rho_{A0} = C_A / C_{A0} = \frac{1}{2}[1 + erf(\frac{(3Y/2X)-1}{2\sqrt{\nu/X}})] \quad (1)$$

where erf is the error function and is given by

$$erf(t) = \frac{2}{\sqrt{\pi}} \int_0^{\pi} e^{-t^2} dt \quad (2)$$

And the other parameters of this model are as follows;
X is bed length parameter, ν is film resistance parameter and their corresponding expressions are given in equations 3 & 4 respectively along with the expression of Y in equation 5.

$$X = \frac{3D_{Ai}K^{*}_{A3i}Z\rho_s}{mU_zR^2} \quad (3)$$

$$\nu = \frac{D_{Ai}K^{*}_{A3i}\rho_s}{RK_{Ai}} \quad (4)$$

$$Y = \frac{2D_{Ai}}{R^2}\left(t - \frac{Z}{U_z}\right) \quad (5)$$

where, $D_{Ai} = 2.60 \times 10^{-5}\, m^2/s$ (diffusion coefficient of water vapor),

$R = 0.0051\, m$ (column radius),

$U_z = \frac{volumetric\ flow\ rate}{\pi R^2} = 0.04125\, m/s$ (superficial vapor velocity),

$Z = 0.057m$ (packing height), m is numerical constant,

$\rho_s = 2200\, kg/m^3$ (density of silica gel),

$K^{*}_{A3i} = 20\, m^3/Kg$ (linear adsorption constant)

And using these parameters, constants we have performed a non-linear optimization on the model to fit the data which implies that calculation for a single data point is not significant to show here and therefore we have provided the flow of calculations to obtain the required results.

Thus we obtain the mass transfer coefficient (K_{Ai}) of silica gel for adsorption, which is listed in the following section. It is to be noted that to perform this optimization we have used the "*lsqnonlin*" function in MATLAB which requires a suitable ordered initial value of the decision variables.

At the end, we estimate the maximum adsorption capacity of the silica gel used using the following expression,

$$W_{max} = VF\int_0^{t_e}(\rho_v(t))\left(1 - \frac{C_{out}(t)}{C_{initial}}\right)dt \quad (6)$$

where, VF is the volumetric flow rate of air,

t_e is the equilibrium time.

Due the change in the relative humidity, the density of vapour will also vary with time and that is captured by the following equation,

$$\rho_v(t) = \frac{RH(t) \times P_{sat}(T)}{R_v \times T} \quad (7)$$

here, $T = 30 + 273.15 = 303.15\, K$ (absolute experiment temperature),

$P_{sat}(303.15K) = 4239.651\, Pa$ (saturation vapor pressure),

$R_v = 461.5\ J/Kg.K$ (gas constant for water vapor).

The saturation vapor pressure can also be computed using Teten's equation,

$$P_{sat}(T) = 611.2 \times exp\left(\frac{17.62(T-273.15)}{T-30.03}\right) \quad (8)$$

where, the pressure is in Pa and the temperature is in K scale.

9. Results & Discussions

This section consists of two important results out of this experiment and they are as follows,

I. Mass Transfer Coefficient for adsorption

At first we show the trend of $C(t)/C_{initial}$ with which gives us the breakthrough curve and this trend is similar to that of $RH(\%)\ vs\ time$. So for simplicity and compactness only one of them is shown in the following figure (6).

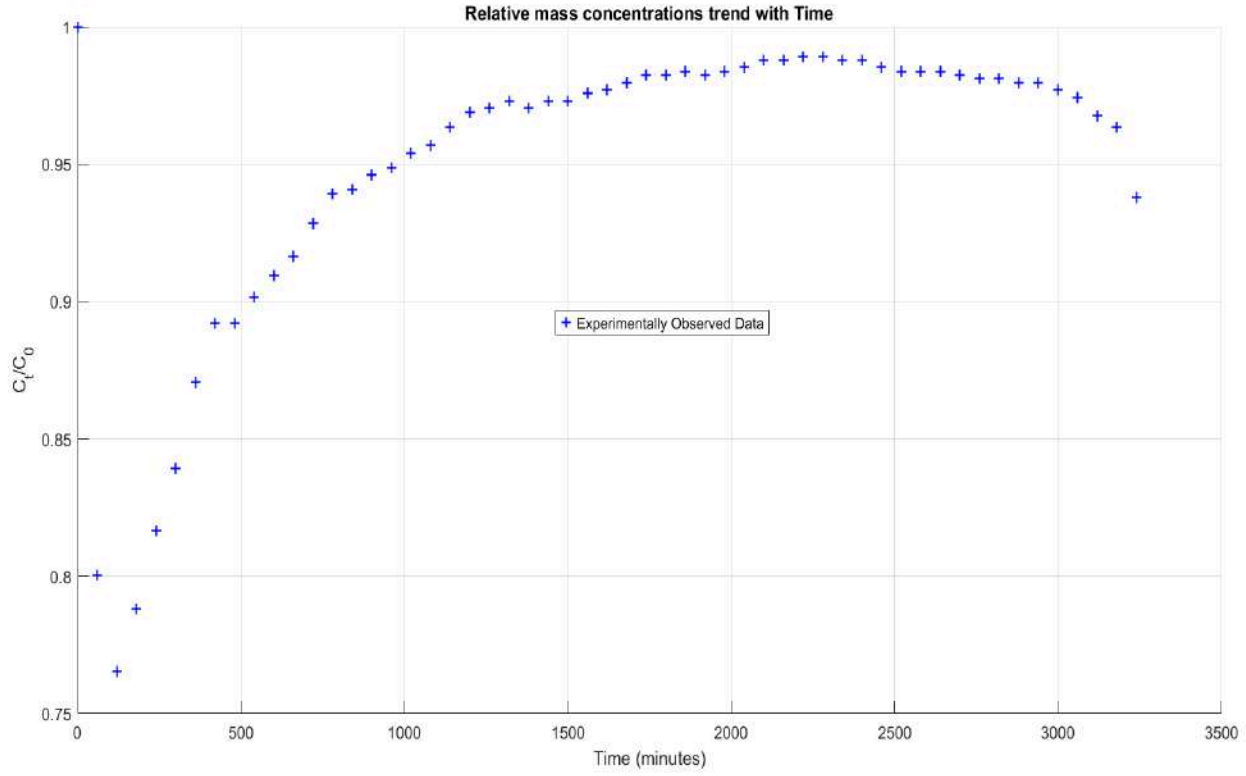


Figure 6: The Breakthrough Curve Trend

The value of overall mass transfer coefficient (K_{Ai}) is coming out to be 0.000340 m/s and the numerical constant value (m) for this fit is coming out to be 100.

II. Estimation of Maximum Adsorption Capacity

The trapezoidal rule is used to estimate the numerical value of the definite integral shown in equation 6. We thus obtain $W_{max} = 0.01073\ g$ as the maximum adsorption capacity.

10. Conclusions

- We observe that the breakthrough curve partially follows the sigmoid like trend and the reason it is not exactly following the same trend is because the volumetric flow rate value we have performed our experiment with was comparatively high for it to capture the portions before breakpoint in the standard breakthrough curve.
- After some point when the complete silica gel gets saturated, we observe the curve becoming flat which means that the complete bed has reached its adsorption limit. The subtle fluctuations at the end might rise because of the equipment's instability.
- From the shape of the adsorption breakthrough curve, we conclude that flow in the fixed bed adsorption experiment is non ideal. Ideally the curve should rise steeply after the breakthrough point and reach near the saturation limit and remain so for a significant amount of time. However in our case the rise is gradual which can be accounted for by the *internal resistance* inside the packed bed.
- Since the amount of water vapor adsorbed by the silica gel is directly proportional to the flow rate of the air, we can conclude that increasing the flow rate will enhance the adsorption process and also the amount adsorbed.

11. References

I. *Representation of Breakthrough Curves for Fixed-Bed Adsorbers and Reactors using Moments of Impulse Response,* article by Linek et. al. to understand the theoretical assumptions and robustness of using the Rosen model for adsorption breakthrough.

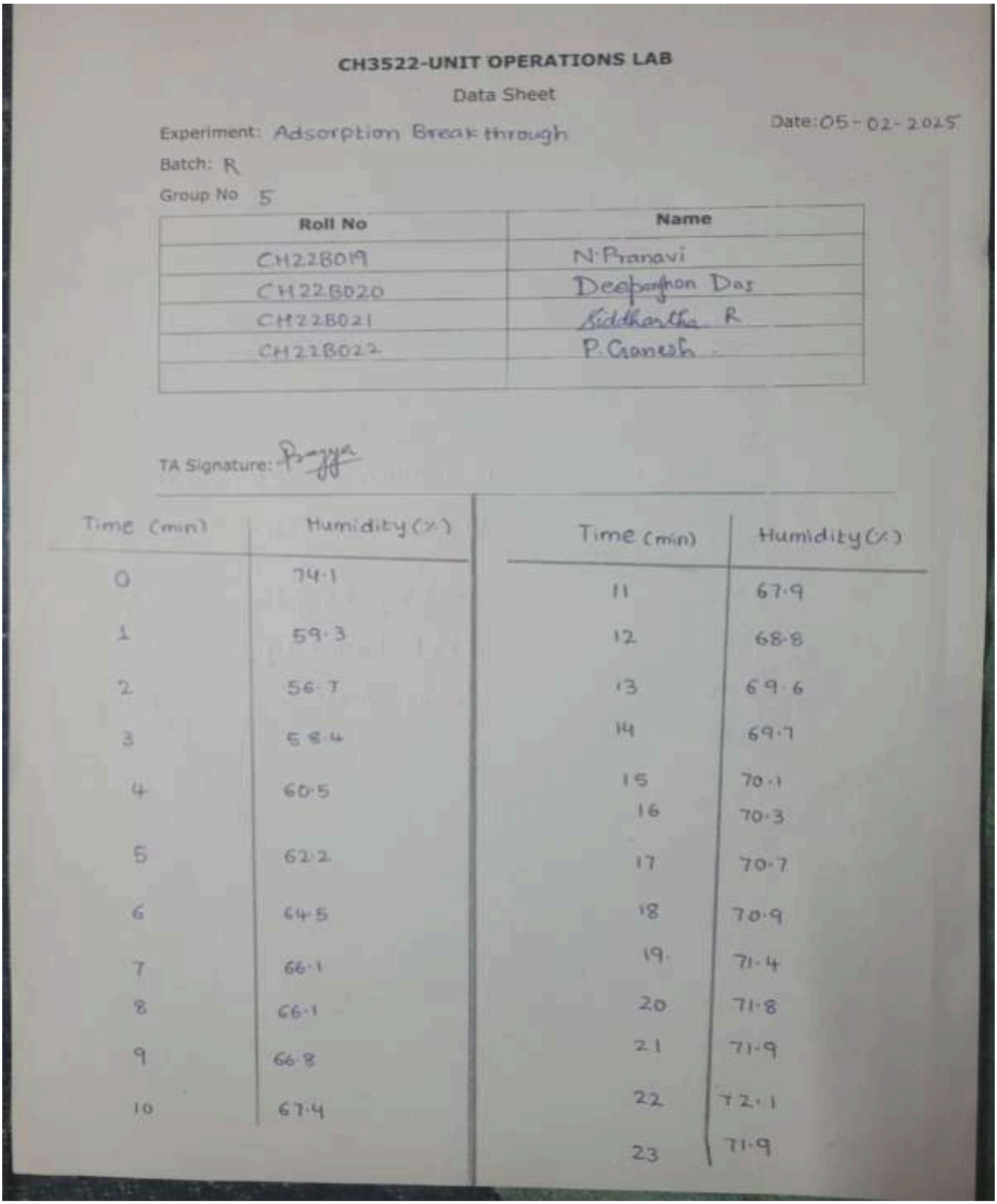
II. Air Density Table to find density at specific temperature.

III. Table provided by Wired Chemist to find vapor pressure of water at specific temperature.

IV. Laboratory exercise: Water vapor and Liquid moisture transport to find the diffusion coefficient of water vapor.

V. Aqua-Calc to find the recorded density of silica gel.

VI. The GitHub repository contains all the related data and coded scripts used for calculations.



CH3522-UNIT OPERATIONS LAB

Data Sheet

Experiment: Adsorption Break through

Date: 05-02-2025

Batch: R

Group No 5

Roll No	Name
CH22B019	N. Pranavi
CH22B020	Deepanjhon Das
CH22B021	Siddhartha R
CH22B022	P. Ganesh

TA Signature: Bajya

Time (min)	Humidity (%)
0	74.1
1	59.3
2	56.7
3	58.4
4	60.5
5	62.2
6	64.5
7	66.1
8	66.1
9	66.8
10	67.4

Time (min)	Humidity (%)
11	67.9
12	68.8
13	69.6
14	69.7
15	70.1
16	70.3
17	70.7
18	70.9
19	71.4
20	71.8
21	71.9
22	72.1
23	71.9

Figure : Laboratory Data 1

Time (min)	Humidity (%)
24	72.1
25	72.1
26	72.3
27	72.4
28	72.6
29	72.8
30	72.8
31	72.9
32	72.8
33	72.9
34	73
35	73.2
36	73.2
37	73.3
38	73.3
39	73.2
40	73.2
41	73.0
42	72.9
43	72.9
44	72.9
45	72.8
46	72.7

Time (min)	Humidity (%)
48	72.7
49	72.6
50	72.6
51	72.4
52	72.2
53	71.7
54	71.4
55	69.5

Parameters :-

D - column diameter : 10.2 mm.

Volume flow rate : 3.37078 ml/s

Packing height : 5.7 cm

Inlet humidity : 74.1 %.

Weight of packing : 1.46 g.

Figure : Laboratory Data 2