



An-Najah National University
Department of Chemical Engineering
(Nablus/Palestine)

Chemical Reactor Engineering Lab, 64428

Instructor:

Dr. ◌Amer EL-Hamouz

Eng. Maha Fuqha

January, 2011

An-Najah National University
Department of Chemical Engineering
Chemical Reactor Engineering Lab, 64428.
Second term 2011

Prerequisites: Chemical Reaction Engineering, Numerical Analysis for Chemical Engineers

Text Book: Prepared Lab Manual

References: 1- Elements of Chemical Reaction Engineering, Fourth edition, 2004,
ISBN 0-13-253220-4
H.Scott Fogler
2- Chemical Reaction Engineering, second edition, 1999,
ISBN 0 471-53019-0
O. Levenspiel

Instructors: Amer M. EL-Hamouz, Associate Professor of Chemical Engineering
Engineer Maha Fuqha, Teaching Assistant

Introduction: This lab is one of the most important labs in the chemical engineering study. In this lab, student will perform experiments to support their theoretical study of Batch, CSTR and PFR reactors.

Objectives:

- 1- To familiarize students with main type of chemical reactors.
- 2- To analyze the experimental data to obtain the reaction rate expression (reaction order and specific reaction rate constant).
- 3- Compare results obtained using different concentration measurement techniques.
- 4- Compare the conversion of reactants during a specification reaction in various types of reactor.
- 5- Observe batch mixing under different operating conditions.

Outline

Part A: Manual measurement of species concentration.

- Safety in Reaction lab.
- Homogeneous Batch Reactor
- Neutralization of Aqueous Solution Of Acetic Acid
- Mixing Behavior under Various Operating Conditions
- Scale up rules in the mixing tank.
- Effect of solution viscosity on mixing behavior in stirred tank reactors
- Mixing degree Measurements using spectrophotometer.

- Plug flow reactor

Part B: Chemical Reactor trainer

- Batch Reactor, Adiabatic Reaction
- Batch Reactor, Isothermal Reaction
- Continuous Stirrer Tank Reactor
- Tubular Flow Reactor
- Stirred Tanks in Series

Comments

- ALL STUDENTS ARE REQUESTED TO ATTEND ALL EXPERIMENTS.
- One formal report should be submitted by each group.
- Final exam will be written exam

Grading :

Quizzes	5%
performance	10%
Reports	35%
Final Exam	50%

An-Najah National University
Department of Chemical Engineering
Chemical Reactor Engineering Lab, 64428.
Second term 2011

Instructions for Preparing Laboratory Reports

The report must be prepared according to the following outline.

1. Title Page

The Title page should be separate from the rest of the report. It should contain:

- a. The title of the experiment.
- b. The number of course.
- c. The names of the writer and his co-worker(s), their ID numbers, and group number.
- d. Name of the instructor to whom the report is submitted
- e. The date when the experiment was run
- f. The date of submission of report

A sample title page is shown on page xv, which can be used when submitting reports.

2. Abstract

The abstract should be informative, and should be written in about three to five sentences.

It should cover all phases of the investigation. It must include the following:

- a. An introductory statement about the subject matter
- b. Brief description of what was done and how it was done.
- c. Selected results (numerical values, if available).
- d. Brief assessment of the results e.g. some percentage errors in
Experimental results in comparison with theoretical values.

While writing the abstract, no reference should be made to graphs, tables or equations inside the report.

3. Introduction

This section should include few sentences discussing the physical and/or chemical principles involved in the experiment. The importance and relevance of the experiment to real applications may also be stated.

4. Theoretical Background

This section should include the theory behind the experiment. It should also contain all those equations, which are used to acquire a certain result. Theoretical correlations, which are used for comparison with experimental results, should also be included.

5. Procedure

Here, you should briefly describe the actual step-by-step procedure you followed in running the experiment. It should be written in your own words, e.g. the needle valve was manipulated in order to adjust the liquid flow rate.

6. Results

The results should be presented in the form of Tables or graphs. The Table should contain the results obtained from experiments and from theoretical knowledge.

Comparisons may also be presented in terms of percent deviation between experimental values and theoretical predictions.

7. Discussion of Results

In this section you should discuss your experimental results and observations. If the results are obtained in terms of graphs, then interpret them also. Describe observed trends and possible relationship between parameters e.g. how a change in one variable affects another. Also show how you make comparison with the values obtained theoretically and discuss the deviation of experimental results from theoretical values. The possible source of errors should also be mentioned.

8. Conclusions and Recommendations

Conclusions are the series of numbered sentences which answer the questions posed at the beginning of each experiments. Conclusions should also include main results (numerical values) and the errors between the experimental and theoretical values. What you have learned from the experiment should be mentioned as well.

Recommendations are the proposals for future work, e.g. suggested changes in equipment, Study of new variables, or possible experiments in relative fields. Like the conclusions, the recommendations are usually listed by numbers, and each consists of only a sentence or two.

9. Literature Cited

Here, you should list the books, Journal's articles, etc. used in writing your report and analyzing the experiment. The reference should be completed (name of the book, author, volume, date of publication, pages, etc.). References should be arranged alphabetically by author's names.

10. Nomenclature

The symbols, which are used in the report, should be defined in the nomenclature in alphabetical order. The accompanying definitions must include proper units.

Appendices

All appendices and graphs should be attached at the end of the report.

A1. Raw Data:

It should contain the raw data (in the log sheet) collected during the experiment.

A2. Analysis of Data and Sample Calculations:

This shows how the collected experimental data are analyzed and transformed into experimental results by using the appropriate equations. Also, how the theoretical results are obtained using theoretical in terms of percentage error. Sample calculations should contain each step, which is used to acquire certain results.

Organization and Neatness

The students must organize their reports in accordance with the format described earlier. It is required/encouraged to use computer packages such as EXCEL, SIGMAPLOT, HARVARD, GRAPHICS or any other suitable engineering software to draw graphs. The neatness will include how the student has organized his report, neat handwriting, accuracy in grammar and spelling, numbering pages, figures, tables and equations. Additional points will be given to those students who use computers in preparing their reports.

An-Najah National University
Department of Chemical Engineering
Chemical Reactor Engineering Lab, 64428.
Second term 2008-2009

Report Grading Form

Name of Student: _____ ID #: _____

Title of Report: _____

Term: _____ Date: _____

Subject	Max Mark	Actual Mark
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1- Title Page	2.	
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2- Abstract	20	
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3- Introduction	5	
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4- Procedure	5	
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5. Results	15	
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6. Discussion of Results	20	
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7. Conclusion and Recommendations	8	
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8. Literature Cited	5	
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9. Nomenclature	2	
-----------------	---	--

10. Organization and Neatness	4	
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Appendix

A1 Raw Data	4	
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A2 Data Analysis and Sample Calculation	10	
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Total	100	
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Signed: _____

Comments:

Sample Cover Page

Kinetics Reaction Laboratory

Homogeneous Batch Reactor

(Experiment no. 1, Performed on: February 20, 2008)

Prepared by: Naser Jaber

Group members: N. Jaber

A. Daqa ✓

M. Badran ✓

Submitted to: Eng. Maha Faqha

Chemical Engineering Department

An-Najah National University

February 26, 2009

III. Criteria for Evaluating Laboratory Reports and Grading Policy

TECHNICALFORMAT

Abstract

Does it stand alone?

Is it understandable?

Does it include a summary of the following- objective, introduction, Theory, Methods and Conclusions?

Objective

Is the objective stated clearly and concisely?

Introduction

Does the introduction explain the relevance of the experiment to the field of engineering?

Theory

Is the theory explained?

Are all the necessary formulas stated and variables defined?

Experimental Methods

Can the experiment be reconstructed from the description given?

Is there a diagram of the experimental set-up?

Results

Is presentation clear and concise? Are all the relevant Tables, Graphs, explanations included? Are the pertinent Sample calculations, References, etc. included in the Appendices?

Conclusion

Are the conclusions derived from the results of the experiment? Were there any discrepancies from expected results? Is the objective of the experiment accomplished?

WRITING QUALITY

Does the report follow the prescribed format?

Is the language of the report easy to read? Is it precise? Does it conform to the conventional standards?

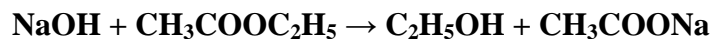
PART 1

Experiment # 1

HOMOGENEOUS BATCH REACTOR

1.1 Objective

To determine the order and value of the rate constant for the liquid reaction of caustic soda and ethyl acetate in a batch reactor:



1.2 Introduction

A batch reactor may be described as a vessel in which chemicals are placed to react. Batch reactors are normally used in small-scale laboratory set-ups to study the kinetics of chemical reactions.

To determine the order and constant of a chemical reaction, the variation of a property of the reaction mixture is observed as the reaction progresses. Data collected usually consist of changes in variables such as concentration of a component, total volume of the system or a physical property like electrical conductivity or refractive index.

The data are then analyzed using pertinent equations to find desired kinetic parameters.

1.3 Theory

For any given reaction in a constant volume system, the rate of the reaction can be represented by:

$$-r_A = kf(C_A) = -\frac{dC_A}{dt} \quad (1.1)$$

Where r_A is the rate of disappearance of reactant A among the reacting species. Equation (1) can be rearranged to give:

$$-\frac{dC_A}{f(C_A)} = kdt \quad (1.2)$$

Integrating equation (1.2) analytically yields

$$-\int_{C_{A_0}}^{C_{A_f}} \frac{dC_A}{f(C_A)} = k \int_0^t dt = kt \quad (1.3)$$

By postulating various forms for $f(C_A)$ in equation (1.3) and correlating the resulting equation with the experimental data, the rate constant k , and order of the reaction can be determined.

1.4 Procedure

1. In the reactor, mix 1.0 liter of the 0.1M Caustic Soda (NaOH) solution with 1.0 liter of the 0.1M ethyl acetate solution at an arbitrary time ($t = 0$) at room temperature. Start the stirrer immediately at an intermediate speed to avoid splashing.
2. After a certain time interval, using a flask or graduated cylinder withdraw a sample of known size (e.g., 25 ml) from the opening in the base of the reactor, and immediately quench this sample with an excess of 0.05M Hydrochloric Acid (HCl) (e.g. 25 ml).
3. Back titrate the quenched sample with 0.05M NaOH solution. The required amount of 0.05M NaOH should be recorded since this will enable you to determine the concentration of NaOH in the reactor at the time you withdrew the sample.
4. Note that a drop or two of phenolphthalein should be added to the quenched sample to detect the end point (i.e., stable new color) before you back titrate. Stirring the flask is also required during back titration.
5. Repeat steps (2) and (3) every 5-10 minutes (and record the time for each new sample) until the amount of NaOH needed in the back titration is nearly equal to the amount of the quenching acid.

Note 1

Make sure to withdraw and analyze at least five samples of the reactor contents during the first 15 minutes of the reaction. To accomplish this it is recommended that you prepare known quantities (25 ml) of the acid indifferent flasks before the start of the reaction. You can then withdraw the samples from the reactor quench, back titrate, etc.

1.5 Report Requirement

1. Calculate the number of moles of unreacted NaOH in each sample withdrawn.
2. Calculate the concentration of unreacted NaOH (mol/lit).
3. Apply the integral method of analysis to determine the reaction order and rate constant of the reaction. This should involve assuming an expression for the reaction rate, substituting into equation 3, integrating, and then plotting.
4. Calculate the half-life of NaOH.
5. Determine the fractional conversion of NaOH at the end of the reaction.
6. Find both the order and rate constant of the reaction from literature and compare between the theoretical and experimental values and give reasons for the deviations.

Note 2

You must give the exact reference i.e author, title and page number of the book from which values were obtained.

1.6 References

1. Levenspiel, O., "Chemical Reaction Engineering", 2nd ed., Wiley and sons, N.Y., p.41 (1977).
2. Smith J.M., " Chemical Engineering Kinetics", 3rd ed., McGraw-Hill Book Comp. N.Y., p.37 (1981).

HOMOGENEOUS BATCH REACTOR

Raw Data

Group Name -----

Temperature :----- °C

Table 1.1Experimental Data

Sample #	Time (min)	Sample Vol. (ml)	Volume of HCl Used In Quenching (ml)	Volume NaOH Used In Titration (ml)

Signature-----

Date-----

Experiment # 2

NEUTRALIZATION OF AQUEOUS SOLUTION OF ACETIC ACID

2.1 Objective

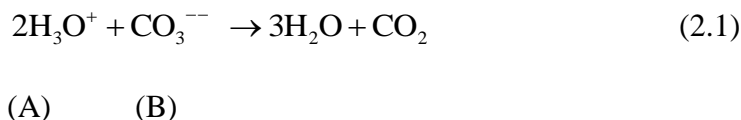
To determine the order of the reaction and the value of combined mass transfer and kinetic rate constant.

2.2 Introduction

Dilute acetic acid (such as vinegar) with a pH of around 3.0 is a reasonably effective and inexpensive of the weak organic acids present in human body. This acid may then be employed for simple laboratory simulation of the biochemical processes associated with neutralization by commercial antacids used to relieve gastric distress. The experiment is based on the analysis of combined mass transfer and reaction kinetics.

2.3 Theory

The overall ionic reaction for the active acid neutralization system is:



This expression is dependent upon the active ingredient.

It is assumed that the instantaneous rate of the overall reaction, measured as the rate of disappearance of the hydronium ion is proportional to the instantaneous remaining surface area of the tablets and the hydronium ion concentration, with the order for the latter as yet unspecified. Thus

$$\frac{1}{V} \frac{dN_A}{dt} = -K_a C_A^n \quad (2.2)$$

The area of a given tablet (disc) is the sum of its two faces plus its edges or

$2\pi r^2 + 2\pi rh$. Assuming that the aspect ratio ($\alpha = \text{height} / \text{radius}$) of a given tablet remains constant throughout the dissolution process, the instantaneous surface area of a tablet is $2\pi r^2 (1 + \alpha)$. Eq. (2.2) then becomes, after assuming constant reaction volume (V).

$$\frac{dC_A}{dt} = -2\pi K m r^2 (1 + \alpha) C_A^n \quad (2.3)$$

It is necessary to relate C_A and r in the above equation. From the stoichiometry of Eq. (2.1)

$$\frac{dN_A}{dt} = 2 \frac{dN_B}{dt} \quad (2.4)$$

Eq. (2.3) then becomes

$$\frac{1}{V} \frac{dN_A}{dt} = -\pi K m r^2 (1 + \alpha) C_A^n \quad (2.5)$$

We further assume that the composition of an antacid tablet remain constant throughout the process. Thus

$$N_B = \frac{\pi m r^2 h \rho X_B}{M_B} = N_B = \frac{\pi m r^3 \alpha \rho X_B}{M_B} \quad (2.6)$$

Eq. (2.5) then becomes

$$\frac{dr}{dt} = \frac{-K(1 + \alpha) M_B V}{3\alpha \rho X_B} C_A^n \quad (2.7)$$

From an overall material balance

$$C_A = C_A^0 - \frac{2\alpha \pi m \rho X_B}{M_B V} (R^3 - r^3) \quad (2.8)$$

Invoking the definition of PH

$$pH = -\log(C_A)$$

$$r = \left[R^3 - \frac{M_B V}{2\alpha\pi m\rho X_B} \left(C_A^0 - C_A \right) \right]^{\frac{1}{3}} \quad (2.9)$$

2.4 Equipment & Material

- A pH meter and electrode with a rapid response time of about 5-10 seconds.
- Two buffer solutions (pH = 1 and 7) for 2 point standardization of the pH meter.
- A magnetic stirrer, stirring bar and a timer.
- A set of vernier caliper to measure the dimensions of the antacids tablets.
- Standard laboratory supplies, such as beakers, graduated cylinders, and reagent bottles.
- Distilled white vinegar.
- Antacids tablets as a neutralization agent.

2.5 Procedure

Following is a step-by-step procedure:

1. Remove protective tip from the pH electrode and rinse it well with distilled water to remove any residue, which may have formed on the electrode tip.
2. Perform 2 points' standardization of the pH meter with the two buffer solutions in accordance with the procedure given in the manufacturer's instruction manual.
3. Prepare a fresh acid solution by mixing 25 ml of distilled white vinegar and 500 ml of distilled water in a 1 liter reagent bottle.
4. Transfer 250 ml of this fresh acid solution from reagent bottle to 500-ml beaker, which is placed, on a magnetic stirrer.
5. Place the beaker below the electrode assembly and lower the electrode tip into acid solution. Ensure that the stirring bar does not hit to electrode tip during the operation.

6. Measure the thickness (h), the diameter (D) and the weight of the antacid tablets using vernier calipers and weight balance.
7. Drop 3 to 5 tablets in the acid and start the timer. Adjust the stirring speed so that a fair degree of homogeneity is achieved and the tablets are just barely lifted off of the bottom of the beaker.
8. Record the pH values of solution at 30- second intervals for the first five minutes, at 1 minute intervals for the next 15 minutes and at 2 minute intervals for the remainder of the run-until the tablets are completely dissolved and the solution pH has off at a constant value (typically 40-50 minutes total).
9. Repeat the above procedure for different type of antacid tablets.

2.6 References

1. Standers, S.A. and Sommerfeld, J.T. "A Laboratory Experiment on Combined Mass Transfer and Kinetics", Chem.Eng. Edu. Vol.22, No.2, Spring 1989,p. 86-91.

2.7 Nomenclature

a	Interfacial
C_A	Concentration of species A
C_B	Concentration of species B
D	Diameter of the tablet
K	Combined mass transfer and kinetic rate constant
M_A	Molecular weight of species A
M_B	Molecular weight of species B
m	Number of tablets
n	Order of reaction
R	Radius of the tablets

V	Volume of the acetic acid solution
X _B	Mass fraction of active ingredient
α	Aspect ratio
ρ	Density of the tablet

NEUTRALIZATION OF AQUEOUS SOLUTION OF ACETIC ACID

1. Data Analysis

Write the characteristics of antacid tablets

Table 2.1 Characteristics of antacid Tablets

Property	Antacid Brand	
Active (Alkaline) Ingredient Typical		
Mass of Tablets, gm.		
Mass of Active Ingredient, mg		
Mass Fraction of Active, (X _B) Ingredient.		
Mol. Wt. of Active Ingredient (M _B)		
Typical Thickness of Tablets(h), cm		
Aspect Ratio ($\alpha = h / R$)		
Density of Tablets g / cm ³		
No. of Tablets Used in the experiment (m)		

2. Determine the order of the reaction

3. Determine the value of combined mass transfer and kinetic rate constant K.

2. Raw Data

[illegible]

Group Name-----

Signature-----

Date-----

EXPERIMENT # 3

Mixing Behavior under Various Operating Conditions

3.1 Objective:

To study the behavior of mixing under various operating.

3.2 Introduction

Mixing is applied to the processes used to reduce the degree of non-uniformity or gradient of a property in a system such as concentration, viscosity, temperature and so on.

3.2.1 Type of Mixing

There are many types of mixing:

1. Single Phase Liquid Mixing.

In this mixing two or more miscible liquid must be mixed to give a product of a desired specification.

2. Mixing of Immiscible Liquids

Two immiscible liquids are stirred together, one phase become dispersed as tiny droplets in the second liquid which forms a continuous phase.

3. Gas- Liquid Mixing

The purpose of mixing here is to produce a high interfacial area by dispersing the gas phase in the form of bubbles into the liquid.

4. Liquid-Solid Mixing

Mechanical agitation may be used to suspend particles in a liquid in order to promote mass transfer or a chemical reaction.

5. Gas-Liquid-Solid Mixing

Like slurry reactors, evaporative crystallization.

3.2.2 Rate and Time for Mixing

Mixing time is the time required producing a mixture or a product of predetermined quality, and the rate of mixing is the rate at which the mixing progresses towards the final state.

For a single-phase liquid in a stirred tank to which a volume of tracer materials is added, the mixing time is measured from the instant the tracer is added to the time when the contents of the vessel have reached the required degree contents of uniformity.

The mixing time will depend upon the process and the following:

- 1) System geometry.
- 2) Impeller Diameter, D.
- 3) Speed of rotation (revs/unit time) N
- 4) Properties of liquids.

$$t = f(N, D, g, \rho, \mu, \text{system geometry})$$

3.2.3 Mixing equipment

In designing mixing equipment there are many factors that must be considered to obtain “desired process results”, among these factors are:

- Vessel size
- Vessel shape
Vessels are typically cylindrical in shape, defined by H/T ratio:
Where: H: vessel height,
T: vessel diameter.
- The base of vessel may be flat, dished, or conical, or specially contoured, depend upon factors such as ease of emptying, or the need to suspend solids.
- Baffles
Baffles are used to prevent cross vortexing, which is detrimental to mixing, particularly in low viscosity systems. The baffles are mounted flush with the wall and these take the form of thin about one-tenth of the tank diameter in width, and typically four equi-spaced baffles may be used.
- Impeller
There are many types of impellers: propellers, turbines, paddles, anchors, helical ribbons and screws which are usually mounted on a central vertical shaft in a cylindrical tank, and they are selected for a particular duty largely on the basis of liquid viscosity.

Propellers, turbine and paddles are generally used relatively low viscosity systems and operate at high rotational speeds.

3.4 Equipment and material

- Water
- dye
- Beakers (2L).
- Stirrer

3.5 Procedure

- A. Effect of impeller position
 - B. Effect of speed
 - C. Effect of aeration
 - D. Effect of baffles
-
1. Fill the beaker with water.
 2. Inject a dye at the top of a beaker.
 3. From time to time take sample and find its concentration by spectrophotometer.
 4. Operate the mixer under laminar flow.
 5. Observe how mixing is carried out and evaluate how long it takes to mix.
 6. Observe the aeration if it happens or not.
 7. Repeat the same experiment but at higher speed that is in turbulent flow.
 8. Repeat the above experiments with different impeller position (1/2 and 1/3 from the bottom).
 9. Repeat the above experiments using baffles.

Mixing Behavior under Various Operating Conditions

Group Name -----

Temperature :----- °C

Table 3.1 Experimental Data

Sample #	Impeller height	Speed (rev/min)	Concentration	Mixing time (sec)	Aeration height (cm)	Aeration height / impeller distance (cm/cm)
	1/3					
	1/2					

	2/3					

Signature-----

Date-----

1. Report on mixing time as a function of impeller speed
2. Plot mixing time vs. time at low speed and high speed.
3. Plot mixing time vs. time at different impeller position.
4. Calculate the power consumption in each case.
5. Compare between using baffles and not using it.
6. Compare your results with theoretical data.

EXPERIMENT # 4

Effect of fluid viscosity on mixing time

4.1 Objective :

To determine the effect of fluid viscosity on the mixing time and mixing quality

4.2 Equipment and material

- CMC solution of different concentration.
- Beakers.
- Stirrer
- dye

4.3 Procedure

Following is a step by step procedure:

Adding non viscous to viscous:

- 1- Prepare a 1L of viscous CMC solution of different concentration. Use a concentrations of 0.2, 0.4 and 0.8%
- 2- Prepare a 5 ml of dye solution of x % concentration for each concentration. This solution when fully mixed in the CMC concentration should give a final concentration of 300ppm.
- 3- Fill the 2L beaker with the viscous fluid and use a low stirrer speed but adequate for mixing (corresponds to the minimum turbulent Reynolds number value).
- 4- Stop the mixer and gently add the dye solution at the top surface of the viscous fluid, switch on the stirrer and observe how the dye moves inside the batch.
- 5- From time to time take sample and find its concentration by spectrophotometer.
- 6- Draw a sketch for the movement of dye in the batch.
- 7- Repeat steps 3 to 5 for other CMC solution.

Adding viscous to non viscous:

- 1- Fill a 2L beaker with 1L water and use a low stirrer speed but adequate for mixing (corresponds to the minimum turbulent Reynolds number value)
- 2- Prepare a 5 ml of CMC solution of different concentration (0.2, 0.4 and 0.8%).
- 3- Add x of dye for each concentration that make the solution when fully mixed in water should give a final concentration of 300ppm.

- 4- Stop the mixer and gently add the dye solution at the top surface of the non viscous fluid, switch on the stirrer and observe how the dye moves inside the batch.
- 5- From time to time take sample and find its concentration by spectrophotometer.
- 6- Draw a sketch for the movement of dye in the batch.
- 7- Repeat steps 3 to 5 for other CMC solution.

Effect of fluid viscosity on mixing time

Group Name -----

Temperature :----- °C

Table 4.1 Experimental Data

	Dye weight (mg)	CMC concentration %	Time (Sec)	Sample concentration	Mixing time
non Viscous to viscous		0.1			
		0.3			
		0.6			
Viscous to non viscous		0.1			
		0.3			
		0.6			

Signature-----

Date-----

- 1- Sketch mixing time vs time for all cases.
- 2- How solution viscosity affect mixing time. Support your results with any existing correlation. (use reference).

EXPERIMENT # 5

Scale up of Stirred tank reactor

5.1 Objective:

To apply different scale up rules on scaling 200ml batch to 2000ml

5.2 Equipment and material

- CMC solution of different concentration.
- Beakers.
- Stirrer
- dye

5.3 Procedure

Following is a step by step procedure:

- 1- prepare a 2% CMC solution.
- 2- Fill the 200 ml beaker with the viscous fluid and use a low stirrer speed but adequate for mixing (corresponds to the minimum turbulent Reynolds number value).
- 3- Stop the mixer and gently add the dye solution at the top surface of the viscous fluid, switch on the stirrer and observe how the dye move inside the batch.
- 4- From time to time take sample and find its concentration by spectrophotometer.
- 5- Draw a sketch for the movement of dye in the batch.
- 6- Use different scale up rules (equal Reynolds number, equal tip speed, and equal power per unit volume) and record the mixing time for the 2000ml batch reactor and 5000 ml batch reactor.
- 7- Repeat steps for another speed.
- 8- Repeat steps for another viscous concentration 0.5%

Scale up of Stirred tank reactor

Group Name -----

Temperature :----- °C

Mixer speed -----

Table 5.1 Experimental Data

Volume of reactor (ml)	H	D	H/D	CMC concentration %	Time (Sec)	Sample concentration	Mixing time
200				2			
				0.5			
2000				2			
				0.5			
5000				2			
				0.5			

Signature-----

Date-----

- 1- Report on mixing time value at different scale (200 ml, 2000 ml and 5000 ml) with same speed.
- 2- Using scale up procedure (Equale mixing time) what are the value of speed at each scale that give same mixing time.

EXPERIMENT # 6

Plug Flow Reactor

Plug flow Reactor

Objectives:

- 1- Study the effect of flow rate on the conversion of acid base reaction (Sodium hydroxide and Ethyl acetate (EA)).
- 2- Determine the conversion of the acid base reaction at different reactor length.
- 3- Study the effect of recycle ratio on the final conversion of the acid base reaction.

Procedure:

- 1- Prepare 2l of 0.1M sodium hydroxide, 0.1 EA and 1 liter 0.1M of HCl solutions.
- 2- Set the peristaltic pump at flow rate of 30ml/minute.
- 3- Fill the pump with EA solution
- 4- Connect the two solutions to peristaltic pump
- 5- Switch on the pump at the desired flow rate.
- 6- Every 1 minute take 10 ml sample and quench it with 10 ml of HCL. Samples should be taken from all sampling points.
- 7- continue the experiment for 15 minutes
- 8- report values of NaOH needed for titration, plot concentration of NaOH at different location with time
- 9- repeat experiment different flow rate, preferable double the previous flow rate.

Experiment 2

1. Prepare 1 liter of 0.1M NaOH and 0.1M HCL
2. Add phenolphthaleine indicator to NaOH solution
3. Pump the solution at low flow rate and take a sample of solution and add HCL of known volume and back titrate with NaOH
4. Record the exit concentration with time and make an observation for color change.

PART II

CHEMICAL REACTORS TRAINER

1. Unit Layout and Function

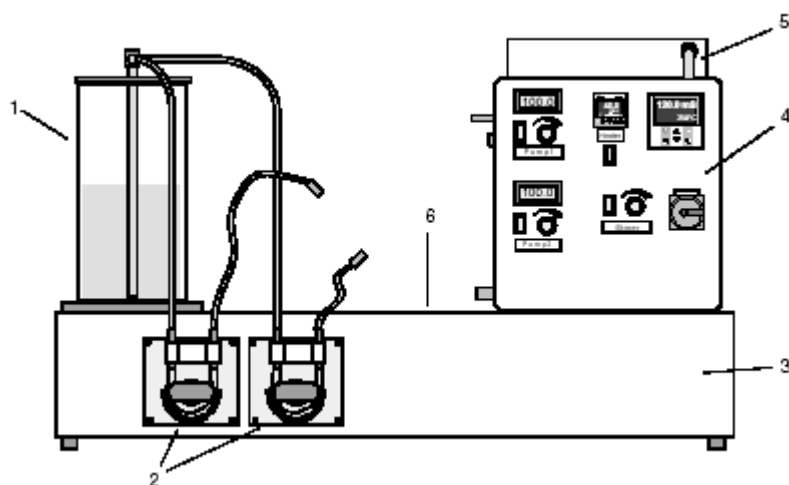


Fig. 1.1 General View of Chemical Reactors Trainer

Figure 1.1 shows the **Chemical Reactors Trainer**, which is used to compare the conversion of reactants during a saponification reaction in various types of reactor. Various reactor models, available as accessories, can be fitted to the **base** (3) of the unit. Two **tanks** (1), one behind the other, contain the liquid chemicals. The two reactants can be fed to the respective experiment reactor using two **hose pumps** (2) fitted to the front of the base. The pump hoses are equipped with self-sealing rapid action couplings so that the objects of the experiments can be interchanged easily. The **switch box** (4) contains switches and adjusters for the pump and stirrer speed, a combined conductivity and temperature measuring unit for the measurement of the conversion of the reactants, and an industrial regulator for provision of hot water. Behind the switch box there is a **water tank** (5) with an electrical heater and a centrifugal pump with which a heating circuit can be run. The product of the reaction produced is collected in a **stainless steel tray** (6) that is let into the base of the unit, and can be disposed of as appropriate after the experiment.

The following reactor models can be used as the object of the experiment:

1. Continuous Stirrer Tank Reactor
2. Tubular Flow Reactor
3. Stirred Tanks in Series
4. Batch Reactor

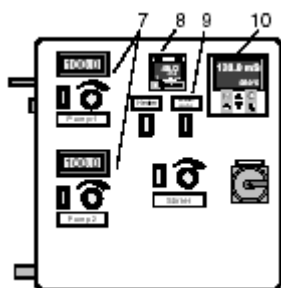


Fig. 1.2 Switch Box

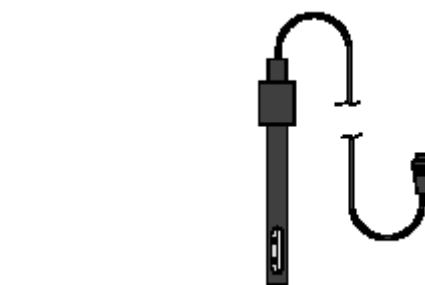
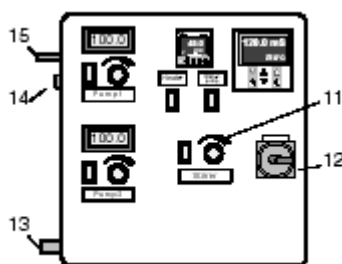


Fig. 1.3 Combined Conductivity and
Temperature Sensor

Figure 1.2 shows the switch box which contains two-control **groups** (7) for the two hose pumps, each group has a switch. An adjusting potentiometer on each group permits the volumetric flow rate of each pump to be adjusted individually from 0% to 100%. Using the associated four digit displays, it is possible to read the corresponding value. An **industrial controller** (8) is used to adjust and regulate the water temperature in the heating circuit. The current water temperature and the heating temperature set are displayed on the display for the regulator. Using the associated switch below the regulator housing, it is possible to switch the heater on or off as required. A hot water pump is installed in the switch box, this can be placed in operation using the **pump switch** (9). The **combined measuring unit** (10) for the **conductivity** and **temperature** of the experimental solution is already adjusted and the parameters set. Further changes are not necessary. As some of the experimental reactors are equipped with a stirrer, on the switch box there is an **adjuster** (11) with a switch and potentiometer using which the

corresponding stirrer speed can be adjusted. The stirrer is connected electrically to the switch box via the socket (14).

The trainer is switched completely on or off, including all components, using the **master switch** (12). The respective reactor can be connected to the hot water system using the hoses supplied via the **water connection** (13) with rapid action couplings. The combined **conductivity** and **temperature measuring sensor** (figure 1.3) is plugged into the **adapter** (15) and is then ready for use without any further action. There are sensor insertion points on the reactors for recording results and ensuring correct measurements.

2. Theoretical Principles and Experiments

2.1 Theory

As a rule, chemical processes are not spontaneous and are incomplete. Indeed, in the majority of cases the reaction products only formed gradually. The speed of the reaction can be very varied, as reactants only react with one another if they meet with a sufficiently large amount of energy. An increase in the temperature therefore increases the conversion of the reactants. The time for which the reactants are in contact is a further criterion.

The conversion of the reactants is thus dependent on:

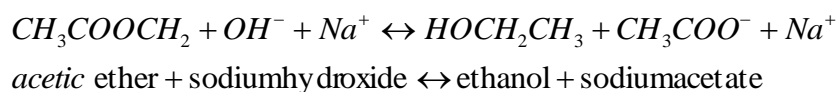
- The nature of the reactants
- The concentration and mixing rate of the reactant
- The time that the reactants are in contact
- The reaction temperature

For each chemical reaction, there exists a process that splits the product of the reaction back into the original reactants. This counter reaction is, in turn, temperature dependent. It thus sets equilibrium between the concentrations of the initial reactants and the product. This situation is termed dynamic, as both reactions occur side by side without interruption; however the reactant concentration does not change.

The response time and position of this equilibrium can be influenced, for example, by:

- Change in the time for which the reactants are in contact or the duration of the period in the reactor
- Change of the reaction temperature

These relationships are to be checked based on the saponification of ether with sodium hydroxide. The following reaction occurs:



The ether molecules are split and each disintegrates into an acetate ion and an ethanol molecule. During this process the hydroxide ions in the sodium hydroxide are consumed. The progress of the reaction and conversion of the reactants can thus be tracked extremely well by the change in the hydroxide concentration. Alternatively, a conductivity measurement or volumetric analysis with acid can be used. From the conductivity values, the percentage conversion of the reactants **S** can be calculated with the aid of the following equation:

$$S = \left(1 - \frac{(k - k_e)}{(k_0 - k_e)} \right) \times 100\%$$

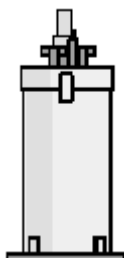
k : Actual measured value for conductivity

k₀ : Initial conductivity of the 2.3% Sodium Hydroxide (NaOH)

k_e : Conductivity of the end product produced

3. Experiments

3.1 BATCH REACTOR, ADIABATIC REACTION



A batch reactor is in general designed as a vacuum sleeve vessel and is closed. It cannot be used continuously. Due to its properties, a reaction in the batch reactor can either be **adiabatic** or **isothermic** using external temperature regulation. In the case of heavily exothermic reactions, there is a risk of uncontrolled acceleration or uncontrolled inhibition of the reaction and thus an unstable reaction. By combining the possible methods of operating the batch reactor, the correct stability conditions can be found for each reaction and thus a runaway of the reaction on the operation of a continuous reactor can be voided.

3.1.1 Performing the Experiments

1. Attach the batch reactor to the trainer above the collecting tray and open it.
2. Prepare a glass beaker with 400 ml of 2.3% Sodium Hydroxide. Measure by using the measuring sensor, the conductivity and temperature of the solution and note.
3. Prepare a glass beaker with 400 ml of 5% Ethyl Acetate solution, measure using the measuring sensor, the conductivity and temperature of the solution and note.
4. Insert the conductivity measuring sensor in the measuring opening on the lid and connect the stirrer
5. Adjust the stirrer to a medium speed, however do not switch it yet.

6. Quickly pour both solutions into the reaction tank.
7. Close lid immediately and switch on the stirrer.
8. Continuously note the conductivity and temperature values for approximately three minutes.
9. Record your data in Table 3.1.

Table 3.1 Measured Values for Batch Reactor, Adiabatic Experiment

[illegible]

Initial Conductivity of 2.3 % NaOH _____mS/cm

Note 1: After the end of the experiment

- Empty the reaction vessel and clean it.
- Rinse measuring sensor with water.
- Dispose of the reaction product correctly.

3.1.2 Report Requirement

1. Convert measured results for conductivity into percentage conversion of reactants.
2. On one graph, plot conversion (%) and temperature (°C) versus time (s).

3.2 BATCH REACTOR, ISOTHERMIC REACTION

3.2.1 Performing the Experiments

1. Attach the Batch Reactor to the trainer above the collecting tray and open it.
2. Prepare a glass beaker with 400 ml of 2.3% Sodium Hydroxide. Measure, using the
3. measuring sensor, the conductivity and temperature of the solution and note.
4. Prepare a glass beaker with 400 ml of 5% Ethyl Acetate solution. Measure, using the
5. measuring sensor, the conductivity and temperature of the solution and note.
6. Insert the conductivity measuring sensor in the measuring opening on the lid and connect the stirrer
7. Adjust the stirrer to a medium speed, however do not switch it yet.
8. Connect the water hoses from the heating system to the connections on the lid and to the switch housing, switch the pump.
9. Quickly pour both solutions into the reaction tank.
10. Close lid immediately and switch on the stirrer.
11. Continuously note the conductivity and temperature values for approximately three minutes.
12. Record your data in Table 3.2

[illegible]

Initial Conductivity of 2.3 % NaOH _____mS/cm

Note 2: After the end of the experiment

- Empty the reaction vessel and clean it.
- Rinse measuring sensor with water.
- Dispose of the reaction product correctly.

3.2.2 Report Requirement

1. Convert measured results for conductivity into percentage conversion of reactants.
2. On one graph, plot conversion (%) and temperature (°C) versus time (s).
3. Compare results with the results from previous experiment (Adiabatic) and give reasons for the deviations.

3.3 CONTINUOUS STIRRER TANK REACTOR

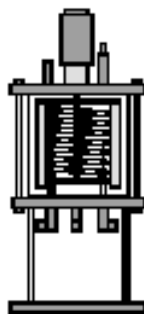


Figure 3.2 Continuous Stirrer Tank Reactor

A key feature of a stirrer tank reactor is that it can work both continuously and also discontinuously in batches. A further advantage is that it is easier to drain in the event of a defective batch or breakdown, as the reaction chamber is directly accessible.

Thus it is possible to quickly set up the experiment. The disadvantages are the poor mixing behavior and less favorable heat transfer to the chemicals used.

3.3.1 Performing the Experiments

1. Attach the continuous stirrer tank reactor to the trainer above the collecting tray
2. Connect the water hoses from the heating system to the connections on the base of the tank and to the switch housing.
3. Connect the chemical hoses from the pumps to the tank using the rapid action connectors.
4. Insert the conductivity measuring sensor in the measuring opening on the lid and connect the stirrer
5. Adjust the overflow on the tank such that the measuring sensor is immersed approximately 50 mm in the liquid with the tank filled.
6. Ensure that all subordinate switches on the main unit are switched off and then switch on the unit at the master switch.

7. Set the required temperature for the heating regulator to 45°C and switch on the heater.
8. Switch on the chemical pumps and adjust both to the same flow rate of approximately 80%
9. When the liquid level reaches the overflow, switch on the stirrer and adjust to a medium speed
10. At regular intervals of approximately 20-30 seconds, the conductivity and reaction temperature indicated on the measuring unit until stable values are reached
11. Then reduce the flow rate of both chemical pumps to the same value of approx. 40%.
12. Then the conductivity and reaction temperature indicated on the measuring unit at regular intervals of approximately 20- 30 seconds until stable values are reached.
13. Check whether the hot water temperature has reached 45°C and switch on the hot water pump.
14. Again the conductivity and reaction temperature indicated on the measuring unit at regular intervals of approximately 20-30 seconds until stable values are reached.
15. Then increase the flow rate of both chemical pumps back to the same value of approximately 80%
16. Again log the conductivity and reaction temperature indicated on the measuring unit at regular intervals of approximately 20-30 seconds until stable values are reached.
17. Switch off chemical pumps, heater, hot water pump and stirrer
18. Record your data in Table 3.3

Table 3.3 Measured Values for Continuous Stirrer Tank Reactor

[illegible]

Initial Conductivity of 2.3 % NaOH _____mS/cm

Note 3: After the end of the experiment

- Undo all connections to the reaction vessel and remove the measuring sensor.
- Empty the reaction vessel and clean it. .
- Rinse measuring sensor with water.
- Dispose of the reaction product correctly
- Drain both chemical tanks and rinse then out.
- Operate both chemical pumps with water to clean them.
- Correctly dispose of the product of the reaction collected in the collecting tray.
- Switch off the master switch.

3.3.2 Report Requirement

1. Convert measured results for conductivity into percentage conversion of reactants.
2. On one graph, plot conversion (%) and temperature (°C) and percentage flow rate values for the pumps versus time (s).
3. Comment on your results.

3.4 TUBULAR FLOW REACTOR

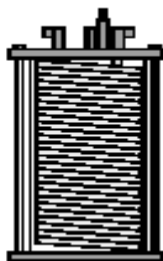


Figure 3.3 Tubular Flow Reactor

Figure 3.3 shows a tubular flow reactor, which is basically comprises a coiled tube or a section of hose in a heat exchanger bath. In the simplest case the bath contains water. Due to the large contact area, very good heat transfer to the reactants or cooling effect on the reactants is achieved Due to the comparatively small cross-sectional aperture, intensive mixing of the chemicals is achieved while the reactants are flowing through the reactor. In comparison to other types of reactors, it therefore offers characteristically higher reactant conversion. In principle, such a reactor can however only be operated continuously and only be cleaned by rinsing, as it cannot be opened.

3.4.1 Performing the Experiments

1. Attach the tubular flow reactor to the trainer above the collecting tray
2. Connect the chemical hoses from the pumps to the tank using the rapid action connectors.
3. Connect the water hoses from the hot water system to the connections on the base of the tank and to the switch housing.
4. Ensure that the end of the overflow hose is in the collecting tray
5. Insert the conductivity measuring sensor in the measuring opening on the lid and connect the stirrer
6. Ensure that all lower level switches on the main unit are switched off and then switch on the unit at the master switch.

7. Set the required temperature for the heating regulator to 45°C and switch on the heater
8. Switch on the chemical pumps and adjust both to the same flow rate of approximately 80%
9. At regular intervals of approximately 20-30 seconds, the conductivity and reaction temperature indicated on the measuring unit until stable values are reached
10. Then reduce the flow rate of both chemical pumps to the same value of approximately 40%.
11. Then the conductivity and reaction temperature indicated on the measuring unit at regular intervals of approximately 20 – 30 seconds until stable values are reached.
12. Check whether the hot water temperature has reached 45°C and switch on the hot water pump.
13. Again the conductivity and reaction temperature indicated on the measuring unit at regular intervals of approximately 20-30 seconds until stable values are reached.
14. Then increase the flow rate of both chemical pumps back to the same value of approximately 80%
15. Again log the conductivity and reaction temperature indicated on the measuring unit at regular intervals of approximately 20-30 seconds until stable values are reached.
16. Switch off chemical pumps, heater, and hot water pump.
17. Record your data in Table 3.4

Table 3.4 Measured Values for Tubular Flow Reactor

Pump 1 (%)	Pump 2 (%)	Time Measured (min.)	Conductivity (mS/cm)	Reactants Conversion (%)	Temperature (°C)

Initial Conductivity of 2.3 % NaOH _____mS/cm

Note 4: After the end of the experiment

- Undo all the connections to the reaction vessel and remove the measuring sensor.
- Rinse the measuring sensor with water.
- Empty reaction vessel and rinse it out.
- Drain both chemical tanks and rinse out.
- Operate both chemical pumps with water to clean them.
- Correctly dispose of the product of the reaction collected in the collecting tray.
- Switch off unit at master switch.

3.4.2 Report Requirement

1. Convert measured results for conductivity into percentage conversion of reactants.
2. On one graph, plot conversion (%) and temperature (°C) and percentage flow rate values for the pumps versus time (s).
3. Comment on your results.

3.5. STIRRED TANKS IN SERIES



Figure 3.4 Stirred Tanks in Series

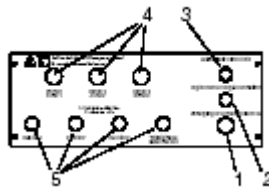


Figure 3.5 Back Panel of the Measuring Point Selector

Figure 3.4 shows a cascade consists of several stirred tanks connected in series. This arrangement offers the advantage that in one setup, solutions with varying degrees of progress of the reaction are available. Figure 3.5 shows the monitoring Panel.

3.5.1 Performing the Experiments

1. Attach the stirred tanks in series to the trainer above the collecting tray
2. Place the measuring point selector on the switch housing on the trainer
3. Connect the **supply connection** (1) (**Figure 3.5**) to the associated socket on the right on the main unit.
4. Connect the **digital connection** (2) (**Figure 3.5**) to the associated socket on the right on the main unit
5. Connect the **conductivity connection** (3) (**Figure 3.5**) to the measuring sensor adapter on the left on the main unit.
6. Connect the three **stirrers** to the associated sockets (4) (**Figure 3.5**).
7. Connect the four **measuring sockets** to the associated receptacles on the reaction tanks and to the corresponding sockets (5) (**Figure 3.5**).
8. Connect the chemical hoses from the pumps to the cascade using the rapid action connectors
9. Ensure that the end of the overflow hose from the delay section is in the collecting tray

10. Place all three-way valves in the correct open position
11. Switch on both chemical pumps and adjust to the same flow rate of approximately 80%
12. Switch on the stirrers one after the other, once the blades are covered with sufficient liquid, and set to a medium speed
13. Once the reaction mixture has reached all tanks and sections, with the aid of the measuring point selector measure all four values for conductivity and reaction temperature and note
14. Reduce the flow rate of both chemical pumps to the same value of around approximately 40%
15. At intervals of one minute, the value at all four measuring points until a stable state is reached.
16. Record your data in Table 3.5

Table 3.5 Measured Values for Stirred Tanks in Series

PUMP 1 80%	PUMP 1 80%	Initial Conductivity Of 2.3 % NaOH----- mS/cm	
	Conductivity (mS/cm)	Reactor Conversion (%)	Temperature (°C)
Tank 1			
Tank 2			
Tank 3			
Delay Section			

Note 5: After the end of the experiment

- Undo all the connections to the reaction vessels and remove the measuring sensors
- Rinse the measuring sensors with water
- Drain the reaction vessels and rinse it out with water
- Drain both chemical tanks and rinse it out
- Operate both chemical pumps with water to clean them
- Correctly dispose of the product of the reaction collected in the collecting tray
- Switch off unit at master switch

3.5.2 Report Requirement

1. Convert measured results for conductivity into percentage conversion of the reactants and compare.

APPENDIX

Technical Data

Conductivity Measure	Range 0...200 mS/cm
Temperature Measure	Range 0...100 °C
Heater, Electrical	2 kW
Volume of Hot Water	approx. 15 l

Hose Pumps:

Speed	0 - 105 min ⁻¹
Pump Flow Rate max.	250 ml/min

Chemicals:

Sodium Hydroxide	2.3%
Acetic Ether (Ethyl Acetate)	
Power Supply	230V, ~50 Hz

Dimensions:

LxWxH	1300 x 680 x 630 mm
Weight approx.	90 kg

Continuous Stirrer Tank Reactor:

Volume	0.4 - 1.5 l
LxWxH	470 x 400 x 380 mm
Weight approx.	13 kg

Tubular Flow Reactor:

Pipe Section Length	20 m
Tube Diameter	5.5 mm
Reactor Volume	0.475 l
LxWxH	470 x 250 x 600 mm

Weight approx. 25 kg

Stirred Tanks in Series:

Tank Volume 1.2 l

Delay Section Length 2.8 m

Delay Section Volume 0.2 l

LxWxH 470 x 460 x 400 mm

Weight approx. 32 kg

Batch Reactor:

Volume 0.95 l

LxWxH 470 x 290 x 380 mm

Weight approx. 8 kg