

# INDIAN INSTITUTE OF TECHNOLOGY

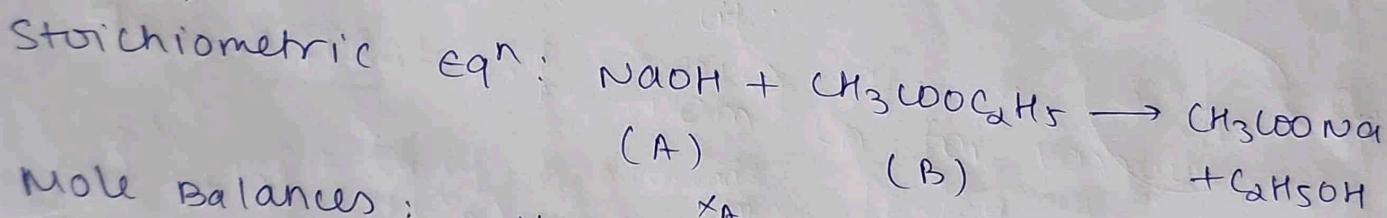
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SHEET NO. \_\_\_\_\_

## Objective:

- 1) To determine the order of reaction between sodium hydroxide & ethyl acetate using a plug flow reactor.
- 2) To find the rate constant at a particular temp.

## Theory:



Mole Balances:

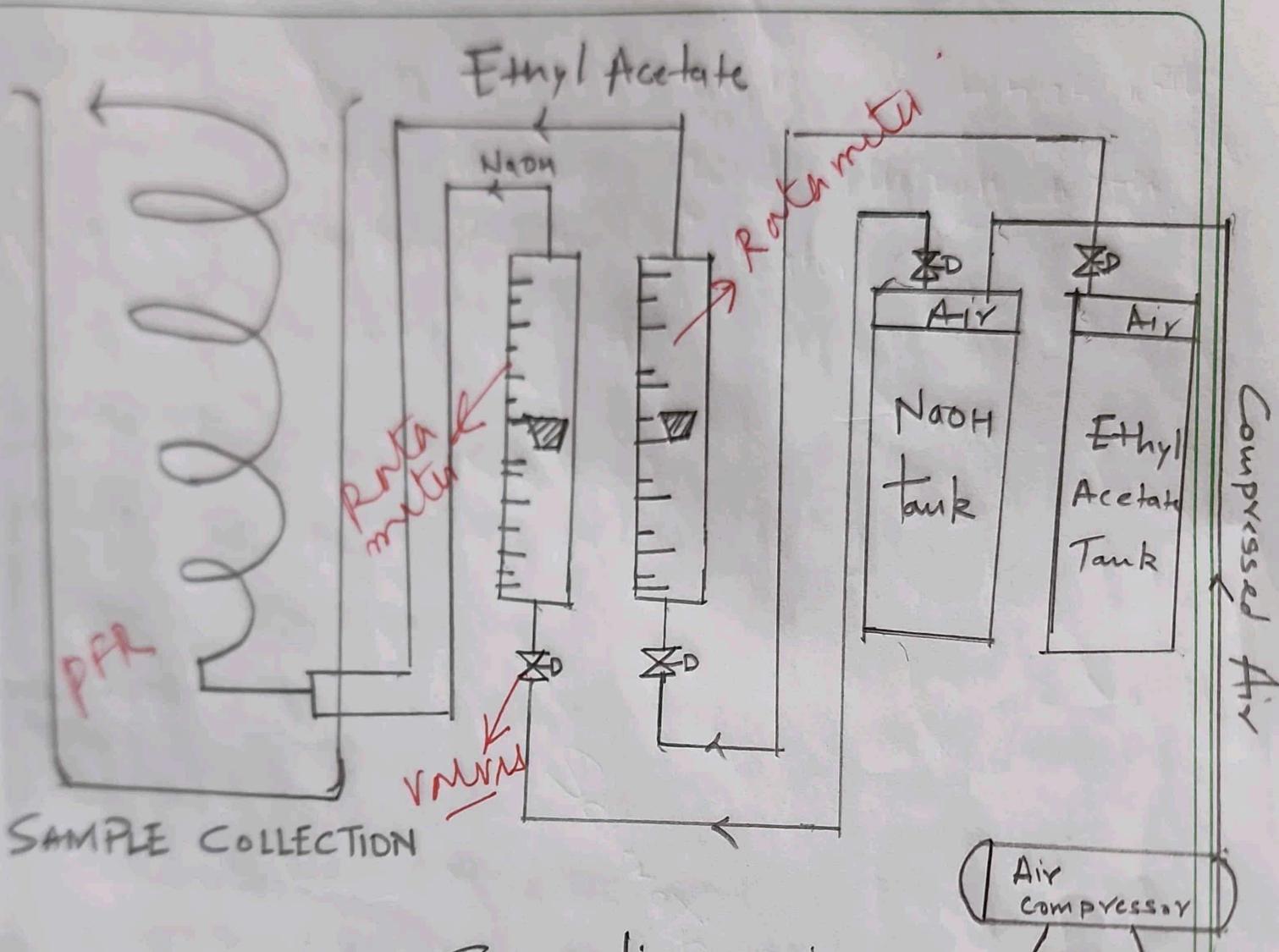
$$\frac{V_R}{F_{A_0}} = \int_0^{x_A} \frac{dx_A}{-r_n}$$

Rate Eqn:  $-r_A = k_1 C_{A_0}^2 (1-x_A) (M-x_A)$

Assuming 2nd order reaction,  
where  $M = \frac{C_{B_0}}{C_{A_0}}$ ,  $x_A$  = conversion of A,  $k_2$  = rate constant

$$\frac{V_R}{F_{A_0}} = \frac{V_R}{V_0 C_{A_0}} = \frac{I}{C_{A_0}} = \frac{1}{k_2 C_{A_0}^2 (M-1)} \ln \left( \frac{M-x_A}{M(1-x_A)} \right) = \frac{1}{k_2} f(x_A)$$

where  $V_0 = V_A + V_B$  &  $I = \frac{V_R}{V_0}$



Schematic Diagram

### Apparatus:

- 1) stainless steel reactor in a constant temp. water bath.
- 2) stop watch

### Dimension of the reactor.

length of the coil : 609.6 cm, inside dia. of the tube = 1.23 cm

volume of the reactor (vol. of tube) : 0.724 liters

### chemicals:

- 1) succinic acid (N/50)
- 2) NaOH (N/20)
- 3)  $\text{CH}_3\text{COOC}_2\text{H}_5$  (N/10)
- 4) phenolphthalein indicator

### Procedure:

- 1) fill both the storage tanks of ethyl acetate & NaOH & calibrate the flow meters.
- 2) Adjust the control values to set the flow rates. Try to keep both flow rates equals.

- 3) Take 5ml of this sample & titrate with the standard succinic acid solution with phenolphthalein as indicator.
- 4) Take 5ml of supplied NaOH & titrate with the standard succinic acid solution to get  $C_A'$  g mol/l/ut.
- 5) calculate  $x_A$  from  $C_A$  for various  $T = \frac{V_R}{V_0}$
- 6) plot  $f(x_A)$  against  $x$  & determine the rate constant from the slope.

N.B.  $\circ$   $C_{A0} = \frac{V_A}{V_A + V_B} C_A' \quad \& \quad C_{B0} = \frac{V_B}{V_B + V_A} C_B'$

### Observations:

Strength of succinic Acid =  $N_{150} = 0.01 \text{ M}$  (dibasic - acid)

Volume of sample = 5 mL

Strength of NaOH (found by titration) =  $N_{18.5} = 0.054 \text{ N}$

$$C_{A_0} = \underline{\underline{0.027 \text{ mol/L}}}$$

Flow rate (LPH)	$C_{A_0}$	Volume (mL)	$X_A$	Average $X_A$
5	0.027	2.1 2.5 2.2	0.688 0.63 0.67	0.664
7.5	0.027	2.8 2.6 2.9	0.585 0.615 0.57	0.59
10	0.027	3.2 3.1 3.0	0.526 0.541 0.555	0.541

### Sample Calculations:

$$N_1 V_1 = N_2 V_2 \quad (\text{meq balance for titration})$$

for  $V = 2.1 \text{ mL}$

$$0.027 \times 2.1 = N_2 \times 5 = N_2 = 0.0084$$

$N_2 = M_2 = 0.0084 \text{ mol/L}$  NaOH is a monoacidic base

$$X_A = \frac{0.027 - 0.0084}{0.027} = 0.688$$

$$\text{Average } x_A = \frac{0.688 + 0.63 + 0.674}{3}$$

$$f(x_A) = \frac{1}{C_{A_0}^2 (M-1)} \ln \left( \frac{M-x_A}{M(1-x_A)} \right)$$

$$M = 2.04$$

$$f(x_A) = \frac{1}{(0.027)^2 (2.04-1)} \times \ln \left( \frac{2.04 - 0.664}{2.04(1-0.664)} \right)$$

$$= 919.75 \text{ L}^2/\text{mol}^2$$

$$\tau = \frac{V_R}{\gamma_0} = \frac{0.724}{5+5} \times 3600 = 260.64 \text{ sec}$$

$T_0 \text{ (s)}$	$f(x_A) (\text{L}^2/\text{mol}^2)$
260.64	919.75
173.76	726.00
130.32	620.13

Results: slope from the plot =  $2.29 \text{ L mol}^{-2} \text{ s}^{-1}$   
rate constant = slope  $\times C_{A_0} = 2.29 \times 0.027$   
=  $0.0621 \text{ mol}^{-1} \text{ s}^{-1}$

Therefore, the rate constant of given reaction at existing temperature conditions is  $0.062 \text{ L mol}^{-1} \text{ s}^{-1}$

### Discussion:

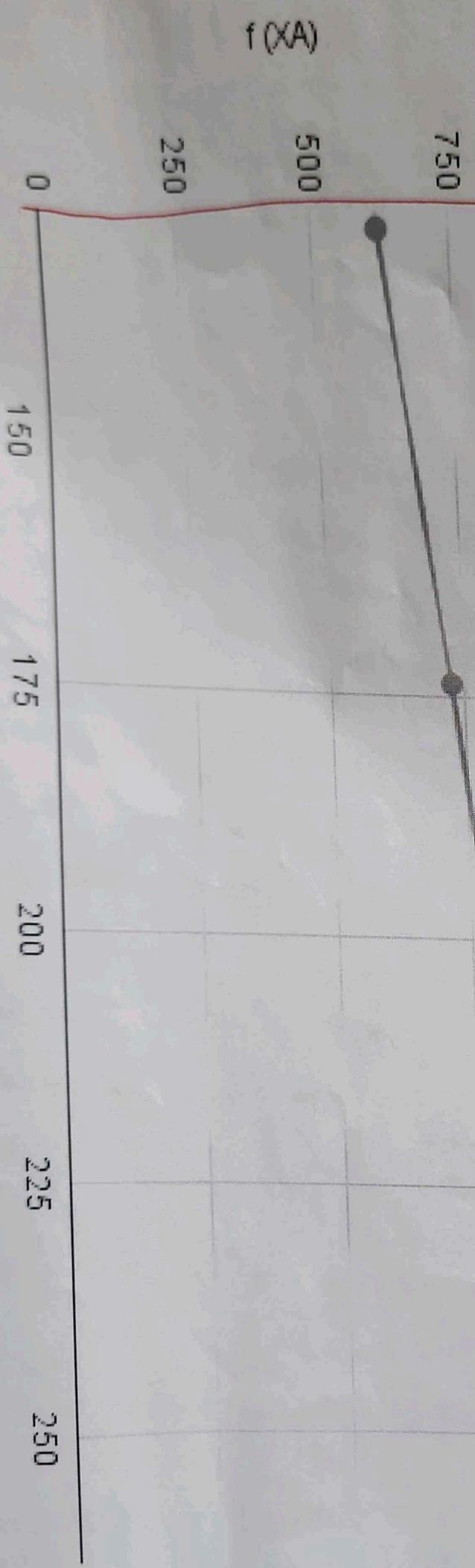
- ① The conversion is seen to decrease with increasing flow rate which seems logical given the residence time is decreasing.
- ② Rotameters are one possible source of error because the marker inside never stays in full equilibrium state therefore, there is always a very small error adding to the system.
- ③ During titration, the final solution is not exactly neutral since phenol phthalein change colour at  $\text{pH} > 7$ .
- ④ care must be taken that the phenol phthalein is not added in excess while titrating to the mixture.

## Conclusion:

With this assumed 2<sup>nd</sup> order reaction taking place in a PFR, we can easily observe the desired results & trends between all relevant values. The rate constant value obtained for the reaction occurring in the PFR is a reasonable value considering the existing temperature condition.

$f(X_A)$  vs  $T$  (sec)

●  $f(X_A) = 2.29^x + 324$



DATE

Exp-2

## Rate study in a CSTR

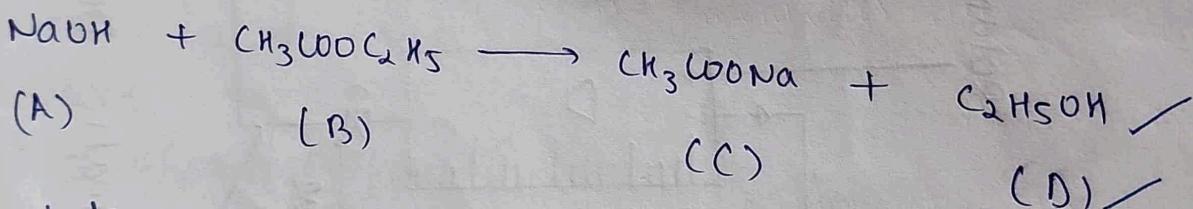
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## Objective:

- 1) To determine the order of Reaction between sodium hydroxide & ethyl acetate using a CSTR reactor
  - 2) To find rate constant at a particular temperature

## Theory:

Stoichiometric eqn:



Mole balance:

$$\frac{V_R}{F_{A_0}} = - \frac{x_A}{y_A} \quad \text{---(1)}$$

$$\text{Rate Eqn : } -r_A = k_2 C_{A_0}^2 (1-x_A) (N-x_A) \quad (2)$$

Assume 2nd order in  $A_1$

Assume 2<sup>nd</sup> order Rxn:

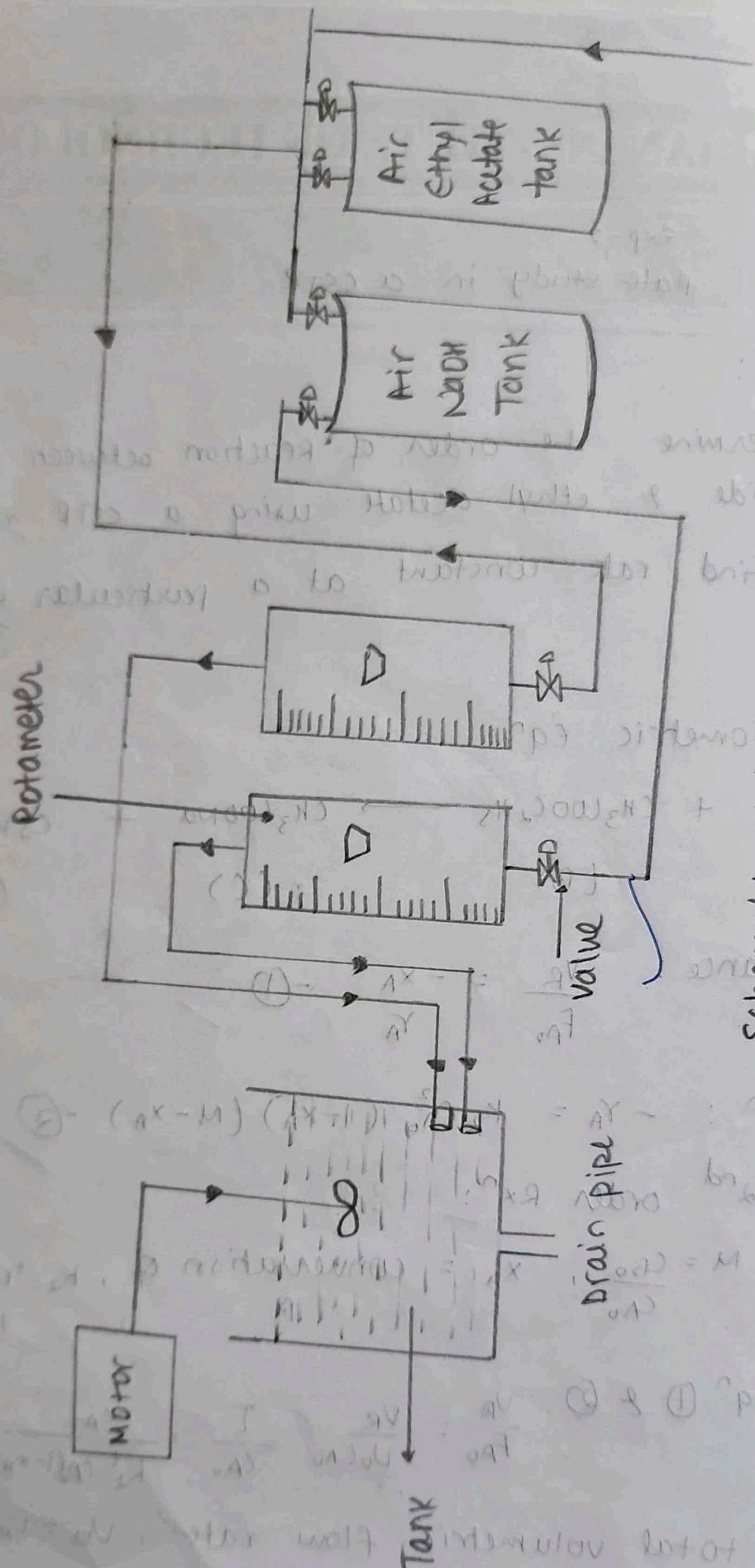
where  $M = \frac{C_{B_0}}{C_{A_0}}$ ,  $x_A$  = conversion of A,  $k_2$  = rate constant

$$\text{from Eq } ① \text{ & } ②, \frac{V_R}{F_{A_0}} = \frac{V_R}{V_0 C_{A_0}} = \frac{I}{C_{A_0}} = \frac{x_A}{k_2^t C_{A_0}(1-x_A)(N-x_A)} = \frac{1}{k_2} f(x_A) \quad \text{in lit/mol min}$$

Where total volumetric flow rate,  $V_B = V_A + V_B$

$$e \quad T = \frac{VR}{V_0}$$

Schematic Diagram



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Apparatus:

- 1) S-S reactor (volume of reactor = 2.815 lit)
- 2) constant temp. water bath
- 3) stop watch
- 4) conical flask

chemicals:

- i) succinic acid (N/50)
- ii) NaOH
- iii)  $\text{CH}_3\text{COOC}_2\text{H}_5$  (N/10)
- iv) phenolphthalein indicator

Procedure:

- 1) fill both the storage tanks of ethyl acetate & NaOH and calibrate the flow meters.
- 2) Adjust the control valves to set the flow rates.  
Try to keep both flow rates equal.
- 3) After attaining steady-state, collect the sample in a flux from the outlet.
- 4) Take 5ml of sample & titrate with the standard succinic acid solution with phenolphthalein as indicator
- 5) Take 5ml of supplied NaOH solution by the

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standard succinic acid solution to get  $C_{A_0}$  g mol/l it

(6) calculate  $x_A$  from  $C_A$  for various  $T = \frac{V_R}{V_0}$

(7) Plot  $f(x_A)$  against  $T$  & determine the rate constant from the slope.

$$\text{N.B} \quad C_{A_0} = \frac{V_A}{V_A + V_B} \times C_{A_0}^1 \quad \text{&} \quad C_{B_0} = \frac{V_B}{V_B + V_A} \times C_{B_0}^1$$

Observation table:

$$N_1 V_1 = N_2 V_2 \Rightarrow \frac{1}{50} \times 12.8 = N_2 \times 5 \Rightarrow N_2 = \frac{N}{19.53}$$

Flow Rate (LPH)	T (s)	$C_{A_0}$ (mol/L)	Titration Volume (mL)	$x_A$	$\log x_A$	$f(x_A)$
5	1013.4		2.9 2.6 2.5	0.547 0.594 0.609	0.583	1507.927
7.5	675.6	0.0256	3.0 2.9 3.0	0.531 0.545 0.531	0.538	1206.597
10	506.7		3.2 3.1 3.4	0.5 0.516 0.469	0.495	992.831

Sample calculation:

$$\text{conc. of succinic acid} = N/50 = 0.02M$$

$$\text{vol. of NaOH} = 5\text{ml}$$

$$\text{vol. of succinic acid} = 12.8\text{ml}$$

$$\text{conc. of NaOH in mixture } (C_{A_0}) = 0.005 \times 12 \times \frac{1}{2} = 0.0256$$

$$\text{For } V = 2.9\text{ml}$$

$$0.02 \times 2.9 = N \times 5 \Rightarrow N = 0.116M$$

$$C_A = 0.0512M$$

$$C_{A_0} = 0.0512 \times \frac{5}{10} = 0.0256$$

$$X_A = \frac{0.0256 - 0.116}{0.0256} = 0.547$$

$$\text{Avg } X_A = \left( \frac{0.547 + 0.594 + 0.609}{3} \right) = 0.583, f(x_A) = 1507.92$$

$$f(x_A) \rightarrow \text{from plot} \Rightarrow y = 1.9021x + 123.92 \\ \text{vs T(S)}$$

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## Results:

- ① The slope of the plot is  $1.9021$
- ② The rate constant is  $\text{slope} \times C_{P_0} = 1.9021 \times 0.0286$   
Thus, the rate constant of the given reaction  
at a temperature  $29^\circ\text{C}$  comes out to be

## Discussion:

- 1) The calculated rate constant value differs significantly from the one determined using a PFR, indicating a deviation of more than 100%.
- 2) possible experimental source of error include the inability of the water bath to maintain a constant temperature leading to non-steady-state conditions and potential impact on  $R^n$  kinetics
- 3) Rotameters pose a source of error as the markers inside may not be in complete equilibrium; however, the use of a calibration curve helps achieve some level of accuracy.
- 4) It is crucial not to add an excess of phenolphthalein to the solution during titration.

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- 5) Careful observation of color change is necessary during titration to accurately identify the titre volume as a slight delay can result in faulty reading.
- 6) Precise handling is required to ensure no drop of succinic acid is wasted during titration, as it can introduce error in the titre volume.
- 7) Vigilance in observing the meniscus during titration is essential to avoid errors in ~~noting~~ noting the titre volume due to parallax.

Conclusion: The experiment aimed to investigate the kinetics of the NaOH-CTOAc Rxn using a CSTR, aiming to determine the reaction order & rate constant at a specific temperature. From the experimental data, the Rxn order with respect to NaOH & CTOAc was determined using graphical analysis, such as the integrated rate laws. The Rxn order provides insights into the stoichiometry & mechanism of the Rxn. Furthermore, by plotting  $f(x_A)$  vs  $t$  and applying appropriate kinetic models, the rate constant ( $k$ ) of the Rxn was calculated at the specific temperature of the experiment. Factors such as mixing efficiency, temperature control, & reactor design influence the observed reaction rates & must be carefully considered in the design & operation of chemical reactors for industrial applications.

$$f(XA) \sqrt{5} T$$

1500  
1400  
1200

$$y = 1.9021x + 123.92$$

1000

$f(XA)$

800

600

400

200

0

200

400

600

800

1000

1200

$T(s)$

Objective: To study physical dissolution of benzoic acid in water & determine the rate constant for physical dissolution.

Theory: The rate of physical dissolution of benzoic acid in water ( $R^1$ ) is given by:

$$R^1 = \frac{(\text{Benzoic Acid in water})_t - (\text{Benzoic Acid in water})_{t=0}}{\text{Time (sec)}} \times \text{vol of water (l)}$$

( $R^1$  in gmol/s or kmol/s)  
specific rate of dissolution ( $R$ )

$$R = \frac{R^1}{A_{\text{ar}}} \text{ kmol/m}^2 \text{ s}$$

In this experiment, a regular shaped benzoic acid solid is dissolved into a sol<sup>n</sup> involving no Rx<sup>n</sup>. The time dependent conc<sup>n</sup> of solid in sol<sup>n</sup> is analysed to obtain the rate of mass transfer in the process & hence the rate constant. This process is very relevant in industrial opr<sup>n</sup> and is usually accompanied by a dissolved solid-liquid Rx<sup>n</sup>, in which case the rate may be enhanced.

Apparatus: Benzoic Acid cylinder, holding rod, vernier screw callipers, stirrer with shaft temperature controller and heater, NaOH ( $\frac{1}{50}$  M), pipette, acid-base titration

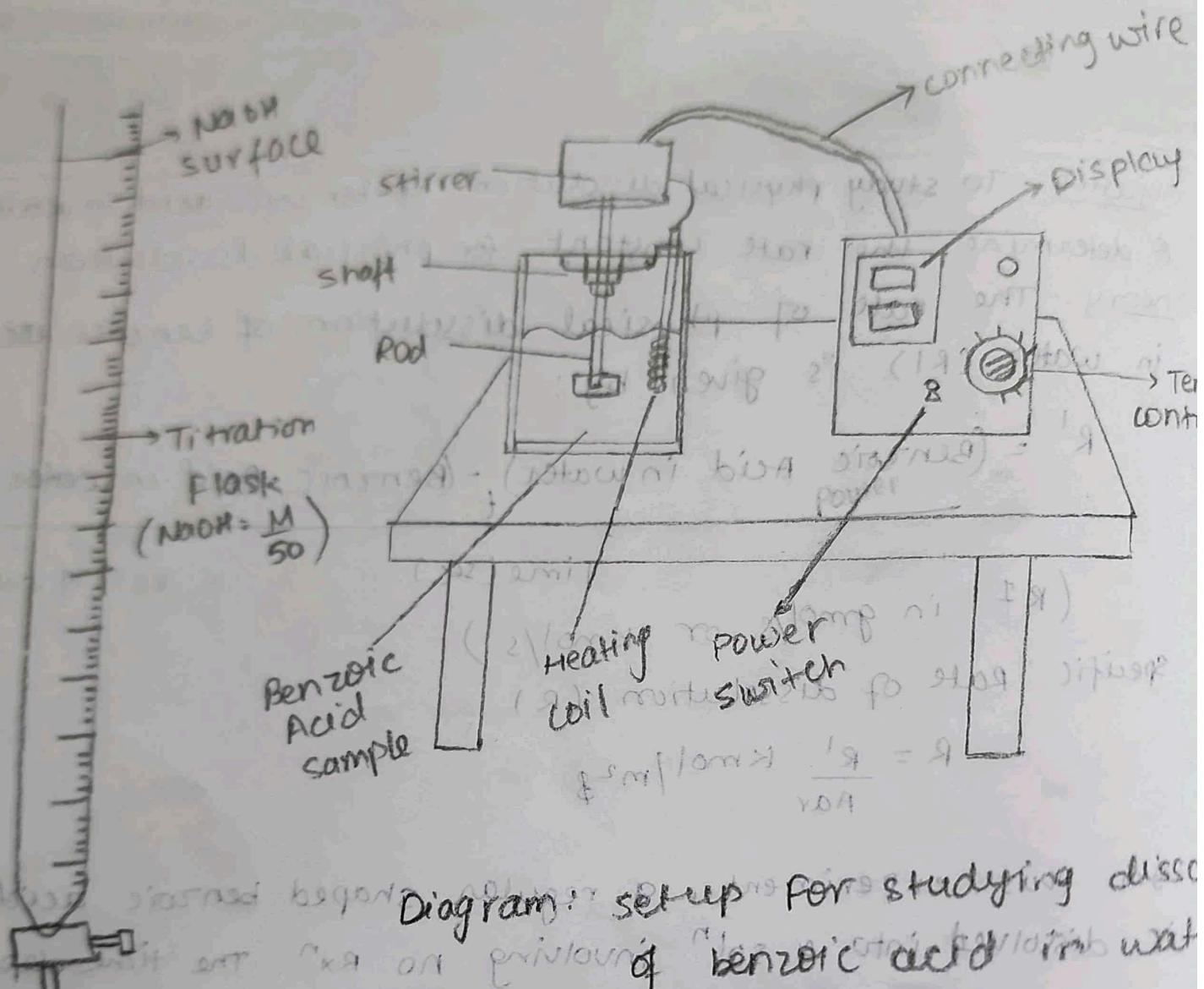


Diagram: setup for studying dissociation constant of benzoic acid in water



conical flask

(with benzoic acid  
CO<sub>2</sub> & phenolphthalein)

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## Procedure:

- 1) Dimensions of the benzoic acid cylinder are measured & it is coupled to the main shaft.
- 2) operating temperature of the water bath is adjusted. The exp. vessel is constant at the desired temp.
- 3) About 400ml of water is added to the inner tank, and the benzoic acid cylinder is adjusted so that a water level of about 3cm above the cylinder surface is maintained.
- 4) speed of the motor is adjusted at the desired level, & the experiment is run for around 3 hrs. After every 10-15min. 5 ml of the sample is titrated and titrated again with the N/50 NaOH sol<sup>n</sup> with phenolphthalein as indicator. Thus the time dependent concn of benzoic acid in water is estimated and the concn vs time data is recorded.
- 5) Motor is stopped & dimension of the cylinder measured (OD, length)
- 6) steps 1-5 are repeated at different rotation speeds & different temperature so that the temperature & speed dependence of the rate constant can be evaluated.

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Observation

(before dissolution)

$$\text{diameter of pellet} = 3.1 + 0.001 \times 39 + 2.54 \text{ cm}$$

$$= 3.19906 \text{ cm}$$

$$\text{thickness of pellet} = 1.4 \times 0.001 \times 20 \times 2.54 \text{ cm}$$

$$= 1.4508 \text{ cm}$$

$$\text{dia. of steel rod} = 0.8 + 0.001 \times 5 \times 2.54 \text{ cm}$$

$$= 0.8127$$

(i) Temperature =  $25^{\circ}\text{C}$

Initial Reading (mL)	final reading (mL)	vol of NaOH (mL)	concn of Benzoic Acid ( $C = \frac{x}{50}$ ) M
0	1	1	0.004
1.3	3.1	1.8	0.0072
3.1	5.4	2.3	0.0092
5.4	8.4	3.0	0.012
8.4	11.9	3.5	0.014
11.9	15.5	3.6	0.0144

After dissolution

$$\text{diameter of pellet} = 3.1 + 0.001 \times 13 \times 2.54$$

$$= 3.13302 \text{ cm}$$

$$\text{thickness of pellet} = 1.3 + 0.001 \times 9 \times 2.54 = 1.32286$$

Temperature = 35°C

Time (min)	Initial volume (ml)	Final volume (ml)	Vol of NaOH (ml)	Conc. of Benzoic Acid (M)
5	15.9	17.1	1.2	0.0048
10	17.1	19.2	2.1	0.0084
15	19.2	22.2	3.0	0.012
20	22.2	25.5	3.3	0.0132
25	25.9	29.5	3.6	0.0144
30	30.4	35.4	5.0	0.02
35	35.4	39.5	5.1	0.0204

After dissolution,

$$\text{Dia of pellet} = 3 + 0.001 \times 9 \times 2.54 = 3.0226 \text{ cm}$$

$$\begin{aligned}\text{Thickness of pellet} &= 1.2 + 0.001 \times 48 \times 2.54 \\ &= 1.32192 \text{ cm}\end{aligned}$$

calculation:

1) Temperature =  $25^\circ\text{C}$

sample used = 5ml

Dimensions:

Initial thickness = 1.4508 cm, initial dia = 3.19906 cm

Final thickness = 1.32286 cm, final diameter = 3.13302 cm  
 $t = 30\text{ min}$

$$\text{Rate of dissolution} = \frac{\left( \frac{\text{conc}^n \text{ Benzoic Acid in water}}{\text{time (s)}} \right)_t - \left( \frac{\text{conc}^n \text{ Benzoic Acid in water}}{\text{time (s)}} \right)_{t=0}}{x \text{ (vol of water)}}$$

$$= \frac{0.0144}{30 \times 60} \times \frac{400}{1000} \Rightarrow 3.2 \times 10^{-6} \text{ mol/s}$$

For first reading ( $t = 5\text{ min}$ )

$$c = 0.004 \text{ mol/L} \quad c^* = 0.0276 \text{ K mol/m}^3$$

(saturation conc.)

$$c^* = 0.0276 \text{ mol/L}$$

$$\therefore 1 - \frac{c}{c^*} = 1 - \frac{0.004}{0.0276} = 0.855$$

$$\therefore \ln\left(1 - \frac{c}{c^*}\right) = \ln(0.855) = -0.15665$$

for area, we have

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$$A_r = \frac{\pi D_o a_r L_{ar}}{\downarrow \text{lateral area}} + 2 \left( \frac{\pi}{4} (D_o^2 a_r - D_{\text{rod}}^2 a_r) \right) \downarrow \text{Disc area}$$

where

$D_o a_r$  and  $D_{\text{rod}}$  inner and outer average diameters of the cylinder respectively. The inner diameter is same as rod's outer diameter.

$L_{ar}$  is avg height.

$$D_o a_r = 3.16604 \text{ cm}$$

~~$$D_{\text{rod}} = 1.38683 \text{ cm}$$~~

$$D_{\text{rod}} = 0.8127 \text{ cm}$$

$$\therefore A_r = \pi \left( \frac{3.16604}{100} \right) \times \left( \frac{1.38683}{100} \right) + 2 \times \frac{\pi}{4} \times \left( \frac{3.16604^2 - 0.8127^2}{100^2} \right)$$

In a similar way, we carry out the calculation of  $\ln(1 - \frac{C}{C_f})$  at every time stamp

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Time (min)	Conc.	$1 - \frac{C}{C^*}$	$\ln\left(1 - \frac{C}{C^*}\right)$
5	0.004	0.855	-0.156
10	0.0072	0.74	-0.302
15	0.0092	0.67	-0.405
20	0.012	0.56	-0.57
25	0.014	0.493	-0.71
30	0.0144	0.478	-0.74

From graph, plotted b/w time &  $\ln\left(1 - \frac{C}{C^*}\right)$  we get,

$$1 - \frac{C}{C^*} = \exp(-0.0228x + 0.0678)$$

$$C = C^* (1 - \exp(-0.0228x + 0.0678))$$

$$R' = \frac{C(-t)}{b_0 t} \times V_{\text{water}}$$

$\hookrightarrow$  in min

$$R = \frac{R'}{A} \quad , \quad \text{slope} = k_{SL} \times \frac{Ar}{V}$$

where  $k_{SL}$  is the rate constant

$$Ar = 28.5055 \text{ cm}^2, V = 0.4 \text{ L} \approx 0.0004 \text{ m}^3$$

$$\therefore k_{SL} = 5.629 \times 10^5$$

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Results:

For temp  $25^{\circ}\text{C}$   $k_{SL} = 5.629 \times 10^{-5} \text{ m/s}$   
(Dissolution  
Rate constant)

For temp  $35^{\circ}\text{C}$   $k_{SL} = 1.05 \times 10^{-4} \text{ m/s}$

Slope of  $\ln\left(1 - \frac{C}{C_0}\right) = -0.0004$

DISCUSSION

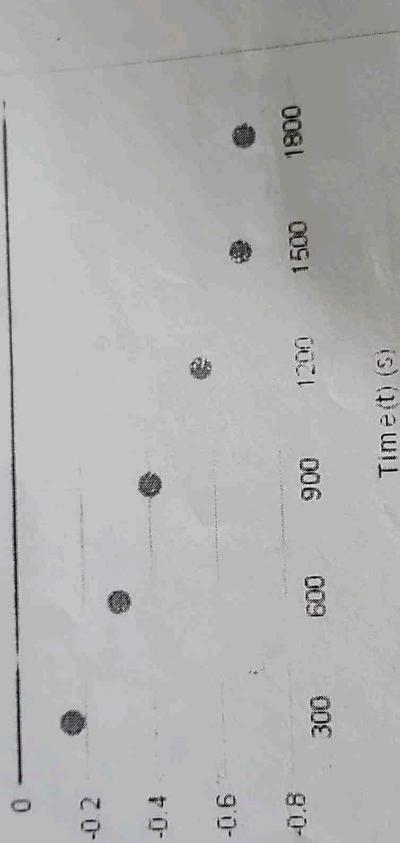
- \* From calculation, the dissolution rate constant has increased when temperature is increased. This is evidence for the fact that the rate of mass transfer increases with temp increases as kinetic energy (on an average) increases for the diffusing molecules ( $E \propto k_B T$ ).  $\rightarrow$  Boltzmann constant
- \* A few ways to make the experiment more efficient and interfential have been suggested below.
  - 1) use of more robust, accurate and lower latency concentration measuring methods than titration, such as gas chromatography.
  - 2) use of compounds other than benzoic acid with more distinct characteristic features.
  - 3) use of more advanced temp controlled mechanisms
- \* While this experiment is a good starting point, it is not suitable to find solubility characteristics on an industrial scale as it is very slow & labour intensive.
- \* The driving force decrease ( $C^* - C$ ), so the rate decreases mathematically  $C = C_0 (1 - e^{-kt}) \Rightarrow \frac{\partial \text{rate}}{\partial t} = \frac{\partial^2 C}{\partial t^2} = -k^2 C_0 e^{-kt} < 0$

### Precautions:

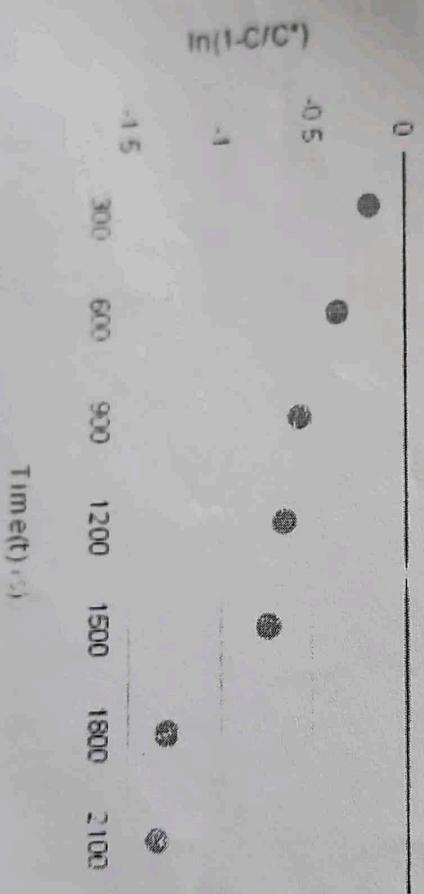
- 1) care should be taken to take small time interval & more number of interval to ensure precision in steady state point.
- 2) care should be taken to keep the titration slow enough so that conc<sup>n</sup> can be precisely known.
- 3) the benzoic acid cylinder should be handled with care, as it is brittle.
- 4) care should be taken to not interact directly with NaOH too much as it is corrosive (as strong base)

Conclusion: This experiment demonstrates the process of physical dissolution & ways to infer properties of this process through indirect methods such as acid-base titration. It also demonstrates how these characteristics change depending on physical properties such as temp. Lastly, we have discussed a few precautions & ways to improve the experiment ✓

$$\ln\left(\frac{1 - \bar{C}}{1 - C^*}\right) \propto t \text{ at Temp} = 25^\circ\text{C}$$



$\ln(1-C/C^*) \sqrt{st}$  at  $T_{el}$   $ip=35C$



Where is the  $en^{N_i}$   
in  
8.5/10

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EXPT: 9 PFR & CSTR in series

Aim: kinetic study in a PFR & calculation at CSTR  
volume connected in series

Objective: ① To find the rate constant of reaction b/w NaOH and ethyl acetate in study in a PFR & CSTR connected in series.

- ② Predict the final conversion from CSTR
- ③ Plot  $-Y_{A1}$  versus conversion ( $x_A$ ) and design the series Reactor.

Theory: Rate eq<sup>n</sup> is given by

$$-Y_A = k_1 C_{A0}^2 (1-x_A) (M-x_A)$$

Mole balance:

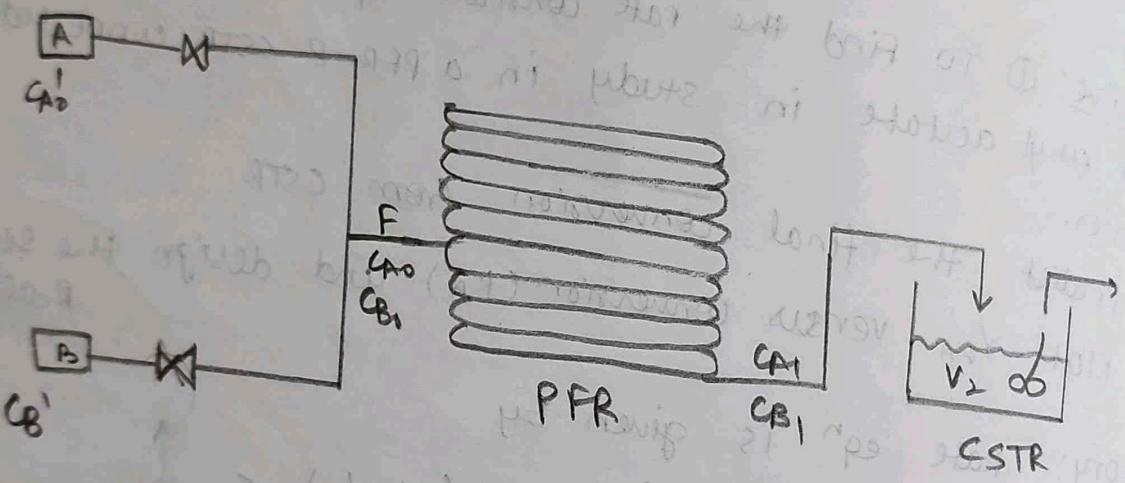
$$\text{In PFR: } \frac{V_1}{F_{A0} \cdot \text{PFR}} = \int_0^{x_{A1}} \frac{dx_{A1}}{-Y_{A1}} = \frac{1}{k_1 (M+1) C_{A0}^2} \times \ln \frac{(M-x_{A1})}{(1-x_{A1})}$$

$$\text{In CSTR: } \frac{V_2}{F_{A0} \cdot \text{CSTR}} = \frac{x_{A2}-x_{A1}}{-Y_{A2}}$$

Chemicals: ① succinic Acid ⑪ NaOH ⑫  $\text{CH}_3\text{COOC}_2\text{H}_5$   
④ phenolphthalein indicator

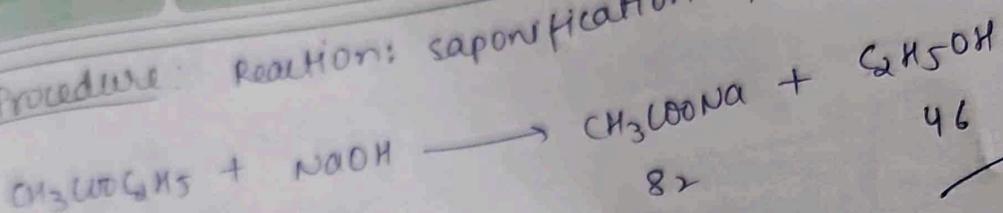
Apparatus:

- ① SS Reactor (PFR & CSTR)
- ② constant temp. water bath
- ③ stop watch
- ④ conical flask.



schematic diagram

Procedure: Reaction: saponification of ethyl acetate with NaOH



- ① each rotameter is calibrated with respective fluid.
- ② A feed rate is fixed for both streams ( $F_A = F_B$ ), such as 1-2 l PM.
- ③ NaOH (A) & Ethyl Acetate (B) streams (Reactants) are allowed to enter PFR First CSTR at equal feed.  $C_{A0}$  &  $C_{B0}$  are calculated.
- ④ Samples of the product are collected from PFR & CSTR respectively.
- ⑤ The samples are analyzed by titration with N/50 succinic acid.
- ⑥ The flow rate is changed two more times, still maintaining  $F_A = F_B$ .
- ⑦ unreacted NaOH at the outlet of both PFR & CSTR is measured using filtration (or conductance measurement of  $\text{SO}_4^{2-}$ ).
- ⑧ For reactor 1 (PFR) rate constant  $k_1$  is estimated using mole balance.
- ⑨  $-\frac{1}{r_A}$  vs  $x_A$  (conversion) is plotted & the CSTR volume is obtained graphically via plotting.

Observation table:

Flow rate	PFR			CSTR			$\Delta V(\text{mL})$
	$v_{\text{initial}}(\text{mL})$	$v_{\text{final}}(\text{mL})$	$\Delta v(\text{mL})$	$v_{\text{initial}}(\text{mL})$	$v_{\text{final}}(\text{mL})$		
$F_A = 5 \text{ LPH}$	0	1.9	1.9	0	1.2		1.2
	1.9	3.7	1.8	1.2	2.3		1.1
	3.7	5.7	2	2.3	3.5		1.2
$F_B = 5 \text{ LPH}$	5.7	8.1	2.4	3.5	5		1.5
	8.1	10.2	2.1	5	6.3		1.3
	10.2	12.3	2.1	6.3	7.7		1.4
$F_A = 10 \text{ LPH}$	10.3	14.3	2	7.7	9.2		1.5
	14.3	16.4	2.1	9.2	10.8		1.6
	16.4	18.6	2.2	10.8	12.2		1.4
$F_B = 10 \text{ LPH}$							

calculation :

$$C_{A0} = \frac{V_A}{V_A + V_B} \cdot C_A' = C_{A/2} = \frac{1}{40} = 0.025 \text{ M}$$

$$C_{B0} = \frac{V_B}{V_A + V_B} C_B' = C_{B/2} = \frac{1}{20} = 0.05 \text{ M}$$

$$M = \frac{C_{B0}}{C_{A0}} = \frac{0.05}{0.025} = 2$$

For  $F_A = F_B = 5 \text{ LPH}$

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Avg volume of succinic acid required (PFR) = 1.9 mL

$$N_1 V_1 = N_2 V_2$$

$$\Rightarrow N_1 = \frac{N_2 V_2}{V_1} \Rightarrow 0.0076 M$$

$$X_{A1} = \frac{C_{A0} - C_{A1}}{C_{A0}} = \frac{0.025 - 0.0076}{0.025} = 0.696$$

Avg vol of succinic acid required (CSTR) = 1.166 mL

$$N_1 V_1 = N_2 V_2$$

$$\Rightarrow N_1 = N_2 V_2 / V_1 \Rightarrow 0.00467$$

$$X_{A2} = \frac{C_{A0} - C_{A2}}{C_{A0}} = \frac{0.025 - 0.00467}{0.025} = 0.81344$$

$$\text{For } K_1 = \frac{V_1}{F_{AO} \cdot PFR} = \frac{1}{K_1 (M-1) C_{A0}^2} \cdot \ln\left(\frac{M-X_{A2}}{1-X_{A1}}\right)$$

$$K_1 = \frac{F_{AO}}{V_1} \cdot \frac{1}{(M-1) C_{A0}^2} \ln\left(\frac{M-X_{A1}}{1-X_{A1}}\right)$$

$$K_1 = \frac{10}{0.724} \cdot \frac{1}{(2-1) 0.025^2} \ln\left(\frac{2 - 0.696}{1 - 0.696}\right)$$

$$K_1 = 804.5105 \text{ mol}^{-1} \text{ L K}^{-1}$$

for  $V_2$  (vol of CSTR)

$$V_2 = F_{AO}, \text{CSTR.} \cdot \frac{X_{A2} - X_{A1}}{-22} = \frac{F_{AO}, \text{CSTR.} (X_{A2} - X_{A1})}{K_1 C_{A0}^2 (1-X_{A2})(M-X_{A2})}$$

$$= \frac{10 \times 0.025 (0.81344 - 0.696)}{804.5105 \times 0.025^2 (1-0.81344) (2-0.81344)} \Rightarrow 0.263 \text{ dm}^3$$

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Results:

Flow rate (LPH)	$C_{A_1}$ (mol/L)	$C_{A_2}$ (mol/L)	$x_{A_1}$	$x_{A_2}$	$k_1$ (mol <sup>-1</sup> Lhr <sup>-1</sup> )	$V_{CSTR}$ (L)
10	0.0076	0.00467	0.696	0.81344	804.5105	0.263776
15	0.0088	0.0056	0.648	0.776	1115.229	0.25117
20	0.0084	0.006	0.664	0.76	1525.22	0.1691

Discussion:

- ① As the Rx<sup>n</sup>, has second order kinetics, the rate constants rise as the flow rates do.
- ② It is the well-mixed nature of CSTR, which simplifies the kinetic eqn for the 2nd order of Rx<sup>n</sup>.
- ③ Because of the varying residence time in the PFR, the residence time distribution in the CSTR also becomes uniform.
- ④ The reactor's non-ideal behaviour (due to manual & equipment errors) also a discrepancy b/w the CSTR's real volume & its estimated value.
- ⑤ In a 2nd order Rx<sup>n</sup>, the quadratic dependence on conc<sup>n</sup> implies that higher initial conc<sup>n</sup> contributes more significantly to conversion.

Precautions: ① wash & properly clean all equipment before  
& after titration.

- ② The indicator i.e phenolphthalein should be carefully dropped.
- ③ It must be ensured to collect sample only when steady state is achieved.

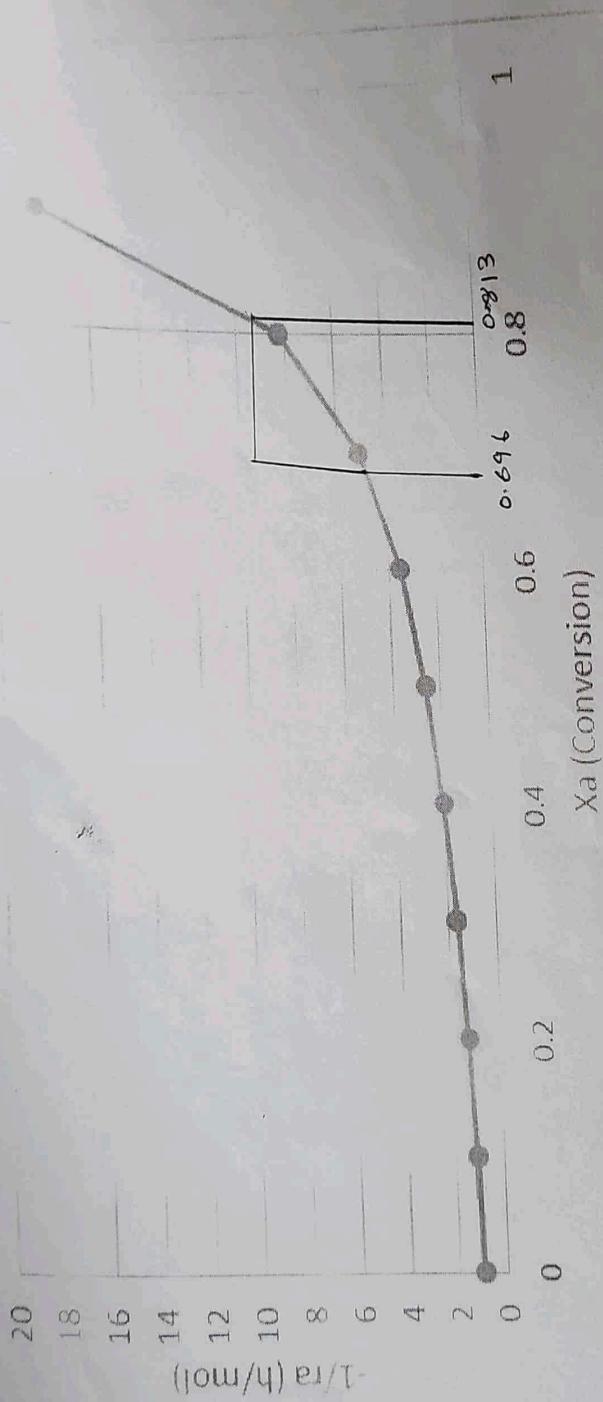
### Source of error:

Human error: Error may seep in owing to various errors such as allowing the system to achieve steady state on flow rate change, imprecise titration, observe effect on distributing the system while taking observation such as distributing the flow dynamics while taking sample etc.

### Conclusion:

This expt. demonstrates Rxn of 2<sup>nd</sup> order carried out on PFR & CSTR connected in series. We perform titration to find out the conversion after each step. We have also discussed how the different manipulated characteristics (such as flow rate) give rise to effects in reaction rate. It was assumed to be 2<sup>nd</sup> order & it was verified as upon increasing value of flow rate, the rate constant also increased.

$-1/r_a$  v/s  $X_a$  for  $F_a = F_b = 5 \text{ LPH}$

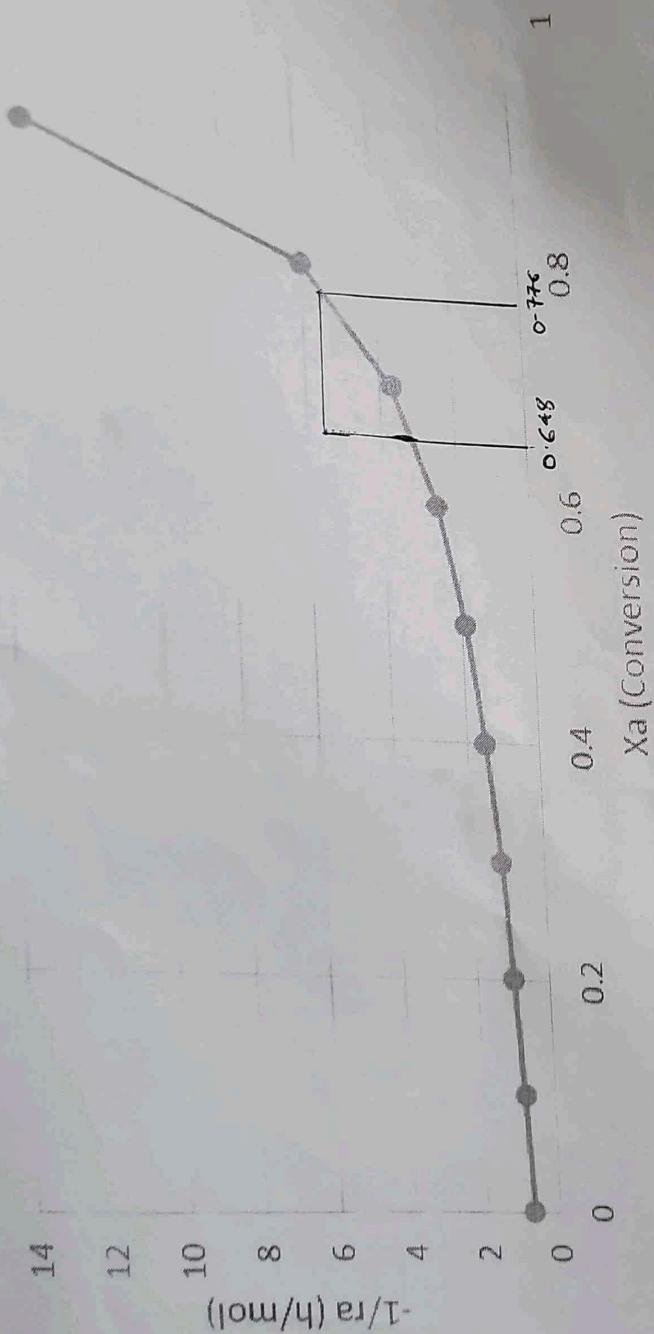


$$K_1 = 804.51 \text{ mol}$$

$$Y_2 = F_{A_0}(\text{CSTR}) \times \text{Area Under the Curve}$$
$$\approx 0.23 \text{ L}$$

- $\frac{1}{ra}$  v/s  $X_a$  for  $F_a = F_b = 7.5$  LPH

Scale: x-axis :  $|_{\text{unit}} = 0.2$   
 y-axis :  $|_{\text{unit}} = 2 \text{ mol}^{-1}$

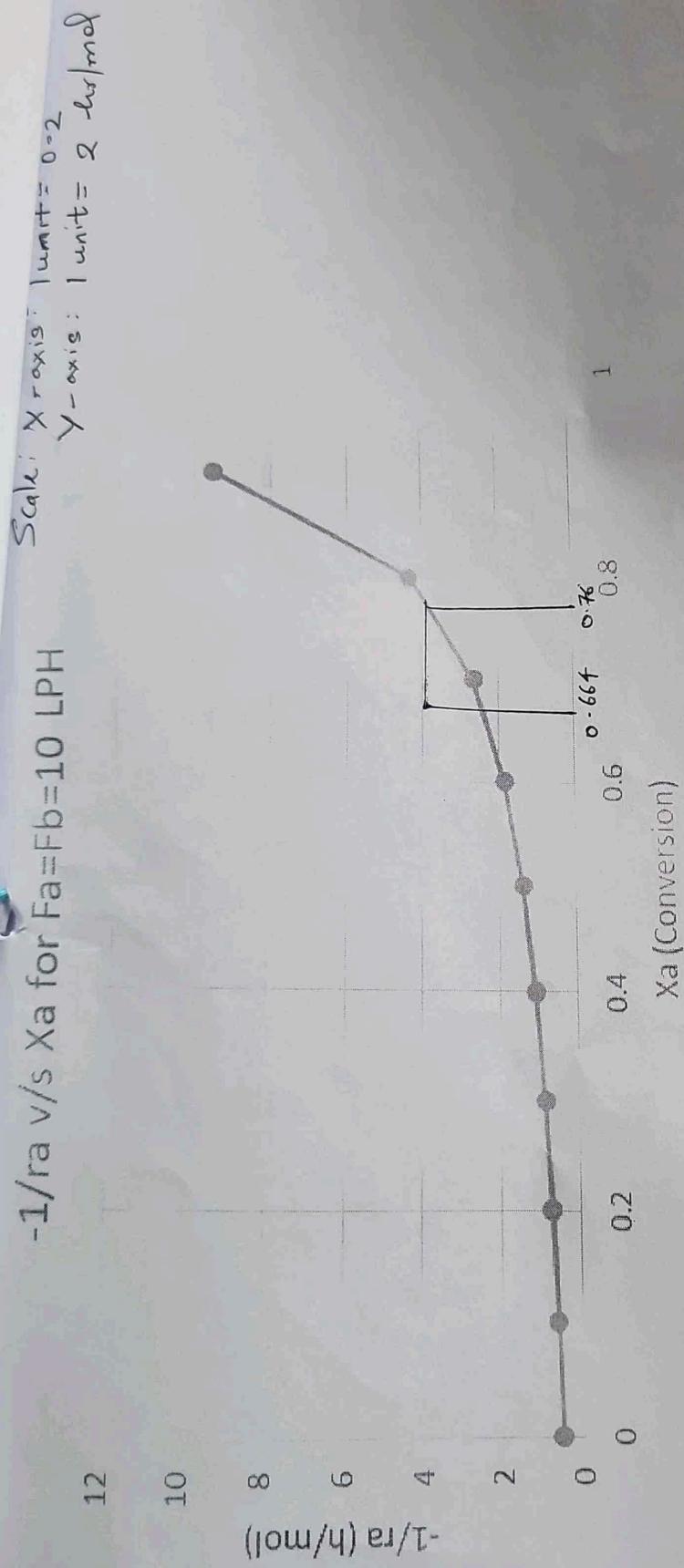


$$K_1 = 1115.229 \text{ mol}^{-1} L h^{-1}$$

$$V_2 = f_{A_0}(CSTR) \times \text{Area under Curve}$$

$$\approx 0.28 L$$

$-1/r_a$  v/s  $X_a$  for  $F_a = F_b = 10 \text{ LPH}$



$$K_1 = 1525.22 \text{ mol}^{-1} \text{L} \text{h}^{-1}$$

$$V_2 = f_{A_0}(\text{CSTR}) \times \text{Area Under Curve}$$

$$\approx 0.21 \text{ L}$$

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Exp: Rate study in a cascade CSTR

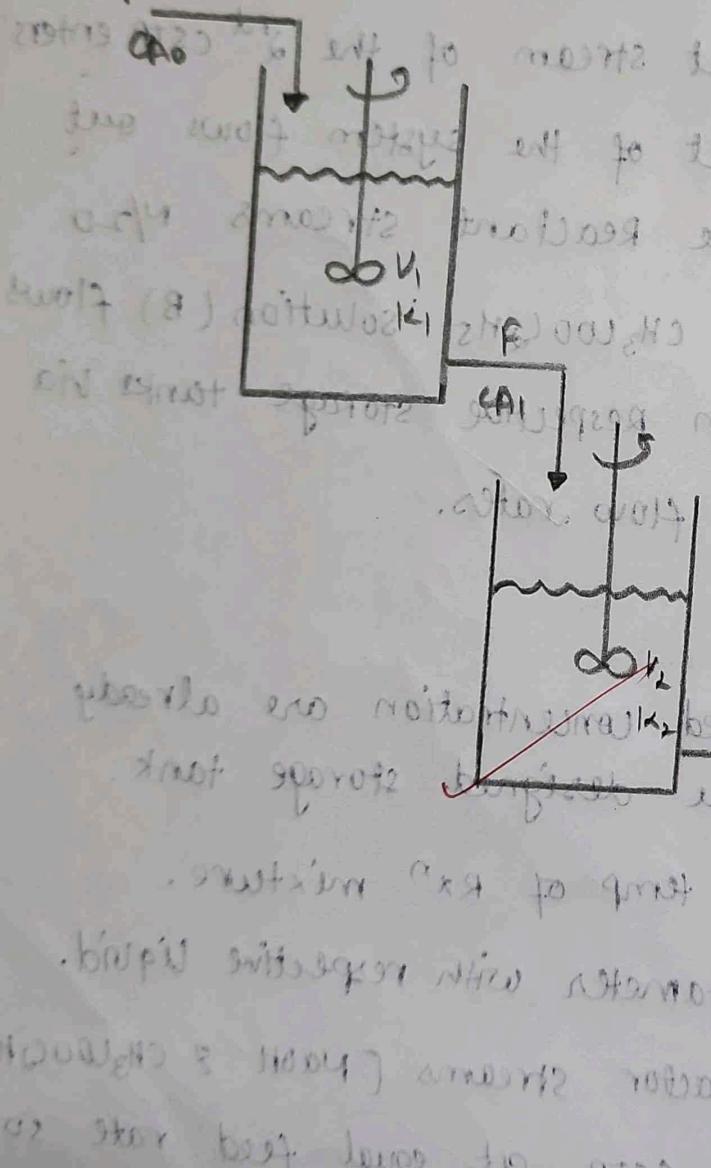
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Aim: Rate study in a cascade CSTR (CSTR in series)

Theory: The cascade consist of 3 identical CSTR's in series where product stream of the 2nd CSTR enters 3rd as a feed & product of the system flows out from the 3rd CSTR. The Reactant streams N/20 NaOH soln. (A) & N/10  $\text{CH}_3\text{COOCH}_3$  solution (B) flows to the 1st CSTR from respective storage tanks via rotameters to control flow rates.

## Procedure:

- ① Reactants of prescribed concentration are already prepared & fed in the designed storage tank.
- ② Record the ambient temp of Rxn mixture.
- ③ calibrate each rotameter with respective liquid.
- ④ Allow the two reactor streams [NaOH &  $\text{CH}_3\text{COOCH}_3$ ] to enter the first CSTR at equal feed rate so that in the reactor  $C_{A0} = C_{B0}$ . start the mixer & wait till the product flows out from the 3rd CSTR



Schematic of cascade CSTR

Give 5-10 minutes to attain steady state.

- ⑤ Fix & record the flow rate  $F_A = F_B$
- ⑥ Collect 5ml sample from each 3 reactor outlets & titrate with N/50 succinic acid soln. to unreacted NaOH.
- ⑦ Repeat step 4-6 for different flow rates.
- ⑧ Record volume of each reactor ( $V_1 = V_2 = V_3$ )

Observation table:

S.No	Flow rate (LPH)	CSTR 1 titration vol (mL)	CSTR 2 titration vol (mL)	CSTR 3 titration vol (mL)	Average CSTR 1	Avg. CSTR 2	Avg. CSTR 3
1	2.5	5.3 5	3.9 3.5	3.1 3.3	5.15	3.7	3.2
2	5	3.8 4.1	2.7 2.7	2.5 2.4	3.95	2.7	2.45
3	7.5	2.9 2.6	2.2 2.3	1.8 1.6	2.75	2.25	1.7

calculations:

$$\text{calculation of } \theta_n \Rightarrow \theta_n = V_n/F$$

for  $f = 5 \text{ LPH}$

$$\theta_n = \frac{1.135}{5+5} = 0.1135 = 6.81 \text{ min}$$

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$$\therefore \theta_1 = \theta_2 = \theta_3 = 6.81 \text{ min}$$

• calculation of unreacted NaOH

$$N_1 V_1 = N_2 V_2$$

$$C_{A_1} V_1 = C_{A_2} V_2$$

Here,

$$C_{A_2} = \frac{1}{50}$$

$V_2$  = volume of acid

$V_1$  = volume of sample (5mL)

$$\text{For CSTR 1 : } \frac{1}{50} \times 3.95 = 5 \times C_{A_1} \Rightarrow C_{A_1} = 0.0158$$

$$\text{For CSTR 2 : } \frac{1}{50} \times 2.7 = 5 \times C_{A_2} \Rightarrow C_{A_2} = 0.0108$$

$$\text{For CSTR 3 : } \frac{1}{50} \times 2.45 = 5 \times C_{A_3} \Rightarrow C_{A_3} = 0.0098$$

for calculation of  $k$ :

$$k = \frac{\left( \frac{C_{A_{n-1}}}{C_{A_n}} \right) - 1}{\theta \times C_{A_n}}, \text{ for } n=1, \theta_n = 6.81 \text{ min}$$

$$k = \left( \frac{\frac{0.025}{0.0158} - 1}{6.81 \times 0.0158} \right) = 5.41 \text{ L/mol min}$$

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Flow rate (LPH)	$t$ (min)	$CA_1$ (mol/L)	$CA_2$ mol/L	$CA_3$ (mol/L)
2.5	13.62	0.0206	0.0148	0.0128
5	6.81	0.0158	0.0108	0.0098
7.5	4.54	0.011	0.009	0.0068

## Results:

Flow rate (LPH)	$k_1$ (L/mol min)	$k_2$ (L/mol min)	$k_3$ (L/mol min)
2.5	0.76	1.94	0.89
5	5.41	6.28	1.53
7.5	25.48	5.44	10.48

## Discussion:

- The series setup of CSTR provide higher conversion than other setup like CSTR parallel. from the results of the experiment, we infer that the concn decreases for subsequent CSTR in series.
- Rate constant is lowest for the third CSTR & highest for the first CSTR, hence the conversion is greater for the 1st CSTR.

- Also we infer that we increases the flow rate of the feed, rate constant increases. This proves the ability of CSTR in series to handle energetic reaction with high flow rates due to superior heat transfer.
- CSTR in series are able to handle higher reactant conc' due to superior properties & provide great advantages.
- Reactor volume is an important factor determining the final conversion from the setup. If we increase the reactor volume of each CSTR conversion increases with time, so it is important to determine an optimal & efficient volume of reactor.

### Conclusion:

A continuous stirred tank reactor (CSTR) is a reactor used in which reagents, reactants & solvent flow into the reactor while product concurrently exist. The CSTR is considered to be a valuable test for continuous crystallisation in continuous mixed suspension, mixed product removal crystallizer but it is not suitable for small scale organic synthesis. The speed of the stirrer is adjusted using regulators in

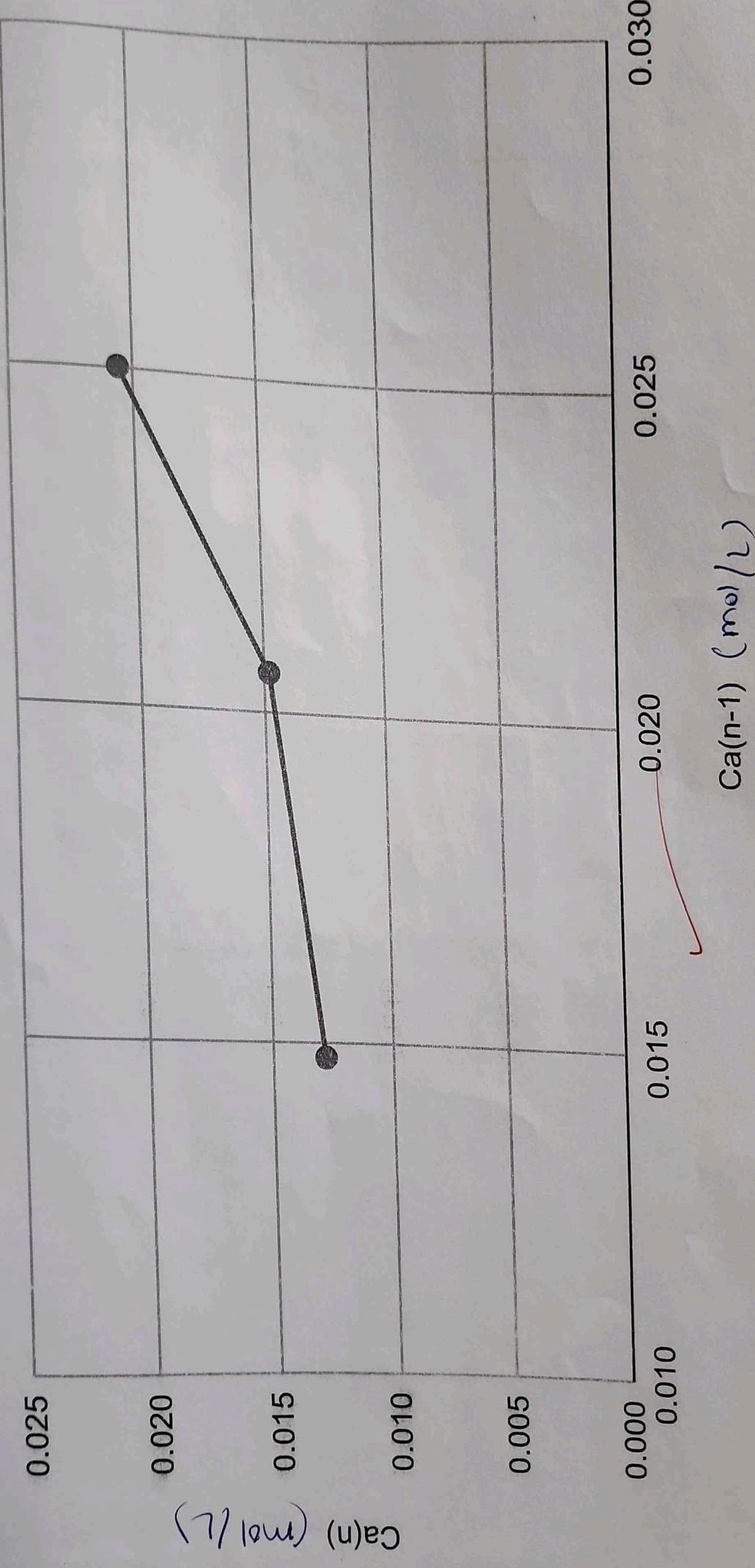
each CSTR connected in series, allowing for control over flow of fluid between reactors in the setup.

Titration is carried out using NaOH sol<sup>n</sup> & succinic acid using phenolphthalein indicator in order to determine the unreacted NaOH which is left

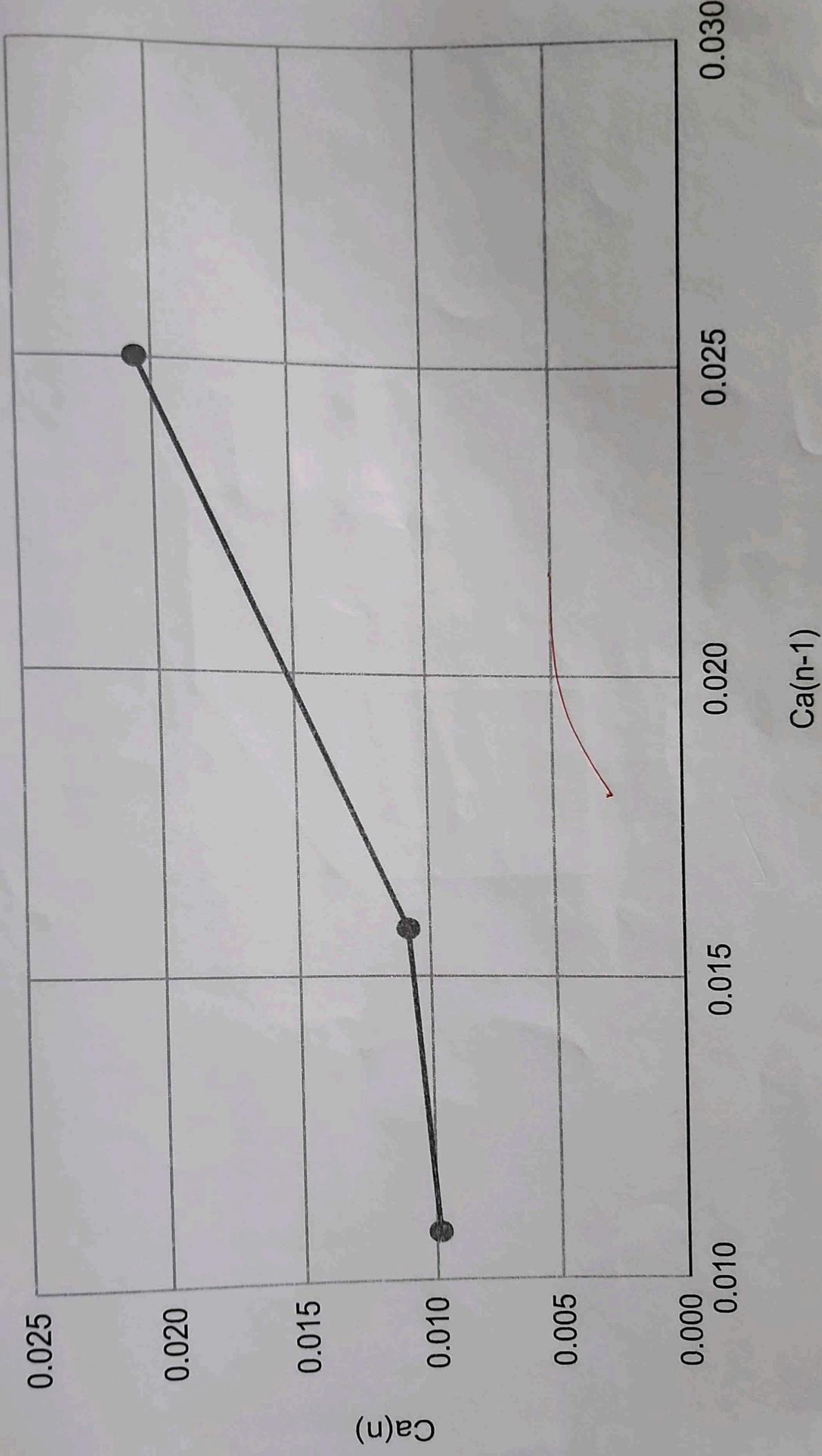
#### Precautions:

- regular monitoring & accurate measurement of flow rate in CSTR from the rotameter along with the measurement is crucial.
- while doing titration, it is necessary to take precise measurement from the pipette, avoiding parallax error.
- solution while titration must be shaken continuously in a beaker to avoid errors.

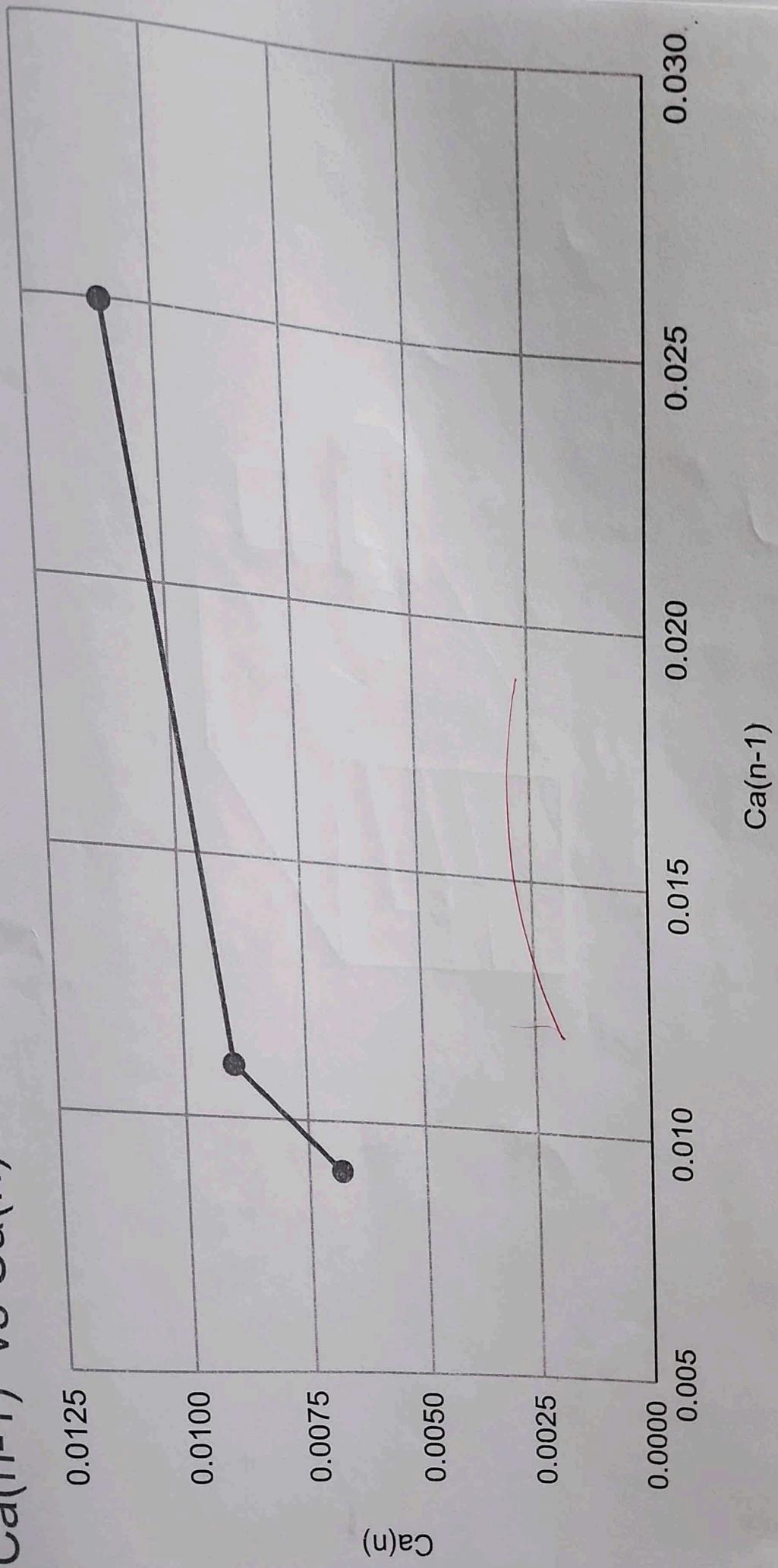
# $\text{Ca}(n-1)$ vs $\text{Ca}(n)$ For 2.5 LPH



# $\text{Ca}(n-1)$ vs $\text{Ca}(n)$ For 5 LPH



# $\text{Ca}(n-1)$ vs $\text{Ca}(n)$ For 7.5 LPH



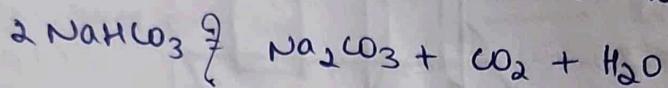
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Study of non-catalytic gas-solid Rx<sup>n</sup>  
by thermogravimetric Technique.

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Objective: To determine the reaction rate constant for the decomposition of cylindrical sodium bicarbonate pellet & find out the activation energy in a temperature range of 100 to 250°C. The stoichiometric eqn,



Theory: For the above reaction the un-reacted core model which has found to satisfy the most of the non-catalytic gas-solid Rx<sup>n</sup> is applied. In case of bicarbonate decomposition chemical resistance at the interface, diffusional resistance of the boundary layer & product layer may individually or simultaneously control the Rx<sup>n</sup> rate.

The rate eqn may be written as:

$$R_{00} f = kt$$

where,

$R_0$  = initial radius of pellet, cm

$\rho_0$  = Density of the reactant pellet, gm/cc

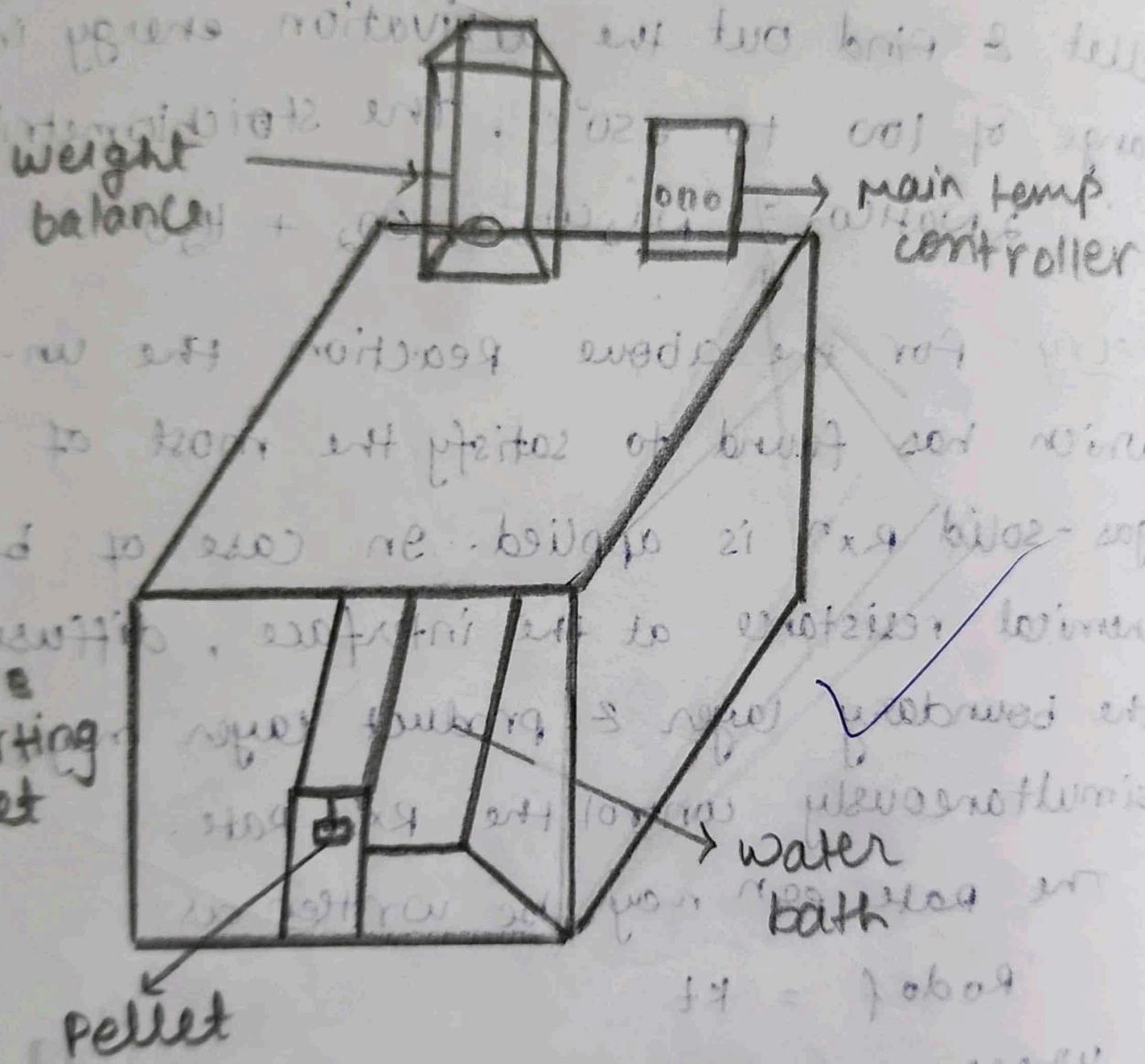
$t$  = Reaction time

$k$  = Modified rate constant

The spherical pellet & the cylindrical pellet with the same length & diameter, the relation b/w conversion  $x$  &  $f$  is:

$$f = 1 - (1-x)^{1/3}$$

for cylindrical pellet with any ratio of length/dia, read



Schematic of the  
 experimental apparatus

Last time had problem with

new technique with a little less drag at

$\text{blw conversion } x \text{ & f is:}$

$$x = 1 - \frac{(a-f)(1-f)^2}{a}$$

so for any value of  $x$  we can get  $f$  from the above reln & calculate  $R_{\text{of}}$  for each time  $t$ . under the isothermal condition  $R_{\text{of}} \text{ vs } t$  plot will give straight line for chemical resistance control. At different temp. we are expected to get different  $R_{\text{of}}$  vs  $t$  straight lines from the slope  $w$  of which we can find out the activation energy by plotting  $g_{\text{nk}}$  vs  $\frac{1}{T}$ .

Apparatus: A thermogravimetric setup used in the present study consist of the following:

- An analytical chainometric (keroy) balance from the left pan of which is suspended a platinum wire carrying sample, made of 200 mesh ss screen.
- A tube furnace consisting of multiple tubes of length 45.72cm & dia 5.08 cm. The furnace bottom is packed with quartz particle made up to some height for preheating the sweeping gas.
- A voltage stabilizer & control device are introduced to avoid the fluctuations in voltage & temp.

Experimental procedure:

- ① The calibration of the thermocouple was done with pure solid substances having definite melting point.
- ② The heating furnace was started two hours before our experiment.

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- ③ The cylindrical pellet was measured using the vernier calliper
- ④ The pellet was then placed into basket & lowered into reactor
- ⑤ We calculated the conversion of 'r' from the loss of pellet.
- ⑥ The temperature recorded by the thermocouple near the basket is assumed to be the pellet temperature. Alternatively the Rxn can be carried out in non-isothermal cond within the range when the temperature is linear with time.
- ⑦ We have to perform these for three different temperatures.

### Observation

Attached

### Sample calculation:

For pellet -1 , t=1min , T=120°C

$$\alpha = \frac{l}{dia} = 0.55673$$

$$\chi = \frac{\Delta W}{\sum \Delta W} = 0.006$$

$$\text{for cylindrical pellet: } \chi = 1 - \left[ \frac{(a-f)(1-f)^2}{(a)} \right]$$

$$f^3 + (a+\chi)f^2 - (2a+1)f + a\chi = 0$$

$$f = 0.00158 \quad \text{and the other time roots are sample conjugates}$$

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$$\text{Density} = d_0 = \frac{3.619}{\frac{\pi d^2}{4}} \text{ g/cc}$$

$$= \frac{3.619}{2.18175} = 1.659 \text{ g/cc}$$

$$Rodof = 0.00218 \text{ at } t = 1 \text{ min}$$

After plotting Rodof vs t we get the k as the slope which is  $0.0008 \text{ g cm}^2 \text{ unit}$ .

Results:

Temperature ( $^{\circ}\text{C}$ )

120  
140  
160

$k (g \text{ cm}^2 \text{ min}^{-1})$

$8.81 \times 10^{-4}$

$10.1 \times 10^{-4}$

$18 \times 10^{-4}$

from the  $\ln(k)$  vs  $\frac{1}{T}$  plot

Slope = -331

$$-\frac{E_a}{R} = -331 \Rightarrow$$

$$E_a = 2.752 \text{ kJ/mol}$$

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## Discussion:

- Thermogravimetric analysis is a method of thermal analysis in which the mass of the sample is measured over time as the temperature changes. These measurements provide information about physical phenomena such as phase transition etc.
- The rate constant was increasing with the increase in temperature. This proves that the  $Rx^n$  is endothermic. Positive value of the activation energy reinforces this.
- since the pellet is composed of  $\text{NaHCO}_3$ , a powdered material therefore it is to be handled with care such that minimum particles are lost. During the experiment, slight breaking of the pellet may occur directly affecting the readings.

## Precautions:

- The sample holder should attain the ambient temperature quickly, which might affect the sample temperature later with increasing time.
- zero error should be taken into consideration after the measurement through vernier calliper.
- The readings should be noted after every 1 minute till the steady state is reached.

Conclusion:

Thermogravimetric analysis of the decomposition of the sodium bicarbonate pellets was carried out. The weight of the sample was noted, after every minute & correspondingly a  $\log \frac{w}{w_0}$  vs time plot was obtained, where slope gave the value of the rate constant of the reaction.

The Reactor's rate constant increased with increase in temperature, indicating an endothermic Rxn. A plot of  $\ln(k)$  vs  $\frac{1}{T}$  was obtained that gave the activation energy.

Sample 1

T 120 C  
D 1.71 cm  
H 0.95 cm  
a 0.55673  
Volume 2.18175 cm<sup>3</sup>

Time (min)	Weight (gm)	x	f	RoDOF	Time (min)	RoDOF
0	3.619	0	0	0	0	0
1	3.529	0.006	0.00158	0.00218	1	0.00218
2	3.409	0.014	0.0037	0.00493	2	0.00493
3	3.27	0.02326	0.00617	0.00789	3	0.00789
4	3.141	0.03186	0.00848	0.01042	4	0.01042
5	3.019	0.03999	0.01067	0.0126	5	0.0126
6	2.911	0.04719	0.01262	0.01437	6	0.01437
7	2.808	0.05405	0.01449	0.01591	7	0.01591
8	2.702	0.06112	0.01642	0.01735	8	0.01735
9	2.61	0.06725	0.01811	0.01848	9	0.01848
10	2.539	0.07198	0.01941	0.01927	10	0.01927
11	2.478	0.07605	0.02054	0.0199	11	0.0199
12	2.424	0.07965	0.02154	0.02042	12	0.02042
13	2.377	0.08278	0.02241	0.02083	13	0.02083
14	2.346	0.08484	0.02298	0.02108	14	0.02108
15	2.325	0.08624	0.02337	0.02125	15	0.02125
16	2.315	0.08691	0.02356	0.02133	16	0.02133
17	2.309	0.08684	0.02354	0.02139	17	0.02139
18	2.304	0.08684	0.02354	0.02144	18	0.02144
19	2.299	0.08684	0.02354	0.02148	19	0.02148
20	2.296	0.08684	0.02354	0.02151	20	0.02151
21	2.295	0.08684	0.02354	0.02152	21	0.02152

## Sample 2

T 140 C  
 D 1.724 cm  
 H 0.83 cm  
 a 0.41844  
 Volume 1.9375 cm<sup>3</sup>

Time (min)	Weight (gm)	x	f	f	RoDOF
0	3.034	0	0	0	0
1	3.011	0.00175	0.00043	0.00058	0.00223
2	2.943	0.00693	0.0017	0.00432	
3	2.853	0.01379	0.0034	0.00635	
4	2.759	0.02095	0.00517	0.00811	
5	2.672	0.02758	0.00682	0.00979	
6	2.583	0.03436	0.00852	0.01143	
7	2.489	0.04152	0.01032	0.01443	
8	2.406	0.04784	0.01191	0.01275	
9	2.323	0.05417	0.01352	0.01397	
10	2.252	0.05958	0.01489	0.01492	
11	2.191	0.06422	0.01608	0.01567	
12	2.132	0.06872	0.01723	0.01634	
13	2.081	0.0726	0.01822	0.01687	
14	2.031	0.07641	0.0192	0.01735	
15	1.995	0.07916	0.01991	0.01767	
16	1.971	0.08098	0.02038	0.01787	
17	1.946	0.08289	0.02088	0.01808	
18	1.94	0.08335	0.02099	0.01812	
19	1.942	0.08319	0.02096	0.01811	
19	1.942	0.08319	0.02096	0.01811	

## Sample 3

160 C

1.734 cm

0.85 cm

0.490196

Time (min)	Volume	Weight (gm)	x	f	RoDof
0	2.00728 cm <sup>3</sup>	3.163	0	0	0.00173
1		3.108	0.00521	0.00129	0.00529
2		2.989	0.01647	0.0041	0.00919
3		2.847	0.02991	0.00747	
4		2.71	0.04288	0.01076	0.01259
5		2.576	0.05556	0.014	0.01558
6		2.446	0.06787	0.01717	0.01814
7		2.351	0.07686	0.0195	0.0198
8		2.268	0.08471	0.02155	0.02111
9		2.174	0.09361	0.02388	0.02242
10		2.111	0.09957	0.02545	0.02321
11		2.061	0.10431	0.02671	0.02378
12		2.033	0.10696	0.02741	0.02407
13		2.021	0.10809	0.02771	0.02419
14		2.022	0.108	0.02769	0.02418

DATE

Study of Adsorption of  $\text{CO}_2$  in agitated contactor.

SHEET NO.

Objective: To find out the mass transfer coefficient  $k_{L,a}$  for absorption of  $\text{CO}_2$  in water in agitated contactor.

2) To observe the effect of speed of agitator on  $k_{L,a}$

Theory: The absorption rate of  $\text{CO}_2$  in a agitated tank can be given as:

$$V \frac{dc_L}{dt} = k_{L,a} (c^* - c_L)$$

where

$V$  = vol of liquid,  $\text{cm}^3$

$c_L$  = concn of  $\text{CO}_2$  in liquid at  $t$ ,  $\text{gmol}/\text{cm}^3$

$c^*$  = solubility of  $\text{CO}_2$  in pure water

On integration of above eq<sup>n</sup> at  $t=0$ ,  $c_L=0$ . & at  $t=t$ ,  $c_L=c$   
& rearranging,  $k_{L,a} = \frac{V}{t} \ln \left[ \frac{c^*}{c^* - c_L} \right]$  in  $\text{cm}^3/\text{min}$  if  $t$  in min

$$c^* = 3.3639 \times 10^{-5} \text{ gmol}/\text{cm}^3 \text{ at } 25^\circ\text{C} \text{ at } 1 \text{ atm}$$

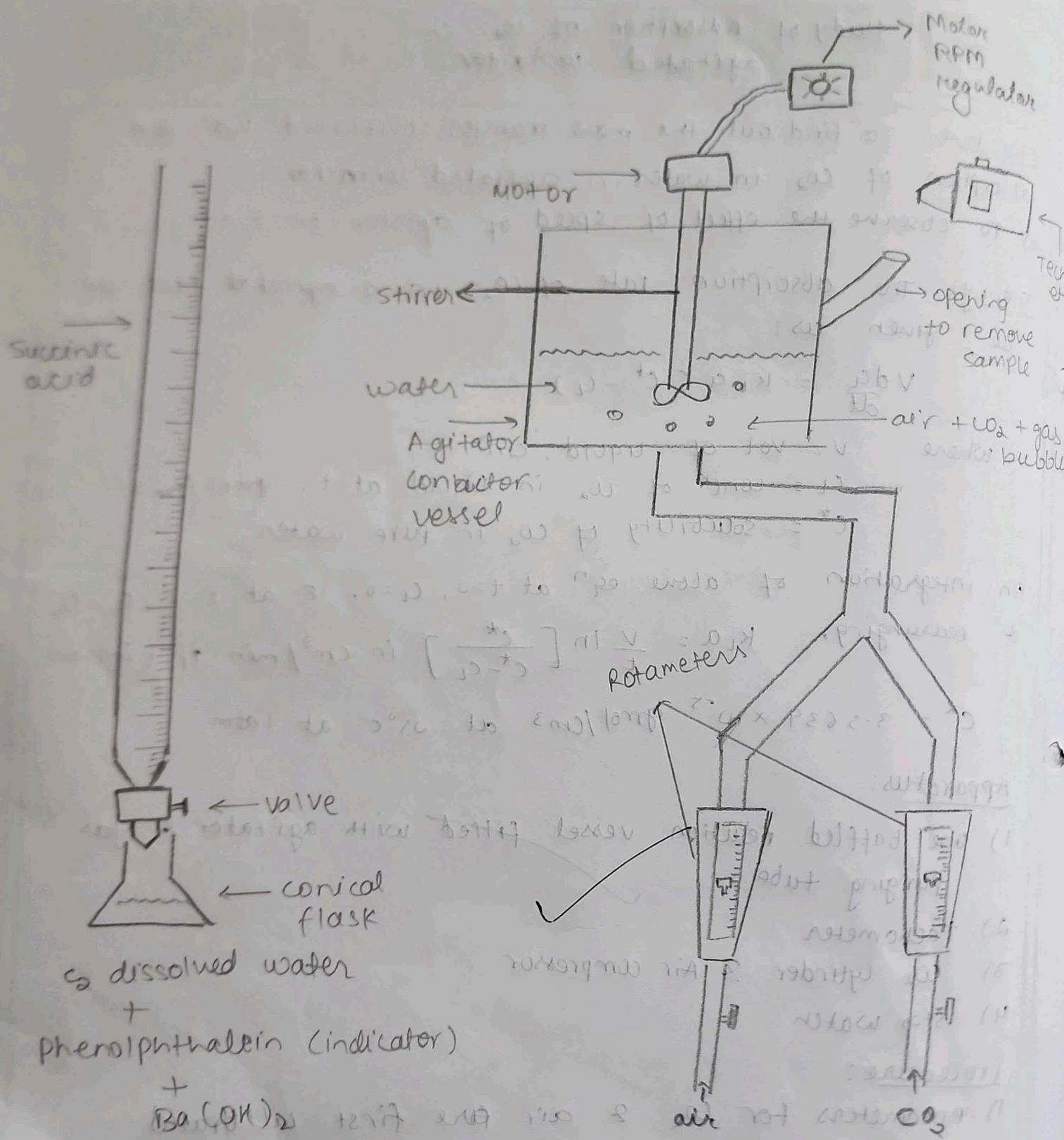
### Apparatus:

- 1) one baffled reaction vessel fitted with agitator & gas purging tube.
- 2) tachometer
- 3)  $\text{CO}_2$  cylinder & Air compressor
- 4) stop watch

### Procedure:

- 1) Rotameters for  $\text{CO}_2$  & air are first calibrated

Schematic diagram: Agitated contractor Apparatus



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- 2) At a constant stirrer speed and temperature,  $\text{CO}_2$  is absorbed in one liter of water in the agitated contractor for a definite period of time (less than 3 min). The conc' of  $\text{CO}_2$  should not be more than 15%. The soln is collected & analysed by titration.
- 3) Repeated the experiment for three RPM
- 4) calculate  $K_{La}$  for these different RPMs & show the plot

### Observation table

flow rate of  $\text{CO}_2 = 200 \text{ cc/min}$

flow rate of air = 1.5 L/min

vol. of  $\text{Ba}(\text{OH})_2$  in each titration sample = 10mL

vol. of water in agitated contractor = 1000 cc

RPM = 165

Time (min)	Vol. of acid (mL)	conc'n of $\text{CO}_2 (\text{mol/L})$	$\ln\left(\frac{C^*}{C^* - a}\right)$	$\Delta V$
0	20.6	$0.088 \times 10^{-5}$	0.039412	<del>0.0020</del>
2	18.5	$0.13 \times 10^{-5}$	0.05688	0.002
4	15.7	$0.186 \times 10^{-5}$	0.06573	0.004
6	14.3	$0.214 \times 10^{-5}$		0.006

RPM = 270

Time (min)	Vol. of acid (mL)	conc'n of $\text{CO}_2 (\text{mol/L})$	$\ln\left(\frac{C^*}{C^* - a}\right)$	$\Delta V$
0	20.5	$0.09 \times 10^{-5}$	0	0
2	18.1	$0.138 \times 10^{-5}$	0.01889	0.002
4	15.5	$0.19 \times 10^{-5}$	0.05814	0.004
6	14.4	$0.212 \times 10^{-5}$	0.065096	0.006

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$$RPM = 370$$

Time (min)	Vol of acid (ml)	Concn of $\text{CO}_2$ (gmol/litre)	$\ln \left( \frac{C^*}{C - u} \right)$	$\Delta V$
0	20.1	$0.098 \times 10^{-5}$	0	0
2	16.5	$0.17 \times 10^{-5}$	0.051858	0.002
4	14	$0.22 \times 10^{-5}$	0.67637	0.004
6	13.6	$0.228 \times 10^{-5}$	0.070185	0.006

calculations:

$$RPM = 165, t = 2 \text{ min}, N(\text{Ba(OH})_2) = \frac{1}{20}, N(\text{succinic acid}) = \frac{1}{50}$$

$$C^* = 3.3639 \times 10^{-5} \text{ gmol/cm}^3$$

$$\begin{aligned} \text{Vol of acid} &= V_1 = 18.5 \text{ ml} & V_2 &= \text{volume of sample} \\ N_1 V_1 &= N_2 V_2 & &= \text{vol. of water} + \text{vol. of Ba(OH)}_2 \\ N_2 &= \frac{18.5}{50 \times 15} & &= 5 + 10 = 15 \text{ ml} \end{aligned}$$

$$N_2 = \text{Normality of Ba(OH)}_2 \text{ in } 15 \text{ ml sample before titration} = \frac{18.5}{50 \times 15}$$

$$n_2 = \text{no. of moles of Ba(OH)}_2 \text{ remaining} = \frac{18.5}{50 \times 15 \times 2} = \frac{18.5}{100}$$

$$n_1 = \text{no. of moles of Ba(OH)}_2 \text{ initially added} = \frac{1}{20} \times \frac{10}{2} = 0.25$$

$$\therefore n_{\text{CO}_2} = \text{no. of moles of CO}_2 \text{ in } 5 \text{ ml sample} = 0.25 - 0.185 = 0.065$$

$$\text{Concn of CO}_2 = \frac{0.065}{5 \times 1000} = 1.3 \times 10^{-5} \text{ mol/litre}$$

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$$k_L a \cdot \frac{t}{V} = \ln \left( \frac{C^*}{C^* - C_L} \right)$$

$$k_L a \cdot \frac{2}{100} = \ln \left( \frac{3.3639 \times 10^{-5}}{3.3639 \times 10^{-5} - 1.3 \times 10^{-5}} \right)$$

$$\Rightarrow k_L a = 19.7061 \text{ cm}^3/\text{min} \quad [\text{for Run 1, } t=2\text{ min}]$$

Result:

For Run 1, RPM = 165,  $k_L a = 12.513 \text{ cm}^3/\text{min}$

For Run 2, RPM = 270,  $k_L a = \underline{12.623} \text{ cm}^3/\text{min}$

For Run 3, RPM = 370,  $k_L a = \underline{14.203} \text{ cm}^3/\text{min}$

Discussion:

- i) The overall mass transfer  $k_L a$  trend with the increase in the contactor. the possible reason showed a particular increasing rotation speed of the agitator for this are:
- i) with increase in the rotating speed of stirrer, the gas bubble breakdown into smaller bubble, which increase the exposed surface area & thus the rate of mass transfer increases.
  - ii) Besides this, the convection which occurs because of stirrer also significantly contributes towards the increase in mass transfer rate.
  - iii) when the rotating speed of the stirrer is increased, there is a shift towards the turbulent flow region, that increases

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$$k_L a \cdot \frac{2}{100} = \ln \left( \frac{3.3639 \times 10^{-5}}{3.3639 \times 10^{-5} - 1.3 \times 10^{-5}} \right)$$

$$\Rightarrow k_L a = 19.7061 \text{ cm}^3/\text{min} \quad [\text{For Run 1, } t=2\text{ min}]$$

Result:

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For Run 3, RPM = 370,  $k_L a = \underline{14.203} \text{ cm}^3/\text{min}$

Discussion:

- i) The overall mass transfer  $k_L a$  trend with the increase in the rotation speed of the agitator showed a particular increasing contactor. the possible reason for this are:
- i) with increase in the rotating speed of stirrer, the gas bubble breakdown into smaller bubble, which increase the exposed surface area & thus the rate of mass transfer increases.
  - ii) Besides this, the convection which occurs because of stirrer also significantly contributes towards the increase in mass transfer rate.
  - iii) when the rotating speed of the stirrer is increased, there is a shift towards the turbulent flow region, that increases

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the convective part of mass transfer.

iv) Higher turbulent promotes greater molecular movement within the liquid increasing the rate of diffusion. This means that more molecules or ions moves rapidly leading to faster mixing & homogenization of substance.

## Conclusion:

In the experiment, the adsorption of  $\text{CO}_2$  was studied in an agitated contractor, three different rotating rates of the stirrers were used and the mass transfer coefficient was found to be increasing with the increase in rotation speed of the stirrer. The amount of  $\text{CO}_2$  adsorbed was found to be titrating the soln with a soln of succinic acid.

Precautions: ① There should not be a gas leak into the agitated vessel.

Hand should be kept steady while using the tachometer to measure the RPM.

③ During titration, succinic acid should be added in a dropwise manner.

## Industrial application:

① Carbon capture and storage (CCS): Agitated contractor  $\text{CO}_2$  adsorption is utilized in industrial application for capturing  $\text{CO}_2$  emmission from various sources such as power plant, cement factories.

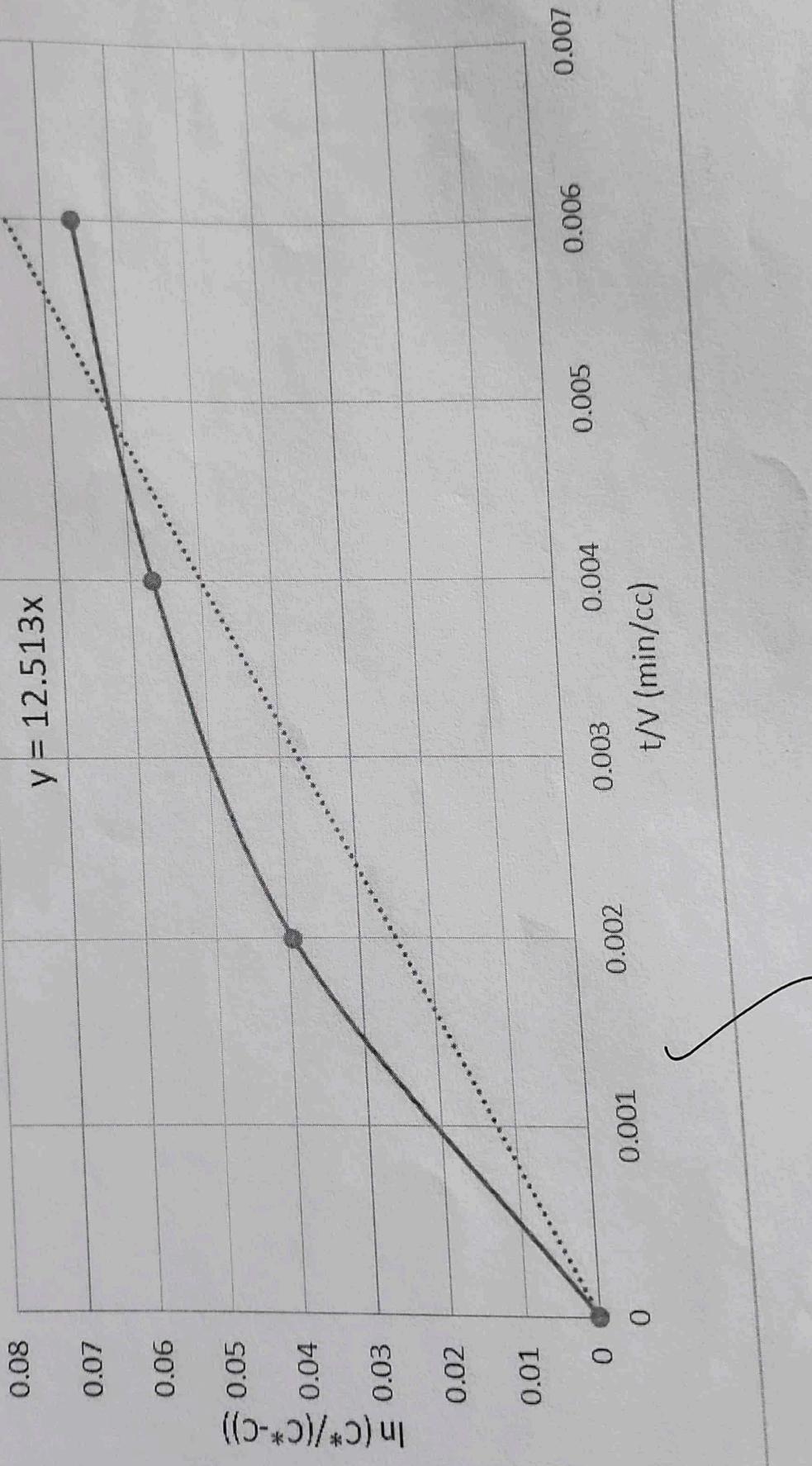
② Gas purification: Agitated contractors enable efficient adsorption of  $\text{CO}_2$  from gas streams, resulting in higher purity of the desired product gas & meeting strongest quality standards for various industrial applications.

A. H. S. K.

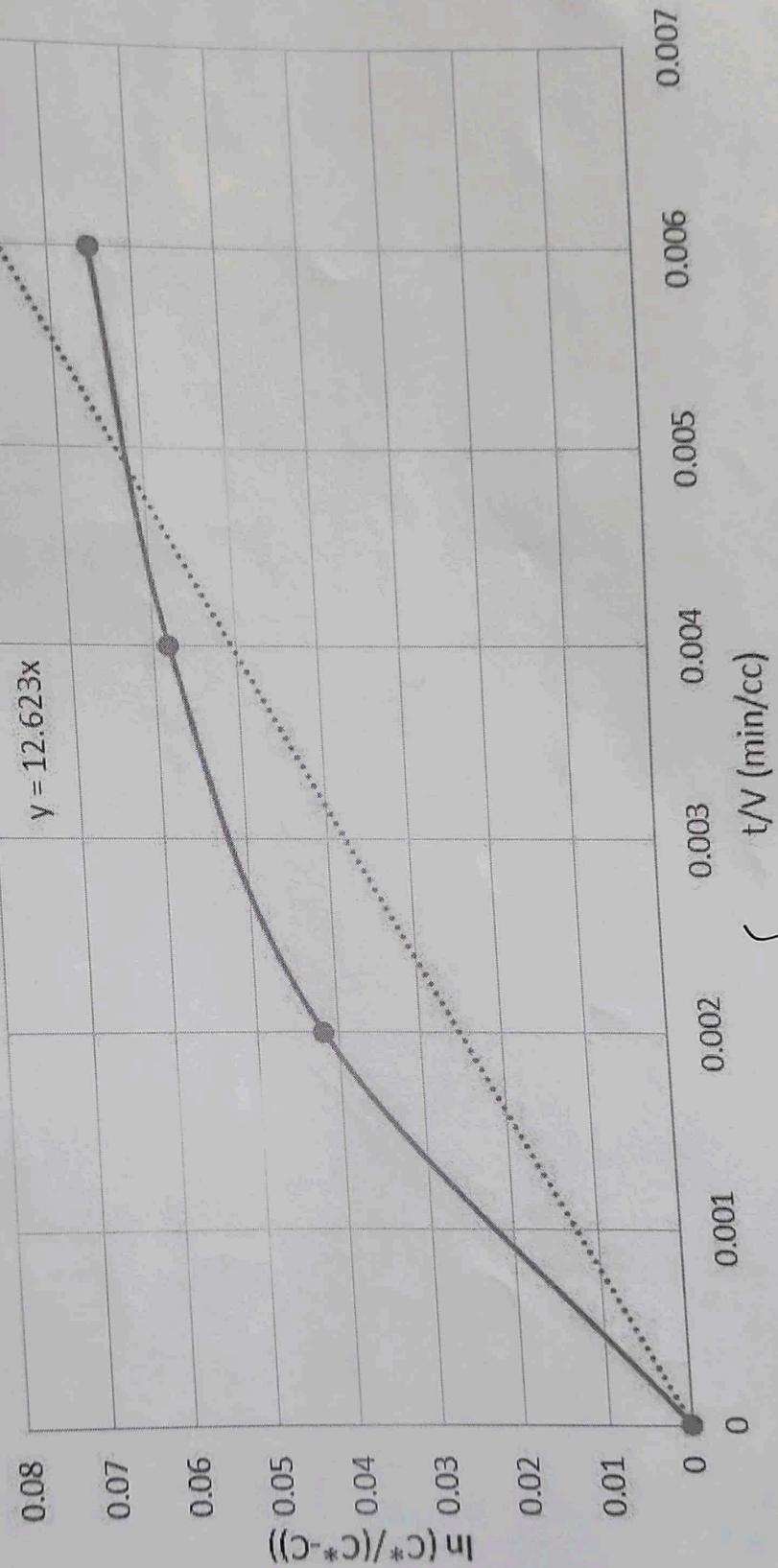
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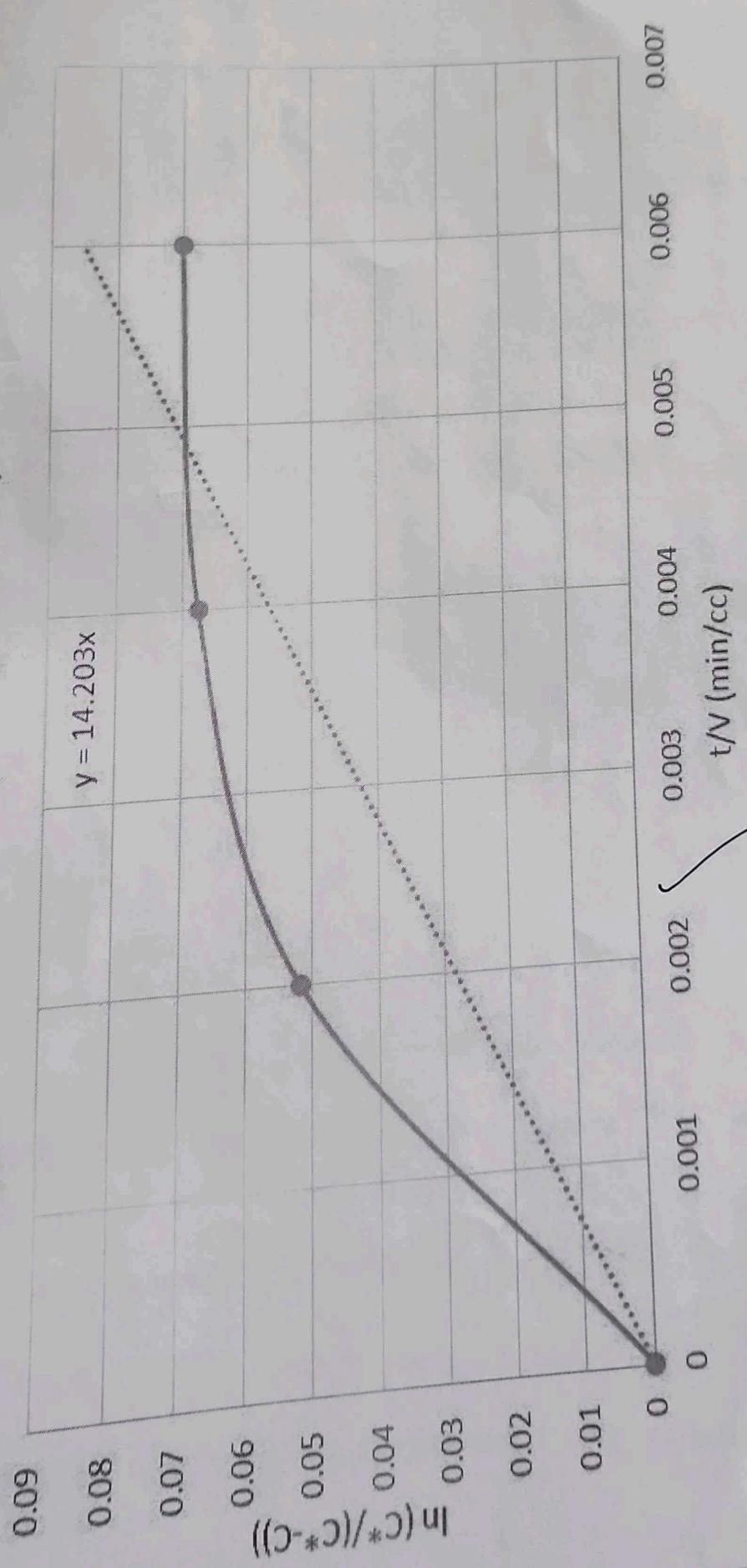
RUN 1:  $\ln(C^*/(C^*-C))$  v/s  $(t/V)$



RUN 2:  $\ln(C^*/(C^*-C))$  v/s  $(t/V)$



RUN 3:  $\ln(C^*/(C^*-C))$  v/s  $(t/V)$



# INDIAN INSTITUTE OF TECHNOLOGY

DATE

Residence Time distribution in a tubular vessel

SHEET NO.

## Objective:

- 1) To construct a curve for pulse input
- 2) To plot age-distribution curve ( $t^{v+}$ )
- 3) To calculate average residence time
- 4) To calculate the vessel dispersion number ( $D/UL$ ) for three different flowrates.

Theory: Real Reactor never fully follow the two idealized flow patterns, plug flow & mixed flow. The deviation from the ideal flow pattern can be determined by residence time distribution. According to Fick's law for molecular diffusion, in the x-direction the differential eq<sup>n</sup> is  $\frac{\partial C}{\partial t} = D \frac{\partial^2 C}{\partial x^2}$ , where  $D$  is axial dispersion coefficient.

In dimensionless form using  $z = \frac{x}{L}$ ,  $\Theta = \frac{t}{t''} = \frac{tu}{L}$ ,

$\frac{\partial C}{\partial \Theta} = \frac{D}{UL} \frac{\partial^2 C}{\partial z^2} - \frac{\partial C}{\partial z}$  where the dimensionless group  $\frac{D}{UL}$  is called vessel dispersion number, is the parameter which measures the extent of axial dispersion.

For  $\frac{D}{UL} \rightarrow 0$ , negligible dispersion, plug flow

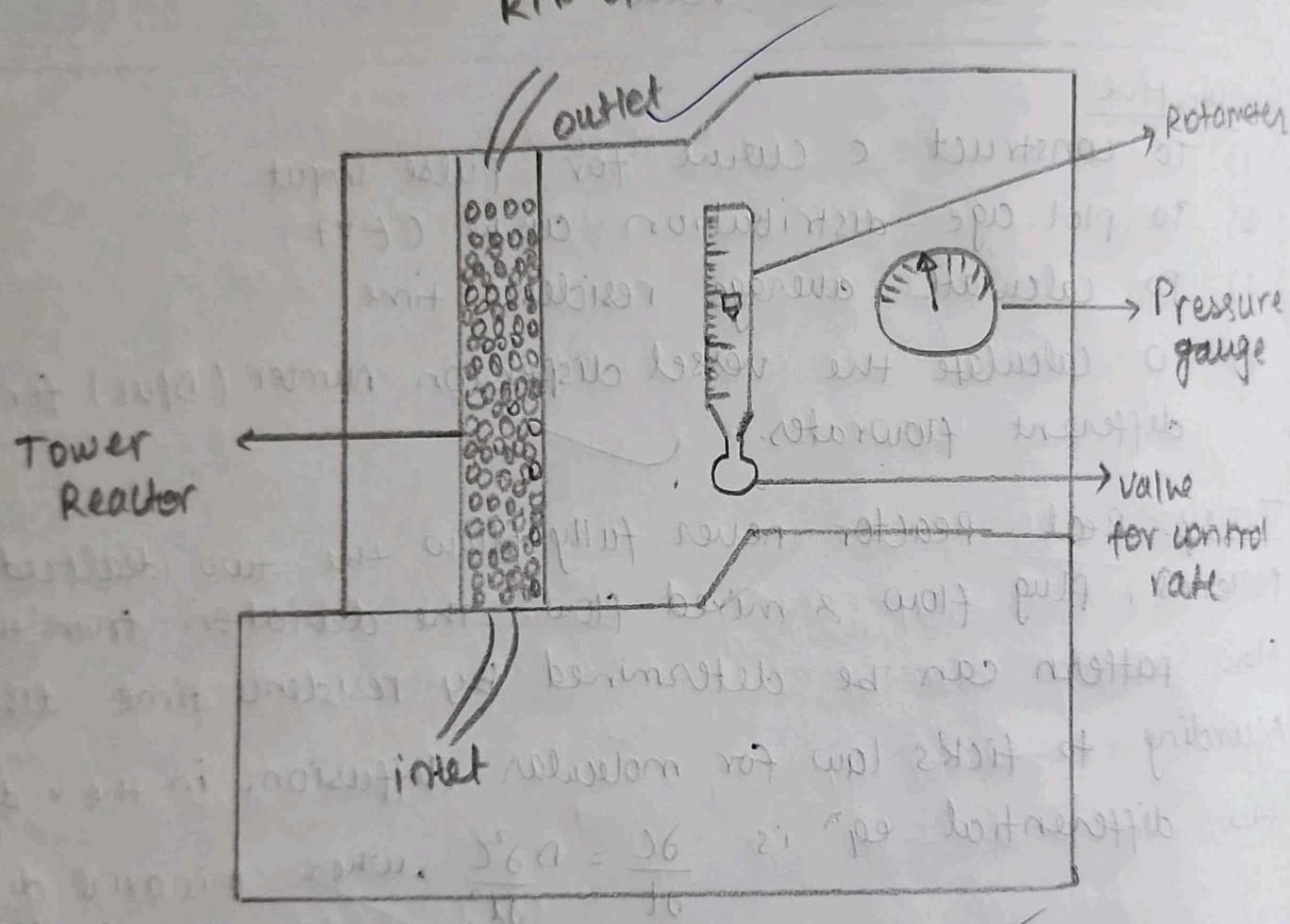
For  $\frac{D}{UL} \rightarrow \infty$ , large dispersion, mixed flow

For closed vessel  $\frac{D}{UL}$  can be determined by following eq<sup>n</sup>:

$$\sigma_0^2 = 2 \frac{D}{UL} - 2 \left( \frac{D}{UL} \right)^2 \left( 1 - e^{-\frac{UL}{D}} \right)$$

$$\text{where, } \sigma_0^2 = \frac{\sigma^2}{t^2}, \quad \sigma^2 = \frac{\sum t_i^2 C_i - \bar{t}^2 \sum C_i}{\sum C_i} \quad \bar{t} = \frac{\sum t_i C_i}{\sum C_i}$$

## RTD of a packed tower



schematic diagram of the experimental setup

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SHEET NO.

## Apparatus:

- 1) one tubular vessel ( $L=81.5\text{ cm}$ ) packed with Rasching rings
- 2) Dye injection system
- 3) stop watch & test tubes
- 4) colorimeter

## Procedure:

- 1) start the water flow by adjusting the valve & metered by calibrated rotameter.
- 2) inject the dye (5 ml of methylene blue) by the hypodermic syringe through the injection port near the inlet.
- 3) collect 10-12 samples from the outlet in different test tubes at one min interval
- 4) Analyze the sample by a spectrophotometer at 664 nm.
- 5) calculate vessel dispersion number from average residence time & variance.
- 6) plot C curve vs t curve.
- 7) plot  $D_{hl}/L$  as a f<sup>n</sup> of Reynold's number

## Observation table:

attached

## Calculations:

$$\text{flow rate} = 15 \text{ LPH}$$

$$\sum C_i = 2.701, \quad \sum t_i C_i = 17.3385$$

$$\sum t_i^2 C_i = 123.61225$$

$$t = \frac{\sum t_i c_i}{\sum c_i} = 6.41928$$

$$\sigma^2 = \left( \frac{\sum t_i^2 c_i}{\sum c_i} \right) - t^2 = 4.33595$$

$$\sigma_0^2 = \frac{\sigma^2}{t^2} = 0.10522$$

length ~~t~~  $\Rightarrow$   $0.815 \text{ m}$ , Dia  $= 0.05 \text{ m}$ , Area  $= \frac{\pi d^2}{4} = 0.00196 \text{ m}^2$

flow rate = area  $\times$  velocity  $= 0.00000416 \text{ m}^3/\text{s}$

velocity  $= 0.0021220 \text{ m/s}$ ,

$$Re = \frac{\rho V D}{\mu} = 106.103 \quad \checkmark \quad \frac{D}{UL} = 0.055715$$

### Results:

Flow Rate (lpm)	$t (\text{min})$	$\sigma^2$	$\sigma_0^2$	Re	$D/UL$
15	6.4193	4.336	0.1052	106.1033	0.0557
17.5	5.9458	4.3137	0.122	123.7872	0.0652
20	4.9791	4.5297	0.1827	141.4711	0.101

### Discussion:

RTD or Residence time distribution is affected by the following factors:

- 1) flow patterns:  $\rightarrow$  Reflects how fluid moves through a system, impacting mixing & dispersion.

- ii) dead zone: These are the stagnant regions within the reactor, leading to elements spending more time than the average residence time
- iii) Axial dispersion: fluid elements don't follow a single path, leading to same mixing along the reactor length.
- The c-curve reflects the distribution of residence times experienced by fluid elements within the reactor. A sharp peak indicates an ideal PFR with all elements exiting at same residence time. A broader peak suggests axial dispersion or mixing, while multiple peaks might indicate channeling or dead zones.
- The  $\epsilon$ -curve is the derivative of the c-curve w.r.t time  $gt$  represents the fraction of liquid leaving the reactor at time ' $t$ '.
- The vessel dispersion number ( $D_{tr}$ ) increases with Reynold's number ( $Re$ ) due to
  - (i) greater fluid movements within the vessel, leading to increased dispersion of the material.
  - (ii) increased turbulence which enhances mixing & dispersion of material.

### Conclusion:

RTD in a tubular vessel was observed for methylene blue as the dye material. The c &  $\epsilon$  curves were plotted for three different flow rates. The mean residence time

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and vessel dispersion number were calculated for these flow rates. The vessel dispersion number increased with Reynold's number as expected.

## Precautions:

- Tubular vessel should be cleaned before every use.
- The calorimeter should be used with care.
- The surface of the sampling container should be cleaned properly.

15 LPH					
Time [ti] (min)	Absorbance [Ci]	tiCi	ti^2Ci	Ei	
0	0	0	0	0	0
0.5	0.012	0.006	0.003	0.008749544295	
1	0.017	0.017	0.017	0.01239518775	
1.5	0.022	0.033	0.0495	0.01604083121	
2	0.03	0.06	0.12	0.02187386074	
2.5	0.039	0.0975	0.24375	0.02843601896	
3	0.042	0.126	0.378	0.03062340503	
3.5	0.05	0.175	0.6125	0.03645643456	
4	0.055	0.22	0.88	0.04010207802	
4.5	0.16	0.72	3.24	0.1166605906	
5	0.275	1.375	6.875	0.2005103901	
5.5	0.335	1.8425	10.13375	0.2442581116	
6	0.39	2.34	14.04	0.2843601896	
6.5	0.263	1.7095	11.11175	0.1917608458	
7	0.211	1.477	10.339	0.1538461538	
7.5	0.172	1.29	9.675	0.1254101349	
8	0.138	1.104	8.832	0.1006197594	
8.5	0.119	1.0115	8.59775	0.08676631425	
9	0.087	0.783	7.047	0.06343419614	
9.5	0.072	0.684	6.498	0.05249726577	
10	0.061	0.61	6.1	0.04447685016	
10.5	0.054	0.567	5.9535	0.03937294933	
11	0.05	0.55	6.05	0.03645643456	
11.5	0.047	0.5405	6.21575	0.03426904849	
12	0.042	0.504	6.048	0.03062340503	

$\Sigma Ci$	2.701
$\Sigma tiCi$	17.3385
$\Sigma ti^2Ci$	123.01225
$t = \Sigma tiCi / \Sigma Ci$	6.419289152
$\sigma^2 = (\Sigma ti^2Ci / \Sigma Ci) - t^2$	4.335951512
$\sigma \theta^2 = \sigma^2 / t^2$	0.1052229661
Length(L)(m)	0.815
Diameter(d)(m)	0.05
Flow Area(m <sup>2</sup> )	0.001963495408
Flow Rate(m <sup>3</sup> /s)	0.000004166666
Velocity(m/s)	0.002122065908
Re	106.1032954
D/uL	0.05571534829

17.5 LPH

Time [ti] (min)	Absorbance [Ci]	tiCi	ti^2Ci	Ei
0	0	0	0	0
0.5	0.005	0.0025	0.00125	0.004205214466
1	0.008	0.008	0.008	0.006728343146
1.5	0.012	0.018	0.027	0.01009251472
2	0.018	0.036	0.072	0.01513877208
2.5	0.024	0.06	0.15	0.02018502944
3	0.035	0.105	0.315	0.02943650126
3.5	0.055	0.1925	0.67375	0.04625735913
4	0.204	0.816	3.264	0.1715727502
4.5	0.345	1.5525	6.98625	0.2901597981
5	0.356	1.78	8.9	0.29941127
5.5	0.314	1.727	9.4985	0.2640874685
6	0.212	1.272	7.632	0.1783010934
6.5	0.161	1.0465	6.80225	0.1354079058
7	0.127	0.889	6.223	0.1068124474
7.5	0.087	0.6525	4.89375	0.07317073171
8	0.078	0.624	4.992	0.06560134567
8.5	0.063	0.5355	4.55175	0.05298570227
9	0.053	0.477	4.293	0.04457527334
9.5	0.049	0.4655	4.42225	0.04121110177
10	0.039	0.39	3.9	0.03280067283
10.5	0.034	0.357	3.7485	0.02859545837
11	0.038	0.418	4.598	0.03195962994
11.5	0.035	0.4025	4.62875	0.02943650126
12	0.026	0.312	3.744	0.02186711522

$$\Sigma Ci = 2.378$$

$$\Sigma tiCi = 14.139$$

$$\Sigma ti^2Ci = 94.325$$

$$t = \Sigma tiCi / \Sigma Ci = 5.945752733$$

$$\sigma^2 = (\Sigma ti^2Ci / \Sigma Ci) - t^2 = 4.313709883$$

$$\sigma \theta^2 = \sigma^2 / t^2 = 0.1220217488$$

$$\text{Length}(L)(m) = 0.815$$

$$\text{Diameter}(d)(m) = 0.05$$

$$\text{Flow Area}(m^2) = 0.001963495408$$

$$\text{Flow Rate}(m^3/s) = 0.000004861111$$

$$\text{Velocity}(m/s) = 0.002475743559$$

$$Re = 123.787178$$

$$D/uL = 0.0652712$$

20 LPH

Time [ti] (min)	Absorbance [Ci]	tiCi	ti^2Ci	Ei
0	0	0	0	0
0.5	0.003	0.0015	0.00075	0.002794597112
1	0.02	0.02	0.02	0.01863064741
1.5	0.004	0.006	0.009	0.003726129483
2	0.007	0.014	0.028	0.006520726595
2.5	0.055	0.1375	0.34375	0.05123428039
3	0.256	0.768	2.304	0.2384722869
3.5	0.396	1.386	4.851	0.3688868188
4	0.308	1.232	4.928	0.2869119702
4.5	0.208	0.936	4.212	0.1937587331
5	0.164	0.82	4.1	0.1527713088
5.5	0.115	0.6325	3.47875	0.1071262226
6	0.093	0.558	3.348	0.08663251048
6.5	0.083	0.5395	3.50675	0.07731718677
7	0.065	0.455	3.185	0.0605496041
7.5	0.057	0.4275	3.20625	0.05309734513
8	0.049	0.392	3.136	0.04564508617
8.5	0.056	0.476	4.046	0.05216581276
9	0.038	0.342	3.078	0.03539823009
9.5	0.037	0.3515	3.33925	0.03446669772
10	0.03	0.3	3	0.02794597112
10.5	0.025	0.2625	2.75625	0.02328830927
11	0.02	0.22	2.42	0.01863064741
11.5	0.019	0.2185	2.51275	0.01769911504
12	0.019	0.228	2.736	0.01769911504
$\Sigma Ci$	2.108			
$\Sigma tiCi$	10.496			
$\Sigma ti^2Ci$	61.8095			
$t = \Sigma tiCi / \Sigma Ci$	4.979127135			
$\sigma^2 = (\Sigma ti^2Ci / \Sigma Ci) - t^2$	4.529687663			
$\sigma \theta^2 = \sigma^2 / t^2$	0.1827097932			
Length(L)(m)	0.815			
Diameter(d)(m)	0.05			
Flow Area(m <sup>2</sup> )	0.001963495408			
Flow Rate(m <sup>3</sup> /s)	0.000005555555			
Velocity(m/s)	0.002829421211			
Re	141.4710605			
D/uL	0.101697			

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Kinetic Study in Batch Reactor

SHEET NO.

## Objective:

- To determine the order of Rxn b/w sodium hydroxide & ethyl acetate
- To find the rate constant at three temp.
- To determine the activation energy & frequency factor.

## Theory:

Stoichiometric eq<sup>n</sup>:



Rate eq<sup>n</sup>:

$$r_A = -\frac{dc}{dt} = k_2 (A_0)^2 (1-x_A)(M-x_A) \text{ assuming 2nd order}$$

where  $M = \frac{C_B}{C_{A_0}}$ ,  $t$  = time in min,  $x_A$  = conversion

$k_2$  = rate constant in  $\text{L/gmol(min)}$

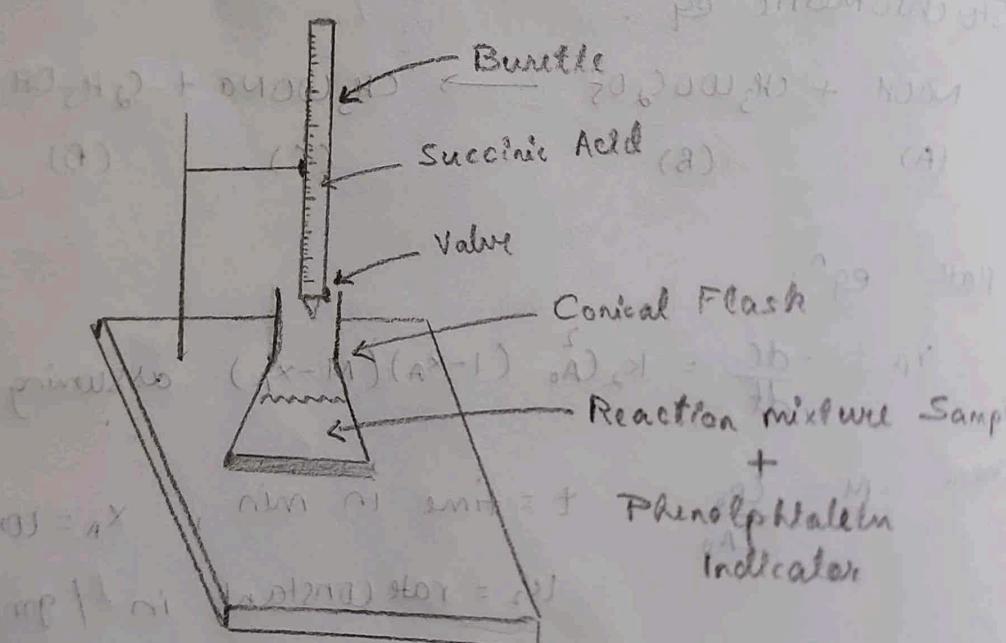
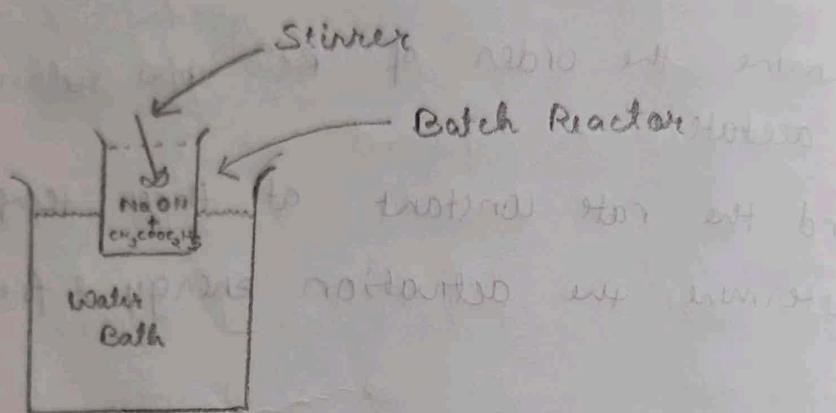
The integral form:

$$\ln \frac{M-x_A}{M(1-x_A)} = C_{A_0}(M-1)k_2 t$$

Apparatus:

- stainless steel batch reactor fitted with stirrer
- constant temperature bath

Digraphs



## Titration Apparatus

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- stop watch
- titration flask

Procedure:

- One liter of NaOH sol<sup>n</sup> is poured in the batch reactor. 9 ml of pure ethyl acetate (as supplied) is added and at the same time start the stop-watch.
- 5ml sol<sup>n</sup> is pipelles out of the Reactor at each min-interval & titrated against given succinic acid sol<sup>n</sup> with phenolphthalein as indicator. At least 5 sample are taken at definite time intervals.
- Repeat experiment for three temp.
- Titrate 5ml of the given NaOH with succinic acid using phenolphthalein indicator to get  $c_{A0}$  gmol/L
- Find rate constant  $k_2$  from the plot of  $\ln \frac{M-x_A}{M(1-x_A)}$  vs  $t$
- Determine  $k_2$  at 3 different temp & plot  $\ln k_2$  against  $\frac{1}{T}$
- Take 5ml of supplied NaOH by the standard succinic sol<sup>n</sup> to get  $c_{A0}$  in (g/mol / lit)

### Observation Table

for 5 ml of NaOH, vol of succinic acid ( $N/50$ ) required  
in 9 ml

Time (min)	vol of succinic acid ( $N/50$ ) for 5ml NaOH		
	$T = 8^\circ\text{C}$	$T = 25^\circ\text{C}$	$T = 45^\circ\text{C}$
2	5.8 mL	2.1 mL	1.9 mL
4	4 mL	1.9 mL	1.5 mL
6	2.9 mL	1.8 mL	0.9 mL
8	2.7 mL	1.5 mL	0.8 mL
10	2.4 mL	1.3 mL	0.6 mL

### Sample calculation:

for  $T = 8^\circ\text{C}$ ,  $t = 2\text{ min}$

vol of succinic acid required to titrate 5ml of  
reaction mixture = 5.8 mL

$$C_A = 5.8 \times \frac{1}{50} = 0.0232$$

$$C_{A0} = 0.0424$$

$$C_B = \frac{5.118}{(88.41 \times 1.09)} = 0.0913$$

$$X_A = \frac{C_{A0} - C_A}{C_{A0}} = 0.4479$$

$$M = \frac{C_B}{C_{A0}} = 2.1536$$

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$$\frac{\ln(n-x_A)}{M(1-x_A)} = 0.36087$$

The plot of  $\ln(k_2)$  vs  $\frac{1}{T}$  has slope =  $-\frac{E_A}{R}$   
 Intercept =  $\ln(A_0)$

$$\therefore \text{slope} = -\frac{E_A}{R} = -1170 \Rightarrow E_A = 9.7274 \text{ kJ/mol}$$

$$\text{Intercept} = \ln A_0 = 5.32 \Rightarrow A = 204.38 \frac{\text{ltr}}{(\text{gmol})(\text{min})}$$

Calculation table: Attached

Results:

Temp (°C)	Rate constant ( $k_2$ )
8	0.165
26	0.175
45	0.269

## Discussion:

Rate constant is a  $f^n$  of temp & activation energy, we see that as temp  $\uparrow$  the rate constant also increases. This is for the endothermic reaction.

The order of Reaction doesn't necessarily depend on the stoichiometric coefficient of reactants. The order of the reaction is determined experimentally. All the reactions we study may not be elementary (single step), therefore it is important that we study the intermediate steps occurs in a reaction, & identify the rate determining step.

Designing a batch Reactor involves the knowledge of Rx<sup>n</sup> kinetics & reaction mechanism. The nature of the Rx<sup>n</sup> should also be known. To ~~able~~ describe the kinetics of a reaction some of the important parameters are rate constant (k), order of the reaction, frequency factor. The determination of these factors will help us optimize the design of various reactors.

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### Precautions:

- The test tubes & flasks need to be washed before titrating other sample.
- The normality of NaOH sol<sup>n</sup> to be used measured before hand.
- Titration should be done properly & readings should be taken with precision.

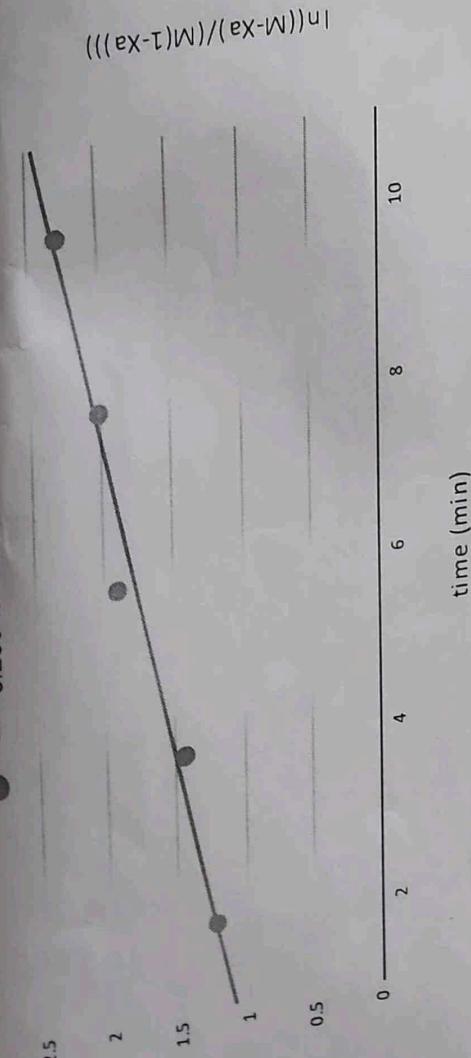
### Conclusion:

In this experiment, NaOH & ethyl acetate were reacted in a typical saponification reaction way. These reaction were carried out at different temperatures. The samples were taken after 2 minutes to calculate the unreacted NaOH in the batch reactor. Then, we plotted the graph of  $(\alpha)$  vs  $t$ , & determined the rate constant for all the cases. Then from a plot of  $\ln k$  vs  $\frac{1}{T}$  graph, we get frequency factor & activation energy.

Time (min)	Volume (mL)	$C_{AO}$	$C_{BO}$	M	Normality	$x_A$	$\ln((M-x_A)/(M(1-x_A)))$	$K_2 \text{ (L/(mol.min))}$
T = 8 C								
2	5.8	0.0424	0.09131301433	2.153608829	0.0232	0.4479056604	0.3608688234	3.373335343
4	4	0.0424	0.09131301433	2.153608829	0.016	0.619245283	0.6265706055	
6	2.9	0.0424	0.09131301433	2.153608829	0.0116	0.7239528302	0.8774724176	
8	2.7	0.0424	0.09131301433	2.153608829	0.0108	0.742990566	0.9355256192	
10	2.4	0.0424	0.09131301433	2.153608829	0.0096	0.7715471698	1.032856902	
T = 26 C								
2	2.1	0.0424	0.09131301433	2.153608829	0.0084	0.8001037736	1.04550952	3.57777991
4	1.9	0.0424	0.09131301433	2.153608829	0.0076	0.8191415094	1.331427614	
6	1.8	0.0424	0.09131301433	2.153608829	0.0072	0.8286603774	1.578336189	
8	1.5	0.0424	0.09131301433	2.153608829	0.006	0.8572169811	1.638869095	
10	1.3	0.0424	0.09131301433	2.153608829	0.0052	0.876254717	1.767175874	
T = 45 C								
2	1.9	0.0424	0.09131301433	2.153608829	0.0076	0.8191415094	1.231427614	5.499558833
4	1.5	0.0424	0.09131301433	2.153608829	0.006	0.8572169811	1.438869095	
6	0.9	0.0424	0.09131301433	2.153608829	0.0036	0.9143301887	1.904639284	
8	0.8	0.0424	0.09131301433	2.153608829	0.0032	0.9238490566	2.014711694	
10	0.6	0.0424	0.09131301433	2.153608829	0.0024	0.9428867925	2.286791829	

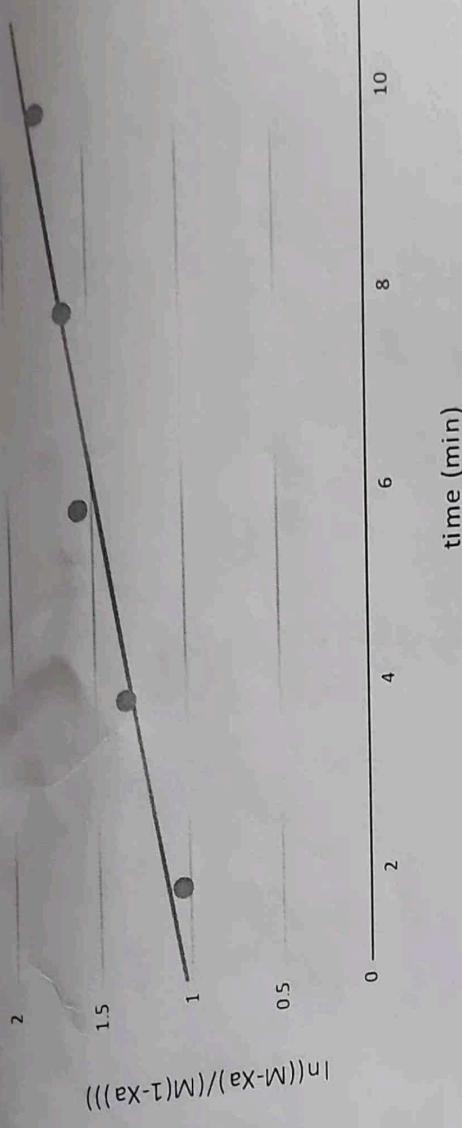
$\ln((M-X_a)/(M(1-X_a)))$  vs time for  $T = 45^\circ C$

$$\bullet - 0.269 \cdot x + 1.24$$



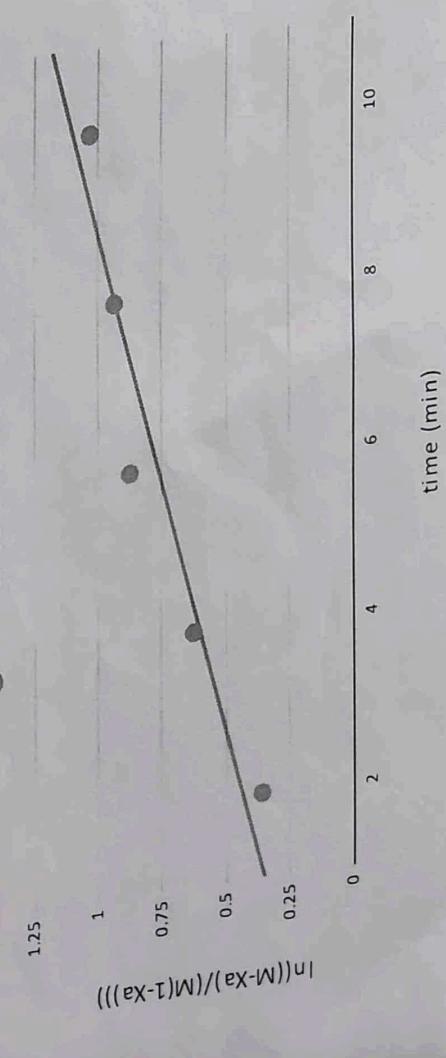
$\ln((M-X_a)/(M(1-X_a)))$  vs time for  $T = 45^\circ C$

$$\bullet - 0.175 \cdot x + 1.12$$



$\ln((M-X_a)/(M(1-X_a)))$  vs time for  $T = 8^\circ C$

$$\bullet - 0.165 \cdot x + 0.436$$



$\ln((M-X_a)/(M(1-X_a)))$

$1/T$

$$0.0033 \quad 0.0034$$

$$0.0032 \quad 0.0033$$

$$0.0034$$