

## CHEMICAL REACTION ENGINEERING LABORATORY LAB MANUAL

### List of Experiments:-

- 1 To determine the order of reaction (n) and the reaction rate constant (k) for the given saponification reaction of ethyl acetate in aqueous sodium hydroxide solution in a Batch Reactor
- 2 To determine the order of reaction (n) and the reaction rate constant (k) for the given saponification reaction of ethyl acetate in aqueous sodium hydroxide solution in a Isothermal Batch Reactor
- 3 To study of a non-catalytic homogeneous second order liquid phase reaction in a C.S.T.R under ambient conditions. To determine the reaction rate constant for saponification of ethyl-acetate
- 4
  1. To study the performance of a cascade of three equal volume CSTRs in series for the saponification of ethyl acetate with NaOH.
  2. To draw the performance chart for the reactor system and evaluate the reaction rate constant at ambient conditions.
- 5 To determine the conversion in PFTR, for Saponification of ethyl acetate with NaOH at ambient conditions.
- 6 To determine the reaction rate constant for saponification of ethyl acetate with NaOH at a fixed temp.  
To study the effect of temp on the reaction rate constant, k and determine the activation energy, (E) for this reaction.

## EXPERIMENT 1

### AIM

To determine the order of reaction (n) and the reaction rate constant (k) for the given saponification reaction of ethyl acetate in aqueous sodium hydroxide solution.

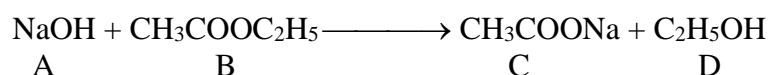
### APPARATUS

1. Batch reactor set up	2. Conical flasks (250 ml) - 6 Nos.(P) + 2 Nos.(G)
3. Stop watch	4. Burette (25 ml) - 1 No
5. Sampling pipette (10 ml) - 1 No.	6. Thermometer (0-110°C) - 1 No.

### CHEMICALS REQUIRED

- |                                  |                           |
|----------------------------------|---------------------------|
| 1. M/10 Sodium hydroxide         | 2. M/10 Ethyl acetate     |
| 3. Phenolphthalein indicator and | 4. N/40 Hydrochloric acid |

### CHEMICAL REACTION



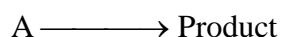
### THEORY

A batch reactor is a closed system such that no stream enters or leaves the reactor. In homogeneous reactions, all reacting species remain in a single phase. The rate of reaction of any reaction component A is defined as

$$-r_A = \frac{1}{V} \left( \frac{dN_A}{dt} \right) = \frac{\text{(moles of A which disappear by reaction)}}{\text{(unit volume) (unit time)}}$$

Rate of reaction is influenced by variables like temperature, pressure, and concentration. The rate of reaction is a function of concentration at constant temperature, i.e.  $-r_A = k C_A^n$  where n is the order of reaction. The interpretation of the kinetic data is a trial and error procedure. A kinetic model is first selected with a corresponding rate equation, and the concentration-time relationship predicted by the model is matched with experimental data.

For a first order unidirectional reaction



Rate of reaction can also be expressed as moles of A disappearing per unit volume per unit time.

$$-r_A = \frac{1}{V} \frac{dN_A}{dt} = \frac{dC_A}{dt} \dots\dots\dots (1)$$

Where,  $C_A$  = concentration of A

$$-r_A = k C_A \dots\dots\dots (2)$$

Combining equations (1) and (2) integration yields

$$\ln C_A = \ln C_{AO} - kt$$

$$\text{or, } C_A = C_{AO} e^{-kt}$$

$$-\ln \frac{C_A}{C_{AO}} = kt$$

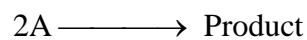
Where,

$C_{AO}$  = Conc. of A at  $t = 0$

$C_A$  = Conc. of A at  $t = t$

If we plot  $-\ln \frac{C_A}{C_{AO}}$  vs  $t$  we must get straight line if the reaction is a first order reaction.

For second order unidirectional reaction



$$\frac{dC_A}{dt} = k C_A^2$$

Integration yields

$$\frac{1}{C_A} = kt + \frac{1}{C_{AO}}$$

If we plot  $1/C_A$  vs.  $t$ , we must get a straight line for a second order reaction with slope =  $k$  and intercept on y-axis =  $1/C_{AO}$ .

The reaction rate constant is a strong function of reaction temperature. The temperature dependence of k is expressed in terms of Arrhenius equation:

$$k = A \exp (-E / RT)$$

Where,

A = frequency factor or Arrhenius constant

E = activation energy

R = ideal gas law constant

This equation can be written in the form:

$$\ln k = \frac{-E}{R} \left[ \frac{1}{T} \right] + \ln k_0$$

Thus, E / R may be evaluated from the slope of a plot of ln k vs. (1/T).

R = 8.314 J/g mole K = 1.98 cal/g mole K

E = J/g mole, cal /g mole

k is known at four temps. : T<sub>1</sub>, T<sub>2</sub>, T<sub>3</sub>, and T<sub>4</sub>

### EXPERIMENTAL SET UP DATA

The experimental set up consists of a batch reactor, constant temperature water bath and stirrer etc.

Volume of reactor	-	1000 ml
Reactor stirrer speed range	-	0 - 75 RPM approximate
Water bath stirrer speed range	-	0 - 150 RPM approximate
Temp. Controller range	-	Ambient to 200 °C with 1 °C resolution.
Control panel	-	Mains ON-OFF switch, Indicator etc, heating coil (SS) provided in reactor, reactor is provided with removable baffles.

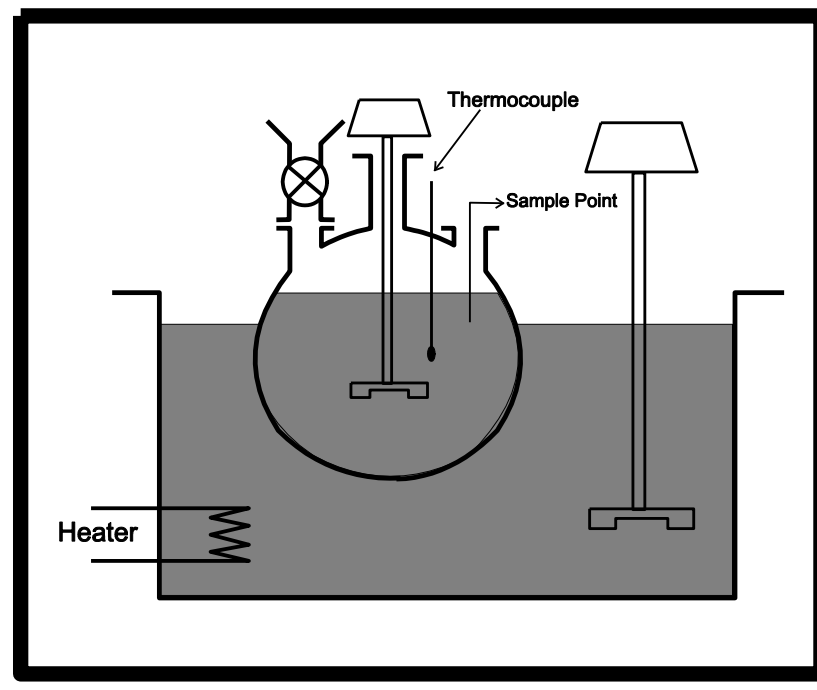


Figure: Batch reactor set up (no water bath)

## PROCEDURE

- Draw a neat schematic diagram of the experimental set up
- Prepare 5 L solution of M/100 ethyl acetate and 5 L solution of M/100 NaOH.
- Prepare a solution of N/40 HCl and N/40 NaOH solutions for the titration.
- Use phenolphthalein as indicator.
- Take 6 Nos. of 250 ml conical flasks and put 20 ml of N/40 HCl in each flask.
- Take N/40 NaOH solution in the burette.
- Adjust the temperature of water bath at 35 °C.
- Take 400 ml each of M/100 CH<sub>3</sub>COOC<sub>2</sub>H<sub>5</sub> and M/100 NaOH in two separate flasks and keep these in the water bath for about 15 min to preheat the solutions.
- Transfer these solutions quickly in the batch reactor. Start immediately the mixer and the stop watch.
- At regular intervals of 3-5 min., withdraw 10 ml of reaction mixture and put it in the conical flask containing N/40 HCl. Take at least 6 samples at regular interval of time.
- Titrate the excess N/40 HCl in each flask using N/40 NaOH from burette and phenolphthalein as indicator.
- Record the reaction temperature.
- In order to study the effect of temp, on k, the above steps may be repeated at temp. = 40 °C and 45 °C. All the reactants must be pre-heated in the water bath before mixing in the reactor.

**OBSERVATIONS AND CALCULATIONS**

**REACTION:**



Reaction temp = °C = K  
 Conc. of NaOH (A) = M/100  
 Conc. of Ethyl acetate (B) = M/100  
 Vol. of Ethyl acetate taken = 400 ml  
 Vol. of Sodium hydroxide = 400 ml  
 Initial Conc. of NaOH (A) in the mixture,  $C_{AO}$  = 0.005 M (g mole/L)  
 Initial Conc. of Ethyl acetate (B) in the mixture,  $C_{BO}$  = 0.005 M (g mole/L)

**Table 1**

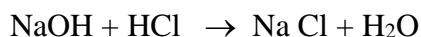
Sl.No	Sampling time, min.	Vol. of sample taken, ml	Vol. of N/40 HCl added to sample, ml	Aliquot of sample taken for titration, ml	Vol. of N/40 NaOH consumed in titration, ml

**Estimation of un-reached NaOH in the reaction mixture ( $C_A$ )**

Volume of N/40 HCl taken in conical flask = 20 ml  
 Volume of reaction mixture sample added = 10 ml  
 Volume of N/40 NaOH used in titration for neutralization of excess N/40 HCl,  
 $V_{NaOH}$  = ml

∴ No. of moles of N/40 NaOH used =  $V_{NaOH} \times 1/40 \times 10^{-3} = 2.5 \times 10^{-5} \times V_{NaOH}$  g moles  
 No. of moles N/40 HCl present initially in conical flask =  $20 \times 1/40 \times 10^{-3}$  g moles =  $5 \times 10^{-4}$  g moles.

Reaction between NaOH and HCl is:



i.e., 1 mole of NaOH reacts with 1 mole of HCl

∴ No. of moles of HCl reacted with excess moles of NaOH in the reaction mixture  
 =  $(5 \times 10^{-4} - 2.5 \times 10^{-5} \times V_{NaOH})$  g moles

∴ No. of moles of NaOH unreacted in the reaction mixture =  $(5 \times 10^{-4} - 2.5 \times 10^{-5} \times V_{NaOH})$  g

moles

Volume of sample collected = 10 ml

∴ Concentration of unreacted NaOH is

$$C_A = \frac{(5 \times 10^{-4} - 2.5 \times 10^{-5} \times V_{\text{NaOH}})}{10 \times 10^{-3}} \text{ g mole /L}$$

**Table 2**

S. No.	Time, t, m	Conc. of unreacted NaOH in the reaction mixture, C <sub>A</sub> (g mole/L)

Plot the curves for 1st order and 2nd order reaction and find out the order of reaction and rate constant.

Tabulate k values at 3 or 4 different temperatures.

**Table 3**

S. No.	Reaction rate constant (k)	Reaction temperature, (T) K	(1/T) K <sup>-1</sup>
1			
2			
3			
4			

Plot k vs. (1/t) on a semi log graph

Where,

$$\text{Slope} = \frac{-E}{R} \quad \text{and Intercept on y-axis} = k_0$$

Express,  $k = k_0 e^{-E/RT}$

### PRECAUTIONS

1. All solutions should be prepared accurately and must be standardized.
2. Titrations should be carried out precisely.
3. The individual reactants must be first pre-heated to the reaction temperature.
4. While taking sample, care should be taken that tip of the pipette does not touch the agitator blade.



5. For setting of reaction temperature, initially the temperature should be set at around 6 °C less than the desired temperature to avoid over shooting. Before starting the flow rate of ethyl acetate, temperature should be set at desired temperature.
6. All the glass wares to be used should be properly rinsed prior to use.

## **CONCLUSIONS**

Write down the points you have concluded from the experiment.

## **REFERENCE**

Levenspiel, O,"Chemical Reaction Engineering," 2nd edition, John Wiley & Sons, Singapore, 1995, pp. 98-99

## EXPERIMENT 2

### AIM

To determine the order of reaction (n) and the reaction rate constant (k) for the given saponification reaction of ethyl acetate in aqueous sodium hydroxide solution in an isothermal batch reactor.

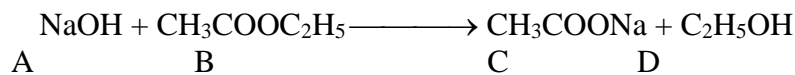
### APPARATUS

1. Batch reactor set up	2. Conical flasks (250 ml) - 6 Nos.(P) + 2 Nos.(G)
3. Stop watch	4. Burette (25 ml) - 1 No
5. Sampling pipette (10 ml) - 1 No.	6. Thermometer (0-110°C) - 1 No.

### CHEMICALS REQUIRED

- |                                  |                           |
|----------------------------------|---------------------------|
| 1. M/10 Sodium hydroxide         | 2. M/10 Ethyl acetate     |
| 3. Phenolphthalein indicator and | 4. N/40 Hydrochloric acid |

### CHEMICAL REACTION



### THEORY

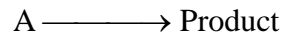
An isothermal batch reactor is a closed system such that no stream enters or leaves the reactor. In homogeneous reactions, all reacting species remain in a single phase. The rate of reaction of any reaction component A is defined as

$$-r_A = \frac{1}{V} \left( \frac{dN_A}{dt} \right) \quad \text{(moles of A which disappear by reaction)}$$

$$= \frac{\text{-----}}{\text{(unit volume) (unit time)}}$$

Rate of reaction is influenced by variables like temperature, pressure, and concentration. The rate of reaction is a function of concentration at constant temperature, i.e.  $-r_A = k C_A^n$  where n is the order of reaction. The interpretation of the kinetic data is a trial and error procedure. A kinetic model is first selected with a corresponding rate equation, and the concentration-time relationship predicted by the model is matched with experimental data.

For a first order unidirectional reaction



Rate of reaction can also be expressed as moles of A disappearing per unit volume per unit time.

$$-r_A = \frac{1}{V} \frac{d N_A}{dt} = \frac{d C_A}{dt} \dots\dots\dots (1)$$

Where,  $C_A$  = concentration of A

$$-r_A = k C_A \dots\dots\dots (2)$$

Combining equations (1) and (2) integration yields

$$\ln C_A = \ln C_{AO} - kt$$

$$\text{or, } C_A = C_{AO} e^{-kt}$$

$$-\ln \frac{C_A}{C_{AO}} = kt$$

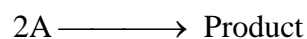
Where,

$$C_{AO} = \text{Conc. of A at } t = 0$$

$$C_A = \text{Conc. of A at } t = t$$

If we plot  $-\ln C_A$  vs.  $t$  we must get straight line if the reaction is a first order reaction.

For second order unidirectional reaction



$$\frac{d C_A}{dt} = k C_A^2$$

Integration yields

$$\frac{1}{C_A} = kt + \frac{1}{C_{AO}}$$

If we plot  $1/C_A$  vs.  $t$ , we must get a straight line for a second order reaction with slope =  $k$  and intercept on y-axis =  $1/C_{AO}$ .

The reaction rate constant is a strong function of reaction temperature. The temperature dependence of  $k$  is expressed in terms of Arrhenius equation:

$$k = A \exp(-E/RT)$$

Where,

A = frequency factor or Arrhenius constant

E = activation energy

R = ideal gas law constant

This equation can be written in the form:

$$\ln k = \frac{-E}{R} \left[ \frac{1}{T} \right] + \ln k_0$$

Thus,  $E/R$  may be evaluated from the slope of a plot of  $\ln k$  vs.  $(1/T)$ .

$R = 8.314 \text{ J/g mole K} = 1.98 \text{ cal/g mole K}$

$E = \text{J/g mole, cal/g mole}$

$k$  is known at four temps. :  $T_1, T_2, T_3,$  and  $T_4$

## EXPERIMENTAL SET UP DATA

The experimental set up consists of a batch reactor, constant temperature water bath and stirrer etc.

Volume of reactor	-	1000 ml
Volume of water Bath	-	13.5 L approximate
Reactor stirrer speed range	-	0 - 75 RPM approximate
Water bath stirrer speed range	-	0 - 150 RPM approximate
Temp. Controller range	-	Ambient to 200 °C with 1 °C resolution.
Control panel	-	Mains ON-OFF switch, Indicator etc

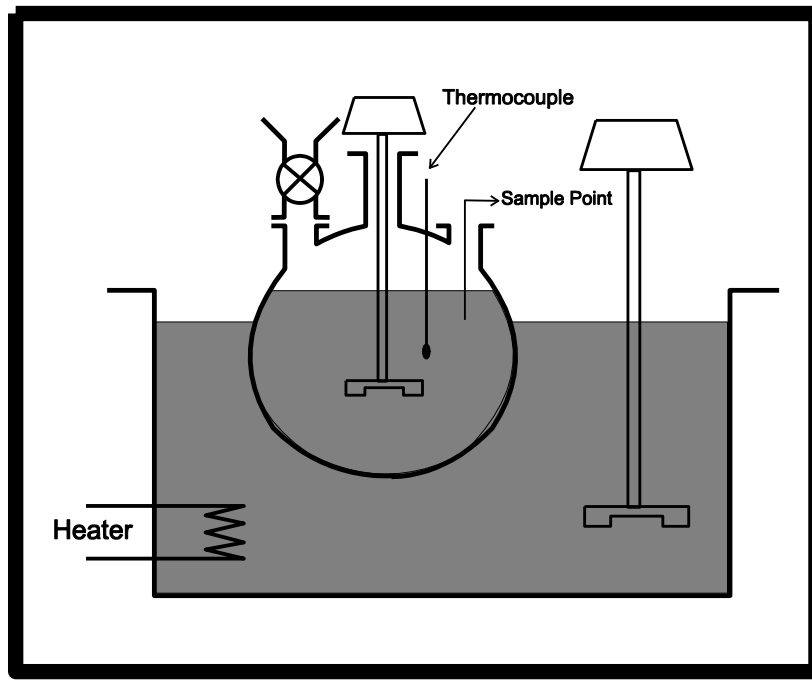


Figure: Isothermal Batch reactor set up.

## PROCEDURE

- Draw a neat schematic diagram of the experimental set up
- Prepare 5 L solution of M/10 ethyl acetate and 5 L solution of M/10NaOH.
- Prepare a solution of N/40 HCl and N/40 NaOH solutions for the titration.
- Use phenolphthalein as indicator.
- Take 6 Nos. of 250 ml conical flasks and put 20 ml of N/40 HCl in each flask.
- Take N/40 NaOH solution in the burette.
- Adjust the temperature of water bath at 35 °C.
- Take 400 ml each of M/10 CH<sub>3</sub>COOC<sub>2</sub>H<sub>5</sub> and M/10 NaOH in two separate flasks and keep these in the water bath for about 15 min to preheat the solutions.
- Transfer these solutions quickly in the batch reactor. Start immediately the mixer and the stop watch.
- At regular intervals of 3-5 min., withdraw 10 ml of reaction mixture and put it in the conical flask containing N/40 HCl. Take at least 6 samples at regular interval of time.
- Titrate the excess N/40 HCl in each flask using N/40 NaOH from burette and phenolphthalein as indicator.
- Record the reaction temperature.
- In order to study the effect of temp, on k, the above steps may be repeated at temp. = 40 °C and 45 °C. All the reactants must be pre-heated in the water bath before mixing in the reactor.

**OBSERVATIONS AND CALCULATIONS**

**REACTION:**



Reaction temp = °C = K  
 Conc. of NaOH (A) = M/10  
 Conc. of Ethyl acetate (B) = M/10  
 Vol. of Ethyl acetate taken = 400 ml  
 Vol. of Sodium hydroxide = 400 ml  
 Initial Conc. of NaOH (A) in the mixture,  $C_{AO}$  = 0.005 M (g mole/L)  
 Initial Conc. of Ethyl acetate (B) in the mixture,  $C_{BO}$  = 0.005 M (g mole/L)

**Table 1**

S.No	Sampling time, min.	Vol. of sample taken, ml	Vol. of N/40 HCl added to sample, ml	Aliquot of sample taken for titration, ml	Vol. of N/40 NaOH consumed in titration, ml

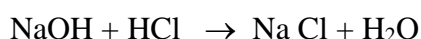
**Estimation of un-reached NaOH in the reaction mixture ( $C_A$ )**

Volume of N/40 HCl taken in conical flask = 20 ml  
 Volume of reaction mixture sample added = 10 ml  
 Volume of N/40 NaOH used in titration for neutralization of excess N/40 HCl,  
 $V_{NaOH}$  = ml

∴ No. of moles of N/40 NaOH used =  $V_{NaOH} \times 1/40 \times 10^{-3} = 2.5 \times 10^{-5} \times V_{NaOH}$  g moles

No. of moles N/40 HCl present initially in conical flask =  $20 \times 1/40 \times 10^{-3}$  g moles =  $5 \times 10^{-4}$  g moles.

Reaction between NaOH and HCl is:



i.e., 1 mole of NaOH reacts with 1 mole of HCl

∴ No. of moles of HCl reacted with excess moles of NaOH in the reaction mixture  
 =  $(5 \times 10^{-4} - 2.5 \times 10^{-5} \times V_{NaOH})$  g moles

∴ No. of moles of NaOH unreacted in the reaction mixture =  $(5 \times 10^{-4} - 2.5 \times 10^{-5} \times V_{NaOH})$  g

moles

Volume of sample collected = 10 ml

∴ Concentration of unreacted NaOH is

$$C_A = \frac{(5 \times 10^{-4} - 2.5 \times 10^{-5} \times V_{\text{NaOH}})}{10 \times 10^{-3}} \text{ g mole /L}$$

**Table 2**

S. No.	Time, t, m	Conc. of unreacted NaOH in the reaction mixture, C <sub>A</sub> (g mole/L)

Plot the curves for 1st order and 2nd order reaction and find out the order of reaction and rate constant.

Tabulate k values at 3 or 4 different temperatures.

**Table 3**

S. No.	Reaction rate constant (k)	Reaction temperature, (T) K	(1/T) K <sup>-1</sup>
1			
2			
3			
4			

Plot k vs. (1/t) on a semi log graph

Where,

Slope =  $\frac{-E}{R}$  and Intercept on y-axis = k<sub>0</sub>

Express,  $k = k_0 e^{-E/RT}$



### **PRECAUTIONS**

1. All solutions should be prepared accurately and must be standardized.
2. Titrations should be carried out precisely.
3. The individual reactants must be first pre-heated to the reaction temperature.
4. While taking sample, care should be taken that tip of the pipette does not touch the agitator blade.
5. For setting of reaction temperature, initially the temperature should be set at around 6 °C less than the desired temperature to avoid over shooting. Before starting the flow rate of ethyl acetate, temperature should be set at desired temperature.
6. All the glass wares to be used should be properly rinsed prior to use.

### **CONCLUSIONS**

Write down the points you have concluded from the experiment.

### **REFERENCE**

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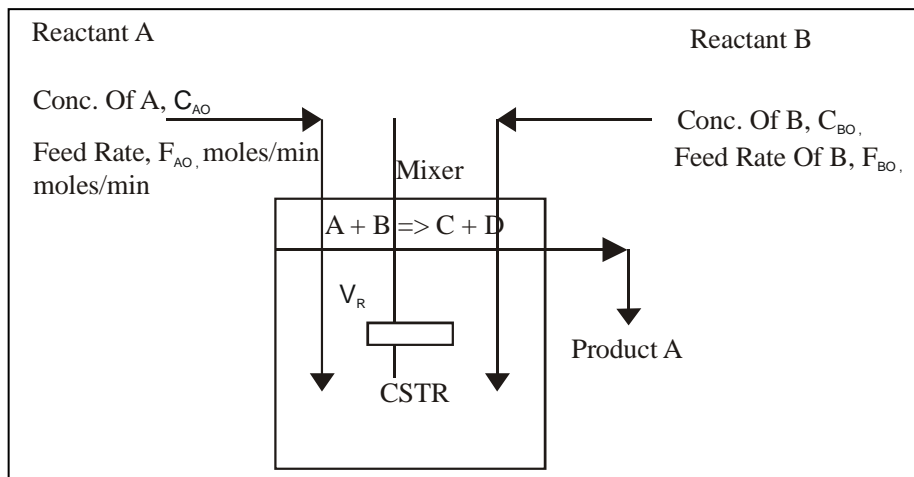
### EXPERIMENT 3

#### AIM:

To determine the reaction rate constant for saponification of ethyl-acetate with NaOH at ambient conditions.

#### THEORY:

In an ideal CSTR (that is an ideal steady state flow reactor) the contents in the reactor are well mixed and have uniform composition throughout. Thus the exit stream has the same composition as the fluid within the reactor. This type of reactor is also known as MIXED FLOW REACTOR.



Reactor volume (volume of fluid in the reactor) =  $V_R$ , Liters,

Volumetric feed rate (volumetric feed rate of A + volumetric feed rate of B) =  $V_0$ , LPM

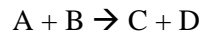
Space time =  $\tau = V_R / V_0$ , min

The performance equation for the mixed flow reactor at steady state is:

$$\tau = \frac{V_R}{V_0} = \frac{C_{A0} X_A}{-r_A} = \frac{C_{A0} - C_A}{-r_A}$$

$X_A$  &  $r_A$  are evaluated at exit stream conditions, which are the same as the conditions within the reactor.

For a second order reaction:



With  $C_{A0} = C_{B0}$  and negligible change in density of reaction mixture

The reaction rate (rate of disappearance of A) =  $-r_A = k C_A^2$

Hence,

$$\tau = \frac{V_R}{V_o} = \frac{C_{A0} X_A}{-r_A} = \frac{C_{A0} - C_A}{-r_A} = \frac{C_{A0} - C_A}{k C_A^2}$$

Degree of conversion,  $X_A = (C_{A0} - C_A) / C_{A0}$

and rate of reaction,  $-r_A = C_{A0} X_A / \tau$ , gmol/in

the rate constant,  $k = (C_{A0} - C_A) / (\tau C_A^2)$ , l/gmol-min

for  $n^{\text{th}}$  order reaction,  $-r_A = k C_A^n$

$$\log (-r_A) = \log k + n \log C_A$$

The order of the reaction,  $n$ , can be obtained from a plot of  $\log (-r_A)$  vs  $\log C_A$ , that yields a straight line with slope =  $n$ , and intercept (at  $C_A = 1$  or at  $\log C_A = 0$ ) shall give the value of  $\log(k)$ .

Also, a plot of  $\tau$  vs  $X_A / (1 - X_A)^2$  shall yield a straight line for an assumed second order reaction with slope =  $1 / (k C_{A0})$ . From this slope rate constant,  $k$  can be obtained.

**DESCRIPTION:**

The reactor is made up of SS -304. The air compressed feed reactants are given through Rotameter. The concentration of feed and the products coming out from the reactors are analyzed by chemical titration and check the effect and the performance of the reactor.

A Continuous stirred tank flow reactor (CSTR) required. 6 No. 50 cc graduated cylinder. No. 100 cc titration flasks, 50 cc burette and one stopwatch, conductivity Cell (optional).

**CHEMICALS:**

1. N/10 NaOH,
2. N/10 HCl,
3. N/10 Ethyl acetate (8.8 gms of ethyl acetate in 1 L of water),
4. Indicator (Phenolphthalein)

**UTILITIES REQUIRED:**

1. Compressed Air Supply at 2 Bar, 0.5 CFM.
2. Water Supply.
3. Drain.
4. Electricity Supply: 1 Phase. 220 V AC. 0.5 kW.
5. Instruments. Laboratory Glassware and Chemicals required for analysis as per the system adopted.

**SPECIFICATIONS:**

<b>Reactor</b>	:	Material Stainless Steel 304 Grade, Capacity 2 Ltrs (Approx).
<b>Stirrer</b>	:	Stainless Steel 304 Grade Impeller and shaft coupled with <b>FHP Motor</b>
<b>Feed Tank (2Nos.)</b>	:	Material Stainless Steel 304 Grade. Capacity - 20 Ltrs.
<b>Feed Circulation</b>	:	By compressed air

<b>Flow Measurement</b>	:	Rota meter 2Nos. (one each for Reactants)
<b>Piping</b>	:	Stainless Steel 304 Grade and PVC
<b>Pressure Regulator</b>	:	0-2 Kg/cm <sup>2</sup>
<b>Pressure Gauge</b>	:	Bourdon type 0-2 Kg/cm <sup>2</sup>
<b>Stop Watch</b>	:	Electronic
<b>Control Panel</b>	:	On / off switch, Mains Indicator etc.

The whole unit is assembled rigidly on a base plate and mounted on a stand.

Most of the parts are powder coated and rests are painted with auto paints.

**PROCEDURE:**

Prepare 20L of N/10 NaOH solution by dissolving 80 gm of NaOH in 20L of water.

1. Prepare 20L of N/10 ethyl acetate solution by mixing 176 gms of ethyl acetate in 20L of water.
2. Fill the respective tanks with these solutions.
3. Adjust the flow rate of the two streams so that in the feed mixture  $C_{A0} = C_{B0}$  i.e. have equal flow rates (e.g. 2 LPH each you may use the flow range: 2 LPH to 16 LPH for each stream) using calibrated rotameters.
4. Pass equimolar feed rates of ethyl acetate and NaOH into the CSTR by allowing equal volumetric feed rates of reactants in to the reactor and simultaneously start the mixer of the CSTR. Ensure constant stirring throughout the experiment.
5. After about 10 min or time equal to the residence time of the reactor (whichever is greater) i.e. when steady state is achieved, collect sample of the liquid at the out let
6. Titrate this solution against N/40 HCl (add HCl from burette).
7. Using phenolphthalein as indicator, note the volume of N/40 HCl used ( $V_{HCl}$ )
8. Note the volumetric flow rate of liquid at the end of the reactor.
9. Note the reaction temperature.
10. For calculating the conversion at equilibrium condition, collect the sample 10 ml, in an empty conical flask and allow the reaction to proceed for completion for two hours. After two hours titrate the solution with N/10 HCl using phenolphthalein as indicator. Note the volume of N/10 HCl used.
11. Repeat all the steps for four to six different flow rates of feed.

Note: report k at the reaction temperature

**STANDARD DATA**

Reaction temp. ,  $T^{\circ}C$   
 Reactor diameter,  $d = \text{-----} \text{ cm}$   
 Effective height of liquid in the reactor,  $L = \text{-----} \text{ cm}$   
 Vol. of the reactor =  $V_R = (\pi / 4) * d^2 * L$ ,  $V_R = \text{-----}L$   
 Volumetric feed rate of feed,  $V_o = \text{-----} \text{ L/min}$   
 Initial Conc. Of NaOH in the feed mixture =  $C_{AO} = \text{-----} \text{ gmol/L}$   
 Initial Conc. of Ethyl acetate in the feed mixture  $C_{BO} = \text{-----} \text{ gmol/L}$   
 Residence Time =  $\tau = V_R / V_o$ , min

$X_A = [C_{AO} - C_A] / C_{AO}$

Concentration of unreacted NaOH in the reaction mixture,  $C_A$

**Estimation of unreacted NaOH in the reaction mixture ( $C_A$ ):**

Titrate the sample collected immediately with N/40 HCl using phenolphthalein as indicator.

Volume of reaction mixture taken as sample =  $V_1$

Volume of acid used to neutralize sample =  $V_{HCl}$

$N/40 \times V_{HCl} = V_1 \times \text{Normality of reaction mixture}$

Normality of reaction mixture =

$C_A = \text{.....Gmole/L}$

Run. No	$\tau$ , min residence time	$C_A$ (conc. NaOH) gmol/L	$X_A$ , degree of conversion	$k = [C_{AO} - C_A] / \tau C_A^2$ .l/gmol-min

Or plot  $\tau$  vs  $X_A / [1 - X_A]^2$  on a simple graph, the slope is =  $1 / (k C_{AO})$ : from the slope find k and report k at the reaction temp.

**PRECAUTION:**

1. Measure the exact volume of water and weigh the chemicals.
2. Always use clean water and good quality chemicals and standard solution for titration.
3. Use electronic balance for weighing of chemicals. Don't mix the droppers of different chemicals.
4. Keep close all the drain valves and vent valve should open while tilling the reactant in feed tanks.
5. Flow should not be disturbed during the experiments.
6. Handle the chemicals carefully.

**TROUBLE SHOOTING:**

1. If any type of suspended particles come in the Rota meter. Remove the Rota meter clean the tube and fit again at its place.
2. If there is any leakage; tight that part or remove that and refix that again after wrapping Teflon tape.
3. If Rota meter fluctuating more than average tight control knob of that.

(Procedure: two nuts are there lose first nut and tight the second slightly, and then first also. Both nuts are on Rota meter)

**REFERENCE:**

1. Chemical Reaction Engineering by Octave Levenspil. Chapter 2 to 5



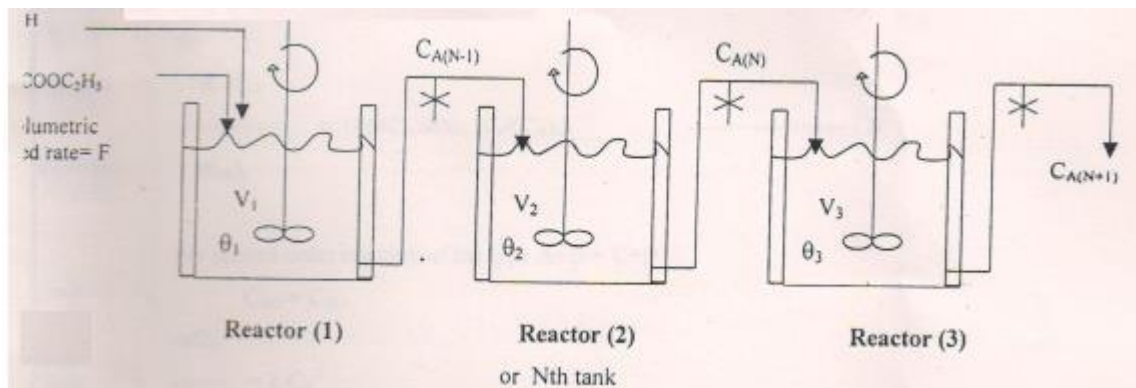
## EXPERIMENT 4

### AIM:

1. To study the performance of a cascade of three equal volume CSTRs in series for the saponification of ethyl acetate with NaOH.
2. To draw the performance chart for the reactor system and evaluate the reaction rate constant at ambient conditions.

### THEORY:

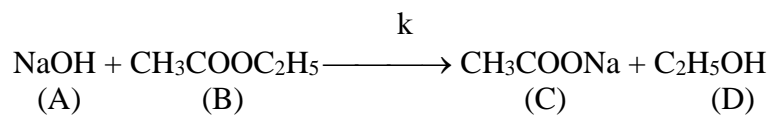
Consider a cascade of 3 CSTR's in series



In this system effluent from one reactor is feed to the next.

### REACTION:

Reaction: Saponification of ethyl acetate with NaOH.



Feed condition is such that:

$$C_{AO} = C_{BO}$$

$$-r_A = \frac{-dC_A}{dt} = k C_A^2$$

Let:

$F$  = volumetric flow rate through the reactor system (constant)

$V_n$  = Volume of reaction mixture in nth reactor.

$(C_A)_n$  = molar conc. of reactant A in the nth reactor.

$(C_A)_{n-1}$  = molar conc. of reactant A in the (n-1) th reactor.

$\theta_n$  =  $V_n/F$  = normal holding in the nth reactor.

A steady flow material balance over nth reactor is:

$$F (C_A)_{n-1} + (dC_A/dt)_n V_n = F (C_A)_n \quad \dots\dots\dots (1)$$

Or

$$\frac{(C_A)_{n-1}}{(C_A)_n} = 1 - (dC_A/dt)_n (\theta_n / (C_A)_n) \quad \dots\dots\dots (2)$$

For a second order reaction of the type  $A + B \rightleftharpoons C + D$

$$C_{AO} = C_{BO}$$

$$-r_A = \frac{-dC_A}{dt} = k C_A^2$$

And the above equation can be written as

$$\frac{(C_A)_{n-1}}{(C_A)_n} = 1 + k \theta_n (C_A)_n \quad \dots\dots\dots (3)$$

**For the conditions:**

Equal volume of tanks ( $V_1 = V_2 = V_3 \dots = V$ )

And  $\theta_1 = \theta_2 = \theta_3 \dots = \theta$

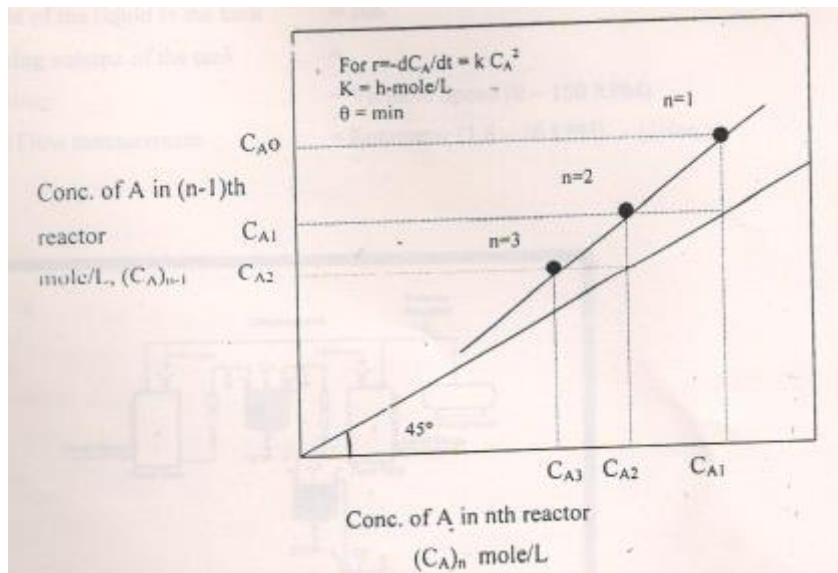
For second order reaction Eq. 2 can also be written as:

$$(C_A)_{n-1} - (C_A)_n = k \theta_n (C_A)_n^2$$

Or

$$\ln [(C_A)_{n-1} - (C_A)_n] = \ln (k \theta_n) + 2 \ln (C_A)_n \dots \dots \dots (4)$$

Performance chart of the cascade of CSTR can be plotted using Eq.3



The value of the reaction rate constant, k, can be obtained from Eq. 4

$$k = \frac{\{[(C_A)_{n-1} / (C_A)_n] - 1\}}{(\theta_n (C_A)_N)}$$

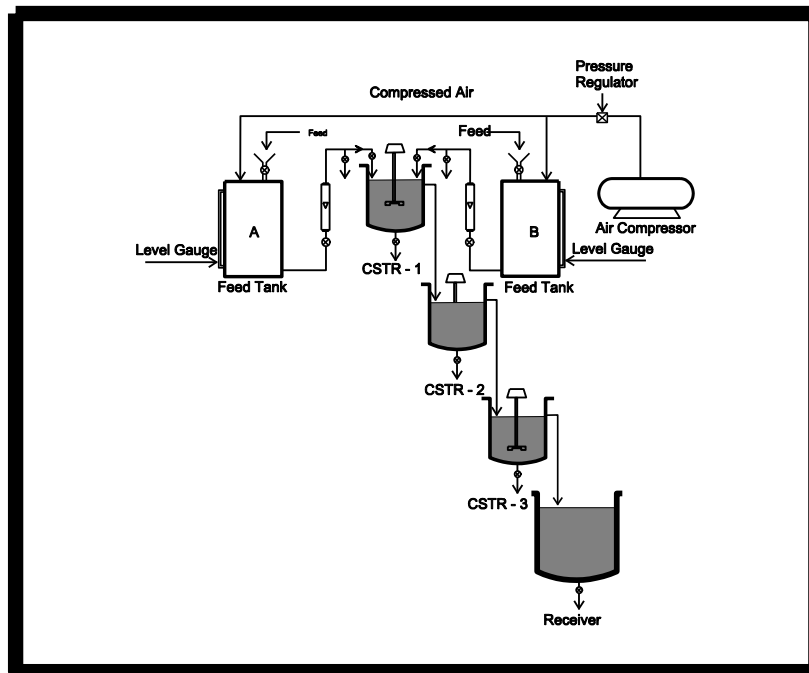
Once the performance chart is prepared, it can be used to predict the unknown intermediate composition of the reactant A for any n (No. of equal sized tanks in series).

As  $n \rightarrow \infty$ , the performance is approximated by a plug flow reactor.

### **EXPERIMENTAL SET UP:**

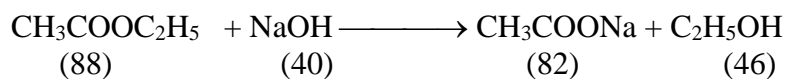
The experimental set up as shown in the figure consists of three identical stirred tanks made of stainless steel. The characteristics and dimensions of the vessel are:

Height of the tank	=	200 mm
Inside diameter of the tanks	=	100 mm
Volume of the tank	=	1.5 liters approximate
Height of the liquid in the tank	=	106 approx
Working Volume of the tank	=	0.9 liters
Agitation	=	Variable Speed
Fluid Flow measurement	=	Rota meter (2 - 20 LPH) – (2 Nos.)
Pressure regulator	=	pressure regulator 0-2 kg/cm <sup>2</sup> , pressure gauge bourdon type 0-2 kg/cm <sup>2</sup> , stop watch electronic



### PROCEDURE:

Reaction: Saponification of ethyl acetate with NaOH.



1. Prepare a solution of M/50 ethyl acetate solution in water (1.76 gm of ethyl acetate in 1L of solution).
2. Prepare a solution of M/50 NaOH in water (0.8 gm in 1L of solution).
3. Fill the feed tanks with the respective solutions.
4. Prepare a solution of M/20 HCl and M/20 NaOH for titration.
5. Use phenolphthalein as indicator.
6. Take 18 conical titration flasks and put 20ml of M/20 HCl in each.
7. Take M/20 NaOH in the burette.
8. Record the ambient temperature of reaction mixture.
9. Calibrate each rotameter with the respective liquid.
10. Fix a feed rate for inlet streams  $F_A = F_B$  (say 1 - 2 LPH).

11. Allow the two reactants streams, (NaOH (A) and CH<sub>3</sub>COOC<sub>2</sub>H<sub>5</sub> (B) to enter the first CSTR at equal feed rate so that in the reactor  $C_{AO} = C_{BO}$ . Start the mixer and wait till you get the liquid out from the 3rd CSTR. Give 5-10 min. for steady state.
12. Collect the 10 ml of sample at the outlet of I CSTR, II CSTR and III CSTR in the conical flasks containing M/20 HCl.
13. Record the volumetric feed rate of ethyl acetate and NaOH
14. Record the volume of each reactor ( $V_1=V_2=V_3$ ).
15. Change the flow rate of each stream (keep  $F_A = F_B$  so that ( $C_{AO} = C_{BO}$ ) and repeat the above steps for 3-4 flow rates.
16. Estimate the unreacted NaOH in each conical flask by titration or by measuring the conductance of the solution.
17. Record space time vs. concentration rate.

### RECORD:

Effective volume of each reactor

$$V_1 = \text{-----} \text{ L}$$

$$V_2 = \text{-----} \text{ L}$$

$$V_3 = \text{-----} \text{ L}$$

In our case  $V_1 = V_2 = V_3 = V$ , (L)

Conc. of NaOH in feed, =  $C_{AO}$  gmole/L

Conc. of CH<sub>3</sub>COOC<sub>2</sub>H<sub>5</sub> in feed,  $C_{BO}$  gmole/L

For equal flow rate of two streams ( $F_A = F_B$ )

$$C_{AO} = C_{BO} = 0.01 \text{ gmole/L}$$

If we start with M/50 solution of each:

S.No.	$V_0$ LPM	V L	$\tau = V/V_0$ min.	$C_{AO}$ gmole/L	$C_{A1}$ gmole/L	$C_{A2}$ gmole/L	$C_{A3}$ gmole/L

--	--	--	--	--	--	--	--

$C_A$  = Conc. of unreacted NaOH in the reactor for space time =  $\tau$

### ESTIMATION OF UN-REACTED NaOH IN THE REACTION MIXTURE:

Volume of M/20 HCl in beaker = 20ml

Volume of reaction mixture added = 10ml

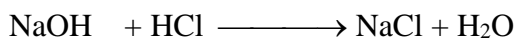
Let the volume of M/20 NaOH used for neutralization of excess HCl =  $V_{\text{NaOH}}$  (ml)

∴ Number of moles of NaOH used =  $V_{\text{NaOH}} (1/20) \times 10^{-3}$  (gmoles)

∴  $n_{\text{NaOH}} = 5 \times 10^{-5}$  gmoles

Number of moles of HCl present initially in the beaker =  $20 \times 10^{-3} \times 0.128$   
=  $1 \times 10^{-3}$  (gmoles)

Reaction between NaOH & HCl is:



∴ 1 mole of NaOH reacts with 1 mole of HCl

∴ Number of moles of HCl reacted with excess moles of NaOH in the reaction mixture  
=  $(1 \times 10^{-3} - 5 \times 10^{-5} V_{\text{NaOH}})$

∴ No. of moles of NaOH unreacted in the reaction mixture  
=  $(1 \times 10^{-3} - 5 \times 10^{-5} V_{\text{NaOH}})$

Volume of sample collected = 10ml

∴ Conc. of un-reacted NaOH is

$$C_A = \frac{(1 \times 10^{-3} - 5 \times 10^{-5} V_{\text{NaOH}})}{10 \times 10^{-3}} \text{ (gmole/L)}$$

$$\% \text{ Conversion} = \frac{C_{\text{AO}} - C_A}{C_{\text{AO}}} \times 100$$

**PRECAUTIONS:**

- Handle all chemicals carefully
- Do not wear loose clothing's in Lab
- Ensure proper ventilation in laboratory

**REFERENCES:**

- Perry's Chemical Engineering Handbook 7<sup>th</sup> Edit



## EXPERIMENT 5

### AIM:

To determine the conversion in PFTR, for Saponification of ethyl acetate with NaOH at ambient conditions.

### INTRODUCTION:

Real reactors do not satisfy the idealized flow patterns, back mix flow or plug flow deviation from ideality can be due to channeling of fluid through the vessel, recycling of fluid within the vessel or due to the presence of stagnant region or pockets of fluid in the vessel.

In an ideal plug flow reactor (PFTR) there is no mixing in the direction of flow and complete mixing perpendicular to the direction of flow. Concentration of the reactant varies along the length of the reactor but not in the radial direction.

### THEORY:

In an ideal plug flow reactor (PFTR) there is no mixing in the direction of flow and Complete mixing perpendicular to the direction of flow. Concentration of the reactant varies along the length of the reactor but not in the radial direction.  $V$  is the volume of the reactor,  $V_0$  is the volumetric feed rate,  $C_{A_0}$  is the feed conc. of A,  $x$  is the degree of conversion. In case of a Coil, turbulence is introduced due to frequent change in direction of flow and presence of secondary flow. So a higher value of  $k$  is expected in case of a coil type PFTR. For steady state rate operation of a PFTR.

### DESCRIPTION:

The set up consists of two feed tanks through which two reactants are fed to the reactor. Rotameters are provided to measure the individual flow of Chemicals. The flow rate can be adjusted by operating the needle valves provided on respective Rotameter. The compressed air is used for circulation of feed. A straight tube type reactor kept horizontally and inclined upward at outlet from where samples are collected for analysis. Pressure Regulator, Pressure Gauge and Safety Valve are fitted in the compressed air line.

### UTILITIES REQUIRED:

- Electricity Supply: Single Phase, 220 VAC, 50 Hz, 1.5 kw, 5-15 amp combined socket with earth Connection.
- Water Supply.
- Floor Drain.
- Floor Area 1.5 m x 1 m.
- Laboratory glass ware :-
  - Conical Flask (250 ml): 02 No.
  - Measuring cylinder (50 ml): 02 No.
  - Burette (50 ml) with stand: 01 No.
  - Beakers (250 ml): 02 No.
  - Funnel: 01 No.
- Chemicals Required:
  - N/10 NaOH: 20 ltrs.
  - N/10 Ethyl Acetate: 20 ltrs.
  - N/10 HCl: 0.5 ltrs.
  - Indicator (Phenolphthalein): Few drops

### EXPERIMENTAL PROCEDURE:

1. Prepare 20L of N/10 NaOH solution by dissolving 80 gm of NaOH in 20L of water.
2. Prepare 20L of N/10 ethyl acetate solution by mixing 176 gm of ethyl acetate in 20L of water.
3. Fill the respective tanks with these solutions.
4. Connect the compresses air supply.
5. Start the compressor.
6. Compress the feed tanks with the help of Pressure Regulator; adjust pressure less than 1 kg/cm<sup>2</sup>.
7. Adjust the flow rate of the two streams so that in the feed mixture  $C_{A0} = C_{B0}$  (i.e. have equal flow rates (e.g. 10 LPH each) using rotameters.
8. Pass equimolar feed rates of ethyl acetate and NaOH into the PFR by allowing equal volumetric feed rates of reactants in to the reactor.

9. After about 10 min or time equal to 1.5 times the residence time of the reactor (which ever is greater) i.e when steady state is achieved, collect exact 20ml of the sample of the liquid at the outlet, in a graduated cylinder that already contains 20 ml of (chilled) N/10 HCl. Transfer this solution (40 ml) in a 250ml or 100 mL titration flask.
10. Using phenolphthalein as indicator, titrate this solution (i.e excess HCl) against N/10 NaOH (add NaOH from burette). & note the volume of N/10 NaOH used ( $V_{NaOH}$ ).
11. Repeat the experiment for different flow rates (before changing the flow rate, drain the reactor first).

### OBSERVATION & CALCULATION:

DATA:

$V_R = \text{----- L}$   
 $R = \text{----- cal/g mole K}$   
 $C_{A0} = \text{----- g mole/L}$   
 $V_{HCl} = \text{----- ml}$

$V_{SAMP} = \text{----- ml}$

OBSERVATION:

$V_0 = \text{----- LPH}$   
 $V_{NaOH} = \text{----- ml}$

CALCULATIONS:

$$H_{CLO} = \frac{V_{HCL} N_{HCL}}{1000}, \text{ g mole} = \text{----- g mole}$$

$$H_{CLR} = H_{CLO} - \frac{(V_{NaOH} N_{NaOH})}{1000}, \text{ g mole} = \text{----- g mole}$$

$$C_A = \frac{H_{CLR}}{V_{SAMP}} \times 1000, \text{ g mole/L} = \text{----- g mole/L}$$

$$X_A = \frac{(C_{A0} - C_A)}{C_{A0}} = \text{-----}$$

$$\tau = \frac{V_R \times 60}{V_o} = \text{-----}$$

$$K = \frac{C_{Ao} - C_A}{\tau \times C_{Ao} \times C_A}, \text{ L/g mole min} = \text{-----}$$

Plot  $\tau$  vs.  $X_A / [1 - X_A]^2$  on a simple graph, the slope is =  $1 / (K C_{Ao})$ ; from the slope

Find K and report K at the room temp.

### NOMENCLATURE:

$C_A$	=	Conc. of unreacted NaOH in the reactor, g mole/L
$C_{Ao}$	=	Initial Conc. of NaOH in the feed mixture, g mole/L
HCLO	=	Amount of HCL taken for quench, g mole
HCLR	=	Amount of HCL reacted with NaOH, g mole
K	=	Rate constant, L/g mol min
$N_{HCL}$	=	Number of moles of HCL used, g mole
$N_{NaOH}$	=	Number of moles of NaOH used, g mole
R	=	Real gas constant, cal/g mol K
$V_R$	=	Reactor volume, L
$V_o$	=	Volumetric feed rate, LPH
$V_{HCl}$	=	Volume of N/10 HCl taken for quench, ml
$V_{SAMP}$	=	Volume of sample taken, ml
$V_{NaOH}$	=	Volume of NaOH used for neutralizing, ml
$X_A$	=	Degree of conversion,
$\tau$	=	Residence time, min

### PRECAUTIONS & MAINTENANCE INSTRUCTIONS:

1. Measure the exact volume of water and weigh the chemicals properly.
2. Always use clean water and good quality chemicals and standard solution for titration.
3. Use electronic balance for weighing of chemicals. Don't mix the droppers of different chemicals.
4. Keep close all the drain valves and vent valve should open while filling the reactant in feed tanks.

5. Flow should not be disturbed during the experiments.
6. Handle the chemicals carefully.

**TROUBLESHOOTING:**

1. If any type of suspended particles are come in the Rota meter. Remove the Rota meter clean the tube and fit that at its place.
2. If there is any leakage tight that part or remove that and fix that again after wrapping Teflon tape.
3. If Rota meter fluctuating more than average tight control knob of that.

**REFERENCES:**

1. Octave Levenspiel, "Chemical reaction engineering,"3rd ed., John Wiley & Sons, NY, 2001.
2. Coulson & Richardson, "Chemical Engineering Vol-3" 4<sup>th</sup> ed., Asian Books Pvt.Lt, ND, 1991.

## EXPERIMENT 6

### AIM:

To determine the conversion in PFTR, for Saponification of ethyl acetate with NaOH at a fixed temperature

To study the effect of temp. on the reaction rate constant,  $k$  and determine the activation energy,  $(E)$  for this reaction..

### INTRODUCTION:

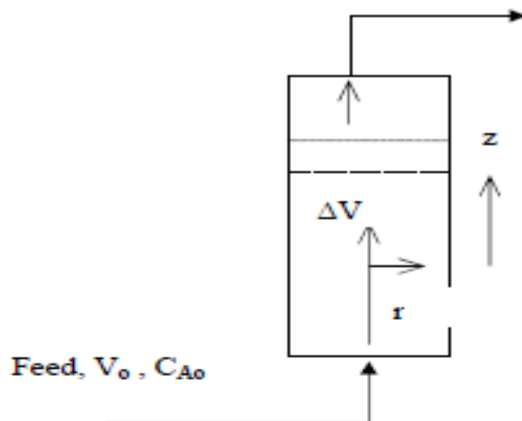
Real reactors do not satisfy the idealized flow patterns, back mix flow or plug flow deviation from ideality can be due to channeling of fluid through the vessel, recycling of fluid within the vessel or due to the presence of stagnant region or pockets of fluid in the vessel.

In an ideal plug flow reactor (PFTR) there is no mixing in the direction of flow and complete mixing perpendicular to the direction of flow. Concentration of the reactant varies along the length of the reactor but not in the radial direction.

### THEORY:-

In an ideal plug flow reactor (PFTR) there is no mixing in the direction of flow and Complete mixing perpendicular to the direction of flow. Concentration of the reactant varies along the length of the reactor but not in the radial direction.  $V$  is the volume of the reactor,  $V_0$  is the volumetric feed rate,  $C_{A0}$  is the feed conc. of A,  $x$  is the degree of conversion.

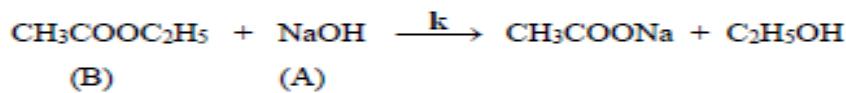
In case of a Coil, turbulence is introduced due to frequent change in direction of flow and presence of secondary flow. So a higher value of  $k$  is expected in case of a coil type PFTR. For steady state rate operation of a PFTR: In case of a Coil, turbulence is introduced due to frequent change in direction of flow and presence of secondary flow. So a higher value of  $k$  is expected in case of a coil type PFTR. For steady state rate operation of a PFTR:



In case of a Coil, turbulence is introduced due to frequent change in direction of flow and presence of secondary flow. So a higher value of  $k$  is expected in case of a coil type PFTR. For steady state rate operation of a PFTR:

$$\frac{V}{F_{A0}} = \int_0^X \frac{dX}{-r_A}$$

For the reaction



A = NaOH

B = CH<sub>3</sub>COOC<sub>2</sub>H<sub>5</sub>

$$-r_A = k C_A C_B$$

For the condition:

$C_{A0} = C_{B0}$  and neglecting change in density.

$$-r_A = k C_A^2$$

$$C_A = C_{A0} (1 - X_A)$$

$$\frac{V}{F_{A0}} = \frac{1}{k C_{A0}^2} \int_0^{X_{AF}} \frac{dX_A}{(1 - X_A)^2}$$

$F_{A0}$  = Molar flow rate of NaOH

$$F_{A0} = V_o C_{A0}$$

$V_o$  = Volumetric flow rate of feed, LPM

$V_R$  = Volume of the PFTR, L

$$\text{or } \frac{V_R}{V_o} = \frac{1}{k C_{A0}^2} \int_0^X \frac{dX_A}{(1 - X_A)^2}$$

$$\text{or } r = \frac{1}{kC_{A0}} \times \frac{X_A}{(1-X_A)}$$

When  $C_{A0} = C_{A0}$ , then,

$$r = \frac{1}{kC_{A0} \left[ \left( \frac{C_{A0}}{C_{A0}} \right) - 1 \right]} \ln \left( \frac{\left( \frac{C_{A0}}{C_{A0}} \right) - X_A}{\left( \frac{C_{A0}}{C_{A0}} \right) (1 - X_A)} \right)$$

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

$C_A$  = Concentration of unreacted NaOH at steady state.

$k$  = Reaction rate constant.

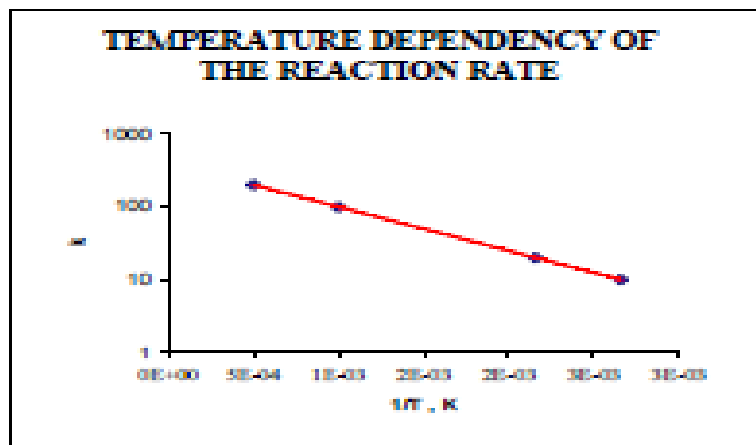
Effect of temperature on reaction rate constant is expressed in terms of Arrhenius' law:

$$k = k_0 e^{-E/RT}$$

$k_0$  is the frequency factor and  $E$  is called the activation energy of reaction.

A plot of  $k$  vs.  $1/T$  yields a straight line with slope =  $-E/R$  from this slope, Activation energy,  $E$  can be estimated.

(We can use a semi - log plot also, log axis for  $k$ , as shown below)



## DESCRIPTION:

The set up consists of two feed tanks through which two reactants are fed to the reactor. Rota meters are provided to measure the individual flow of Chemicals. The flow rate can be adjusted by operating the needle valves provided on respective Rota meter. The compressed air is used for circulation of feed. A straight tube type reactor kept horizontally and inclined upward at outlet from where samples are collected for analysis. Pressure Regulator, Pressure Gauge and Safety Valve are fitted in the compressed air line.

## UTILITIES REQUIRED:



Electricity Supply: Single Phase, 220 VAC, 50 Hz, 1.5 kw, 5-15 amp combined socket with earth Connection.

- Water Supply.
  - Floor Drain.
  - Floor Area 1.5 m x 1 m.
  - Laboratory glass ware :-
    - Conical Flask (250 ml): 02 No.
    - Measuring cylinder (50 ml): 02 No.
    - Burette (50 ml) with stand: 01 No.
    - Beakers (250 ml): 02 No.
    - Funnel: 01 No.
  - Chemicals Required:
    - N/10 NaOH: 20 ltrs.
    - N/10 Ethyl Acetate: 20 ltrs.
    - N/10 HCl: 0.5 ltrs.
- Indicator (Phenolphthalein): Few drops

#### **EXPERIMENTAL PROCEDURE:**

1. Prepare 20L of N/10 NaOH solution by dissolving 80 gm of NaOH in 20L of water.
2. Prepare 20L of N/10 ethyl acetate solution by mixing 176 gm of ethyl acetate in 20L of water.
3. Fill the respective tanks with these solutions.
4. Fill the water bath with water.
5. Set the reaction temp. Start the heater and stirrer of the bath and wait till steady state temp. in the water bath is attained. Record the reaction temperature, T.
6. Start the pumps of feed tanks to flow the feed to overhead tanks and wait till overflow starts from overhead tanks..
7. Adjust the flow rate of the two streams so that in the feed mixture  $C_{A_0} = C_{B_0}$  (i.e. have equal flow rates (e.g. 10 LPH each) using rota meters.
8. Pass equimolar feed rates of ethyl acetate and NaOH into the CSTR by allowing equal volumetric feed rates of reactants in to the reactor

9. After about 10 min or time equal to twice the residence time of the reactor (which ever is greater) i.e when steady state is achieved, collect exact 20ml of the sample of the liquid at the out let, in a graduated cylinder that already contains 20 ml of (chilled) N/10 HCl. Transfer this solution (40 ml) in a 250ml or 100 mL titration flask.
10. Using phenolphthalein as indicator, titrate this solution (i.e excess HCl) against N/10 NaOH (add NaOH from burette). & note the volume of N/10 NaOH used (  $V_{NaOH}$  )
11. Repeat all the steps for four different reaction temperatures at a single flow rate of feed.
12. Repeat all steps for different flow rates at a constant temperature.

### OBSERVATION & CALCULATION:

DATA:

$V_R$  = ----- L  
 $R$  = ----- cal/g mole K  
 $C_{A0}$  = ----- g mole/L  
 $V_{HCl}$  = ----- ml  
  
 $V_{SAMP}$  = ----- ml  
  
 $V_0$  = ----- LPH  
 $V_{NaOH}$  = ----- ml

CALCULATIONS:

$$HClO = \frac{V_{HCl} N_{HCl}}{1000}, \text{ g mole} = \text{----- g mole}$$

$$HCLR = HClO - \frac{(V_{NaOH} N_{NaOH})}{1000}, \text{ g mole} = \text{----- g mole}$$

$$C_A = \frac{HCLR}{V_{SAMP}} \times 1000, \text{ g mole/L} = \text{----- g mole/L}$$

$$X_A = \frac{(C_{A0} - C_A)}{C_{A0}} = \text{-----}$$

$$\tau = \frac{V_R + 60}{V_0} = \text{----- min}$$

$$K = \frac{C_{A0} - C_A}{\tau C_{A0} C_A}, \text{ L/g mole min} = \text{----- L/g mole min}$$

Plot  $\tau$  vs.  $X_A/[1-X_A]^2$  on a simple graph, the slope is  $= 1/(K C_{A0})$ ; from the slope find K and report K at the reaction temp.

**CALCULATION FOR ACTIVATION ENERGY (E):**

un. o	, K	1/T, K <sup>-1</sup>	$\tau$ , L/g mol min

Plot K vs 1/T on a semi-log graph. Draw a smooth line (best fit line) through all the data points and measure the slope and intercept of the straight line.

Slope =  $-\frac{E}{R} = \text{-----}$

$= \text{slope} \cdot R, \text{ cal/g mol} = \text{-----} \text{ cal/g mol}$

**9. NOMENCLATURE:**

$C_A$  = Conc. of unreacted NaOH in the reactor, g mole/L

$C_{A0}$  = Initial Conc. of NaOH in the feed mixture, g mole/L

$E$  = Activation energy, cal/g mol

$N_{HCL}$  = Amount of HCL taken for quench, g mole  
Amount of HCL reacted with NaOH, g

$N_{HCLR}$  = mole

$K$  = Rate constant, L/g mol min

$N_{HCL}$  = Number of moles of HCL used, g mole/L

$N_{NaOH}$  = Number of moles of NaOH used, g mole/L

$R$  = Real gas constant, cal/g mol K

T	=	Reaction temperature, K
V <sub>R</sub>	=	Reactor volume, L
V <sub>O</sub>	=	Volumetric feed rate, LPH
V <sub>HCl</sub>	=	Volume of N/10 HCl taken for quench, ml
V <sub>SAMP</sub>	=	Volume of sample taken, ml
V <sub>NaOH</sub>	=	Volume of NaOH used for neutralizing, ml
X <sub>A</sub>	=	Degree of conversion,
T	=	Residence time, min

**NOMENCLATURE:**

C <sub>A</sub>	=	Conc. of unreacted NaOH in the reactor, g mole/L
C <sub>Ao</sub>	=	Initial Conc. of NaOH in the feed mixture, g mole/L
HCL <sub>O</sub>	=	Amount of HCL taken for quench, g mole
HCL <sub>R</sub>	=	Amount of HCL reacted with NaOH, g mole
K	=	Rate constant, L/g mol min
N <sub>HCL</sub>	=	Number of moles of HCL used, g mole
N <sub>NaOH</sub>	=	Number of moles of NaOH used, g mole
R	=	Real gas constant, cal/g mol K
V <sub>R</sub>	=	Reactor volume, L
V <sub>o</sub>	=	Volumetric feed rate, LPH
V <sub>HCl</sub>	=	Volume of N/10 HCl taken for quench, ml
V <sub>SAMP</sub>	=	Volume of sample taken, ml
V <sub>NaOH</sub>	=	Volume of NaOH used for neutralizing, ml
X <sub>A</sub>	=	Degree of conversion,
τ	=	Residence time, min

**PRECAUTIONS & MAINTENANCE INSTRUCTIONS:**

7. Measure the exact volume of water and weigh the chemicals properly.
8. Always use clean water and good quality chemicals and standard solution for titration.
9. Use electronic balance for weighing of chemicals. Don't mix the droppers of different chemicals.

10. Keep close all the drain valves and vent valve should open while filling the reactant in feed tanks.
11. Flow should not be disturbed during the experiments.
12. Handle the chemicals carefully.

### **TROUBLESHOOTING:**

1. If any type of suspended particles are come in the Rotameter. Remove the Rotameter clean the tube and fit that at its place.
2. If there is any leakage tight that part or remove that and fix that again after wrapping Teflon tape.
3. If Rotameter fluctuating more than average tight control knob of that.
4. If D.T.C display '1' on display board it means sensor connection is not OK tight that.
5. If switch ON the heater but temperature can't rise but panel LED is ON it means bath had burned replace that

### **REFERENCES:**

1. Octave Levenspiel "Chemical reaction engineering" 3rd ed, Wiley, NY, 2001, Page 101-102, 111-112.
2. Coulson & Richardson, "Chemical Engineering Vol-3" 4th ed, Asian Books Pvt.Ltd, 1. ND, 1991, Page 34-37.



PLUG FLOW REACTOR COIL TYPE



CONTINUOUS STIRRER TANK REACTOR (CSTR)



CASCADE CSTR



ISOTHERMAL BATCH REACTOR



PFR (STRAIGHT TUBE TYPE)