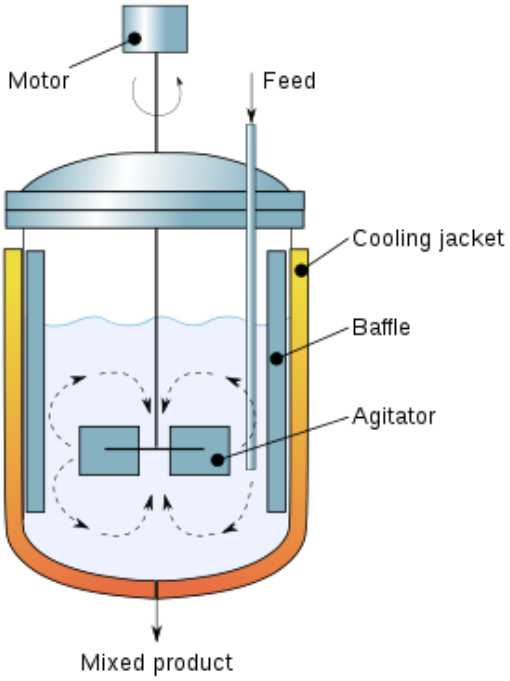
Laboratory Manual
For

Chemical Kinetics and Reaction Engineering (CHC304)

AY: 2022-23



Department of Chemical Engineering
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Index:-

Things to Do

- Be on time. Follow all written and verbal instructions carefully
- Conduct yourself in a responsible manner at all times in the laboratory
- Store your backpacks in the place mentioned by the Instructor and /or TA
- Familiarize yourself with all labs procedure before doing lab experiments
- Bring your lab notebook and an open mind to every lab
- Be aware of others in the lab. Areas of the room may be crowded at times and you should take care not to disturb the experiments of others in the lab
- Be aware of all the safety devices
- Dress properly during a laboratory activity. Personal protective equipment (PPE), such as gloves, safety glasses and lab coats, is an important factor in your safety when working in the laboratory. Proper PPE should be worn during laboratory experiments
- Keep clutter to a minimum. Work areas should be kept clean and tidy at all times
- Inform the instructor and/or TA if there is a problem. You will have their immediate attention if you have cut yourself (even if you consider it minor), if something broke and needs cleaning up, or if you are on fire
- Use extreme care when handling sharp objects
- Heated glassware remain very hot for a long time, picked up with caution. Use tongs or heat protective gloves if necessary
- Read the chemical safety information (MSDS).
- Keep all materials away from open flames
- Dispose of all chemicals, broken glass and other lab materials as directed
- Always be sure that electrical equipment is turned in the "OFF" position before plugging it into an electrical socket
- Always Turn OFF all electrical switches of the equipment before leaving laboratory
- Report ALL incidents, accidents, injuries, hazards or chemical spills to the instructor (no matter how trivial it seems)
- Wash your hands before you leave the lab for the day

Things Not to Do

- DO NOT eat, drink beverages, chew gum in the lab
- DO NOT touch any equipment, chemicals, or other materials in the laboratory area until you are instructed to do so
- NEVER do any experiment on your own and NEVER work in the lab alone
- NEVER use electrical instrument around water
- DO NOT use the mobile
- NEVER smell, taste or touch chemicals
- DO NOT work with chemicals until you are sure of their MSDS that includes some awareness of their flammability, reactivity, toxicity, and disposal
- NEVER return unused chemicals to the original container/bottle
- DO NOT casually dispose of chemicals down the drain
- NEVER use chipped, cracked, or dirty glassware.
- DO NOT panic
- NEVER leave the lab without washing your hands

EXPERIMENT-1

KINETICS IN BATCH REACTOR (EQUIMOLAR FEED)

AIM: To determine the rate constant for the saponification reaction by integral analysis for equimolar feed

APPARATUS REQUIRED: Reaction vessel with stirrer, conical flasks, burette, and pipette

CHEMICALS REQUIRED: NaOH solution, Ethyl Acetate solution, HCl solution, Phenolphthalein indicator

THEORY: A batch reactor may be described as a vessel in which any chemicals are placed to react. Batch reactors are normally used in studying the kinetics of chemical reactions, where the variation of a property of the reaction mixture is observed as the reaction progresses. The experimental batch reactor is usually run isothermally at constant volume, because it is easy to predict the result of such run. The stoichiometry of the saponification reaction between sodiumhydroxide and Ethyl Acetate is:

$$CH_3COOC_2H_5 + NaOH \rightarrow CH_3COONa + C_2H_5OH$$

The starting point for all design is the material balance expressed for any reactant and product over a small element of volume.

$$INPUT = OUTPUT + DISAPPEARENCE\ BY\ REACTION + ACCUMULATION$$

$$F_{jo} - F_j - \int^{v} r_j\, dv = \frac{dN_j}{dt}$$

A batch reactor has neither inflow nor outflow of reactants or products while the reaction is being carried out.

$$F_{jo} = F_j = 0$$

The resulting general mole balance on species j is

$$-\frac{dN_j}{dt} = \int^{v} r_j\, dv$$

If the reaction mixture is perfectly mixed so that there is no variation in the rate of reaction throughout the reactor volume, r_j can be taken out of the integral and the mole balance can be written as

$$-\frac{dN_j}{dt} = r_j dv$$

Consider a reaction

$$A + B \rightarrow C + D$$

Assume a rate equation like:

$$-r_A = k\, C_A C_B$$

$$-r_A = -\frac{dC_A}{dt} = k\, C_A C_B$$

Where,

k is the rate constant

C_A = concentration of reactant A at any time

C_B = concentration of reactant B at any time

If M=1 i.e. $C_{A0} = C_{B0}$ then,

The rate equation may be approximated

$-r_A = k\, C^2_A$

Now substituting the value for $-r_A$ and integrating, we get the expression of the form :

$$\frac{1}{C_A} - \frac{1}{C_{A0}} = kt$$

In terms of conversion X_A with respect to the reactant A is :

$$\frac{1}{C_{AO}} \frac{X_A}{1-X_A} = kt$$

Let ϕ = $\frac{1}{C_{A0}} \frac{X_A}{1-X_A}$

Plot a graph between ϕ vs. t (time). The slope of the straight line obtained will give the rate constant of the reaction.

PROCEDURE:

- Prepare 1 litre of NaOH solution.
- Estimate the strength of NaOH solution by using standard Oxalic acid.
- Estimate the strength of HCl solution by using standardised NaOH solution.
- Transfer nearly 750 ml of NaOH solution into batch reactor.
- Take nearly 15 conical flasks and transfer 15 ml of HCl solution into conical flask.
- Switch on the stirrer.
- Add some amount of ethyl acetate to the reactor such that Ethyl acetate is maintained as limiting reagent.
- Transfer the reaction mixture into conical flask, which contains 15 ml of known HCl solution for every one minute time interval.

- Take the samples up to 10 minutes.
- Note down the room temperature.
- Titrate the contents of the conical flask with NaOH solution to estimate the concentrations of ethyl acetate and NaOH solutions.

OBSERVATIONS

Standardization NaOH

Volume of Oxalic acid taken = V_{oxalic} = …… ml

Normality of Oxalic acid taken = N_{oxalic} = …… N

Volume of NaOH rundown for neutralization of Oxalic acid = V_1 = …… ml

Standardization of HCl

Volume of HCl taken = V_2 = …… ml

Volume of NaOH rundown for neutralization of HCl = V_3 = …… ml

Volume of reactor mixture taken into a conical flask = V_{sample} = …… ml

Volume of HCl taken into conical flask = V_{HCl} = …… ml

Volume of Ethyl acetate added to the reactor = V_{Ethyl} = …… ml

Volume of NaOH taken into the reactor = O_{NaOH} = …… ml

S.No.	Time (min)	V_{NaOH} (ml)

SAMPLE CALCULATIONS

Standardization

Normality of NaOH solution = N_{NaOH} = (V_{oxalic} × N_{oxalic}) / V_1 = (10 × 0.1) / 11.4 = 0.088 N

Normality of HCl solution = N_{HCl}= (V_3 × N_{NaOH}) / V_2 = (10 × 0.088) / 10 = 0.091 N

For Reactor

Volume of NaOH = 250 ml

Volume of Ethyl Acetate = 250 ml

Total volume of reactor = 500 ml

Initial Concentration of NaOH = C_{Bo} = (250 × 0.088) / 500 = 0.044 mol/l

Initial Concentration of Ethyl acetate = C_{A0} = (250 × .1) / 500 = 0.05 mol/l

M = 0.05/.044 = 1.13 ~ 1.00

At time = 2 min

Gram moles of NaOH present in the reaction = $V_{HCl} \times N_{HCl} - V_{NaoH} \times N_{NaOH}$

= 15 × 0.091 – 11.8 × 0.088

= 0.327

C_B = 0.0327 mol/l

$C_A = C_{A0} - (C_{B0} - C_B)$ = 0.05 – (0.044 – 0.0327) = 0.0387 mol/l

$X_A = 1 - C_A / C_{A0}$ = 1- 0.0387 /0.05 = 0.226

$\phi = \frac{1}{C_{A0}} \frac{X_A}{1-X_A} = \frac{1}{0.05} \frac{0.226}{1-0.226}$ = 5.83

CALCULATION TABLE

Obtain the below values at different interval of time.

S.No	C_A	C_B	X_A	ϕ	time

RESULT

Plot a graph between ϕ vs time (t)

A straight line passing through the origin will be obtained. Calculate the slope of the straight line from the graph.

The rate constant for the reaction of Ethyl Acetate with NaOH is equal to the slope of the straight line with equals …….

CONCLUSIONS

The rate constant for the reaction of Ethyl acetate with NaOH with non-equimolar feed was found out to be ……..

In batch reactor a stirrer is constantly used to maintain same concentration of reactants and products at all the regions in the reactor at any time.

Final conversion for equimolar and non-equimolar feed will be different for same value of limiting reagent in both cases but the rate constant k will be same always same for the two cases.

The reaction was of order two, hence after a long time the conversion obtained was significantly large but not equal to one.

EXPERIMENT-02

KINETICS IN BATCH REACTOR (NON-EQUIMOLAR FEED)

AIM: To determine the rate constant for a saponification reaction by using integral analysis

APPARATUS REQUIRED: Reactor vessel with stirrer, conical flask, burette, and pipette

CHEMICALS REQUIRED: NaOH solution, ethyl acetate solution, HCl, Phenolphthalein indicator

THEORY: A batch reactor may be described as a vessel in which any chemicals are placed to react. Batch reactors are normally used in studying the kinetics of chemical reactions, where the variation of a property of the reaction mixture is observed as the reaction progresses. The stoichiometry of the saponification reaction between sodium hydroxide and Ethyl Acetate is:

$$CH_3COOC_2H_5 + NaOH \rightarrow CH_3COONa + C_2H_5OH$$

The starting point for all design is the material balance expressed for any reactant and product over a small element of volume.

$$INPUT = OUTPUT + DIAPPEARENCE\ BY\ REACTION + ACCUMULATION$$

$$F_{jo} - F_j - \int^{v} r_j\ dv = \frac{dN_j}{dt}$$

A batch reactor has neither inflow nor outflow of reactants or products while the reaction is being carried out.

$$F_{jo} = F_j = 0$$

The resulting general mole balance on species j is

$$-\frac{dN_j}{dt} = \int^{v} r_j\ dv$$

If the reaction mixture is perfectly mixed so that there is no variation in the rate of reaction throughout the reactor volume, r_j can be taken out of the integral and the mole balance can be written as

$$-\frac{dN_j}{dt} = r_j dv$$

Consider a reaction $A + B \rightarrow C + D$

Assume a rate equation like:

$$-r_A = k\, C_A C_B$$

$$-r_A = -\frac{dC_A}{dt} = k\, C_A C_B$$

Where, k is the rate constant.

In terms of conversion and using the

$$-r_A = C_{A0}\left(\frac{dX_A}{dt}\right) = KC^2_{A0}\,(1- X_A)\,(M - X_A)$$

Where $M=C_{B0}/C_{A0}$ and X_A is the conversion of A.

M is the initial ratio of concentrations of B (C_{B0})to A(C_{A0}) in the reactor.

After breakdown into partial fractions, integration and rearrangement, the final result is

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

Where,t= time , M = C_{B0}/C_{A0},

X_A is conversion of A at given time

The above equation is valid for M>1.

Let ϕ= $ln[\ (M - X_A / M(1 - X_A)\ ] / C_{A0}(M - 1)$

Plot the graph of ϕ *vs.t*. The slope of the straight line obtained will give the rate constant of the reaction.

PROCEDURE:

- Prepare 1 litre of NaOH solution.
- Estimate the strength of NaOH solution by using standard Oxalic acid.
- Estimate the strength of HCl solution by using standardized NaOH solution.
- Transfer nearly 750 ml of NaOH solution into batch reactor.
- Take nearly 15 conical flasks and transfer 15 ml of HCl solution into each conical flask.
- Switch on the stirrer.
- Add some amount of ethyl acetate to the reactor such that Ethyl acetate is maintained as limiting reactant.
- Transfer the reaction mixture into conical flask, which contains 15 ml of known HC1 solution for every one minute time interval.
- Take the samples up to 10 minutes.

- Note the room temperature.
- Titrate the content of the conical flask with NaOH solution to estimate the concentrations of Ethyl Acetate and NaOH solutions.

OBSERVATIONS

Standardization NaOH

Volume of Oxalic acid taken = V_{oxalic} = …… ml

Normality of Oxalic acid taken = N_{oxalic} = …… N

Volume of NaOH rundown for neutralization of Oxalic acid = V_1 = …… ml

Standardization of HCl

Volume of HCl taken = V_2 = …… ml

Volume of NaOH rundown for neutralization of HCl = V_3 = …… ml

Volume of reactor mixture taken into a conical flask = V_{sample} = …… ml

Volume of HCl taken into conical flask = V_{HCl} = …… ml

Volume of Ethyl acetate added to the reactor = V_{Ethyl} = …… ml

Volume of NaOH taken into the reactor = Q_{NaOH} = …… ml

S.No	Time (min)	V_{NaOH} (ml)

SAMPLE CALCULATION

Standardization

Normality of NaOH solution = N_{NaOH} = (V_{oxalic} × N_{oxalic}) / V_1 = (10 × 0.1) / 9.5 = 0.105 N

Normality of HCl solution = N_{HCl}= (V_3 × N_{NaOH}) / V_2 = (9 × 0.105) / 10 = 0.0945 N

For Reactor

Volume of NaOH = 500 ml

Volume of Ethyl Acetate = 250 ml

Total volume of reactor = 750 ml

Initial Concentration of NaOH = C_{Bo} = (500 × 0.105) / 750 = 0.07 mol/l

Initial Concentration of Ethyl acetate = C_{A0} = (250 × 0.1) / 750 = 0.033 mol/l

M = 0.07/.033 = 2.12

At time = 0.5 min

Gram moles of NaOH present in the reaction = $V_{HCl} \times N_{HCl} - V_{NaoH} \times N_{NaOH}$

= 15 × 0.0945 – 7.8 × 0.105

= 0.5985

C_B = 0.05985 mol/l

$C_A = C_A - (C_{B0} - C_B)$ = 0.033 – (0.07 – 0.05985) = 0.02285 mol/l

$X_A = 1 - C_A / C_{A0}$ = 1- 0.02285/0.033 = 0.31

$\Phi = \ln[(2.12 - 0.31) / \{ 2.12 \times (1 - 0.31) \}] / \{ 0.033 \times (2.12 -1) \}$ = 5.98

CALCULATION TABLE

Obtain the below values at different interval of time.

S.No	C_A	C_B	X_A	ϕ	time

RESULT

Plot a graph between ϕ vs. time (t)

A straight line passing through the origin will be obtained. Calculate the slope of the straight line from the graph.

The rate constant for the reaction of Ethyl Acetate with NaOH is equal to the slope of the straight line with equals …….

CONCLUSIONS

The rate constant for the reaction of Ethyl acetate with NaOH with non-equimolar feed was found out to be ……..

In batch reactor a stirrer is constantly used to maintain same concentration of reactants and products at all the regions in the reactor at any time.

Final conversion for equimolar and non-equimolar feed will be different for same value of limiting reagent in both cases but the rate constant k will be same always same for the two cases. Ethyl acetate served as limiting reagent in case of non-equimolar feed.

EXPERIMENT-03

ISOTHERMAL BATCH REACTOR (DETERMINATION OF ACTIVATION ENERGY)

Aim: To study of a non-catalytic homogeneous reaction in an isothermal batch reactor.

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

To determine the effect of temperature on k and determine the activation energy.

THEORY:

A batch reactor is a closed system with no input and output streams. A batch reactor can operate under the following conditions:

Isothermal (temperature of reaction mass remains constant)

Perfectly mixed (composition of the reaction mixture is uniform throughout)

Constant volume (volume of the reaction mixture within the reactor remains constant; there is no appreciable change in the density of reaction mass)

In a homogeneous reaction, all the reactants remain in single phase. The rate of reaction for such a reaction is expressed as:

Moles of Product formed per unit volume of reaction mixture per unit time.

For a first order unidirectional reaction $A \rightarrow Product$

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

Also

$$-r_A = KC_A = KN_A / V$$

Combining these two equations yield.

$$ln\ N_A = ln\ N_{A0} - kt$$

$$N_A = N_{A0}\, e^{-kt}$$

Or, in terms of Concentrations

$$C_A = C_{A0}\, e^{-kt}$$

And

N_{A0} = Number of moles of A at $t = 0$

N_A = Number of moles of A at $t = t$

For second order unidirectional reaction

$$2A \quad \rightarrow \quad Product$$

If we plot $1/C_A$ Vs t we must get a straight line with slope = k and intercept = $1/C_{A0}$

For second order reaction:

$$A + B \quad \rightarrow \quad Product$$

(If $N_{A0} = N_{B0}$ then $N_A = N_B$ for all t)

It reduces to

$$2A \rightarrow product$$

Degree of conversion X is defined as:

Equation

And $N_B = N_{B0} - N_{A0}\, X$ *for* $N_{A0} \neq N_{B0}$

Equation

The effect of concentration on the rate of reaction is generally determined experimentally in a batch reactor by studying the rate of reaction at constant temperature

The interpretation of the kinetic data involves a trial and error procedure. A kinetic model is first assumed and the experimental observed conversion – time rate is matched with the selected model. The reaction rate constant is a strong function of reaction temperature. The temperature dependence of k is expressed in terms of Arrhenius equation: $k = A\, exp(-Ea/RT)$

A = frequency factor of Arrhenius constant

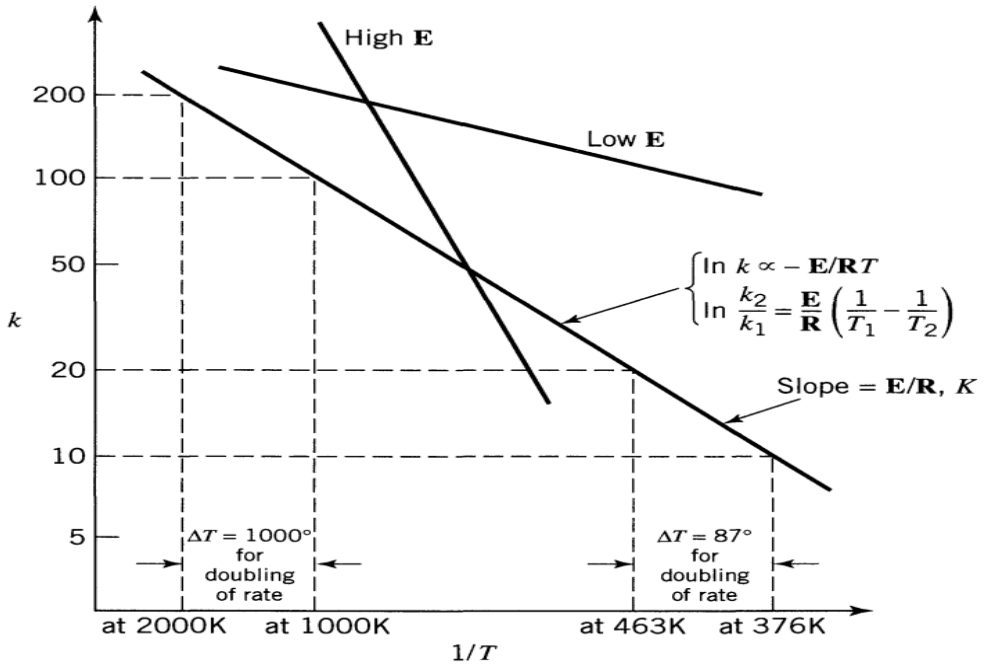
E_a = Activation Energy

R = gas law constant

This equation can be written in the form:

$$ln\, k = lnA - E_a/(RT)$$

A plot of $ln\, k$ *Vs* $1/T$ shall yield E_a and A.



In case of second order reversible reaction with $C_{A0} = C_{B0}$ Equation

Equation

X_{Ae} = fractional conversion at equilibrium Saponification of Ethyl acetate & NaOH

$$CH_3COOC_2H_5 + NaOH \longrightarrow CH_3COONa + C_2H_5OH$$

88g 40 82 46

B + A C + D

Description

A batch reactor is closed system with no input and output streams. A batch reactor can operate under conditions like isothermal (Temperature of reaction Mass remains constant), perfectly mixed (composition of reaction mixture is uniform throughout), and constant volume (volume of the reaction mixture within the reactor remains constant, there is no appreciable change in the density of reaction mass). This set – up is used to study a non – catalytic homogeneous reaction under isothermal condition. The set up consists of a reactor fitted in a constant temperature water bath. One stirrer is fitted for mixing the reactants in reactor and other is fitted in water batch to keep the uniform temperature throughout in the bath. The temperature of bath can be maintained from ambient to 90 ^{0}C with the help of Digital temperature indicator cum controller. Samples can be taken out with the help of a sampling pipette.

Volume of reactor = 2.815 lt

Volume of bath = 14.3lt

Reactor stirrer =100rpm

Bath stirrer = 100rpm

Temperature controller – ambient to 200 ^{0}C with 1 ^{0}C resolution Utilities required

1. Water supply
2. drain
3. electricity supply 1 phase 220V AC,1.5kw
4. Instruments, laboratory glass ware and chemicals required for analysis as per the system adopted.

Chemicals:

1. N/10 NaOH
2. N/10 HCl
3. N/10 Ethyl acetate (8.8gms of ethyl acetate in 1 lits of water), indicator (phenolphthalein)

EXPERIMENTALPROCEDURE:

1. Prepare a solution of N/10 ethyl acetate by mixing 8.8gms (or 9.8ml at 20 ºC) of ethyl acetate in 1L of solution.
2. Prepare a solution of N/10 NaOH by dissolving 4gms / 1L of solution
3. Prepare a solution of N/10 HCl
4. Use phenolphthalein as indicator
5. Take 6Nos of 250ml beakers and put 20ml of N/10HCl in each beaker
6. Take N/10 NaOH in the burette
7. Adjust the temperature of water bath at25 ºC
8. If the volume of reactor is 1L then take 400ml each of N/10 $CH_3COOC_2H_5$ and N/10 NaOH in two separate flasks and keep these in the water bath for about 15min
9. Transfer these solutions quickly in the batch reactor. Start immediately the mixer and the stopwatch
10. At regular intervals of 3-5min withdraw 100ml of reaction mixture and put it in the marked beakers containing N/10 HCl. Take at least 6 samples at regular intervals of time.
11. Transfer the solution from beakers to the conical flasks, and titrate the excess N/10 HCl in each flask using N/10 NaOH from burette and phenolphthalein as indicator
12. Record the reaction temperature
13. Equilibrium conversion can be determined at reaction time of ½hr
14. In order to study the effect of temperature on k the above steps may be repeated at temperature = 35 ºC and 50 ºC. All the reactants must be preheated in the water batch before mixing in the reactor.

Standard Data

Reactor	:Material stainless steel 304 grade, Volume 2.1 L
Water Bath	: Material stainless steel 304 grade, double wall, insulated with ceramic wool
Heater	: Nichrome wireHeater
Stirrers (2Nos)	: stainless steel 304 grade impeller and shaft coupled with FHPmotor

Stopwatch : electronic

Temperature sensor : RTD PT – 100type

Control panel comprises of Digital Temperature controller cum – indicator (For Batch) = 0 – 1999.9 ºC, RTD PT – 100 type

With standard make on/off switch, mains indicator fuse etc.

The whole unit is assembled rigidly on a base plate and mounted on a stand Most of the parts are powder coated and rest are painted with auto paints

FORMULAE:

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

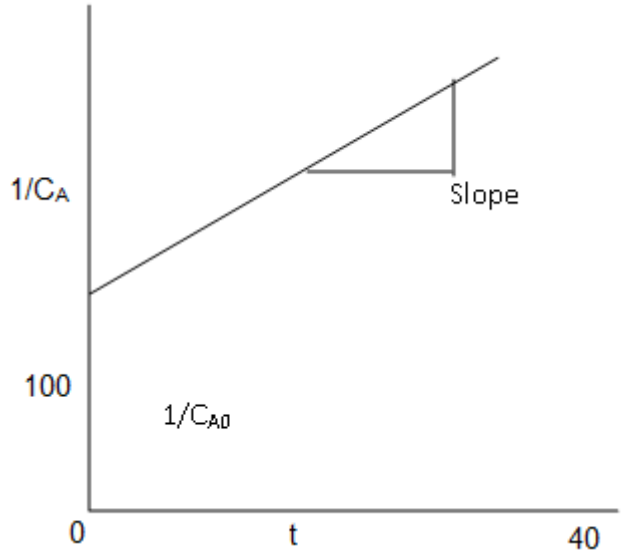
Amount of HCl taken as quench, HCl = $(V_{HCl}).(N_{HCl}) / 1000$, gmol

Amount of HCl reacted with the un-reacted NaOH of the reaction sample = HCLR = HCLO – (V* N_{NaOH})/1000), gmol

Concentration of unreacted NaOH in the reaction mixture, C_A= (HCLR / V_{Samp})*1000, gmol/L

Time, t (min)	C_A Concentration of NaOH (gmol/L)	$1/C_A$ (L/gmol)

Plot t Vs $1/C_A$



Slope = k, L/gmole – min

C_A = concentration of NaOH in gmole/L

t =time in min

Plot ln k Vs 1/T, which yields $Slope = -\frac{E_a}{R}$ and

Intercept = ln k_0

Observations and Calculation Reaction:

$A+B \longrightarrow C+D$ $(C_{A0} = C_{B0})$

Reaction temperature=

Concentration of Ethyl acetate = N/10

Concentration of NaOH = N/10 Volume of

Ethyl acetate taken =400ml

Volume of Sodium Hydroxide = 400ml

Initial concentration of Ethyl acetate (B) in the mixture = 0.01N = C_{B0} (gmol/L) Initial concentration of NaOH (A) in the mixture = 0.01N = C_{A0} (gmol/L)

At Temperature T_1=

S. No	Time, t min.	Concentration of unreacted NaOH in the reaction mixture, C_A (gmol/L)	$1/C_A$

At Temperature T_2=

S. No	Time, t min.	Concentration of unreacted NaOH in the reaction mixture, C_A (gmol/L)	$1/C_A$

At Temperature T_3=

S. No	Time, t min.	Concentration of unreacted NaOH in the reaction mixture, C_A (gmol/L)	$1/C_A$

Tabulate the time concentration data (t Vs C_A)

Determine the rate constant k by assuming the reaction to be of second order and plotting $1/C_A$ Vs t

$$\frac{1}{C_A} = kt + \frac{1}{C_{A0}}$$

PRECAUTIONS & MAINTANANCE INSTRUCTION:

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 filling the reactant in feed tanks
5. Flow should not be disturbed during the experiments
6. Handle the chemicals carefully
7. Don't ON heater switch before filling the water in the bath
8. There should be no air in the flow during experiment

TROUBLE SHOOTING:

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 (Procedure: two nuts are there lose first nut and tight the second lightly, and then first also, both nuts are on rotameter)
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

REFERENCE:

1. Chemical Reaction Engineering by Octave Levenspiel, Chapter 2.

Experiment-04

Kinetics in plug flow reactor (PFR)

AIM: To determine the rate constant for saponification reaction of ethyl acetate and sodium hydroxide in a straight tube type plug flow reactor at ambient conditions.

APPARATUS REQUIRED: Reactor vessel with stirrer, conical flask, burette, and pipette

CHEMICALS REQUIRED: NaOH solution, ethyl acetate solution, HCl, Phenolphthalein indicator

THEORY: In a plug flow reactor the concentration of the reactant decreases progressively and that of the product increases. In an ideal PFR there is no mixing in the direction of the flow but there is complete mixing in the direction perpendicular to the flow. Therefore the concentration of the reactants and products vary long the length of the reactor but not along the radial direction. The stoichiometry of the saponification reaction between sodiumhydroxide and Ethyl Acetate is:

$$NaOH + CH_3COOC_2H_5 \rightarrow CH_3COONa + C_2H_5OH$$

(A) (B) (C) (D)

The order of the above reaction is 2. The rate of the reaction for equimolar feed rate of A & B can be expressed as follows:

$$-r_A = -\frac{dC_A}{dt} = kC_A^2 = kC_{A0}^2(1 - X_A)^2$$

For a plug flow reactor the performance equation at steady state can be expressed as follow:

$$\int_0^{X_{Af}} \frac{dX_A}{-r_A} = \frac{\tau}{C_{AO}}$$

τ is space time.

Now substituting the value for $-r_A$ and integrating we get the following expression:

$$\tau = \frac{1}{k\,C_{AO}} \frac{X_A}{(1-X_A)}$$

Plot the graph of τ vs $X_A / (1 - X_A)$. The plot will give a straight line and the slope of this line equals to

$1 / kC_{A0}$. From this value of slope the rate constant k can be calculated.

PROCEDURE:

- Prepare 20 L of 0.1 N NaOH solution by dissolving 80 gm of sodium hydroxide in 20 L of distilled water.
- Prepare 20 L 0f 0.1 N ethyl acetate solution by mixing 176 gm of ethyl acetate in 20 L of distilled water.
- Take 20 ml of 0.1 N HCl in measuring cylinder.
- Fill the burette by 0.1 N NaOH solution.
- Close all the valves.
- Open the valve V_4 and V_6, fill NaOH in feed tank A.
- Close valve V_4 and V_6.
- Open valve V_5 and V_7, fill ethyl acetate in feed tank B.
- Close valve V_5 and V_7.
- Connect air supply to the setup at valve V_1.
- Open valve V_1, and set air pressure 0.5 to 1 kg/cm^2 by pressure regulator and pressure gauge.
- Pass sodium hydroxide and ethyl acetate into the reactor by allowing equal flow rate controlled by the valves.
- After about 10 min or time equal to the residence time of the reactor collect exact 10 ml of the sample from the outlet in measuring cylinder that already contain 20 ml of HCl.
- Transfer the sample solution in a conical flask.
- Titrate the solution, using phenolphthalein as an indicator against 0.1 N sodium hydroxide.
- Repeat the experiment for different flow rates of feed.

OBSERVATIONS

Standardization NaOH

Volume of Oxalic acid taken = V_{oxalic} = …… ml

Normality of Oxalic acid taken = N_{oxalic} = …… N

Volume of NaOH rundown for neutralization of Oxalic acid = V_1 = …… ml

Standardisation of HCl

Volume of HCl taken = V_2 = …… ml

Volume of NaOH rundown for neutralization of HCl = V_3 = …… ml

Volume of reactor mixture taken into a conical flask = V_{sample} = …… ml

Volume of HCl taken into conical flask = V_{HCl} = …… ml

For Reactor

Working volume of the reactor = V_R = …… Lt

$C_{A0} = N_{NaOH} / 2$ mol / l

$HCl_0 = (V_{HCl} \times N_{HCl}) / 1000$ mol

$HCl_R = HCl_0 - (V_1 \times N_1) / 1000$ mol

$C_A = (HCl_0 \times 1000) / V_{sample}$ mol / l

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

$T = (V_R \times 60) / (V_A + V_B)$ min

S.N0	V_A (LPH)	V_B (LPH)	V_1 (ml)

SAMPLE CALCULATIONS

Standardization

Normality of NaOH solution = $N_{NaOH} = (V_{oxalic} \times N_{oxalic}) / V_1 = (10 \times 0.1) / 9.6 = 0.104$ N

Normality of HCl solution = $N_{HCl} = (V_3 \times N_{NaOH}) / V_2 = (9.4 \times 0.104) / 10 = 0.1$ N

For Reactor

$C_{A0} = N_{NaOH} / 2 = 0.104 / 2 = 0.052$ mol / l

$HCl_0 = (V_{HCl} \times N_{HCl}) / 1000 = (20 \times 10) / 1000 = 0.002$ mol

$HCl_R = HCl_0 - (V_1 \times N_1) / 1000 = 0.002 - (16.9 \times 0.104) / 1000 = 0.0002424$ mol

$C_A = (HCl_0 \times 1000) / V_{sample} = (0.0002424 \times 1000) / 10 = 0.02424$ mol / l

$X_A = (C_{A0} - C_A) / C_{A0} = (0.052 - 0.02424) / 0.052 = 0.534$

$$\frac{X_A}{(1-X_A)} = \frac{0.534}{(1-0.534)} = 1.145$$

$\tau = (V_R \times 60) / (V_A + V_B) = (0.750 \times 60) / (5 + 5) = 4.5$ min

CALCULATION TABLE

S.No	τ (min)	X_A	k (L/mol min)	$X_A / (1- X_A)$

RESULT

Plot a graph between τ vs $X_A / (1- X_A)$ and find the slope (S) of the straight line obtained.

We get S = …… min

Then $k = 1 / (C_{A0} S)$

CONCLUSIONS

The rate of reaction for saponification reaction for a PFR is found out to be ……

The PFR took some time to operate at steady state. The concentration of the reactant decreases progressively along the length of the reactor unlike the CSTR where it decreases to a lower value as soon as the reactants are fed to the reactor.

The PFR favours high conversion for reactions which has rate greater than 1 unlike CSTR which favours reactions having rate less than 1.

EXPERIMENT-05

KINETICS IN MIXED FLOW REACTOR (MFR)

AIM: To study of a non-catalytic homogenous second order liquid phase reaction in a CSTR under ambient conditions.

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

APPARATUS REQUIRED: CSTR, conical flask, burette, pipette

CHEMICALS REQUIRED: NaOH solution, ethyl acetate solution, HCl, Phenolphthalein indicator

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. The stoichiometry of the saponification reaction between sodiumhydroxide and Ethyl Acetate is:

$$CH_3COOC_2H_5 + NaOH \rightarrow CH_3COONa + C_2H_5OH$$

Reactor volume = Vr

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

Space time = $\tau = Vr / V_0$ min

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

$$\tau = Vr/V_0 = C_{A0}X_A / (-r_A) = C_{A0} - C_A / (-r_A)$$

Xa and r_A are evaluated at exit stream conditions, which are the same as conditions within the reactor.

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

The reaction rate (Rate of disappearance of A) = $-r_A = kC_A^2$

Hence,

$\tau = Vr/V_0 = C_{A0}X_A / (-r_A) = C_{A0} - C_A / (-r_A) = C_{A0} - C_A / kC_A^2$

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

And rate of reaction, $-r_A = C_{Ao}X_A/\tau$, gmol/l-min

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

For n^{th} order reaction, $-r_A = kC_A^n$

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

PROCEDURE:

- Prepare 20L of N/10 NaOH solution by dissolving 80 gm of NaOH in 20L of water.
- Prepare 20L of N10 ethyl acetate solution by mixing 176 gms of ethyl acetate in 20L of water
- Fill the respective tanks with these solutions.
- Adjust the flow rate of the two streams so that in the feed mixture $C_{Ao} = C_{Ho}$ le have equal flow rates (eg 2 LPH each you may use the flow range 2 LPH to 16 LPH for each stream) using calibrated rotameters.
- Pass equimolar feed rates of ethyl acetate and NaOH into the CSTR by allowing equal volumetric feed rate of reactants in to the reactor and simultaneously start the mixer of the CSTR Ensure constant stirring throughout the experiment
- After about 10 min or time equal to the residence time of the reactor (whichever is greater Te when steady state is achieved, collect sample of the liquid at the outlet
- Titrate the solution against N/40 HCI (and HCI from burette).
- Using phenolphthalein as Indicator not the volume of N/40 HCI used (Vc)
- Note the volumetric flow rate of liquid at the end of the reactor
- Note the reaction temperature.
- 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 HCI using phenolphthalein as indicator Note the volume of N /10 HCI used.

- Repeat all the steps for four to six different flow rates of feed

OBSERVATION TABLE:

Standardisation of NaOH

S. No.	Volume of Oxalic Acid (ml)	Volume of NaOH (ml)
1	10	9.5

$$N_1V_1 = N_2V_2$$

$$N_1(9.5) = 0.1 \times 10$$

$$N1 = 0.105 = \text{Normality of NaOH}$$

Standardization of HCl

S. No.	Volume of NaOH (ml)	Volume of HCl (ml)
1	9.5	10

$$N_1V_1 = N_2V_2$$

$$N_1(10) = 9.5 \times 0.1$$

$$N_1 = 0.095 = \text{Normality of HCl}$$

S. No.	V_A (LPH) NaOH	V_B (LPH) Ethyl Acetate	Initial Vol. Of NaOH (ml)	Final Vol. Of NaOH (ml)
1	5	5	0	5.9

SAMPLE CALCULATION:

$\upsilon = 5$

C_{B0} = initial concentrate of NaOH

$= (Q_{NaOH} \times N_{NaOH}) / (Q_{NaOH} + N_{NaOH})$

$= 5 \times 0.105/10 = 0.0525$

Gram mol of HCl added = $15 \times 10^{-3} \times 0.095$

= 0.001425

Gram mol of NaOH remaining in the reaction mixture $= 15 \times 10^{-3} \times 0.095 - 10.9 \times 10^{3} \times 0.105$

= 0.0002805

$C_A = 0.02805$

$X_A = 1 - 0.02805/0.0525 =$ **0.465**

CALCULATION TABLE:

S.No	C_B	X	υ (LPH)
1	0.0280	0.465	5

GRAPHS AND DATA TO BE ANALYSED:

The order of 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 τ 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.

CONCLUSIONS: In a CSTR, the concentration of the reactant drops immediately to a certain value which is same throughout the CSTR at steady state. A high conversion for reaction whose rate is less than 1 can be achieved using CSTR when compared to a PFR.
The rate constant calculated for a reaction using a CSTR and a PFR will be approximately same at same temperature.

EXPERIMENT-06

KINETICS IN SEMI-BATCH REACTOR

OBJECTIVE:

Study of a non-catalytic homogeneous reaction in a Semi-batch reactor.

AIM:

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

APPARATUS:

1. Semi batch reactor set up,
2. Conical flasks (250 ml) – Nos.(P) +2 Nos. (G)
3. Stopwatch – 1 No
4. Burette – 1 No
5. Volumetric (Sampling) pipette (10 ml) – 1 No.

CHEMICALS REQUIRED:

1. M/100 and N/40 Sodium hydroxide,
2. M/100 Ethyl acetate
3. Phenolphthalein indicator, and
4. N/40 Hydrochloric acid

CHEMICALS REACTION:

$$NaOH + CH_3COOC_2H_5 \rightarrow CH_3COONa + C_2H_5OH$$

A B → C D

THEORY:

A semi batch reactor can be considered as a continuously operated tank reactor where incoming and outgoing mass flows are not equal to each other; consequently, the total mass of reaction mixture is not constant.

In a semi-batch reactor one reactant (A) is added initially and the other reactant (B) is added continuously at constant rate. The continuous feeding of one of the reactants causes change in the composition in addition to the changes due to the reaction itself.

Consider the reaction

$A + B \xrightarrow{k} C + D$

In which B is fed to the vat containing only A initially. Mass balance for reactant A gives (at time t)

$$\begin{bmatrix} Number\ of\ moles\ of\ A \\ in\ the\ vat\ at\ time\ t \end{bmatrix} = \begin{bmatrix} Number\ of\ moles\ of\ A \\ in\ the\ vat\ initially \end{bmatrix} - \begin{bmatrix} Number\ of\ moles\ of\ A \\ reacted\ upto\ time\ t \end{bmatrix}$$

$$N_A = N_{A0} - N_{A0}X_A$$

Where X_A is the moles of A reacted per mole of A initially in the vat.

Similarly, material balance for B gives

$$\begin{bmatrix} Number\ of\ moles\ of\ B \\ in\ the\ vat\ at\ time\ t \end{bmatrix}$$

$$= \begin{bmatrix} Number\ of\ moles\ of\ B \\ in\ the\ vat\ initially \end{bmatrix} + \begin{bmatrix} Number\ of\ moles\ of\ B \\ added\ to\ the\ vat \end{bmatrix} - \begin{bmatrix} Number\ of\ moles\ of\ B \\ reacted\ upto\ time\ t \end{bmatrix}$$

$$N_B = N_{Bi} + \int_0^t F_{B0}\, dt - N_{A0}X_A$$

Where, FB0 = molar feed rate of B.

For constant molar feed rate:

$$N_B = N_{Bi} + F_{B0}t - N_AX_A$$

Rate equation is given by:

$$(-r_A)V = N_{A0}\frac{dX_A}{dt}$$

Fluid volume at any time t is

$$V = V_0 + v_0t$$

where, V_0= initial volume of sodium hydroxide, v_0=flow rate of ethyl acetate.

For a second order irreversible reaction, rate of reaction can be written as,

$$-r_A = k\, C_AC_B$$

when, $C_{A0} = C_{B0}$

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

$$C_A = \frac{N_A}{V} = \frac{N_{A0}(1 - X_A)}{V_0 + v_0 t}$$

$$C_B = \frac{N_B}{V} = \frac{N_{Bi} + F_{B0}t - N_{A0}X_A}{V_0 + v_0 t}$$

$$(-r_A) = N_{A0}\frac{dX_A}{dt} = \frac{k[N_{A0}(1 - X_A)][N_{Bi} + F_{B0}t - N_{A0}X_A}{(V_0 + v_0 t)^2}$$

$$\frac{dX_A}{dt} = \frac{k[(1 - X_A)][N_{Bi} + F_{B0}t - N_{A0}X_A}{(V_0 + v_0 t)^2}$$

$$X_A = k\int_0^t \frac{(1 - X_A)(F_{B0}t - N_{A0}X_A)}{(V_0 + v_0 t)^2}dt$$

where, $N_{Bi} = 0$ (no B present in the vat initially)

$$X_A = 1 - \frac{C_A(V_0 + v_0 t)}{N_{A0}}$$

EXPERIMENTAL SET UP DATA:

The experimental set up consists of a semi batch reactor made of stainless steel. The dimensions of the vessel are as under:

STIRRED TANK:

Height of the tank = 220 mm.

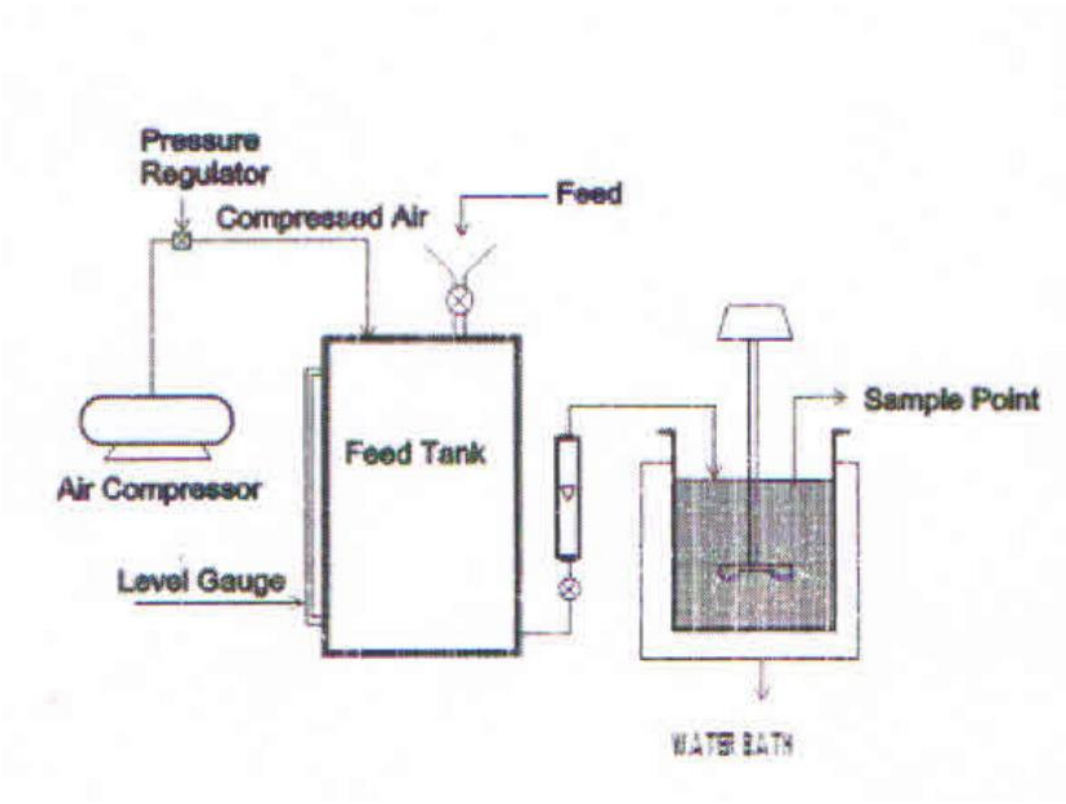
Inside diameter = 145 mm.

Volume of the tank = 3.63 L

Working volume of the tank = 3.13 L apprx

Agitator speed (Variable) = (0 – 3—RPM apprx).

Fluid Flow measurement range Rotameter = (2.0 – 20 LPH)



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 solutions of N/40 HCl and N/40 NaOH.
- Take 6 Nos. of 250 ml conical flasks and put 20 ml of N/40 HCl in each flask.
- Take N/40 NaOH in the burette.
- Adjust the reaction temperature as desired. Adjust the mixer speed so that stirring starts.
- Add measured quantity (500 ml) of aqueous solution of NaOH in the reaction vessel, start stirrer and wait till the temperature equals the set temperature.
- Adjust the volumetric flow rate of ethyl acetate solution with the help of Rotameter to desired value (2 - 4 LPH) without adding any ethyl acetate solution to reaction vessel
- At time t = 0, start adding ethyl acetate solution (without disturbing its flow rate) to reaction mixture; simultaneously start the stop watch.
- At regular interval of 3 min., withdraw 10 ml of solution (reaction mixture) using sampling pipette and put it in the marked conical flask containing N/40 HCl.

- Take at least 6 samples.
- Titrate the excess N/40 HCl in each flask using N/40 NaOH from burette and phenolphthalein as indicator.
- Record the reaction temperature.
- Above procedure is repeated for second different flow rate of ethyl acetate also.

OBSERVATION AND CALCULATIONS:

Reaction temperature = _____°C

Concentration of ethyl acetate, C_{B0} = M/100 = ____ gmol/L

Concentration of sodium hydroxide C_{A0} = M/100 = _____ gmol/L

Volume of NaOH taken (V_0) = 500 ml

Number of moles of NaOH (A) initially present, $N_{A0} = C_{A0}V_0$ __ gmol

Flow rate of ethyl acetate (v_0) = ___LPH

Molar flow rate of ethyl acetate, $F_{B0} = v_0C_{B0}$ = gmol/h

Table 1

S.NO.	Measured flow rate of reagent B, LPH	Sampling time, min	Vol. of sample taken, ml	Vol. of N/40 HCl added, ml	Vol. of sample taken, ml	Vol. of N/40 NaOH consumed in titration, ml

Estimation of un-reacted 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 = $V_{NaOH} \times 1/40 \times 10^{-3} \times V_{NaOH}$ gmoles

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

Reaction between NaOH and HCl is:

$$NaOH + HCl \rightarrow NaCl + H_2O$$

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.

$$= (5 \times 10^{-4} - 2.5 \times 10^{-5} \times V_{NaOH}) \text{ g moles}$$

∴ No. of moles of NaOHun-reacted 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 un-reacted NaOH is

$$C_A = \frac{5 \times 10^{-4} - 2.5 \times 10^{-5} V_{NaOH}}{10 \times 10^{-3}} \; g\, mol/L$$

$$X_A = 1 - \frac{C_A(V_0 + v_0 t)}{N_{A0}}$$

$$X_A = k \int_0^t \frac{(1 - X_A)(F_{B0} t - N_{A0} X_A)}{(V_0 + v_0 t)^2} dt \quad \ldots\ldots\ldots\ldots (1)$$

Table 2

S. NO.	Time, min	C_A, g mole/L	X_A	Value of Integrand

Calculate the value of above definite integral in eqn. (1) using Simpson's rule for numerical integration and then find out the value of k.

PRECAUTIONS:

1. All solutions should be prepared accurately and must be standardized.
2. Titrations should be carried out precisely.
3. While taking samples, care should be taken that tip of the pipette does not touch the agitator blade.
4. For setting of reaction temperature, initially the temperature should be set at around 6 ° C less than the desired temperature to avoid shooting. Before starting the flow rate of ethyl acetate, temperature should be set at desired temperature.
5. While filling the supply (feed) tanks, care should be taken that valve of compressed air is closed and vent is open.
6. If supply tank is to be re-filled, care should be taken that compressed air valve should remain closed and vent should be opened slowly to avoid spraying of chemical present in the tank. Refilling should be started only when pressure of the tank is released.
7. Rotameters should be periodically monitored to ensure constant flow rates through them.
8. After completion of experiment, chemicals should be removed from the feed tank and the air pressure should be released.

CONCLUSIONS:

Write down the points you have concluded from the experiment.

REFERENCE:

Fogler, H.S., "Elements of Chemical Reaction Engineering," 2nd edition, Prentice-Hall of India, New Delhi, 1995, pp, 152-155.

EXPERIMENT-07

RTD IN PLUG FLOW REACTOR (PFR)

OBJECTIVE:

RTD Studies in PFR

AIM:

1. To plot the RTD curve for a PFTR using a pulse tracer
2. To determine the dispersion number (D/uL)

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 fluids in the vessel.

To predict the exact behaviors of a vessel as a chemical reactor, RTD or stimulus response technique is used.

THEORY:

The exit age distribution function of fluid leaving a vessel or RTD of a fluid in vessel is called E-CURVE.

The normalized curve is such that

$$\int_0^\infty E dt = 1 \qquad \text{------(1)}$$

In stimulus response experimentation the system is perturbed and then sees how the system reacts or responds to this stimulus. The analysis of the response gives the desired information. A pulse tracer input signal could be used as a stimulus.

A pulse tracer input signal could be used as the stimulus.

The concentration time curve for pulse signal at the vessel outlet is called C-CURVE.

Considering steady state flow of fluid through a closed vessel.

C = E -----(2)

Where $E(t)=C_i / \Sigma \Delta t_i C_i$

The mean age of the exit stream or mean residence-time is:

$\tau = \tau_E = \tau_C = \int_0^\infty tEdt = \sum tE\Delta t$ ------(3)

Also,

$\tau = [\Sigma\ t_iC_i\ /C_i]^2$

And the variance of E or C distribution is

$\sigma_t^2 = \int_0^\infty t^2Edt - \tau^2 = \sum t^2E\Delta t - \tau^2$

Or $\sigma^2 = [\Sigma\ t_i^2Ci\ /\ \Sigma\ C_i] - [\Sigma\ t_iC_i\ /\ \Sigma\ C_i]^2$

Definitions: (dimensions quantities)

$\theta = t/\tau$ *and*

$E_\theta = \tau E$

$C_\theta = \tau C$

$\sigma_\theta^2 = \sigma^2 / \tau^2$

Thus, σ_θ^2 can be evaluated from the experimental data on *C vs t* for known values of σ_θ^2 the dispersion number (1/Pe) can be obtained from

(D/uL=1/Pe)

For a closed vessel:

(Dispersion model)

$\sigma_\theta^2 = 2\ (D/uL) - 2(D/uL)^2(1- e^{uL/D})$

Dispersion no= D/uL

If D/uL (the dispersion number) → 0, it corresponding to negligible dispersion, hence, Plug Flow.

If D/uL (the dispersion number) → ∞ , it corresponding to large, hence mixed flow.

D is the axial or longitudinal dispersion coefficient.

For open vessel condition:

$$C_\theta = \frac{1}{2\sqrt{\pi\theta\left(\frac{D}{UL}\right)}} \exp\left[-\frac{(1-\theta)^2}{4\theta\left(\frac{D}{UL}\right)}\right]$$

From the experimental data using a pulse of tracer, we obtain the concentration of the tracer (C_i) Vs the time (t) data.

$E(t)=Ci\ /\ \Sigma\ \Delta t_iC_i$

$\sum C_i$

$\sum C_i\, t_i$

and

$\sum C_i\, t_i^2$

Then calculate:

$\tau = [\Sigma\ t_i C_i / C_i]$

And obtain the corresponding values of E_θ

Plot: C_i vs t_i; E_i vs t_i; and E_θ *vs* θ

$$\sigma^2 = [\Sigma\ t_i^2 C_i \times \Sigma C_i] - \tau^2 = \frac{\sum C_i t_i^2}{\sum C_i} - \left[\frac{\sum C_i t_i}{\sum C_i}\right]^2$$

$$\sigma_\theta^2 = \sigma^2 / \tau^2$$

Using this value of σ_θ, calculate the dispersion Co-efficient, D/uL from Equation 5 by hit and trail method. First neglect the second term on RHS of the equation and obtain the approximate value of D/uL, then Improve upon this value till you get LHS of equation 5 equal to RHS

DESCRIPTION

The setup consists of one feed tank through which water is fed to the reactor. The flow rate can be adjusted by opening the needle valve and measured by rotameter. The compressed air used for circulation of feed. Helical coil tube type plug flow reactor made of stainless-steel pipe is provided. Reactants enter at lower end and exit at the top of coil from where samples are collected for analysis. For understanding the RTD characteristics, a special arrangement to inject tracer into the lower end of reactor, using a syringe is provided. Pressure Regulator & Pressure Gauge are filled at the compressed air line.

UTILITIES REQUIRED

1. Compressed air supply at 2 Bar, 0.25 CFM
2. Water Supply
3. Drain
4. Laboratory Glassware

CHEMICAL REQUIRED

1. 1N Acetic acid

2. N/10 NaOH
3. Phenolphthalein indicator

EXPERIMENTAL PROCEDURE

- Fill the feed tank with water and connect compressed air line to the apparatus.
- Place the syringe containing known amount of 1N Acetic acid at Tracer inlet
- Start the supply of water to the reactor at particular flow rate with the help of rotameter
- Allow water to flow through the PFTR and attain steady state.
- Inject 1N Acetic acid into the system as a pulse signal from the tracer inlet.
- At regular time intervals (say 1 minute), collect the samples at the outlet in pre-marked beakers/measuring cylinders (about 20ml) until all tracer leaves the vessel.
- Analyze these samples with N/10 NaOH using phenolphthalein as indicator.
- Repeat the experiment for different flow rates.

SPECIFICATION

Reactor	:	Material stainless steel, capacity 0.7 Ltrs. (Approx)(helical coiled tube type)
Feed Tank	:	Material stainless steel, capacity 20 Ltrs.
Feed Circulation	:	By compressed air
Flow measurement	:	Rotameter
Piping	:	Material stainless steel and PVC
Pressure Regulator	:	0-2 Kg/cm^2
Pressure Gauge	:	Bourdon type 0-2 Kg/cm^2
Stop watch	:	Electronic

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

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

FORMULAE

1. Concentration of Acetic acid in sample,

$$C_i = \frac{N_1 V_1}{V_2} \times \frac{40}{1000} gm/ml$$

2. Experimental mean residence time,

$$\tau = \frac{\sum C_i t_i}{\sum C_i}$$

3. Theoretical mean residence time,

$$\tau_t = \frac{V_R}{V_o}$$

4. $\sigma_\theta^2 = 2\,(D/uL) - 2(D/uL)^2(1- e^{uL/D})$

OBSERVATION & CALCULATION

For calculating the concentration of Acetic acid in the exit stream, take 10 ml of the solution in titration flask. Add 2-3 drops of phenolphthalein and titrate against N/10 NaOH (taken in a burette).The end point is light pink.

Let the volume of N/10 NaOH used = V_1

Normality of N/10 NaOH used = N_1

Volume of sample taken = V_2

V_1N_1 = V_2N_2

$[V_1\,(N/20)]_{NaOH}$ = $(10\times N_2)_{Acetic\ acid}$

Normality of Acetic acid in the solution, N_2 = $V_1/200$-gram eq/L

Concentration of Acetic acid = $[(V_1/200) \times 40.0]$ gram eq/L

Concentration of Acetic acid at exit = 0.2 V_1 gm eq/L

Time min	Volume of N/20 NaOH used per 10ml of Solution	Conc. Of Acetic acid gm/L, C_i	$t_i \times C_i$	$t_i^2 \times C_i$
		ΣC_i=	$\Sigma\ t_i \times C_i$=	$\Sigma\ t_i^2 \times C_i$=

ΣC_i =

$\Sigma\ t_i \times C_i$ =

$\Sigma\ t_i^2 \times C_i$ =

τ = $[\Sigma\ t_i^2 C_i \times \Sigma C_i]$

σ^2 = $[\Sigma\ t_i^2 C_i \times \Sigma C_i] - \tau^2$

σ_θ^2 = σ^2 / τ^2

using the dispersion model:

σ_θ^2 = $2\ (D/uL) - 2(D/uL)^2(1- e^{uL/D})$

For an initial trial method, neglect the second term on RHS

$2(D/uL)$ = σ_θ^2

By hit and trial method (improve upon the previous values exact values of D/uL is:

The dispersion number D/uL =

Theoretical mean residence time is

τ_t = V_R/V_o, $(L/LPM) \rightarrow$ min

Calculate the experimental mean residence time is

τ = $\Sigma\ t\ E\ \Delta t = \tau = [\Sigma\ t_i C_i \times \Sigma C_i]$

Compare τ_t and τ and discuss your results

To plot RTD curve (E curve)

Time min	C_i, gm/L	$E_i = \frac{\Sigma C_i}{\Delta t}$	$\theta = t/\tau$	$E_\theta = \tau E_i$

Plot E curve, θ *vs* E_θ

NOMENCLATURE

θ	=	reduced time
E	=	activation energy
τ	=	Experimental mean residence time
τ_t	=	theoretical mean residence time
σ_θ	=	variance at time, θ
t	=	time
V_1	=	volume of N/10 Acetic acid
V_R	=	Volume of reactor
V_o	=	Volumetric flow rate
D/UL	=	dispersion number

PRECAUTIONS & MAINTENANCE INSTRUCTIONS

- Always use clean water, good quality chemicals and standard solutions for titration.
- Keep close all the drain valves. Vent valve should open while filling the water in feed tank
- Air pressure should not more than 1 Kg/cm^2
- Flow should not be disturbed during the experiments
- Handle the chemicals carefully
- If any types of suspended particles are in the rotameter, stop the flow, the drain the water tank and reactor. Fill the tank with clean water after proper cleaning of feed tank and reactor.

EXPERIMENT-08

RTD IN MIXED FLOW REACTOR (MFR)

OBJECTIVE:

RTD studies in mixed flow reactor (MFR).

AIM:

- To plot the RTD curve for a MFR using a pulse tracer.
- To determine the dispersion number.

INTRODUCTION:

Real reactors do not satisfy the idealized flow patterns, back mix 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. To predict the exact behavior of a vessel as a chemical reactor, RTD or stimulus response technique is used.

THEORY:

The exit age distribution function of fluid leaving a vessel or RTD of fluid in a vessel is called the E-CURVE. The normalized curve is such that

$\int_0^\infty Edt = 1$ ……. (1)

In stimulus -response experimentation the system is perturbed and then observes how the system reacts or responds to this stimulus. The analysis of the response gives the desired information. A pulse tracer input signal could be used as a stimulus.

The concentration – time curve for pulse signal at the vessel outlet is called the C-CURVE. Considering steady-state flow of fluid through a closed vessel:

C = E ……... (2)

The mean age of the exit stream or mean residence time is:

$\tau = \int_0^\infty tEdt = \sum tE\Delta t$ ……... (3)

$\tau = \frac{\sum(t_i C_i)}{\sum C_i}$ …… (4)

And the variance of the E or C distribution is

$$\sigma_t^2 = \int_0^\infty t^2\, E dt - \tau^2 = \sum t^2\, E \Delta t - \tau^2 \qquad \text{……. (5)}$$

$$\sigma_t^2 = \frac{\sum(t_i^2 C_i)}{\sum C_i} - \left[\frac{\sum(t_i C_i)}{\sum C_i}\right]^2 \qquad \text{…….. (6)}$$

Variance, or a measure of the spread of the curve at time θ.

$$\sigma_\theta^2 = \frac{\sigma_t^2}{\tau^2} \qquad \text{……. (7)}$$

Models are useful for representing flow in real vessels, for scale up, and for diagnosing poor flow. We have different kind of models depending on whether flow is close to plug, mixed, or somewhere in between. For small deviations from plug flow dispersion model is used. Suppose an ideal pulse of tracer is introduced into the fluid entering a vessel. The pulse spreads as it passes through the vessel, and to characterize the spreading this model, we assume a diffusion like process superimposed on plug flow. We call this dispersion, the dispersion coefficient D represents the spreading process. (D/uL) is the dimensionless group characterizing the spread in the whole vessel.

For closed vessel

$$\sigma_\theta^2 = 2\left(\frac{D}{uL}\right) - 2\left(\frac{D}{uL}\right)^2 \left[1 - e^{-\frac{uL}{D}}\right] \qquad \text{…… (8)}$$

Ignoring the second term of the above equation we get

$$\frac{D}{uL} = \frac{\sigma_\theta^2}{2} \qquad \text{……. (9)}$$

Defining the reduced time as:

$$\theta = \frac{t}{\tau} \qquad \text{……. (10)}$$

Exit age distribution at time i

$$E_i = \frac{C_i}{\sum C_i \Delta t} \qquad \text{…….. (11)}$$

Exit age distribution at time i

$$E_\theta = \tau \times E_i \qquad \text{…… (12)}$$

Plot a graph between θ vs E_θ.

DESCRIPTION:

The setup consists of one feed tank through which water is fed to the reactor. The flow rate can be adjusted by the needle valve and measured by rotameter. The compressed air is used for circulation of feed. The continuous stirred tank reactor made of stainless steel is provided for understanding the RTD characteristics. A pipette is used for dozing the tracer into the C.S.T.R. pressure regulator & pressure gauge re fitted at the compressed air line.

UTILITIES REQUIRED:

Electricity supply: Single phase, 220 V AC, 50Hz, 5-15 Amp. Combined socket with earth connection.

Compressed air supply: 0.25 CFM at 1 Bar.

Water supply (Initial fill).

Floor drain required.

Laboratory glassware required: -

Burette (50 ml) : 01 No.
Conical flasks (250 ml) : 01 No.
Pipette (20 ml) : 01 No.
Measuring cylinder (250 ml) : 01 No.

Chemicals: -

N/10 NaOH : 200 ml
Concentrated Acetic acid : 100 ml
Phenolphthalein indicator: Few drops

EXPERIMENTAL PROCEDURE:

STARTING PROCEDURE:

- Close all the valves V_1-V_6.
- Open the valve V_3-V_4 and fill the feed tank with water.
- Connect compressed air supply to the set up at valve V_1.
- Connect electric supply to the set up.
- Open valve V_1 and set air pressure 0.5 to 1 kg/cm^2 by pressure regulator and pressure gauge.
- Start the supply of water to the reactor at particular flow rate with the help of valve V_2.
- Fill N/10 sodium hydroxide in burette.
- Switch ON the stirrer and wait till the water comes out from the outlet.
- Fill the concentrated Acetic acid (10 ml or 20 ml) in the pipette.
- Input concentrated Acetic acid into the system with the help of pipette.
- At regular time intervals (say 30 sec for high water rate, 1min for low water rate), collect 20 ml sample from the outlet in measuring cylinder.
- Transfer the sample solution in conical flask.
- Titrate the sample solution, using phenolphthalein as an indicator against N/10 sodium hydroxide (add NaOH from burette).
- Repeat the experiment for different flow rates (before changing the flow rate, drain the reactor first).

CLOSING PROCEDURE:

- When experiment is over stop the flow of water by close the valve V_2.
- Switch OFF the stirrer.
- Drain the reactor and feed tank by open the valve V_6-V_5.

OBSERVATION & CALCULATION:

DATA:
Working volume of reactor V_R = _____L
Volume of sample V_2 = 20 ml
Normality of NaOH used for titration N_1 = 0.1 g eq/L

OBSERVATION TABLE:			
S. No.	t_i (min)	V_1 (ml)	V_0 (LPH)

CALCULATIONS:

$N_2 = \frac{V_1 N_1}{V_2}$ (g eq/L)

$C_i = \frac{N_2}{2}$ (mol/L)

$\tau_t = \frac{V_R * 60}{V_0}$ (min)

$\Delta t = t_i - t_{i-1}$ (min)

CALCULATION TABLE:1						
S. No.	t_i (min)	C_i (mol/lit)	t_iC_i	$t_i^2C_i$	Δt (min)	$C_i\Delta t$
		$\sum C_i$	$\sum t_iC_i$	$\sum t_i^2C_i$		

$\tau = \frac{\sum t_iC_i}{\sum C_i}$ (min)

$$\sigma_t^2 = \left(\frac{\sum t_i^2C_i}{\sum C_i}\right) - \tau^2$$

$$\tau_\theta^2 = \frac{\sigma_t^2}{\tau^2}$$

$$\frac{D}{uL} = \frac{\sigma_\theta^2}{2}$$

$$E_i = \frac{C_i}{\sum C_i\,\Delta t}$$

$$\theta = \frac{t}{\tau}$$

$$E_\theta = \tau \times E_i$$

CALCULATION TABLE:2		
θ	E_i	E_θ

Plot a graph between θ vsE_θ.

NOMENCLATURE:

Nom	Column Heading	Units	Type
C_i	Concentration of H_2SO_4 in sample	mole/L	Calculated
D/uL	Dispersion number	*	Calculated
E_i	Exit age distribution at time i	*	Calculated
E_θ	Exit age distribution at time θ	*	Calculated
N_1	Normality of NaOH used for titration	g eq/L	Given
N_2	Normality of H_2SO_4 in sample solution	g eq/L	Calculated
t_i	Time	min	Measured
V_1	Volume of NaOH used for titration	ml	Measured
V_2	Volume of sample	ml	Given
V_0	Volumetric flow rate	LPH	Measured

V_R	Working volume of reactor	Lit	Given
T	Experimental mean residence time	min	Calculated
τ_t	Theoretical mean residence time	min	Calculated
Θ	Reduced time	*	Calculated
σ_θ	Variance at time θ	*	Calculated
σ_t	Variance at time t	min_2	Calculated
Δt	Average time difference	min	Calculated

PRECAUTION & MAINTENANCE INSTRUCTIONS:

- Always use distilled water, good quality chemicals and standard solution for titration.
- Keep close all the drain valves V_5-V_6, and vent valve V_4 should be open while filling water in the feed tanks.
- Air pressure must be set below 1 kg/ cm^2.
- Flow should not be disturbed during the experiments.
- Handle the chemicals carefully.

TROUBLESHOOTING:

- If any type of suspended particles are come in the rotameter, remove the rotameter clean the tube and fit it at its place.
- If there is any leakage tight that part or fix that again after wrapping Teflon tape.
- If rotameter fluctuating more than average tight the control knob properly.

REFERENCES:

Levenspiel. Octave (2001). Chemical Reaction Engineering. 3rd Ed. NY: John Wiley & Sons. Pp 293 – 294, 299-301, 305.

Fogler H. Scoot (2008). Elements of Chemical Reaction Engineering. 4th Ed. ND: Prentice-Hall of India Pvt. Ltd. Pp 871-873,879,887-888.

EXPERIMENT-09

RTD IN PACKEDBED REACTOR (PBR)

OBJECTIVE:

Study of a Non-Catalytic Homogeneous reaction in Packed Bed Reactor.

AIM:

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

INTRODUCTION:

A packed-bed reactor shown in figure. When a reactor ispacked with catalyst, the reacting fluid usually does not flow through the reactor uniformly. Rather, there may be sections in the packedbed that offer littleresistance to flow, and as a result a major portion of the fluid may channelthrough this pathway. Consequently, the molecules following this pathway donot spend as muchtime in the reactor as those flowing through the regions ofhigh resistance to flow. We see thatthere is a distribution of times that molecules spend in the reactor in contact with the catalyst.

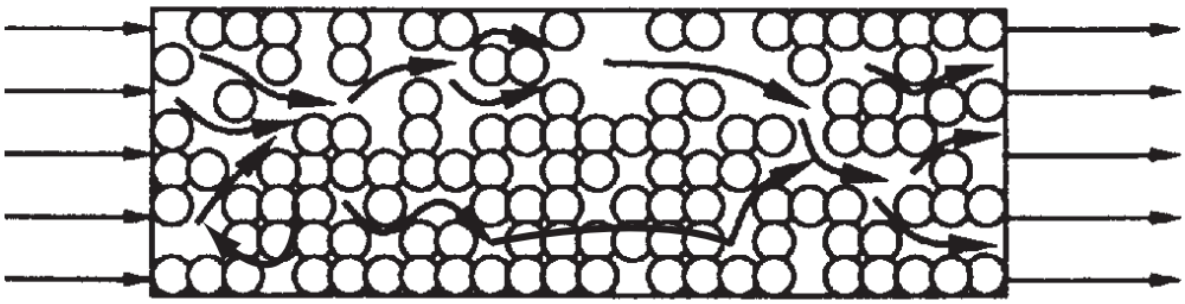


Figure. Packed-bed reactor.

RTD in Batch and Plug-Flow reactors

The RTDs in plug-flow reactors and ideal batch reactors are the simplest toconsider. All the atoms leaving such reactors have spent precisely the sameamount of time within the reactors.The distribution function in such a case isa spike of infinite height and zero width, whose area isequal to 1; the spikeoccurs at t = V/ν, or Θ = 1. Mathematically, this spike is represented by the Dirac delta function:

$$E(t) = \delta\,(t - \tau)$$

The Dirac delta function has the following properties:

$$\delta(x) = \begin{cases} 0 & when\ x \neq 0 \\ \infty & when\ x = 0 \end{cases}$$

$$\int_{-\infty}^{\infty} \delta(x)dx = 1$$

$$\int_{-\infty}^{\infty} g(x)\delta(x-\tau)dx = g(\tau)$$

To calculate τ the mean residence time, we get g(x) = t

$$t_m = \int_0^{\infty} tE(t)dt = \int_0^{\infty} t\delta(t-\tau)dt = \tau$$

But we already knew this result. To calculate the variance we set, $g(t) = (t - \tau)^2$, and the variance σ^2, is

$$\sigma^2 = \int_0^{\infty} (t-\tau)^2\, \delta(t-\tau)dt = 0$$

All material spends exactly a time τ in the reactor, there is no variance.

The cumulative distribution function F(t) is

$$F(t) = \int_0^{t} E(t)dt = \int_0^{t} \delta(t-\tau)dt$$

The E(t) function is shown in figure (a), and F(t) is shown in figure (b).

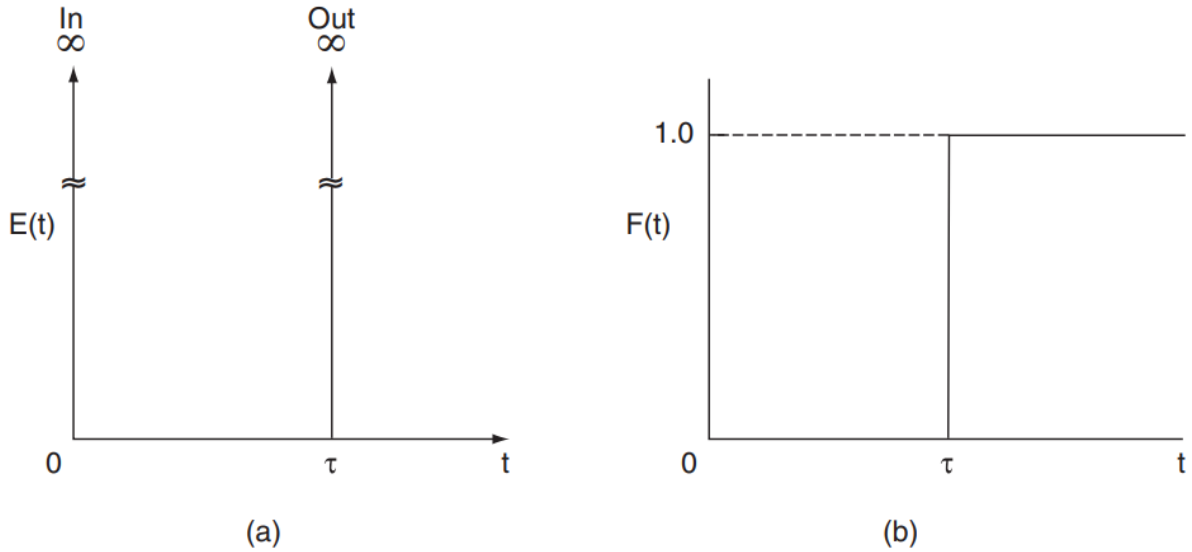


Figure. Ideal plug-flow response to a pulse tracer input.

RTD in CSTR

In an ideal CSTR the concentration of any substance in the effluent stream isidentical to the concentration throughout the reactor. Consequently, it is possible to obtain the RTD fromconceptual considerations in a fairly straightforward manner. A material balance on an inerttracer that has been injected as apulse at time t = 0 into a CSTR yields for t >0

$$\text{In} - \text{Out} = \text{Accumulation}$$

$$0 - \nu C = V\frac{dC}{dt}$$

Because the reactor is perfectly mixed, C in this equation is the concentrationof the tracer eitherin the effluent or within the reactor. Separating the variablesand integrating with $C = C_0$ at t = 0 yields

$$C(t) = C_0 e^{-t/\tau}$$

This relationship gives the concentration of tracer in the effluent at any time t.

To find E(t) for an ideal CSTR,

$$E(t) = \frac{C(t)}{\int_0^\infty C(t)dt} = \frac{C_0 e^{-t/\tau}}{\int_0^\infty C_0 e^{-t/\tau}\,dt} = \frac{e^{-t/\tau}}{\tau}$$

Evaluating the integral in the denominator completes the derivation of the RTDfor an ideal CSTR given by Equations,

$$E(t) = \frac{e^{-t/\tau}}{\tau}$$

$$E(\Theta) = e^{-\Theta}$$

Recall $\Theta = t/\tau$ and $E(\Theta) = \tau E(t)$.

Response of an ideal CSTR

$E(\Theta) = e^{-\Theta}$
$F(\Theta) = 1 - e^{-\Theta}$

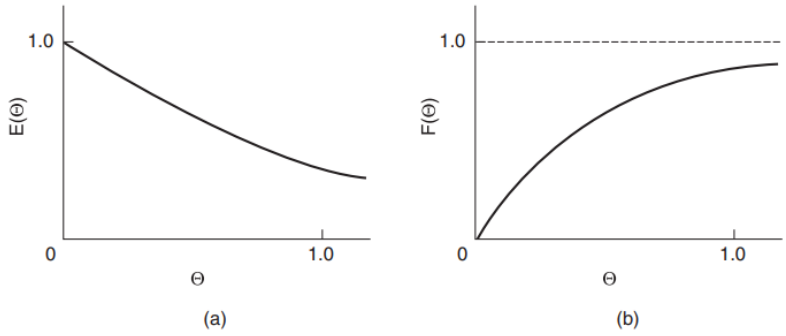


Figure. E(Θ) and F(Θ) for an Ideal CSTR.

The cumulative distribution F(Θ) is

$$F(\Theta) = \int_0^\Theta E(\Theta)\,d(\Theta) = 1 - e^{-\Theta}$$

The E(Θ) and F(Θ) functions for an Ideal CSTR respectively.

Earlier it was shown that for a constant volumetric flow rate, the mean residence time in a reactor is equal to V/ν, or τ. This relationship can be shown in a simpler fashion for the CSTR. Applying the definition of the mean residence time to the RTD for a CSTR, we obtain

$$t_m = \int_0^\infty tE(t)dt = \int_0^\infty \frac{1}{\tau} e^{-t/\tau} dt = \tau$$

Thus, the nominal holding time (space time) τ = V/ is also the mean residence time that the material spends in the reactor.

The second moment about the mean is a measure of the spread of the distribution about the mean. The variance of residence times in a perfectly mixed tank reactor is (let x = t/τ)

$$\sigma^2 = \int_0^\infty \frac{(t-\tau)^2}{\tau} e^{-t/\tau} dt = \tau^2 \int_0^\infty (x-1)^2 e^{-x} dx = \tau^2$$

Then σ = τ. The standard deviation is the square root of the variance. For a CSTR, the standard deviation of the residence-time distribution is as large as the mean itself!!

UTILITIES REQUIRED

1. Compressed air supply at 2 Bar,0.25CFM
2. Water Supply
3. Drain
4. Laboratory Glassware

CHEMICAL REQUIRED

5. 1N Acetic acid
6. N/10 NaOH
7. Phenolphthalein indicator

EXPERIMENTAL PROCEDURE

- Fill the feed tank with water and connect compressed air line to the apparatus.
- Place the syringe containing known amount of 1N Acetic acid at Tracer inlet
- Start the supply of water to the reactor at particular flow rate with the help of rotameter
- Allow water to flow through the PFTR and attain steady state.
- Inject 1N Acetic acid into the system as a pulse signal from the tracer inlet.

- At regular time intervals (say 1 minute), collect the samples at the outlet in pre-marked beakers/measuring cylinders (about 20ml) until all tracer leaves the vessel.
- Analyze these samples with N/10 NaOH using phenolphthalein as indicator.
- Repeat the experiment for different flow rates.

SPECIFICATION

Reactor		:Material stainless steel, capacity 0.7 Ltrs. (Approx.)(helical coiled tube type)
Feed Tank	:	Material stainless steel, capacity 20 Ltrs.
Feed Circulation	:	By compressed air
Flow measurement	:	Rotameter
Piping	:	Material stainless steel and PVC
Pressure Regulator	:	0-2 Kg/cm^2
Pressure Gauge	:	Bourdon type 0-2 Kg/cm^2
Stop watch	:	Electronic

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

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

FORMULAE

1. Concentration of NaOH in sample,

$$C_i = \frac{N_1 V_1}{V_2} \times \frac{40}{1000} gm/ml$$

2. Experimental mean residence time,

$$\tau = \frac{\sum C_i t_i}{\sum C_i}$$

3. Theoretical mean residence time,

$$\tau_t = \frac{V_R}{V_o}$$

4. $\sigma_\theta^2 = 2\,(D/uL) - 2(D/uL)^2(1 - e^{uL/D})$

OBSERVATION & CALCULATION

For calculating the concentration of Acetic acid in the exit stream, take 10 ml of the solution in titration flask. Add 2-3 drops of phenolphthalein and titrate against N/10 NaOH (taken in a burette).The end point is light pink.

Let the volume of N/10 NaOH used = V_1

Normality of N/10 NaOH used = N_1

Volume of sample taken = V_2

V_1N_1 = V_2N_2

$[V_1 (N/20)]_{NaOH}$ = $(10\times N_2)_{Acetic\ acid}$

Normality of Acetic acid in the solution, N_2 = $V_1/200$-gram eq/L

Concentration of Acetic acid = $[(V_1/200) \times 40.0]$ gram eq/L

Concentration of Acetic acid at exit = $0.2\ V_1$ gm eq/L

Time min	Volume of N/10 NaOH used per 10ml of Solution	Conc. Of Acetic acid gm/L, C_i	$t_i \times C_i$	$t_i^2 \times C_i$
		ΣC_i=	$\Sigma\ t_i \times C_i$=	$\Sigma\ t_i^2 \times C_i$=

ΣC_i =

$\Sigma\ t_i \times C_i$ =

$\Sigma\ t_i^2 \times C_i$ =

τ = $[\Sigma\ t_i^2 C_i \times \Sigma C_i]$

σ^2 = $[\Sigma\ t_i^2 C_i \times \Sigma C_i] - \tau^2$

σ_θ^2 = σ^2 / τ^2

using the dispersion model:

σ_θ^2 = $2\ (D/uL) - 2(D/uL)^2(1- e^{uL/D})$

For an initial trial method, neglect the second term on RHS

$2(D/uL) = \sigma_\theta^2$

By hit and trial method (improve upon the previous values exact values of D/uL is:

The dispersion number D/uL =

Theoretical mean residence time is

$\tau_t = V_R/V_o$, (L/LPM) → min

Calculate the experimental mean residence time is

$\tau = \Sigma\ t\ E\ \Delta t = \tau = [\Sigma\ t_i C_i \times \Sigma C_i]$

Compare τ_t and τ and discuss your results

To plot RTD curve (E curve)

Time min	C_i, gm/L	$E_i = \Sigma C_i \Delta t$	$\theta = t/\tau$	$E_\theta = \tau E_i$

Plot E curve, θ vs E_θ

NOMENCLATURE

θ	=	reduced time
E	=	activation energy
τ	=	Experimental mean residence time
τ_t	=	theoretical mean residence time
σ_θ	=	variance at time, θ
t	=	time
V_1	=	volume of N/10 NaOH
V_R	=	Volume of reactor
V_o	=	Volumetric flow rate
D/UL	=	dispersion number

PRECAUTIONS & MAINTENANCE INSTRUCTIONS

- Always use clean water, good quality chemicals and standard solutions for titration.
- Keep close all the drain valves. Vent valve should open while filling the water in feed tank
- Air pressure should not more than 1 Kg/cm^2
- Flow should not be disturbed during the experiments
- Handle the chemicals carefully
- If any types of suspended particles are in the rotameter, stop the flow, the drain the water tank and reactor. Fill the tank with clean water after proper cleaning of feed tank and reactor.

EXPERIMENT-10

RTD IN MIXED FLOW REACTORS IN SERIES

AIM: To determine the non-ideality of reactor through its dispersion number and comparison of residence time distribution with ideal reactors.

APPARATUS: Reactor vessels with stirrer, conical flasks, Burette, Pipette, test tubes.

CHEMICALS REQUIRED: NaOH solution, Ethyl Acetate, HCl, Phenolpthaline indicator

THEORY: The residence time distribution for N continuous stirred tank reactors in series is given by

$$E(t) = \frac{t^{N-1}}{(N-1)!\tau_i^{N}} e^{-\left(\frac{t}{\tau_i}\right)}$$

EXPERIMENTAL SET-UP DATA:

The experimental set up 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 = 122 mm

Height of the liquid in the tank = 175 mm

Agitation speed range = Variable Speed (0-300) RPM (apprx).

Fluid Flow measurement range = (2.0 – 20 LPH) (Rotameter)

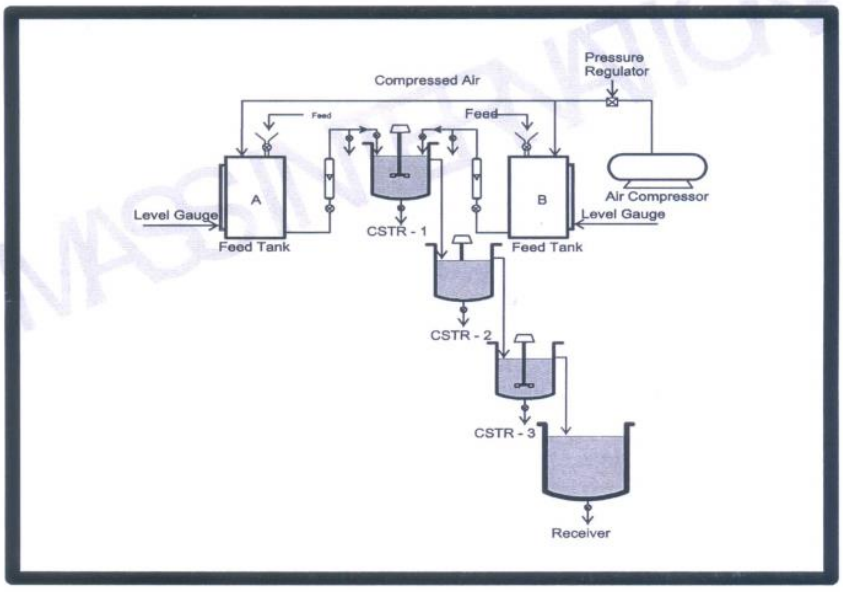


Figure: CSTRs in series set up.

PROCEDURE:

1. Standardize the NaOH solution using Oxalic acid.
2. Switch on the stirrer
3. Adjust some flow rate of water to the reactor and allow the system for steady state.
4. After the system reaches steady state, note down the flow rate of stream into the reactor.
5. Inject Glacial acetic acid as a tracer.
6. Collect 10 ml sample from exit stream for each 1min time interval.
7. Titrate each sample against NaOH, and note down the volume of NaOH rundown.
8. Collect the samples till there is no acetic acid present in the exit stream.
9. Note down the volume of reactor.

OBSERVATIONS AND CALCULATIONS:

Standardization of NaOH:

Volume of Oxalic acid taken (V_1) = ml

Normality of Oxalic acid taken (N_1) = N

Volume of NaOH rundown for neutralization = V = ml.

Time, t min	Volume of NaOH Rundown V_{NaOH}

Volume of Sample collected from exit stream = 10 ml.

Volume of mixed flow reactor = V_M = lt.

Volumetric flow rate to the reactor V_O = LPH.

Calculations:

Normality of NaOH = $N_{NaOH} = \frac{V_1 N_1}{V}$

Concentration of Acetic acid in the exit stream at time t, $C(t) = \frac{V_{NaOH} N_{NaOH}}{10}$

Plot a graph C(t) Vs t

Q = $\int_0^\infty C(t)dt$ = Area under the Curve C(t) Vs t

The Exit age distribution function at time *t*, *E(t)* = $\frac{C(t)}{Q}$

Plot a graph of E(t) Vs t

The mean Residence time, $\bar{t} = \int_0^\infty tE(t)dt$

Plot a graph of tE(t) Vs t

$\bar{t} = \int_0^\infty tE(t)dt$ = Area under the curve of *tE(t) Vs t*

The ***variance***, $\sigma^2 = \int_0^\infty \left(t - \bar{t}\right)^2 E(t)dt$

Plot a graph of $\left(t - \bar{t}\right)^2 E(t)$ *Vs t*

$\sigma^2 = \int_0^\infty \left(t - \bar{t}\right)^2 E(t)dt$ = Area under the Curve $\left(t - \bar{t}\right)^2 E(t)$ Vs t

Calculate, $\sigma_\theta^{\ 2} = \frac{\sigma^2}{\bar{t}^2}$

Number of ideal equal to this variance is given by

$N = 1/\sigma_\theta^2$

Calculate *E(t)* theoretical by

Residence time in the mixed flow reactor $\tau_i = \frac{V_M}{V_O} x60$ min.

N = 3.

$$E(t) = \frac{t^{N-1}}{(N-1)!\tau_i^{\ N}} e^{-\left(\frac{t}{\tau_i}\right)}$$

Time	*C(t)*	*E(t)*	*t E(t)*	$t^2 E(t)$	$E(t)_{ideal}$

RESULT:

1. Number of ideal equal to this variance is _____.
2. The experimental Exit age distribution is compared with ideal distribution.

PRECAUTIONS:

While filling the supply (feed) tanks, care should be taken that valve of compressed and vent is open.

If supply tank is to be refilled, care should be taken that compressed air valve should remain closed and vent should be opened slowly to avoid spraying of chemical present in the tank. Refilling should be started only when pressure of the tank is released.

Rotameters should be periodically monitored to ensure constant flow rated through them.

After completion of experiment, chemicals should be removed from the feed tank and release the pressure.

All the solutions should be prepared accurately and must be standardized.

Titrations should be carried out precisely 2-3 concordant readings should always be taken.

All the glass wares to be used should be properly rinsed prior to use.

CONCLUSIONS:

Write down the points you have concluded from this experiment.

REFERENCE:

Levenspil, O., "Chemical Reaction Engineering," 2nd edn., John Wiley & Sons, Singapore, 1995, pp. 134-137.