



# **Chemical Technology Lab. I**

## **(10626478)**

### **CHEMICAL REACTORS TRAINER**

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

Revised by:

Dr. Majd Eshtaya

**Fall 2023**

## 2. Chemical Reactors Trainer

### 2.1 Unit Layout and Function



Figure. 2.1: General View of Chemical Reactors Trainer (Mod. CRBS/EV)

Figure 2.1 shows the **Chemical Reactors Trainer**, which is used to compare the conversion of reactants during a saponification reaction in various types of reactors.

The chemical reactors service unit provides feed tanks, feed pumps, temperature control system and instrumentation for continuous stirred-tank reactor (CSTR), two plug flow reactors (PFR), a batch reactor (BR), three reactors in series and a laminator flow reactor (LFR), interchangeable on the same service unit mod. CRB/EV). Each of the four chemical reactors modules is mounted on a base to stand on service unit.

The reaction proposed is the classical saponification of ethyl acetate with sodium hydroxide, checked in real time with a conductivity meter.

Simply choose which reactor will be studied, place on the service unit and connect to the appropriate services as reported in the relative reactor instruction manual. The unit consists of a stainless-steel framework to be bench mounted and upon which the necessary components to investigate on four types of chemical reactor can be mounted. The unit incorporates a basin in which any spillage is collected.

The main components of the unit can be described as follow:

- Two 5.0 litre capacity AISI 316 stainless steel vessels for the reagents, code D1 and D2, provided of drain valve and connected with the pumps using silicon tube.
- Two variable speed feed pumps, code G1 and G2, peristaltic type, range 0-95 ml/min. The speed of the pumps can be adjusted using the relative potentiometer on the switchboard and read on the relative display (range 0-100%).
- Variable speed agitator control. The reactor accessories mod. CR1, CR3 and CR4 are equipped with agitators driven by electric motor. The speed of reactor stirrer motor is adjusted using the relative potentiometer on the switchboard and read on the relative display (range 0-100% proportional to 0-400 rpm). A socket is used to power the agitator motor of the reactor accessories mod. CR1, CR3 and CR4.
- Self-contained hot water circulator for operation of reactors at temperatures above room temperature. Flexible tubes are used to connect the circulator to each reactor. Water, heated by electrical heating elements, is pumped by a circulating pump *via* flexible pipe through the desired reactor. The water is returned to the feeding vessel after heating. Temperature control is achieved by circulation of heated water through submersed coils in the case of the CSTR (CR1) and batch (CR3) and by circulation in a jacket in the case of the tubular reactor (CR2). A temperature sensor (Pt100) is used individually with any of the reactors in conjunction with the automatic temperature controller of the service unit which modulates the power to the heating element.
- The process temperature can be settled on the thermostat (TW1) by pressing the key in the upper left corner and simultaneously pressing the UP (↑) or DOWN (↓) keys. The hot water circulator is equipped of a level safety switch, code LSW1, and a temperature safety switch, code TWS1.
- A conductivity meter with probe, code CE1, like the temperature sensor, is used individually with any of the reactors. The conductivity is displayed in units of milliSiemens.

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

## 2.2. Theoretical Principles and Experiments

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:

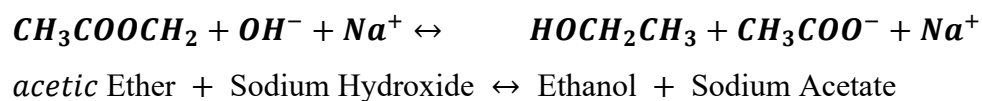
- The nature of the reactants
- The concentration and mixing rate of the reactant
- The time that the reactants are in contact
- The reaction temperature

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.

## 2.3. Experiments

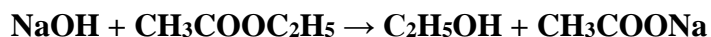
### Batch Reactor



Figure. 2.2: Batch Reactor

The batch reactor (Figure 2.2) is designed to demonstrate the mechanism of a chemical reaction in this type of reactor and the effects of the operative conditions, such as reaction temperature, concentration, stirring rate on reaction rate in isothermal and adiabatic conditions. Isothermal condition requires a water chiller or cold tap water in case of exothermal reactions.

The reaction chosen is the hydrolysis of ethyl acetate by sodium hydroxide (saponification); this reaction can be carried out under safe conditions of temperature and pressure and it is well documented in literature.



As the reaction proceeds, hydroxyl ions are consumed and acetate ions are produced. Both sodium hydroxide and sodium acetate contribute conductance to the reaction solution whilst ethyl acetate and ethyl alcohol do not. Hydroxyl ion has a very much larger specific conductance than acetate ion. Hence, the alkaline hydrolysis of ethyl acetate may be monitored by following the change in the conductance of the reaction mixture with time. By determination of the conductivity as a function of time, the rate constant is found. If the reaction is carried out at different temperatures, the activation energy can also be found.

The following correlations allow calculating the concentration of NaOH, using the measured conductivity, and then the conversion.

At infinite time

$$C_{NaAc}^{\infty} = C_{AcEt}^{in} \text{ if } C_{AcEt}^{in} < C_{NaOH}^{in}$$

or

$$C_{AcNa}^{\infty} = C_{NaOH}^{in} \text{ for } C_{AcEt}^{in} \geq C_{NaOH}^{in}$$

$$\text{if } C_{AcEt}^{in} \leq C_{NaOH}^{in} \text{ then } C_{NaOH}^{\infty} = C_{NaOH}^{in} - C_{AcEt}^{in}$$

The following correlations allow to calculate the conversion at time t:

$$C_{NaOH}^t = \left[ \frac{K_o - K_t}{K_o - K_{\infty}} \right] (C_{NaOH}^{\infty} - C_{NaOH}^o) + C_{NaOH}^o$$

$$X_A = \frac{C_{NaOH}^o - C_{NaOH}^t}{C_{NaOH}^o}$$

Where K is the conductivity.

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 **isothermal** 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.

## EXPERIMENT # 1

***Objective: determining of the rate law (the order and value of the rate constant) for the reaction with the integral method***

### **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 0.05M 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 0.05M 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 (40%), however do not switch it yet.
6. Connect the water hoses from the heating system to the connections on the lid and to the switch housing, switch the pump.
7. Quickly pour both solutions into the reaction tank.
8. Close lid immediately and switch on the stirrer.
9. Continuously note the conductivity and temperature values.
10. Record your data in Table 2.1.

**Note:** After the end of the experiment

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

### **Report Requirement**

1. Calculate the concentration of NaOH (mol/lit).
2. Convert measured results for conductivity into percentage conversion of reactants.
3. Apply the integral method of analysis to determine the reaction order and rate constant of the reaction.
4. Plot conversion (%) versus time (s).
5. 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.

6. Compare results with the results from previous experiment (Homogenous Batch Reactor) and give reasons for the deviations.
7. Comment on your results.

**Table 2.1 Measured Values for Batch Reactor, Isothermal Experiment**

<b>Initial NaOH Concentration (M)</b>			<b>Conductivity (mS)</b>	
<b>Initial AcEt Concentration (M)</b>			<b>Conductivity (mS)</b>	
<b>Stirrer Speed</b>				
<b>Sample #</b>	<b>Time (sec)</b>	<b>Conductivity (mS)</b>		<b>Temperature (°C)</b>
1.				
2.				
3.				
4.				
5.				
6.				
7.				
8.				
9.				
10.				
11.				
12.				
13.				
14.				
15.				
16.				
17.				
18.				
19.				
20.				
21.				
22.				
23.				
24.				
25.				
26.				



## EXPERIMENT # 2

Produce a calibration curve conversion vs. conductivity, at  $T = 20^{\circ}\text{C}$ ,  $25^{\circ}\text{C}$ ,  $30^{\circ}\text{C}$  and  $35^{\circ}\text{C}$ , using the following three solution: for  $X = 0$  use 0.05 M NaOH, for  $X = 0.5$  use 0.025 M NaOH and 0.025 M sodium acetate, for  $X = 1$  use 0.05 M of sodium acetate.

*Note: select on the conductivity meter the manual compensation for the temperature*

## EXPERIMENT # 3

***Objective: determining of the rate law for the reaction with the method of initial rates***

One of the first steps in studying the kinetics of a chemical reaction is to determine the rate law for the reaction.

One method for determining the rate law is to use the method of initial rates.

The Method of Initial Rates involves measuring the rate of reaction,  $r$ , at very short times before any significant changes in concentration occur. Suppose one is studying a reaction with the following stoichiometry:



While the form of the differential rate law might be very complicated, many reactions have a rate law of the following form:

$$r = k[\text{A}]^{\alpha}[\text{B}]^{\beta}$$

The initial concentrations of A and B are known; therefore, if the initial reaction rate is measured, the only unknowns in the rate law are the rate constant,  $k$ , and the exponents  $\alpha$  and  $\beta$ . Maintain constant the concentration of a reagent and varying the concentration of the other, it is possible to determine the order of reaction respect this reagent.

One typically measures the initial rate for several different sets of concentrations and then compares the initial rates.

1. Fill the reactor with 1 litre of NaOH 0.05 M (2 g in 1 litre)
2. Start the stirrer at 50% of the speed
3. Start the hot water circulator
4. Reach the desired temperature (e.g.  $25^{\circ}\text{C}$ ) and switch off the circulator
5. Weigh 4.4 g of ethyl acetate (MW = 88) in an Erlenmeyer flask to obtain the desired concentration (4.4 g in 1 litres equal to 0.05 M)
6. Pour the ethyl acetate in the reactor (use the stopper on the lid) and at the same time start the data acquisition with a sampling time of 10 sec. for 4 minutes (in any case, until to the plot of conductivity vs. time is linear).

7. Drain the reactor and repeat the experiment following the next table:

Test	c°NaOH (M)	c°AcEt (M)
1	0.05	0.03
2	0.05	0.05
3	0.05	0.07
4	0.07	0.05
5	0.03	0.05

#### EXPERIMENT # 4

**Objective: determining of the rate law for the reaction with the Isolation Method of initial rates**  
**Another strategy for determining of the rate law is the Isolation Method.**

In this method the concentration of one reactant is made much smaller than the concentrations of the other reactants. Under this condition, all reactant concentrations except one are essentially constant, and the simple zero-, first-, and second-order kinetic plots can usually be used to interpret the concentration-time data.

Operating as in experiment # 3, collect the data with a sampling time of 10 sec. for 4 minutes using the following concentrations:

Test	c°NaOH (M)	c°AcEt (M)
1	0.07	0.01
2	0.05	0.5

#### EXPERIMENT # 4

Objective: determining of the rate constant, frequency factor and activation energy

From the kinetic law, the following correlation is obtained:

$$K_t = K_f + \left( \frac{1}{C^0 K} \right) \left( \frac{K_o - K_t}{t} \right)$$

Plotting  $K_t$  vs.  $\frac{K_o - K_t}{t}$ , we obtain a straight line with slope  $1/(C^0 K)$ ; it is easy, from this value, to calculate the value of rate constant  $K$  at the reaction temperature. With test at different temperature, using the Arrhenius correlation, it is possible the determining of frequency factor and activation energy.

Repeat experiment # 3 at 30°C and 35°C and calculate the rate constant, frequency factor and activation energy.