

SATHYABAMA UNIVERSITY
(Established Under Section 3 Of UGC Act 1956)
DEPARTMENT OF CHEMICAL ENGINEERING
Jeppiaar Nagar, Chennai – 119.



SCHX4008 – Chemical Reaction Engineering Lab

Name : _____

Register Number : _____

Batch Number : _____

Dos and Don'ts in the laboratory

Life threatening injuries can happen in the laboratory. For that reason, students need to be informed of the correct way to act and things to do in the laboratory. The following is a safety checklist that can be used as a handout to students to acquaint them with the safety.

Conduct

- Do not engage in practical jokes or boisterous conduct in the laboratory.
- The use of mobile phone and programmable calculators / Laptop is prohibited.
- The performance of unauthorized experiments is strictly forbidden.
- Do not sit on laboratory benches while performing experiments
- Never run in the laboratory. Wear the personal protective equipment.

General Work Procedure

- Never work in the laboratory without the supervision of a teacher.
- Always perform the experiments or work precisely as directed by the teacher.
- Immediately report any spills, accidents, or injuries to Lab in-charge.
- Never leave experiments while in progress.
- Be careful when handling hot glassware and apparatus in the lab. Hot glassware looks just like cold glassware.
- Never fill a pipette using mouth suction. Always use a pipetting device. If you suffering from any mouth injury use measuring jars after getting permission from Lab in-charge.
- Turn off all heating apparatus, gas valves, and water faucets when not in use.
- Coats, bags, and other personal items must be stored in designated areas, not on the bench tops or in the aisle ways.
- Notify your teacher of any sensitivity that you may have to particular chemicals if known.
- Keep the floor clear of all objects (e.g., ice, small objects, and spilled liquids).

Housekeeping

- Keep work area neat and free of any unnecessary objects.
- Thoroughly clean your laboratory work space at the end of the laboratory session.
- Do not block the sink drains with debris.
- Inspect all equipment for damage (cracks, defects.) prior to use; do not use damaged equipment.
- Never pour chemical waste into the sink drains or wastebaskets.
- Place chemical waste in appropriately labeled waste containers.
- Properly dispose of broken glassware and other sharp objects.

Apparel in the Laboratory

- Wear disposable gloves, as provided in the laboratory, when handling hazardous materials. Remove the gloves before exiting the laboratory.
- Wear a full-length, long-sleeved laboratory coat or chemical-resistant apron.
- Wear shoes that adequately cover the whole foot; low-heeled shoes with non-slip soles are preferable. Do not wear sandals, open-toed shoes, Open-backed shoes or high-heeled shoes in the laboratory.
- Secure long hair and loose clothing from the rotating parts. (especially loose long sleeve)

CONTENTS

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STANDARDIZATION OF NaOH

Take NaOH in the burette.

Pipette out 10 ml (V_A) Oxalic acid into 100 ml conical flask.

S. No.	Burette Reading (ml)		Difference = I – F (ml)	Normality of NaOH (N_A) mol/l
	Initial (I)	Final (F)		
1				
2				

$$N_A = \frac{N_{COOH_2} \times V_{COOH_2}}{V_A} \quad N$$

OBSERVATION TABLE

S. No.	Time (t) min	Titer value (y) ml
1		
2		
3		
4		
5		
6		
7		

CALCULATIONS**ANALYTICAL METHOD**

- Normality of $CH_3COOCH_2CH_3$ $N_B = 0.05$ N
- Normality of NaOH $N_A = \underline{\hspace{2cm}}$ N
- Initial Concentration of NaOH $C_{A0} = \frac{N_A V_A}{V_A + V_B}$ mol/l
 $C_{A0} = \underline{\hspace{2cm}}$ mol/l
- Initial Concentration of $CH_3COOCH_2CH_3$ $C_{B0} = \frac{N_B V_B}{V_A + V_B}$ mol/l
 $C_{B0} = \underline{\hspace{2cm}}$ mol/l
- Initial molar ratio of the reactants $M = \frac{C_{B0}}{C_{A0}}$ No unit

Experiment No:**Date:****INTERPRETATION OF BATCH REACTOR****AIM**

To determine second order rate constant for saponification reaction between ethyl acetate and sodium hydroxide.

APPARATUS

Batch Reactor Setup	-	1 unit
100 ml standard flask	-	4 No.
500 ml standard flask	-	2 Nos.
100 ml conical flasks	-	5 Nos.
50 ml burette	-	1 No.
10 ml pipette	-	1 No.

CHEMICALS AND REAGENTS

Ethyl acetate	500 ml	0.05 N
Sodium hydroxide	600 ml	0.05 N
Oxalic acid	100 ml	0.05 N
Hydrochloric acid	100 ml	0.05 N
Phenolphthalein indicator	--	--

THEORY

Equipment by which empirical information is obtained can be divided into two types, the batch and *flow* reactors. The batch reactor is simply a container to hold the contents while they react. All that has to be determined is the extent of reaction at various times, and this can be followed in a number of ways, for example:

1. By following the concentration of a given component.
2. By following the change in some physical property of the fluid, such as the electrical conductivity or refractive index.
3. By following the change in total pressure of a constant-volume system.
4. By following the change in volume of a constant-pressure system.

The experimental batch reactor is usually operated isothermally and at constant volume because it is easy to interpret the results of such runs. This reactor is a relatively simple device adaptable to small-scale laboratory set-ups, and it needs but little auxiliary equipment or instrumentation. Thus, it is used whenever possible for obtaining homogeneous kinetic data

There are two procedures for analyzing kinetic data, the *integral* and the *differential* methods. In the integral method of analysis we guess a particular form of rate equation and, after appropriate integration and mathematical manipulation, predict that the plot of a certain concentration function versus time should yield a straight line. The data are plotted, and if a reasonably good straight line is obtained, then the rate equation is said to satisfactorily fit the data.

6. Unreacted concentration of NaOH at any time

$$C_A = N_{\text{HCl}} - \frac{N_{A \times Y}}{10} \quad \text{mol/l}$$

$$C_A = \underline{\hspace{2cm}} \quad \text{mol/l}$$

7. Fractional conversion of NaOH

$$X_A = \frac{C_{A0} - C_A}{C_{A0}} \quad \text{No unit}$$

$$X_A = \underline{\hspace{2cm}} \quad \text{No unit}$$

Sl. No.	Time (t in min)	X_A	$\ln \frac{M - X_A}{M(1 - X_A)}$	Rate Constant k in l/mol.min
1				
2				
3				
4				
5				
6				
7				

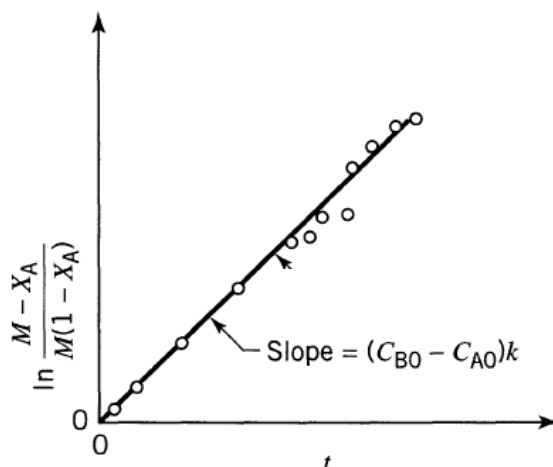
Average of k values = $\underline{\hspace{2cm}}$ l/mol/min

8. From the integrated rate equation of bimolecular irreversible reaction,

$$\text{Rate constant } k = \frac{1}{C_{B0} - C_{A0} t} \ln \frac{M - X_A}{M(1 - X_A)} \quad \text{l/mol.min}$$

GRAPHICAL METHOD

Plot 't' in x-axis and $\ln \frac{M - X_A}{M(1 - X_A)}$ in y-axis



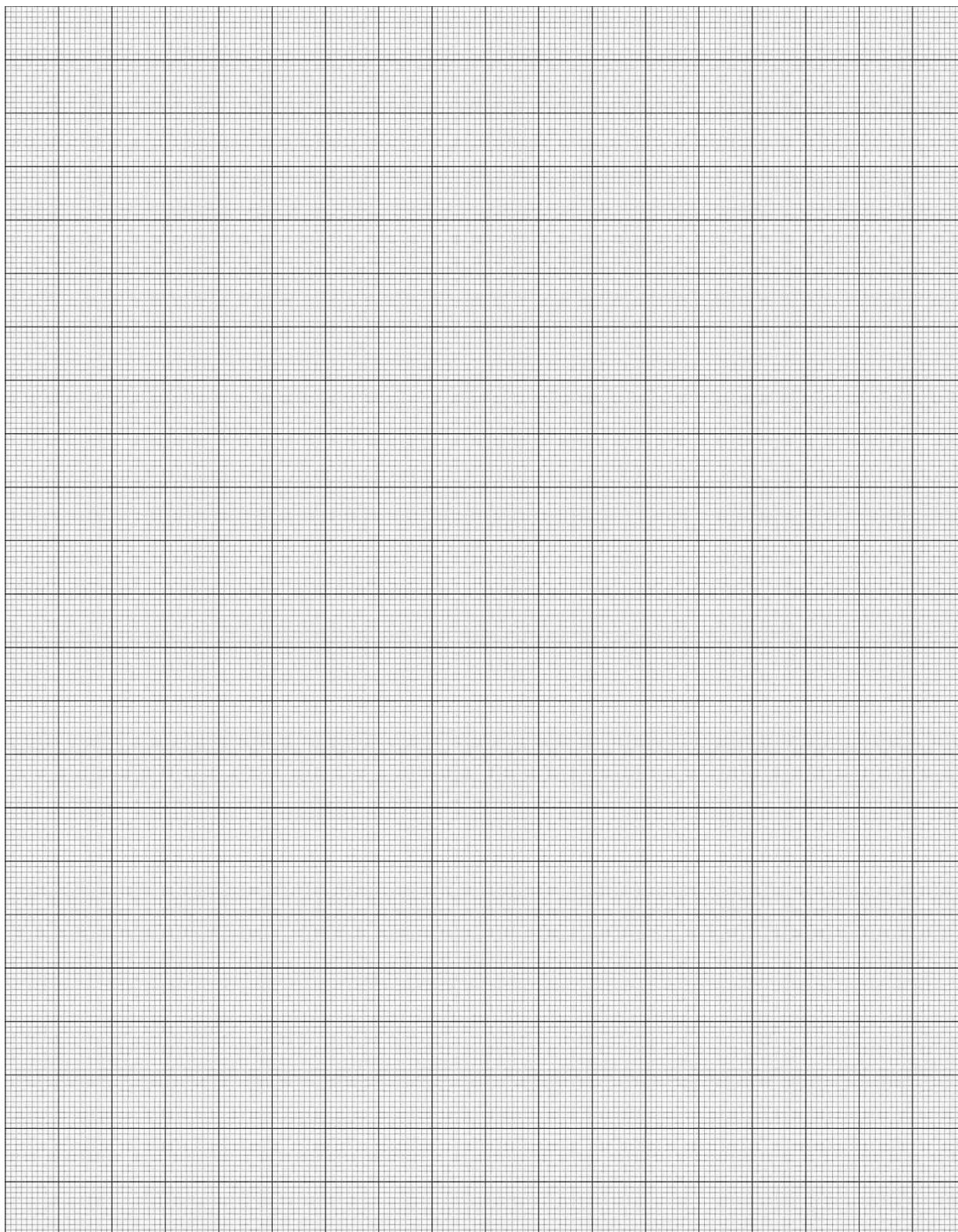
From graph, slope

$$m = \underline{\hspace{2cm}}$$

So, Rate constant

$$k = \frac{m}{C_{B0} - C_{A0}} \quad \text{l/mol.min}$$

Scale
In x-axis 1cm =
In y-axis 1 cm =



PROCEDURE

1. Standardize NaOH and HCl.
2. Pipette out 10 ml of 0.05 N HCl and transfer it to 100 ml conical flask.
3. Transfer 500 ml of NaOH into the reactor and switch on the stirrer.
4. Add 500 ml of ethyl acetate into the reactor and simultaneously start the stop watch.
5. Pipette out 10 ml reaction mixture at the end of every one minute and transfer into the flask containing HCl.
6. Titrate the contents with NaOH using phenolphthalein indicator.
7. Note the end point (y).

RESULT

Second order rate constant for saponification reaction between ethyl acetate and sodium hydroxide was found to be

$$k = \text{_____ } l/mol.min \quad (\text{Analytical method})$$

$$k = \text{_____ } l/mol.min \quad (\text{Graphical method})$$

OBSERVATION TABLEFlow rate of the water is (v) = _____ lph

Sl. No.	Time (t) S	Titer value (y) ml
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		

CALCULATIONS

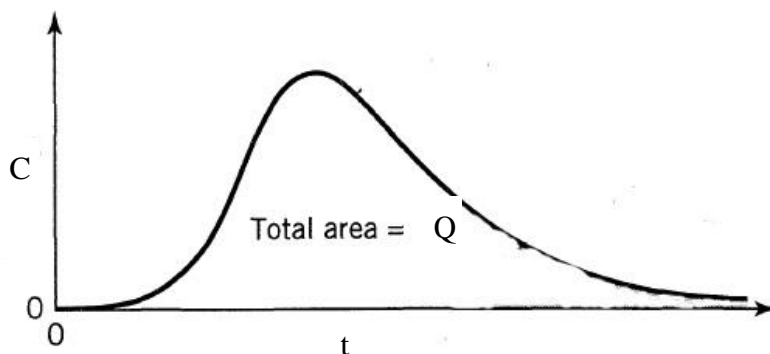
Concentration of NaOH in exit stream

$$C = \frac{N_{\text{COOH}_2} \times y}{10} \quad \text{mol/l}$$

$$C = \underline{\hspace{2cm}} \quad \text{mol/l}$$

C-Curve

Plot time in x-axis and concentration of NaOH in y-axis

From the graph, area under C-curve $Q = C \Delta t = \underline{\hspace{2cm}}$

Residence Time Distribution can be calculated as

$$E = \frac{C}{\text{Area under C-curve}} = \frac{C}{Q} \quad \text{time}^{-1}$$

Experiment No:**Date:****RTD STUDIES IN PLUG FLOW REACTOR****AIM**

To plot RTD curve and to determine the mean residence time for the given plug flow reactor.

APPARATUS

Plug Flow Reactor Setup	-	1 unit
100 ml standard flask	-	1 No.
50 ml standard flask	-	1 No.
100 ml conical flasks	-	8 Nos.
50 ml burette	-	1 No.
10 ml pipette	-	1 No.

CHEMICALS AND REAGENTS

Distilled Water	15 liter	--
Sodium hydroxide as tracer	50 ml	0.3 N
Oxalic acid	100 ml	0.1 N
Phenolphthalein indicator	--	--

THEORY

Plug flow reactor is characterized by the fact that the flow of fluid through the reactor is orderly with no element of fluid overtaking or mixing with any other element ahead or behind. The necessary and sufficient condition for plug flow is for the residence time in the reactor to be the same for all elements of fluid. But real equipment always deviates from these ideals. Three somewhat interrelated factors make up the contacting or flow pattern:

1. The RTD or residence time distribution of material which is flowing through the vessel
2. The **state of aggregation** of the flowing material, its tendency to clump and for a group of molecules to move about together
3. The **earliness and lateness of mixing** of material in the vessel.

The elements of fluid taking different routes through the reactor may take different lengths of time to pass through the vessel. The distribution of these times for the stream of fluid leaving the vessel is called the exit age distribution E , or the residence time distribution RTD of fluid. E has the units of time^{-1} .

PROCEDURE

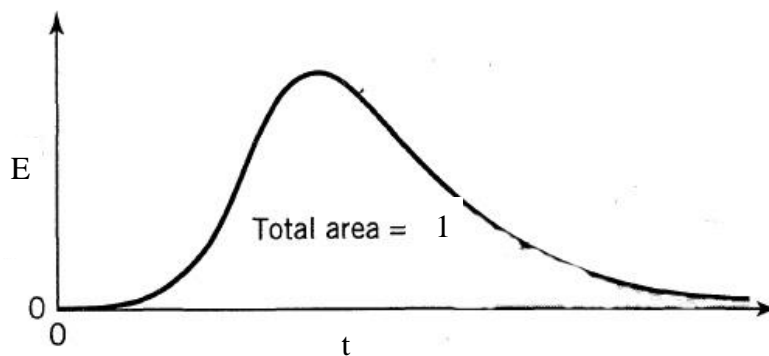
1. Prepare standard solution of oxalic acid and NaOH.
2. Fill the reactor and the feed tank with distilled water.

CALCULATION TABLE

S. No.	time (t) S	Conc. of NaOH (C) mol/l	$E = C / Q$ S^{-1}	$t E \Delta t$
1				
2				
3				
4				
5				
6				
7				
8				
9				
10				

 $E \Delta t =$ $t E \Delta t =$ **E-Curve**

Plot time (t) Vs RTD (E)



Mean residence time

$$t = \frac{t E \Delta t}{E \Delta t}$$

S

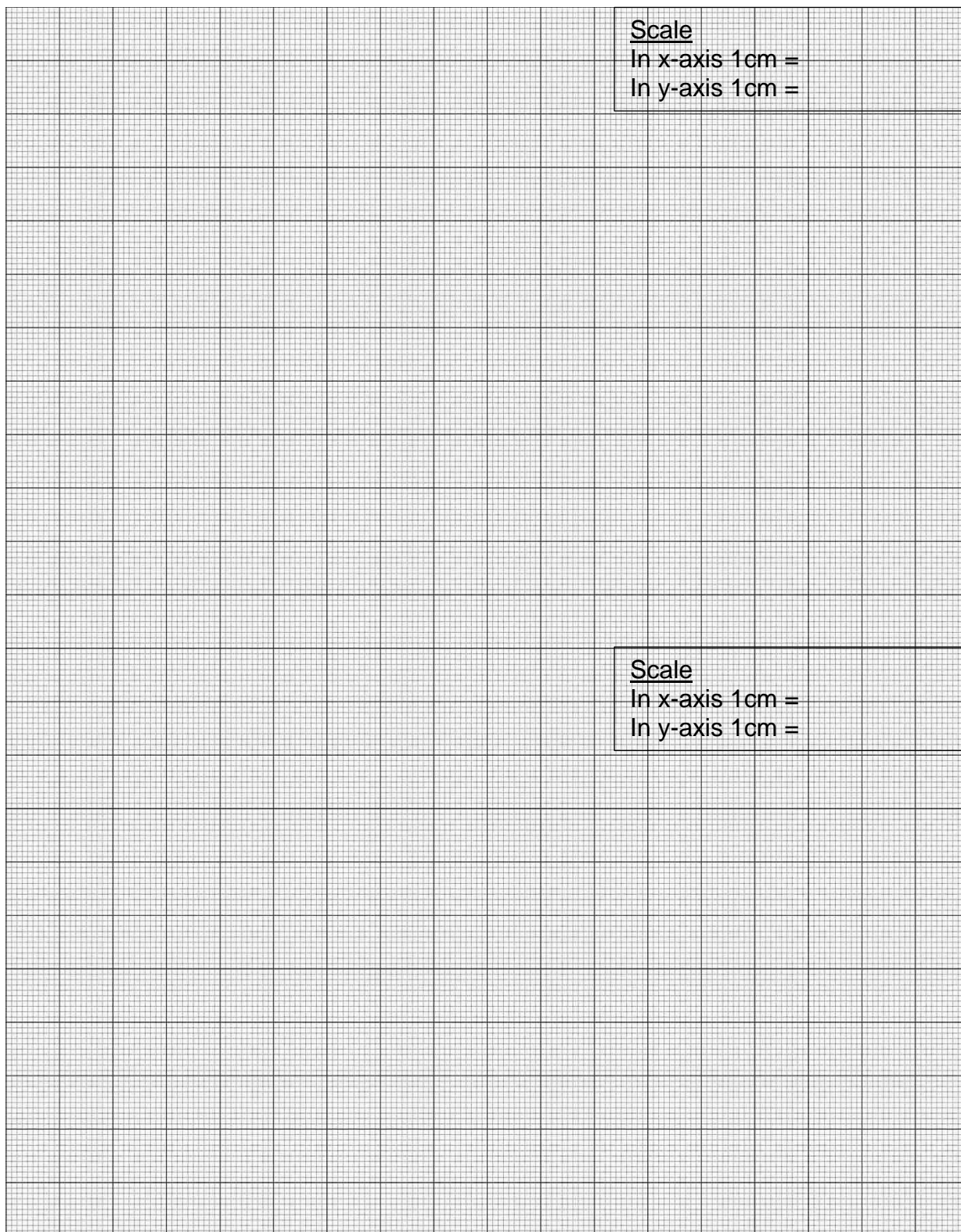
$$t = \underline{\hspace{2cm}}$$

S

Space time

$$\tau = \frac{V_R}{v}$$

S



CALCULATION

CALCULATION

CALCULATION

3. Start to run distilled water at a constant flow rate (v) into the reactor. Ensure that the water flows out from the reactor doesn't contain any NaOH and the water flows through the reactor covering entire cross section. Note down the flow rate.
4. When the reactor starts over flowing inject 10 ml of the tracer into the feed stream in as rapidly as it is possible and start the stop watch.
5. Collect the out flowing sample from the reactor for every 30 seconds (0-30, 30-60, 60-90, and so on) in a conical flask and pipette out 10 ml of this into 100 ml conical flask and titrate with oxalic acid.
6. Continue the experiment till all the injected NaOH leaves the reactor.

OBSERVATION

Volume of the reactor $V_R = \frac{\pi}{4} \times d^2 \times L \quad m^3$

Where

d – diameter of reactor in m

L – Length of the reactor in m

$$V_R = \underline{\hspace{2cm}} m^3 = \underline{\hspace{2cm}} l$$

RESULTS

Residence Time Distribution curve was plotted for the given PFR at a specified feed rate $\underline{\hspace{2cm}}$ lph. The mean residence time for the given plug flow reactor was found to be $\underline{\hspace{2cm}}$ S and space time is $\underline{\hspace{2cm}}$ S

STANDARDIZATION OF NaOH

Take NaOH in the burette.

Pipette out 10 ml (V_A) Oxalic acid into 100 ml conical flask.

S. No.	Burette Reading (ml)		Difference = I – F (ml)	Normality of NaOH (N_A) mol/l
	Initial (I)	Final (F)		
1				
2				

$$N_A = \frac{N_{COOH_2} \times V_{COOH_2}}{V_A} \quad N$$

OBSERVATION TABLE

Volume of the reactor = _____ l

Sl. No.	Flow Rates (lph)		Space Time (min) τ	Titer value of NaOH (ml)		Concentration (mol/l)			Fractional Conversion (No unit)		
	v_A NaOH	v_B EA		PFR (y)	MFR (z)	C_{A0}	C_{A1}	C_{A2}	X_{A1}	X_{Af}	X_{A2}
1											
2											
3											
4											
5											

CALCULATIONS

1. Normality of $CH_3COOCH_2CH_3$ $N_B = 0.05$ mol/l

2. Normality of NaOH $N_A = \underline{\hspace{2cm}}$ mol/l

3. Initial Concentration of NaOH $C_{A0} = \frac{N_A v_A}{v_A + v_B}$ mol/l

$C_{A0} = \underline{\hspace{2cm}}$ mol/l

4. Unreacted concentration of NaOH after reaction in PFR

$C_{A1} = N_{HCl} - \frac{N_A \times y}{10}$ mol/l

$C_{A1} = \underline{\hspace{2cm}}$ mol/l

Experiment No:**Date:****COMBINED REACTORS (PFR FOLLOWED BY MFR)****AIM**

To study the performance of combined reactors (PFR followed by MFR) and to calculate the conversion achieved by this multiple reactor system.

APPARATUS

Combined Reactor Setup	-	1 unit
100 ml standard flask	-	4 No.
500 ml standard flask	-	2 Nos.
100 ml conical flasks	-	5 Nos.
50 ml burette	-	1 No.
10 ml pipette	-	1 No.

CHEMICALS AND REAGENTS

Ethyl acetate	10 liter	0.05 N
Sodium hydroxide	10 liter	0.05 N
Oxalic acid	100 ml	0.05 N
Hydrochloric acid	100 ml	0.05 N
Phenolphthalein indicator	--	--

THEORY

For a single reaction, total size of multiple reactors connected in series is usually less than the size of single reactor. In other words, for a given size of reactor the conversion in a multiple reactor system is more than in a single reactor. But in PFR connected in series will give the same performance as single PFR. Sometimes different types of flow reactors can be connected in series. For the most effective use of a given set of ideal reactors we have the following general rules:

1. For a reaction whose rate-concentration curve rises monotonically (any n -order reaction, $n > 0$) the reactors should be connected in series. They should be ordered so as to keep the concentration of reactant as high as possible if the rate-concentration curve is concave ($n > 1$), and as low as possible if the curve is convex ($n < 1$). As an example, the ordering of units should be plug, small mixed, large mixed, for $n > 1$; the reverse order should be used when $n < 1$.
2. For reactions where the rate-concentration curve passes through a maximum or minimum the arrangement of units depends on the actual shape of curve, the conversion level desired, and the units available. No simple rules can be suggested.
3. Whatever may be the kinetics and the reactor system, an examination of the $1/(-r_A)$ vs. C_A curve is a good way to find the best arrangement of units.

5. Unreacted concentration of NaOH after reaction in PFR and MFR

$$C_{A2} = N_{HCl} - \frac{N_A \times Z}{10} \quad \text{mol/l}$$

$$C_{A2} = \underline{\hspace{2cm}} \quad \text{mol/l}$$

6. Fractional conversion of NaOH in PFR

$$X_{A1} = \frac{C_{A0} - C_{A1}}{C_{A0}} \quad \text{No unit}$$

$$X_{A1} = \underline{\hspace{2cm}} \quad \text{No unit}$$

1. Fractional conversion of NaOH in PFR & MFR

$$X_{Af} = \frac{C_{A0} - C_{A2}}{C_{A0}} \quad \text{No unit}$$

$$X_{Af} = \underline{\hspace{2cm}} \quad \text{No unit}$$

2. Fractional conversion of NaOH in MFR

$$X_{A2} = X_{Af} - X_{A1} \quad \text{No unit}$$

CALCULATION

CALCULATION

EXPERIMENTAL PROCEDURE

1. Prepare 10 l of 0.05 N NaOH solutions and transfer it to the feed tank.
2. Prepare 10 l of 0.05 mol/l ethyl acetate and transfer it to the feed tank.
3. Standardize NaOH and HCl.
4. Pipette out 10 ml of 0.05 N HCl and transfer it to 100 ml conical flask.
5. Start running NaOH at a constant flow rate and switch on the stirrer. Note down the flows rate.
6. Start running ethyl acetate at a constant flow rate and note down same. Wait until the reactor reaches steady state. (τ minutes after overflow)
7. Collect about 15ml of sample from MFR and then from PFR.
8. Pipette out 10 ml reaction mixture and transfer into the flask containing HCl. Titrate the contents with NaOH using phenolphthalein indicator.
9. Change the flow rate of NaOH and ethyl acetate alternatively and repeat the experiment.

OBSERVATION

Volume of the reactor

$$V_R = \frac{\pi}{4} \times d^2 \times L \quad \text{m}^3$$

Where

d – diameter of reactor in m

L – Length of the reactor in m

$$V_R = \underline{\hspace{2cm}} \text{ m}^3 = \underline{\hspace{2cm}} \text{ l}$$

RESULT

Performance of combined reactors (PFR followed by MFR) was studied and the conversion achieved by this multiple reactor system was calculated at different flow rates.

STANDARDIZATION OF NaOH

Take NaOH in the burette.

Pipette out 10 ml (V_A) Oxalic acid into 100 ml conical flask.

S. No.	Burette Reading (ml)		Difference = I – F (ml)	Normality of NaOH (N_A) mol/l
	Initial (I)	Final (F)		
1				
2				

$$N_A = \frac{N_{COOH_2} \times V_{COOH_2}}{V_A} \quad N$$

OBSERVATION AND CALCULATION

- Normality of HCl $N_{HCl} = \underline{\hspace{2cm}}$ N
- Normality of NaOH $N_A = \underline{\hspace{2cm}}$ N
- Total reaction time $= \underline{\hspace{2cm}}$ min
- Volumetric flow rate of NaOH $v_A = \underline{\hspace{2cm}}$ ml/min
- Volumetric flow rate of $CH_3COOC_2H_5$ $v_B = \underline{\hspace{2cm}}$ ml/min
- Initial Concentration of NaOH $C_{A0} = \frac{N_A v_A}{v_A + v_B}$ mol/l
 $C_{A0} = \underline{\hspace{2cm}}$ mol/l
- Volume of the reaction mixture $= \underline{\hspace{2cm}}$ ml
- Volume of HCl used to arrest reaction $V_0 = \underline{\hspace{2cm}}$ ml
- Volume of NaOH consumed to neutralize the mixture
 $V_1 = \underline{\hspace{2cm}}$ ml
- Volume of HCl consumed while arresting the reaction
 $= V_0 - V_1$ ml
 $= \underline{\hspace{2cm}}$ ml
- Moles of HCl consumed while arresting the reaction
 $= \frac{V_0 - V_1}{1000} N_{HCl}$ mole

Experiment No:**Date:****SEMI BATCH REACTOR****AIM**

To study the performance of semi batch reactor in which two reactants are added continuously and the product collected as a batch.

APPARATUS

Semi batch reactor setup	-	1 unit
250 ml Conical flask	-	1 No
100 ml beaker	-	1 No
50 ml burette	-	1 No
50 ml measuring cylinder	-	1 No

CHEMICALS

Ethyl acetate	10 liter	0.05 N
Sodium hydroxide	10 liter	0.05 N
Oxalic acid	100 ml	0.05 N
Hydrochloric acid	100 ml	0.05 N
Phenolphthalein indicator	--	--

THEORY

Equipment in which homogeneous reactions are effected can be one of three general types; the batch, the steady-state flow, and the unsteady-state flow or semi-batch reactor. The last classification includes all reactors that do not fall into the first two categories.

The semi-batch reactor is probably the most frequent type of reactor in the chemical industry, particularly in the fine chemical branch, in organic chemistry laboratories and in biotechnological processes.

Motivations for Using Semi-Batch Reactors:

1. Control of concentration of reactants to improve the selectivity of a reaction.
2. Addition of reactants in small increments to control the composition distribution of the product (e.g polymerization).
3. Control heat production of reaction (exothermic reactions).
4. Avoid toxicity of substrates for producing organisms or isolated enzymes.
5. Removal of product to increase conversion and selectivity.
6. Avoid accumulation of reactants prone to thermal decomposition.
7. Simulate continuous production especially for small scale.

In a surprising contrast, the semi-batch reactor is the least covered in the chemical and biochemical industry. The major reason for this discrepancy is the difficulty in getting analytical solutions of the differential equations describing such a type of

12. Moles of NaOH unreacted in the reaction

= Moles of HCl consumed

= _____ mole

13. Unreacted concentration of NaOH
in the reaction mixture at the time of
arresting the reaction

$$C_A = \frac{\text{moles of NaOH unreacted} \times 1000}{\text{Volume of reaction mixture}}$$

C_A = _____ mol/l

14. Fractional conversion of NaOH

$$X_A = \frac{C_{A0} - C_A}{C_{A0}} \quad \text{No unit}$$

CALCULATION

reactor. Additionally, in semi-batch reactors everything is usually varying, concentrations, temperature and volume.

PROCEDURE

1. Fill the overhead tanks with 0.05N of sodium hydroxide and 0.05N of ethyl acetate.
2. Feed the reactor with 0.05N of NaOH and 0.05N of ethyl acetate at a constant and equal flow rate (____lph) for a period of ____ min.
3. Arrest the reaction by adding 30ml of HCl to the reaction mixture
4. Titrate the mixture against 0.05N of NaOH and note the volume of NaOH required for neutralizing.

RESULT

The conversion of NaOH is ____% in the given semi batch reactor to which the reactants were added at a flow rate of ____ lph for ____min.

STANDARDIZATION OF NaOH

Take NaOH in the burette.

Pipette out 10 ml (V_A) Oxalic acid into 100 ml conical flask.

S. No.	Burette Reading (ml)		Difference = I – F (ml)	Normality of NaOH (N_A) mol/l
	Initial (I)	Final (F)		
1				
2				

$$N_A = \frac{N_{COOH_2} \times V_{COOH_2}}{V_A} \quad N$$

OBSERVATION TABLE

Sl. No.	Flow Rates, lph		Space Time min τ	Titer value of NaOH (y) ml
	v_A (NaOH)	v_B (EA)		
1				
2				
3				
4				
5				

CALCULATIONS**ANALYTICAL METHOD**

- Normality of $CH_3COOCH_2CH_3$ $N_B = 0.05$ N
- Normality of NaOH $N_A = \underline{\hspace{2cm}}$ N
- Initial Concentration of NaOH $C_{A0} = \frac{N_A v_A}{v_A + v_B}$ mol/l
 $C_{A0} = \underline{\hspace{2cm}}$ mol/l
- Initial Concentration of $CH_3COOCH_2CH_3$ $C_{B0} = \frac{N_B v_B}{v_A + v_B}$ mol/l
 $C_{B0} = \underline{\hspace{2cm}}$ mol/l
- Initial molar ratio of the reactants $M = \frac{C_{B0}}{C_{A0}}$ No unit

Experiment No:**Date:****KINETIC STUDIES IN A MIXED FLOW REACTOR****AIM**

To study the kinetic behavior of the given saponification reaction between ethyl acetate and sodium hydroxide in MFR and to determine second order rate constant of the reaction.

APPARATUS

Mixed Flow Reactor Setup	-	1 unit
100 ml standard flask	-	2 No.
100 ml conical flasks	-	5 Nos.
50 ml burette	-	1 No.
10 ml pipette	-	1 No.

CHEMICALS AND REAGENTS

Ethyl acetate	10 liter	0.05 N
Sodium hydroxide	10 liter	0.05 N
Oxalic acid	100 ml	0.05 N
Hydrochloric acid	100 ml	0.05 N
Phenolphthalein indicator	--	--

THEORY

The ideal steady-state mixed flow reactor is also called as backmix reactor or ideal stirred tank reactor, C^* (meaning C-star), CSTR, or the CFSTR (constant flow stirred tank reactor), and, as its names suggest, it is a reactor in which the contents are well stirred and uniform throughout. Thus, the exit stream from this reactor has the same composition as the fluid within the reactor. We refer to this type of flow as mixed flow, and the corresponding reactor the mixed flow reactor, or MFR.

The performance equation of mixed flow reactor is

$$\frac{V}{F_{A0}} = \frac{\tau}{C_{A0}} = \frac{X_A}{-r_A} = \frac{\Delta X_A}{-r_A} \quad \text{any } \varepsilon_A$$

For constant density systems

$$\frac{V}{F_{A0}} = \frac{\tau}{C_{A0}} = \frac{C_{A0} - C_A}{C_{A0}(-r_A)} \quad \varepsilon_A = 0$$

EXPERIMENTAL PROCEDURE

1. Prepare each 100 ml of 0.05 N HCl and 0.05 N Oxalic acid respectively.
2. Prepare 10 l of 0.05 N NaOH solution and transfer it to the feed tank.
3. Prepare 10 l of 0.05 mol/l ethyl acetate and transfer it to the feed tank.
4. Standardize NaOH and HCl.
5. Pipette out 10 ml of 0.05 N HCl and transfer it to 100 ml conical flask.
6. Start running NaOH at a constant flow rate and switch on the stirrer. Note down the flows rate.

6. Space time $\tau = \frac{V_R}{v_A + v_B}$ min

7. Unreacted concentration of NaOH at any time

$$C_A = N_{\text{HCl}} - \frac{N_A \times y}{10} \quad \text{mol/l}$$

$$C_A = \underline{\hspace{2cm}} \quad \text{mol/l}$$

8. Fractional conversion of NaOH

$$X_A = \frac{C_{A0} - C_A}{C_{A0}} \quad \text{No unit}$$

$$X_A = \underline{\hspace{2cm}} \quad \text{No unit}$$

9. Concentration of EA at any time

$$C_B = C_{B0} - C_{A0}X_A \quad \text{mol/l}$$

$$C_B = \underline{\hspace{2cm}} \quad \text{mol/l}$$

10. Performance equation of MFR for bimolecular irreversible second order reaction is

$$\frac{\tau}{C_{A0}} = \frac{X_A}{k C_A C_B}$$

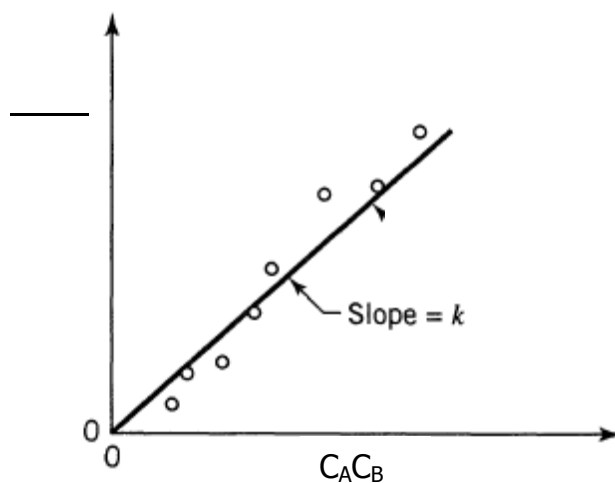
Rearrange the above equation straight line form

$$\frac{C_{A0} X_A}{\tau} = k C_A C_B$$

CALCULATION TABLE

S. No	C_{A0} mol/l	C_{B0} mol/l	M No unit	C_A mol/l	C_B mol/l	X_A No unit	$C_A C_B$	$\frac{C_{A0} X_A}{\tau}$
1								
2								
3								
4								
5								

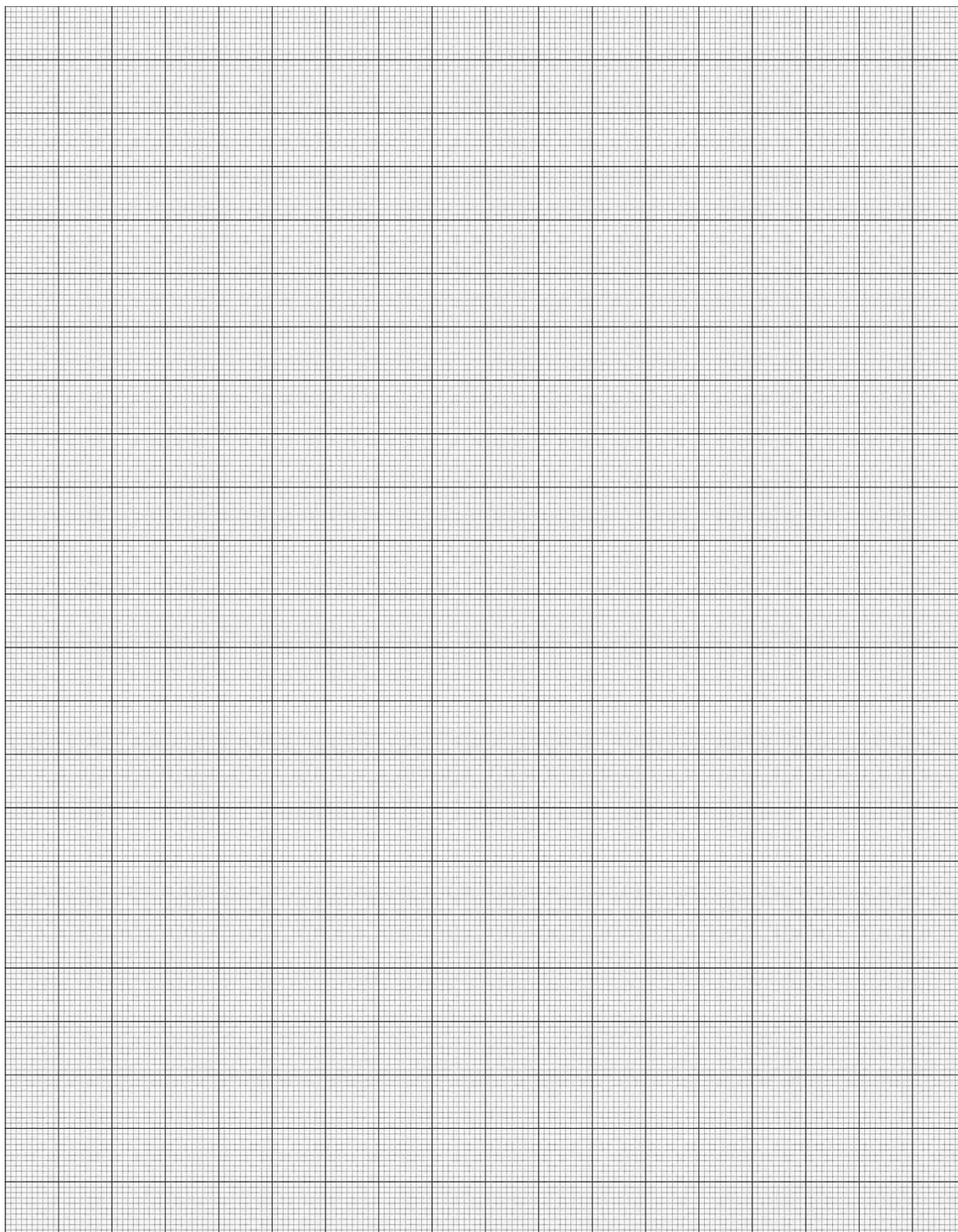
11. Plot a graph of $\frac{C_{A0} X_A}{\tau}$ versus $C_A C_B$ and determine the slope k



Scale

In x-axis 1cm =

In y-axis 1 cm =



CALCULATION

CALCULATION

CALCULATION

7. Start running ethyl acetate at a constant flow rate and note down same. Wait until the reactor reaches steady state (τ min after overflow)
8. After a reaction time of 2.5 min collect about 15ml of sample.
9. Pipette out 10 ml reaction mixture and transfer into the flask containing HCl. Titrate the contents with NaOH using phenolphthalein indicator.
10. Change the flow rate of NaOH and ethyl acetate alternatively and repeat the experiment.

OBSERVATION

Volume of the reactor $V_R = \frac{\pi}{4} \times d^2 \times L$ m^3

Where

d – diameter of reactor in m

L – Length of the reactor in m

$$V_R = \text{_____ } m^3 = \text{_____ } l$$

RESULT

The kinetic behavior of the given saponification reaction between ethyl acetate and sodium hydroxide in MFR was studied and second order rate constant of the reaction was found to be _____ $l/mol.min$.

STANDARDIZATION OF NaOH

Take NaOH in the burette.

Pipette out 10 ml (V_A) Oxalic acid into 100 ml conical flask.

S. No.	Burette Reading (ml)		Difference = I – F (ml)	Normality of NaOH (N_A) mol/l
	Initial (I)	Final (F)		
1				
2				

$$N_A = \frac{N_{COOH_2} \times V_{COOH_2}}{V_A} \quad N$$

OBSERVATION TABLE

Sl. No.	Flow Rates, lph		Space Time min	Titer value of NaOH (y) ml
	v_A (NaOH)	v_B (EA)	τ	
1				
2				
3				
4				
5				

CALCULATIONS**ANALYTICAL METHOD**

- Normality of $CH_3COOCH_2CH_3$ $N_B = 0.05$ N
- Normality of NaOH $N_A = \underline{\hspace{2cm}}$ N
- Initial Concentration of NaOH $C_{A0} = \frac{N_A v_A}{v_A + v_B}$ mol/l
 $C_{A0} = \underline{\hspace{2cm}}$ mol/l
- Initial Concentration of $CH_3COOCH_2CH_3$ $C_{B0} = \frac{N_B v_B}{v_A + v_B}$ mol/l
 $C_{B0} = \underline{\hspace{2cm}}$ mol/l
- Initial molar ratio of the reactants $M = \frac{C_{B0}}{C_{A0}}$ No unit

Experiment No:**Date:****KINETIC STUDIES IN A PLUG FLOW REACTOR****AIM**

To study the kinetic behavior of the given saponification reaction between ethyl acetate and sodium hydroxide in PFR and to determine second order rate constant of the reaction.

APPARATUS

Plug Flow Reactor Setup	-	1 unit
100 ml standard flask	-	2 No.
100 ml conical flasks	-	5 Nos.
50 ml burette	-	1 No.
10 ml pipette	-	1 No.

CHEMICALS AND REAGENTS

Ethyl acetate	10 liter	0.05 N
Sodium hydroxide	10 liter	0.05 N
Oxalic acid	100 ml	0.05 N
Hydrochloric acid	100 ml	0.05 N
Phenolphthalein indicator	--	--

THEORY

The first of the two ideal steady-state flow reactors is variously known as the plug flow, slug flow, piston flow, ideal tubular, and unmixed flow reactor. It can be referred as the plug flow reactor, or PFR, and to this pattern of flow as plug flow. It is characterized by the fact that the flow of fluid through the reactor is orderly with no element of fluid overtaking or mixing with any other element ahead or behind. Actually, there may be lateral mixing of fluid in a plug flow reactor; however, there must be no mixing or diffusion along the flow path. The necessary and sufficient condition for plug flow is for the residence time in the reactor to be the same for all elements of fluid.

The performance equation of plug flow reactor is

$$\frac{V}{F_{A0}} = \frac{\tau}{C_{A0}} = \int_0^{X_{Af}} \frac{dX_A}{-r_A} \quad \text{any } \varepsilon_A$$

For constant density systems

$$\tau = - \frac{C_A}{C_{A0}} \frac{dC_A}{-r_A} \quad \varepsilon_A = 0$$

EXPERIMENTAL PROCEDURE

1. Prepare each 100 ml of 0.05 N HCl and 0.05 N Oxalic acid respectively.
2. Prepare 10 l of 0.05 N NaOH solution and transfer it to the feed tank.
3. Prepare 10 l of 0.05 mol/l ethyl acetate and transfer it to the feed tank.
4. Standardize NaOH and HCl.
5. Pipette out 10 ml of 0.05 N HCl and transfer it to 100 ml conical flask.
6. Start running NaOH at a constant flow rate and note down the flows rate.

6. Space time $\tau = \frac{V_R}{v_A + v_B}$ min

7. Unreacted concentration of NaOH at any time

$$C_A = N_{\text{HCl}} - \frac{N_{\text{AY}}}{10} \quad \text{mol/l}$$

$$C_A = \underline{\hspace{2cm}} \quad \text{mol/l}$$

8. Fractional conversion of NaOH

$$X_A = \frac{C_{A0} - C_A}{C_{A0}} \quad \text{No unit}$$

$$X_A = \underline{\hspace{2cm}} \quad \text{No unit}$$

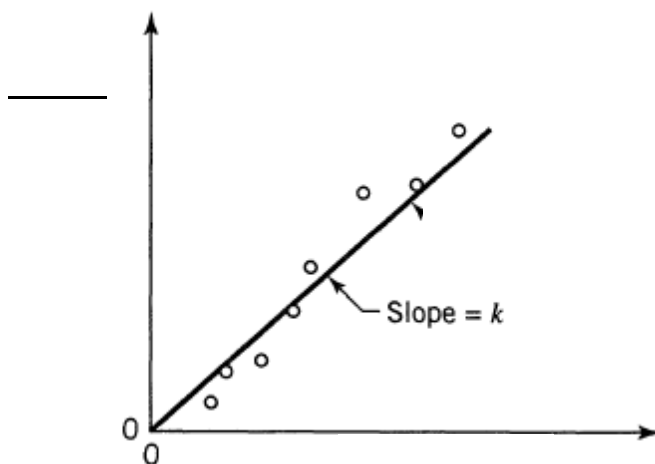
9. Performance equation of PFR for bimolecular irreversible second order reaction is

$$\ln \frac{M - X_A}{M(1 - X_A)} = k C_{A0} M - 1 \tau$$

CALCULATION TABLE

S. No	C_{A0} mol/l	C_{B0} mol/l	M No unit	C_A mol/l	C_B mol/l	X_A No unit	$C_{A0} M - 1 \tau$	$\ln \frac{M - X_A}{M(1 - X_A)}$
1								
2								
3								
4								
5								

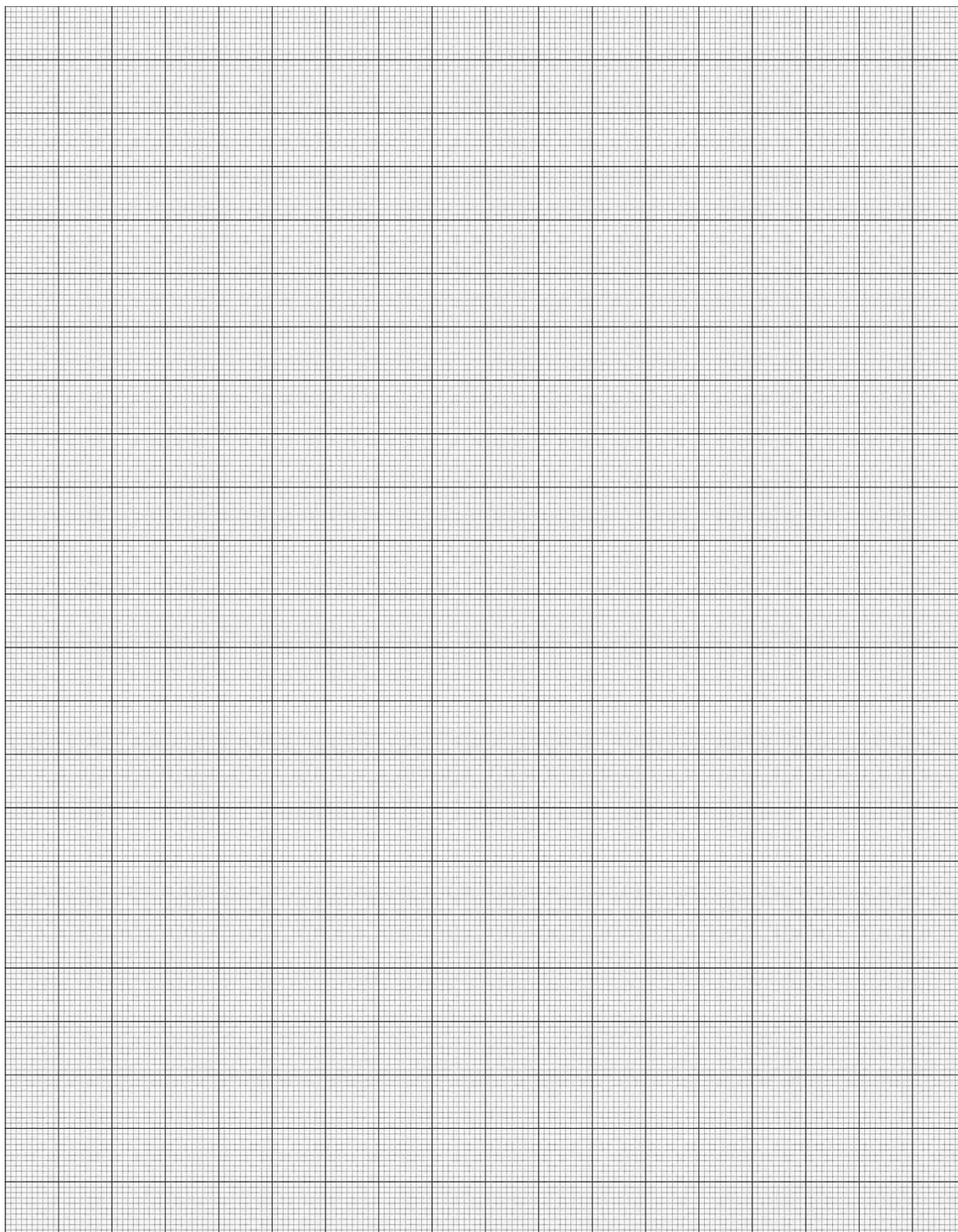
8. Plot $\ln \frac{M - X_A}{M(1 - X_A)}$ versus $C_{A0} M - 1 \tau$ graph and determine the slope (k)



Scale

In x-axis 1cm =

In y-axis 1 cm =



CALCULATION

CALCULATION

CALCULATION

7. Start running ethyl acetate at a constant flow rate and note down same. Wait until the reactor reaches steady state (τ min after overflow)
8. Collect about 15ml of sample and change the flow rates of NaOH and Ethyl acetate.
9. Pipette out 10 ml reaction mixture and transfer into the flask containing HCl. Titrate the contents with NaOH using phenolphthalein indicator.
10. Change the flow rate of NaOH and ethyl acetate alternatively and repeat the experiment.

OBSERVATION

Volume of the reactor $V_R = \frac{\pi}{4} \times d^2 \times L \quad m^3$

Where

d – diameter of reactor in m

L – Length of the reactor in m

$$V_R = \underline{\hspace{2cm}} m^3 = \underline{\hspace{2cm}} l$$

RESULT

The kinetic behavior of the given saponification reaction between ethyl acetate and sodium hydroxide in PFR was studied and second order rate constant of the reaction was found to be $\underline{\hspace{2cm}} l/mol.min$.

OBSERVATIONS & CALCULATION**INITIAL CONCENTRATION OF NaOH**

Take Oxalic acid in the burette.

Pipette out 10 ml (V_A) freshly prepared NaOH into 100 ml conical flask.

S. No.	Burette Reading (ml)		Difference = I – F (ml)	Normality of NaOH (N_A) mol/l
	Initial (I)	Final (F)		
1				
2				

$$[NaOH_i] = \frac{N_{COOH_2} \times V_{COOH_2}}{V_A} \quad N$$

FINAL CONCENTRATION OF NaOH

Take Oxalic acid in the burette.

Pipette out 10 ml (V_A) NaOH from the reactor at the end of reaction into 100 ml conical flask.

S. No.	Burette Reading (ml)		Difference = I – F (ml)	Normality of NaOH (N_A) mol/l
	Initial (I)	Final (F)		
1				
2				

$$[NaOH_f] = \frac{N_{COOH_2} \times V_{COOH_2}}{V_A} \quad N$$

Dimensions of solid

Initial Diameter

$$D_i = \text{_____ m}$$

Initial Length

$$L_i = \text{_____ m}$$

Diameter of SS rod

$$D_r = \text{_____ m}$$

Final Diameter

$$D_f = \text{_____ m}$$

Final Length

$$L_f = \text{_____ m}$$

Average Diameter

$$D_{avg} = \frac{D_i + D_f}{2} = \text{_____ m}$$

Average Length

$$L_{avg} = \frac{L_i + L_f}{2} = \text{_____ m}$$

Average Area

$$A_{avg} = \pi D_{avg} L_{avg} + \frac{\pi}{4} D_{avg}^2 - D_r^2$$

$$= \text{_____ m}$$

Experiment No:**Date:****SOLID – LIQUID NON CATALYTIC REACTOR****AIM**

To determine the specific rate of dissolution of Benzoic acid in sodium hydroxide solution with instantaneous reaction.

APPARATUS

Cylindrical mold of Benzoic acid	1 No
Dissolution set	1 unit
250 ml Conical flask	2 Nos
50 ml burette	1 No
20 ml pipette	1No

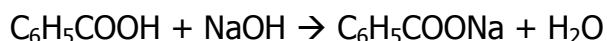
CHEMICALS AND REAGENTS

Sodium hydroxide as tracer	1000 ml	0.3 N
Oxalic acid	100 ml	0.1 N
Phenolphthalein indicator	--	--

THEORY

Solid-Liquid mass transfer plays an important role in many industrial operations. The dissolution may be accompanied by a dissolved solid-liquid or dissolved solid-gas reaction. In such cases, it is desirable to know the enhancement in the mass transfer due to instantaneous reaction.

In this study the reaction taking place is as follows,



Thus for each mole of Benzoic acid dissolved and reacted, one mole of sodium hydroxide is consumed. The dissolution rate can therefore be followed by determining the fall of NaOH concentration. The rate of dissolution of Benzoic acid is given by

$$R = \frac{\text{NaOH}_i - \text{NaOH}_f}{\text{Time}} \times \text{Volume of NaOH}$$

Where R is the rate of dissolution, $[\text{NaOH}_i]$ & $[\text{NaOH}_f]$ are initial and final sodium hydroxide concentrations respectively. The specific rate is then obtained by $R' = R/A_{\text{avg}}$ where A_{avg} is the average surface area which is given by,

$$A_{\text{av}} = \pi D_{\text{avg}} L_{\text{avg}} + \left\{ \frac{\pi}{4} (D_{\text{avg}}^2 - D_r^2) \right\}$$

Dissolution with Reaction

The rate of dissolution of Benzoic acid $R = \frac{NaOH_i - NaOH_f}{Time} \times Volume\ of\ NaOH$

Specific Dissolution rate $R' = \frac{R}{A_{avg}} = \text{_____ mol/m}^2\text{S}$

CALCULATION

PROCEDURE

- 1) Prepare the solid Benzoic acid by melting the acid and pouring the cylindrical mold with glass rod/SS rod in the center.
- 2) Measure the dimensions of the cylinder.
- 3) Prepare 1000 ml of 0.3N sodium hydroxide and 100 ml of 0.1 N oxalic acid accurately.
- 4) Standardize sodium hydroxide by titrating against standard oxalic acid using phenolphthalein as indicator.
- 5) Take 750 ml of 0.3 N sodium hydroxide solution in the beaker fix the benzoic acid mold in the setup and start the motor. Allow the dissolution to proceed for 15 minutes.
- 6) Stop the stirrer, take a sample of the solution and titrate against 0.1 N oxalic acid using phenolphthalein indicator to find final normality of NaOH.
- 7) Measure the final dimensions of the cylinder.

RESULT

The specific rate of dissolution of Benzoic acid in sodium hydroxide solution with instantaneous reaction was found to be _____ mol/m²S.

OBSERVATION TABLEFlow rate of the water is (v) = _____

lph

Sl. No.	Time (t) S	Titer value (y) ml
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		

Sl. No.	Time (t) S	Titer value (y) ml
11		
12		
13		
14		
15		
16		
17		
18		
19		
20		

CALCULATIONS

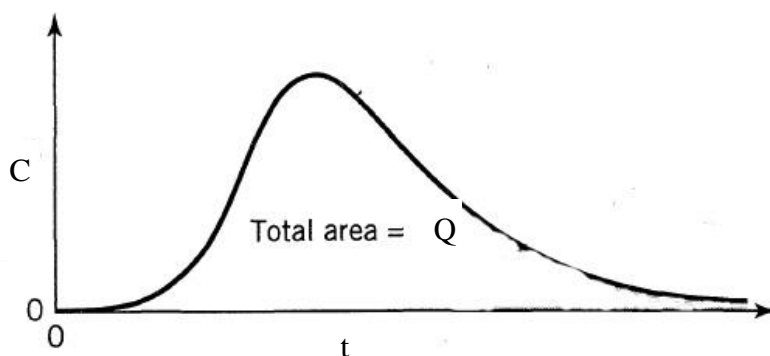
Concentration of NaOH in exit stream

$$C = \frac{N_{\text{COOH}_2} \times y}{10} \quad \text{mol/l}$$

$$C = \underline{\hspace{2cm}} \quad \text{mol/l}$$

C-Curve

Plot time in x-axis and concentration of NaOH in y-axis

From the graph, area under C-curve $Q = C \Delta t = \underline{\hspace{2cm}}$

Residence Time Distribution can be calculated as

$$E = \frac{C}{\text{Area under C-curve}} = \frac{C}{Q} \quad \text{time}^{-1}$$

Experiment No:**Date:****RTD STUDIES IN MIXED FLOW REACTOR****AIM**

To plot RTD curve and to determine the mean residence time for the given mixed flow reactor.

APPARATUS

Mixed Flow Reactor Setup	-	1 unit
100 ml standard flask	-	1 No.
50 ml standard flask	-	1 No.
100 ml conical flasks	-	8 Nos.
50 ml burette	-	1 No.
10 ml pipette	-	1 No.

CHEMICALS AND REAGENTS

Distilled Water	15 liter	--
Sodium hydroxide as tracer	50 ml	0.5 N
Oxalic acid	100 ml	0.2 N
Phenolphthalein indicator	--	--

THEORY

What is happening in the reactor depends on how exactly the reaction are mixed in it. Mixing is a process too complex to develop mathematics to describe it. To complicate it further, there is reaction going on. In order to simplify this problem, the analysis is done in two stages. First we consider only reaction with certain assumption in regard to mixing. Then we conduct on experiment to study the flow or mixing behaviour in order to quantity the mixing process and then club these two pieces of information to get to know what exactly happening in the reactor. The later experimental part is called RTD (Residence Time Distribution) studies.

During the operation of a flow reactor, we inject a traces into the feed stream to study how exactly it comes out of the reactor. We measure the concentration of the traces at the outlet at different intervals of time. This information is used to determine Mean Residence Time, later the kinetics information it clubbed with this to get actual performance of the reactor.

PROCEDURE

7. Prepare standard solution of oxalic acid and NaOH.
8. Fill the reactor and the feed tank with distilled water.
9. Start to run distilled water a t a constant flow rate (v) into the reactor. Ensure that the water flows out from the reactor doesn't contain any NaOH. Note down the flow rate.

CALCULATION TABLE

S. No	time (t) S	C mol/l	$E = C/Q$ S^{-1}	t EΔt	S. No	time (t) S	C mol/l	$E = C/Q$ S^{-1}	t EΔt
1					11				
2					12				
3					13				
4					14				
5					15				
6					16				
7					17				
8					18				
9					19				
10					20				

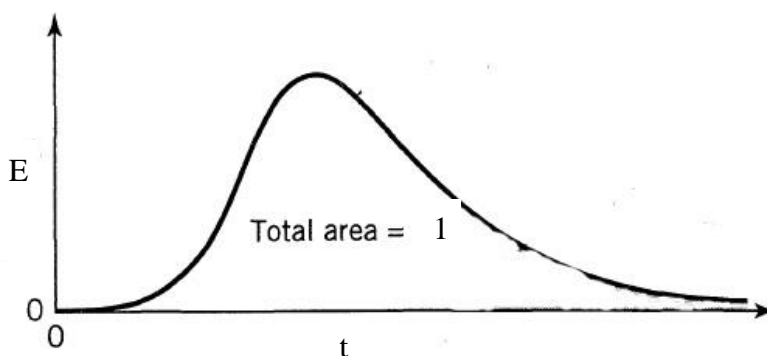
From the above table

$$E \Delta t =$$

$$tE \Delta t =$$

E-Curve

Plot time (t) Vs RTD (E)



Mean residence time

$$t = \frac{tE\Delta t}{E\Delta t}$$

S

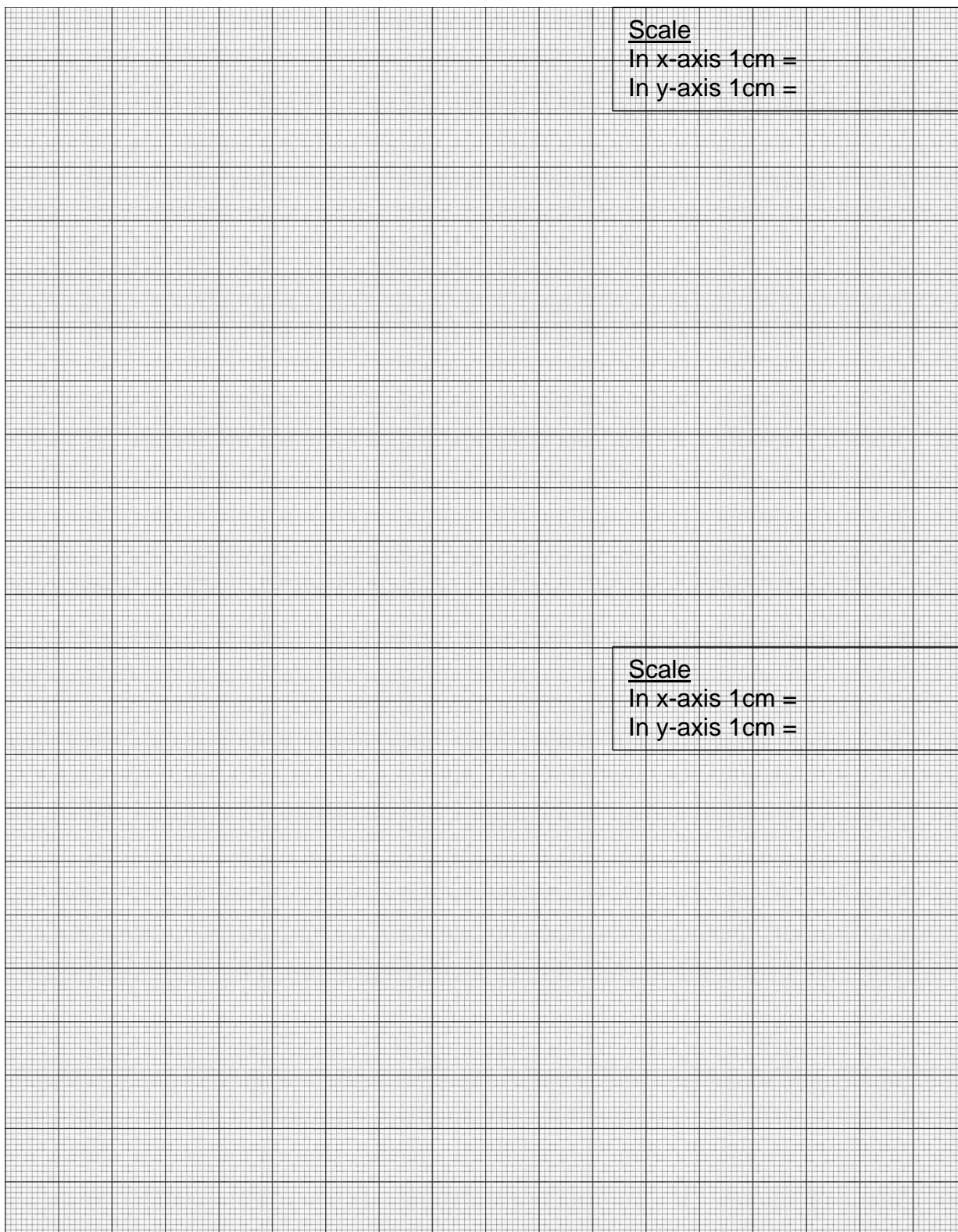
$$t = \underline{\hspace{2cm}}$$

S

Space time

$$\tau = \frac{V_R}{v}$$

S



CALCULATION

CALCULATION

CALCULATION

10. When the reactor starts over flowing inject 10 ml of the tracer near to the feed entry in the reactor, as rapidly as it is possible and start the stop watch.
11. Collect the out flowing sample from the reactor for every 30 seconds (0-30, 30-60, 60-90, and so on) in a conical flask and pipette out 10 ml of this into 100 ml conical flask and titrate with oxalic acid.
12. Continue the experiment till all the injected NaOH leaves the reactor.

OBSERVATION

Volume of the reactor $V_R = \frac{\pi}{4} \times d^2 \times L \quad m^3$

Where

d – Diameter of reactor in m

L – Length of the reactor in m

$$V_R = \text{_____ } m^3 = \text{_____ } l$$

RESULTS

Residence Time Distribution curve was plotted for the given MFR at a specified feed rate _____ lph. The mean residence time for the given plug flow reactor was found to be _____ S and space time is _____ S

POINTS TO REMEMBER

1. Specifications of Reactors

Mixed Flow Reactor		Plug Flow Reactor	
Diameter	10 cm	Diameter	1 cm
Height	13 cm	Length	4 m

In Non-catalytic reactor, Dia of SS rod $D_r = 10\text{mm}$

2. Normality = (gram equivalent of solute / liter solution)

3. Molarities = (g.mole of solute / liter solution)

4. Molality = (g.mole of solute / Kg solvent)

5. Equivalent weight = Molecular weight / valency

6. Preparing 1N solution:

- To make a 1.0 N (= 1.0 M since valency is 1) solution of NaOH in water, you will want to weigh out 40.0 grams of NaOH, dissolve it in about some liters of water, and then add water to the solution to take the total volume up to exactly 1.0 liters.
- To make a 1.0 N (not equal to 1.0 M since valency is 2) solution of Oxalic acid in water, you will want to weigh out 63.5 (=127/2) grams of Oxalic acid (oxalic acid available in hydrated condition), dissolve it in about some liters of water, and then add water to the solution to take the total volume up to exactly 1.0 liters.

S. No	Compound / Chemical	Molecular weight (g/mol)	Eq. weight
1.	Na	23.00	
2.	O	16.00	
3.	H	01.00	
4.	Ca	40.00	
5.	C	12.00	
6.	Cl	35.50	
7.	NaOH	40.00	40.00
8.	HCl	36.50	36.50
9.	Acetic Acid	60.05	60.05
10.	Oxalic acid	126.1	63.50
11.	Sodium Carbonate	106.0	53.00
12.	Ethyl Acetate	88.10	88.10