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Kinetic study of hydrolysis of ethyl acetate using caustic soda

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Abstract

We report here, the hydrolysis of ethyl acetate by using caustic soda which is followed by means of conductance measurements which is widely used in chemical industry. The main aim of this research is to study the parameters of production of ethyl acetate by chemical reaction kinetics using an anion ion-exchange acting as a catalyst and acid-base titrations. The reaction of ethyl acetate and sodium hydroxide (caustic-soda) is done in a plug-flow reactor (steady-state tubular reactor) under the effect of different parameters including temperature, concentration and flow-rate, which allows the determination of activation energy and rate constants, due to large number of experiments. Factorial design method is used for the calculations of the experiment. It was determined that the order of the reaction is a second-order reaction.

Keywords: Hydrolysis; Chemical Kinetics; Alkali; Ethyl Acetate; Reaction Rate

1. Introduction

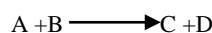
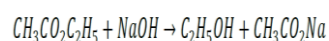
Chemical kinetics is the part of physical chemistry that studies reaction rates of the reaction. As in this project we study the kinetics of saponification reaction by hydrolysis of ethyl acetate with caustic soda (sodium hydroxide) to produce ethanol and sodium acetate which takes place in a liquid plug flow reactor as this reaction is considered one of the most known reactions in the chemical industry, where ethanol is considered as a great major in chemical industries as it can react with acetic acid to produce ethyl acetate, also sodium acetate which is used in many applications such as soap. [1-12] It is well established that kinetics is a decisive factor in investigating chemical reactions used in several synthetic routes.[13-19]

The chemical kinetics of this reaction is studied by rate of the reaction which depends on the speed of the reaction, where we have three mainly used rates: zero-order reactions, first-order reactions and second-order reactions, as in this saponification reaction we use second-order reaction which depends on the concentrations of one second-order reactant. [20-28] The kinetics of the reaction of ethyl acetate with sodium hydroxide depends on main three parameters: temperature, flow rate and concentration of the reactants as the values of these parameters are continuously changed in-order to get the best results.[29-37]

Ethanol is applied to a batch distillation to separates it from sodium acetate, as in saponification reaction ethanol can react in a continuous flow reactor with acetic acid where ethanol is produced again. During experiments cost and time should be reduced, the best technique is applied in order to reduce the number of facing problems.

2. Experimental

2.1. Saponification reaction of ethyl acetate using caustic soda



This equation represents the reaction between the Sodium hydroxide (A) and Ethyl Acetate (B) and that results in the formation of the Sodium Acetate (C) and Ethyl Alcohol (D). This reaction is considered to be an equi- molar one and the first order with respect to the sodium hydroxide and the ethyl acetate and within the limits of the concentration as well as temperature in the lab it is the second order overall. As for the second order it's the saponification of ethyl acetate $CH_3COOC_2H_5$ with dilute sodium hydroxide NaOH and it is an irreversible reaction.

The conductivities of the ethyl acetate CH_3COONa and the ethyl alcohol C_2H_5OH could be ignored in this reaction as the OH ion is the most highly conductive species, Cole-Palmer Model 19100-00 conductivity meter has been used in the lab and is designed to display the conductivity of the solution in terms of the specific conductivity.

Within the batch, the apparatus takes place and within a continuous stirred tank reactor as well, the batch mode pours about 50 ml each of 0.01 N NaOH solution as well as 0.01 N ethyl acetate solutions within the vessel while using a funnel. A motor driven stirrer is used for mixing liquids within the reactors and the vessel is being comprised of a glass cylinder and a steel base and a removable PVC top cover is used for access and some cleaning purposes. The reactors liquid temperature is controlled by the hot water that is

being circulated by a coil that is being immersed in this reactor, the cold water is admitted in the heater tank through the solenoid valve and when the operating level is being reached it closes automatically and then the water is heated through a heating element in this tank and is being circulated in the reactor vessel through a pump and then it returns back to the tank again through the return tank.

The setting of the cooler in the cooling mood once done, the solenoid valve opens then cold water is pumped directly to the reactor vessel and then the hot water is being discharged through the drain pipe outflow. The outflow pipe is operating only when the reactor is set in the cooling mode.

2.2. Conductivity measurements

After calibrating the conductivity cells, the conductivity meter becomes ready for the measurement as 500 ml of 0.01 M solution of NaOH and ethyl acetate are mixed together in a batch reactor at a fixed temperature while applying appropriate till the cell temperature becomes equal to the mixture temperature and then the conductivity is measured.

2.3. Methodology

In the experimental work, a flow-rate of a mixture containing 50% ethyl acetate and 50% sodium hydroxide was used while using a concentration of 0.1 for ethyl acetate and 0.1 for NaOH. Then, temperature was changed within the range 20 °C- 60 °C.

15 ml of HCl is added to 10 ml of samples taken every 5 minutes to prevent further reaction between ethyl acetate and sodium hydroxide. Then, one or two drops of phenolphthalein indicator were added and titration is implemented using NaOH till the sample color turns pink. The resulting NaOH volume is used to get the rate constant by calculating the concentration of the mixture by the equation:

$$c = \frac{V_{\text{HCl}}c_{\text{HCl}} - V_{\text{NaOH}}c_{\text{NaOH}}}{V}$$

Where:

C_{HCl} , C_{NaOH} concentrations of HCl and NaOH.

V_{HCl} volume of HCl (10 ml).

V_{NaOH} ... volume of NaOH (ml).

V .. Volume of the reaction mixture used in titration (10 ml).

In typical calculations, the value of $1/C$ is calculated and plotted against a given temperature, where the slope shows the value of desired rate constant. Hence, the activation energy is calculated by using the equation:

$$\ln \frac{k_1}{k_2} = \frac{E_a}{R} \left(\frac{1}{T_2} - \frac{1}{T_1} \right)$$

The temperature during the experiment is kept constant and changing the other parameters of flow-rates and concentrations. Where, each one of these parameters is used in different values, to determine the better conversion for ethyl acetate.

3. Results and discussion

From the concentration results, namely ($1/C$), the rate constant was calculated as shown in Figure 1. The obtained values at Temperature = 30 °C, by plotting t vs. $1/C$ are summarized in Table 1.

Table 1: Calculated Results at Different Reaction Times at 30°C

t (min)	V (NaOH)	C	1/C
10	7.2	0.078	12.82051
15	7.3	0.077	12.98701
20	7.5	0.075	13.33333
25	7.6	0.074	13.51351
30	7.7	0.073	13.69863

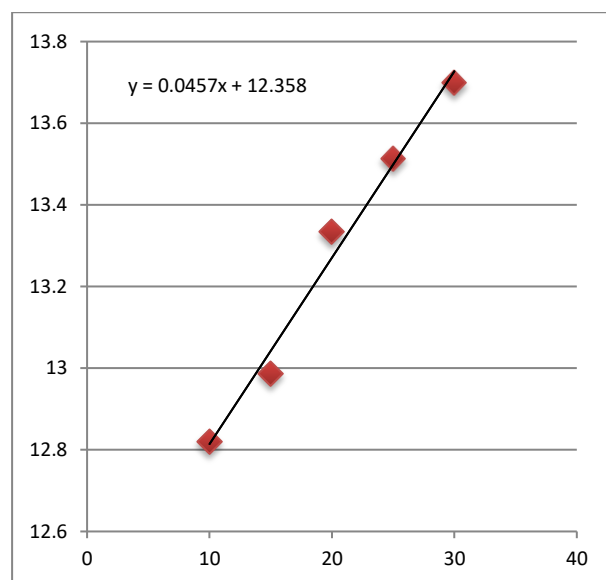


Fig. 1: Representation of Concentration – Time Data at 30 °C.

From the equation of the line in figure 1, the slope could be calculated as follows:

$$K = \text{slope} = 0.0457 \text{ dm}^3/\text{mol} \cdot \text{min} = 0.000762 \text{ L/mol} \cdot \text{s}$$

$$\frac{1}{c} - \frac{1}{c_0} = 0.000762t$$

Similarly, from the concentration results, namely ($1/C$), the rate constant was calculated as shown in Figure 2. The obtained values at Temperature = 40 °C, by plotting t vs. $1/C$ are summarized in Table 2.

Table 2: Calculated Results at Different Reaction Times at 40°C

t (min)	V (NaOH)	C	1/C
10	7.5	0.075	13.33333333
15	7.6	0.074	13.51351351
20	7.7	0.073	13.69863014
25	7.8	0.072	13.88888889
30	8.3	0.067	14.92537313

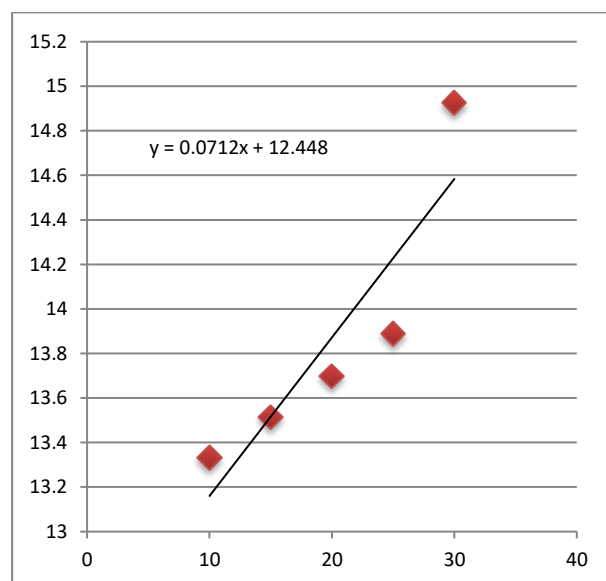


Fig. 2: Representation of Concentration – Time Data at 40 °C.

From the equation of the line in figure 2, the slope could be calculated as follows:

$$K = \text{slope} = 0.0712 \text{ dm}^3/\text{mol} \cdot \text{min} = 0.001187 \text{ L/mol} \cdot \text{s}$$

$$\frac{1}{c} - \frac{1}{c_0} = 0.0001187t$$

Also, from the concentration results, namely (1/C), the rate constant was calculated as shown in Figure 3. The obtained values at Temperature = 50 °C, by plotting t vs. 1/C are summarized in Table 3.

Table 3: Calculated Results at Different Reaction Times at 50°C

t (min)	V (NaOH)	C	1/C
10	8	0.07	14.28571
15	8.4	0.066	15.15152
20	8.5	0.065	15.38462
25	8.7	0.063	15.87302
30	9	0.06	16.66667

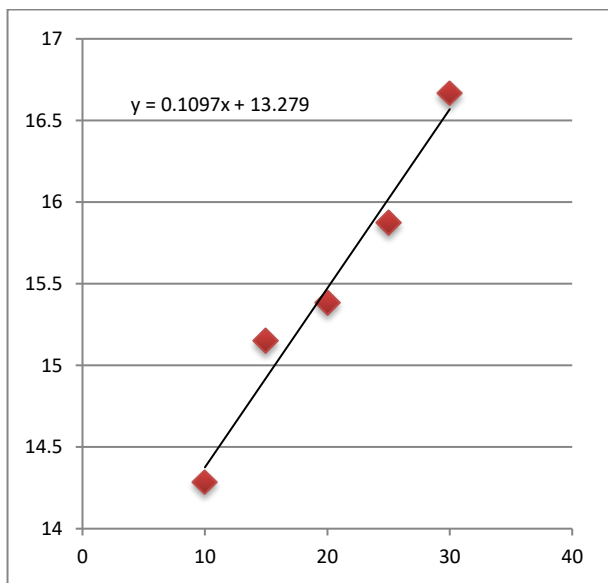


Fig. 3: Representation of Concentration – Time Data at 50 °C.

From the equation of the line in figure 3, the slope could be calculated as follows:

$$K = \text{slope} = 0.0712 \text{ dm}^3/\text{mol}\cdot\text{min} = 0.0022 \text{ L/mol}\cdot\text{s}$$

$$\frac{1}{c} - \frac{1}{c_0} = 0.0022t$$

Similarly, from the concentration results, namely (1/C), the rate constant was calculated as shown in Figure 4. The obtained values at Temperature =55 °C, by plotting t vs. 1/C are summarized in Table 4.

Table 4: Calculated Results at Different Reaction Times at 55°C

t (min)	V (NaOH)	C	1/C
10	8.2	0.068	14.70588
15	8.5	0.065	15.38462
20	8.7	0.063	15.87302
25	8.9	0.061	16.39344
30	9.3	0.057	17.54386

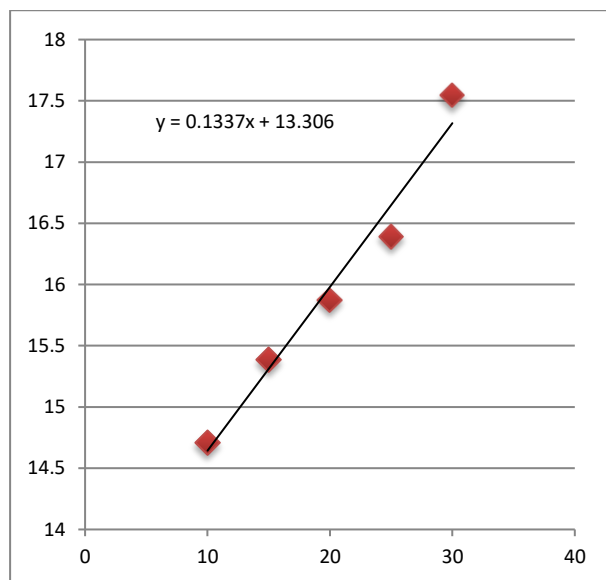


Fig. 4: Representation of Concentration – Time Data at 55 °C.

From the equation of the line in figure 4, the slope could be calculated as follows:

$$K = \text{slope} = 0.0712 \text{ dm}^3/\text{mol}\cdot\text{min} = 0.001187 \text{ L/mol}\cdot\text{s}$$

$$\frac{1}{c} - \frac{1}{c_0} = 0.001187t$$

Plot between ln K vs. 1/T to ensure that the reaction is second order reaction.

Table 5: Calculated Results at Different Reaction Temperatures.

T	K	1/T	ln K
303	0.000762	0.0033	-7.17956
313	0.001187	0.003195	-6.73633
323	0.0018	0.003096	-6.31997
328	0.002	0.003049	-6.21461

Arrhenius equation was used to calculate the activation energy of the reaction under investigation. The relation between ln(k) and (1/T) for second order kinetic model is shown in Figures 5.

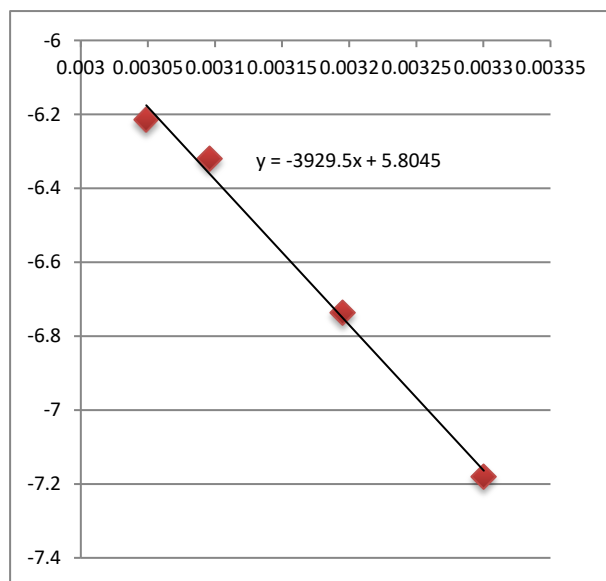


Fig. 5: Activation Energy for the Reaction According to the Second Order Model.

Since the plotted data gives a straight line between ln k vs. 1/T, then the experiments follow Arrhenius correlation and the reaction is second order.

Slope = - E/R = - (-3929.5), So, E = (8.314) (3929.5) = 32669.86 kJ/mol

After 20 minutes of taking all the samples the reaction has reached the steady state and no more changes in conversion

Then, Intercept = ln A = 5.8045, A = 331.7893

Table 6: Different Flow Rates at Temperature = 30 °C and 0.1 M Etac, 0.1 M Naoh

FEtAc, FNaOH	VNaOH	CNaOH (initial)	CNaOH (outlet)	X	molesNaOH (initial)	molesNaOH (outlet)	molesNaOH (reacted)
30%, 50%	9.5	0.05	0.005	0.9	0.27	0.00005	0.26995
50%, 70%	6	0.045678	0.04	0.12	0.26663	0.0004	0.24626
70%, 50%	8.6	0.03	0.014	0.533	0.162	0.00014	0.16186
50%, 30%	7.4	0.026495	0.026	0.0187	0.143071	0.00026	0.14281

Table 7: Different Flow Rates at Temperature = 50 °C and 0.1 M Etac, 0.1 M Naoh

FEtAc, FNaOH	VNaOH	CNaOH (initial)	CNaOH (outlet)	X	molesNaOH (initial)	molesNaOH (outlet)	molesNaOH (reacted)
30%, 50%	9.6	0.05	0.004	0.92	0.27	0.00004	0.26996
50%, 70%	9.1	0.04568	0.009	0.803	0.3784	0.00009	0.37831
70%, 50%	10.8	0.03	-0.008	1.2667			
50%, 30%	9.2	0.02649	0.008	0.6980	0.1622	0.00008	0.16212

Table 8: Comparison between the Maximum Conversions and Yields at All Conditions

	Best Conversion	Best Yield
T = 30 °C and 0.1 M EtAc, 0.1 M NaOH	90% (at 30% EtAc, 50%NaOH)	0.26995 moles (at 30% EtAc, 50%NaOH)
T = 50 °C and 0.1 M EtAc, 0.1 M NaOH	92% (at 30% EtAc, 50%NaOH)	0.37831 moles (at 50% EtAc, 70%NaOH)
T = 30 °C and 0.1 M EtAc, 0.2 M NaOH	28.3% (at 70% EtAc, 50%NaOH)	0.53957 moles (at 70% EtAc, 50%NaOH)
T = 50 °C and 0.1 M EtAc, 0.2 M NaOH	30% (at 70% EtAc, 50%NaOH)	0.5396 moles (at 70% EtAc, 50%NaOH)
T = 30 °C and 0.2 M EtAc, 0.1 M NaOH	41.33% (at 50% EtAc, 50%NaOH)	0.37804 moles (at 30% EtAc, 70%NaOH)
T = 50 °C and 0.2 M EtAc, 0.1 M NaOH	29.7% (at 70% EtAc, 30%NaOH)	0.37799 moles (at 70% EtAc, 30%NaOH)

4. Conclusion

In conclusion, the flow rate was monitored experimentally from twenty to thirty minutes, until it reaches the steady state after adding equal amounts of ethyl acetate and sodium hydroxide in each tank. The conversion increases as a result of the reactants takes a long time to react due to increasing of residence time.

It was determined that the order of the reaction is a second-order reaction.

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