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Evaluation of Ethyl Acetate Saponification Reaction: Comparison of the Accuracy of Conductometric and Titrimetric Methods

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Abstract

This study evaluated the kinetic characteristics of the ethyl acetate saponification reaction using two analytical approaches, namely conductometry and titrimetry, in a stirred batch reactor system at various temperature conditions. Both methods showed that the reaction followed an overall second-order pattern, in line with the theory of saponification reaction kinetics. However, significant differences were found in the conversion values and activation energy obtained. The titrimetric method showed higher reproducibility and accuracy, with an estimated activation energy of 37.40 kJ/mol. This study also confirmed that increasing the reactant concentration accelerated the reaction rate by increasing the frequency of effective intermolecular collisions. Overall, the titrimetric method is recommended as a more reliable approach in the study of saponification kinetics in this experimental system.

Keywords: Saponification, ethyl acetate, titrimetry, conductometry, batch reactor

1 Introduction

Ethyl acetate is an important organic solvent extensively used in numerous industrial applications, especially in the chemical sector. Its versatility has led to a steady increase in global demand. Ethyl acetate has a low level of toxicity and, most importantly, is biodegradable [1]. Ethyl acetate can react spontaneously with sodium hydroxide at low temperatures and ambient pressures with Gibbs reaction value ($\Delta G_r^o{}_{,298K}$) -146.74 kJ/mol, making this reaction suitable for studying reactions occurring in the chemical industry [2].

The ethyl acetate saponification is a fundamental reaction that is widely studied in chemistry and is widely used as a classical model to describe the mechanism of second-order reactions in the study of chemical kinetics. The reaction presented with **Eq. 1** [3–5].

$$NaOH + CH_3COOC_2H_5 \rightarrow CH_3COONa + C_2H_5OH \quad \text{(Equation 1)}$$

The kinetics performance of the ethyl acetate saponification reaction with sodium hydroxide is very dependent on some crucial operational parameters. The three main parameters that significantly affect the rate of this reaction are the system temperature, the velocity of the reactant solution flow in the reactor, and the initial concentration of each reactant. These three variables are not static; Each is systematically modified in order to evaluate its effect on reaction speed and conversion efficiency. By doing variations on temperature, flow rate, and reactant concentration, it is possible to identify the most optimal operational conditions to achieve the maximum reaction rate and the stability of the reaction system. Therefore, the right control of these parameters becomes an important aspect in experimental design and in industrial applications that require high reaction efficiency [6].

Studies on this reaction have been carried out extensively by various researchers through experimental approaches at different temperature ranges, with the use of various measurement methods to obtain accurate kinetic data. The volumetric titration method has been used independently by Alime eta al and Ikhazuangbe et al to monitor the progress of a reaction, through periodic sampling of the reactor. The main limitations of this approach lie in the precision of the sample volume that must be taken at a fixed time, as well as the potential bias due to the use of a color indicator in the titration process [7,8]. The conductometric technique reported by Ahmad et all, Afzal et al, and Walker [9–11] allows in-situ monitoring of reaction dynamics, without sampling intervention. Measurements are carried out manually, with accuracy highly dependent on the speed of the instrument's response during the conductometric process, so that response delays can cause significant deviations to the data obtained. Acidimetric and microcalorimetric techniques have been used to determine rate constants in ester saponification reactions. Although both methods provide high precision, their procedural complexity often limits practical implementation. To simplify this process, an alternative approach has been developed that incorporates online data acquisition and conductometric measurements, allowing kinetic parameters to be estimated with reduced operational complexity [12].

Although a number of studies have been conducted by various researchers, the data obtained regarding the rate constant and activation energy in the saponification reaction of ethyl acetate with sodium hydroxide show quite significant variations. Therefore, this study aims to compare two approaches - titrimetric and conductometric - in studying the kinetics of the saponification reaction of ethyl acetate.

2 Material and Method

The materials used in this study included ethyl acetate, sodium hydroxide (NaOH), distilled water, hydrogen chloride (HCl), and phenolphthalein indicator. The ethyl acetate samples and sodium hydroxide were obtained from the Merck.

In this study, the saponification reaction between ethyl acetate and sodium hydroxide was carried out in a stirred batch reactor system with a stoichiometric ratio of 1:1, each at a concentration of 0.1, 0.05 and 0.01M. 500 mL of ethyl acetate was added into the 1000 ml reactor with contain 500mL NaOH solution and prepared at a uniform temperature before the process began. The reaction proceeded under constant stirring using a magnetic stirrer at a speed of 200 rpm to ensure the homogeneity of the mixture [13]. As the reaction progressed, the concentration of OH^- ions decreased due to the consumption of reactants, while CH_3COO^- ions were formed as products. These changes in ionic composition were monitored in real-time by measuring the conductivity of the solution using a conductivity meter. The conductivity values were recorded at specific time intervals until a steady state was reached, indicating the completion of the reaction. In the titrimetric procedure, a 10 mL aliquot of the reaction mixture was transferred into a 50 mL Erlenmeyer flask, homogenized, and subsequently titrated with 0.05 M HCl, using phenolphthalein to determine the endpoint. To investigate the influence of temperature on the reaction kinetics, the experiments were repeated at temperatures between 25 °C and 50 °C, with the actual reaction temperature carefully monitored and controlled using an alcohol thermometer while heating on a hot plate. A schematic of the experimental system can be seen in **Fig. 1**.

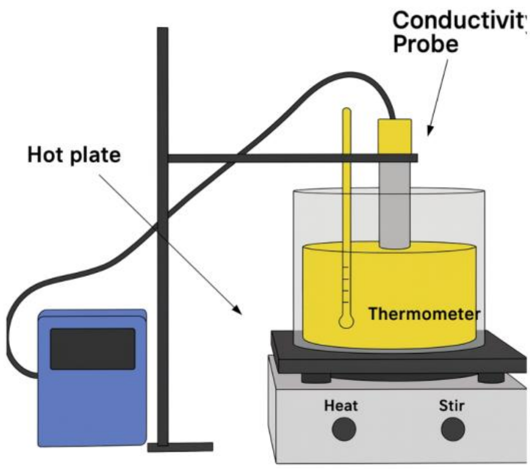

Figure 1. Experimental setup of saponification ethyl acetate

The conversion value of NaOH is determined by calculation using **Eq. 2**.

$$X_{\mathrm{NaOH}} = \frac{c_{\mathrm{NaOH,o}} - c_{\mathrm{NaOH}}}{c_{\mathrm{NaOH,o}}} \cdot 100\% \qquad \text{(Equation 2)}$$

where $c_{\mathrm{NaOH,0}}$ is the initial concentration of NaOH, and c_{NaOH} is the NaOH concentration at the final.

3 Result and Discussion

3.1 Calibration Data

Conductivity measurements of the reactants and products were carried out to determine the specific conductivity values of each compound involved, namely sodium hydroxide, ethyl acetate, sodium acetate, and ethanol. Measurements were carried out on each compound in the concentration range of 1 M to 0.01 M, using a conductivity meter that had been previously calibrated with standard solutions. The conductivity measurement results are presented in **Table 1 - 4**.

Table 1. Conductivity of pure water

Conductivity (mS)	Temperature (ºC)
0.376	25.6

Table 2. Conductivity of NaOH solution

Concentrations (M)	Conductivity (mS)	Temperature (ºC)
0.10	15.93	26.7
0.05	9.30	26.9
0.01	2.21	26.2

Table 3. Conductivity of ethyl acetate solution

Concentrations (M)	Conductivity (mS)	Temp. (ºC)
0.10	0.189	26.9
0.05	0.351	27.3
0.01	0.371	26.8

Table 4. Conductivity of ethanol solution

Concentrations (M)	Conductivity (mS)	Temp. (ºC)
0.10	0.305	27.5
0.05	0.348	27.5
0.01	0.371	26.8

Table 5. Conductivity of sodium acetate solution

Concentrations (M)	Conductivity (mS)	Temp. (ºC)
0.10	4.61	25.9
0.05	2.62	26.6
0.01	0.88	26.8

Electrical conductivity measurements performed separately on each solution showed that sodium hydroxide (NaOH) had the highest conductivity compared to the other compounds. At the same concentration of 0.1 M, the NaOH solution showed a conductivity of 15.93 mS, while sodium acetate, ethyl acetate, and ethanol showed values of 4.61 mS, 0.371 mS, and 0.371 mS, respectively.

This difference in conductivity is consistent with the basic principle of electrochemistry, where strong electrolytes produce a greater number of ions in solution, thus conducting electric current more efficiently than weak electrolytes. Sodium hydroxide, as a strong electrolyte, undergoes almost complete ionic dissociation in solution, producing highly mobile Na^+ and OH^- ions, which contribute significantly to the increased electrical conductivity of the solution.

3.2 Conversion

The conversion value reflects the efficiency of reactant utilization in producing products. High conversion indicates optimal reactor performance in facilitating chemical reactions. Therefore, conversion evaluation is an important indicator in analyzing reaction process performance.

In this study, conversion analysis was carried out using two analytical approaches, namely conductometric and titrimetric methods, at six variations of reaction temperatures: 25°C, 30°C, 35°C, 40°C, 45°C, and 50°C. The conversion values obtained are presented in **Table 6**. Based on the test results using the conductometric method, the highest conversion value was obtained at 79.3% at a temperature of 50°C, while the lowest value was recorded at 76.4% at a temperature of 25°C. In contrast, the titrimetric method showed a higher conversion gain, with a maximum value of 97.0% at 50°C and a minimum of 91.0% at 25°C.

Table 6. Conversion of sodium hydroxide with initial concentration of sodium hydroxide 0.05M

Temp. (ºC)	X_{NaOH}(%)	
	conductometric	titrimetric
25	76.4	91.0
30	79.3	92.0
35	77.2	93.0
40	78.8	93.0
45	78.9	95.0
50	79.3	97.0

The difference in conversion values between the conductometric and titrimetric methods, which ranges from 12.65% to 17.65%, is due to the limitations of each method. Conductometric is susceptible to ionic interference from the sodium acetate product, which has a relatively high conductivity, resulting in lower conversion readings. In contrast, titrimetric is more accurate because it is based on a selective and precise stoichiometric reaction.

Despite differences in absolute values between the methods, both methods consistently show a trend of increasing conversion with increasing reaction temperature. This is in line with the principle of chemical reaction kinetics, where increasing temperature increases the kinetic energy of molecules, thereby increasing the frequency of effective collisions and increasing the reaction rate. Consequently, at higher temperatures, more reactants react at the same time, resulting in higher conversions.

3.3 Kinetics

This kinetic study was conducted through experimental measurements in a batch reactor, as previously described. Since the saponification reaction of ethyl acetate is a second-order reaction with an equivalent molar ratio, the sodium hydroxide concentration was determined based on the kinetic formula **Eq. 3-6**.

$$-r_A = -\frac{dC_A}{dt} = -\frac{dC_B}{dt} = kC_AC_B \qquad \text{(Equation 3)}$$

$$-\frac{dC_A}{dt} = kC_A^2 \qquad \text{(Equation 4)}$$

$$\int_{C_{A0}}^{C_A} \frac{-dC_A}{C_A^2} = k\int_{t_0}^{t} dt \qquad \text{(Equation 5)}$$

$$\frac{1}{C_A} = kt + \frac{1}{C_{A0}} \qquad \text{(Equation 6)}$$

where,

$-r_A = -\frac{dC_A}{dt}$ = sodium hydroxide reaction rate (mol/L.s)

$-\frac{dC_B}{dt}$= ethyl acetate reaction rate (mol/L.s)

k = forward reaction rate constant (L/mol.s)

C_A= sodium hydroxide concentration (mol/L)

C_B= ethyl acetate concentration (mol/L)

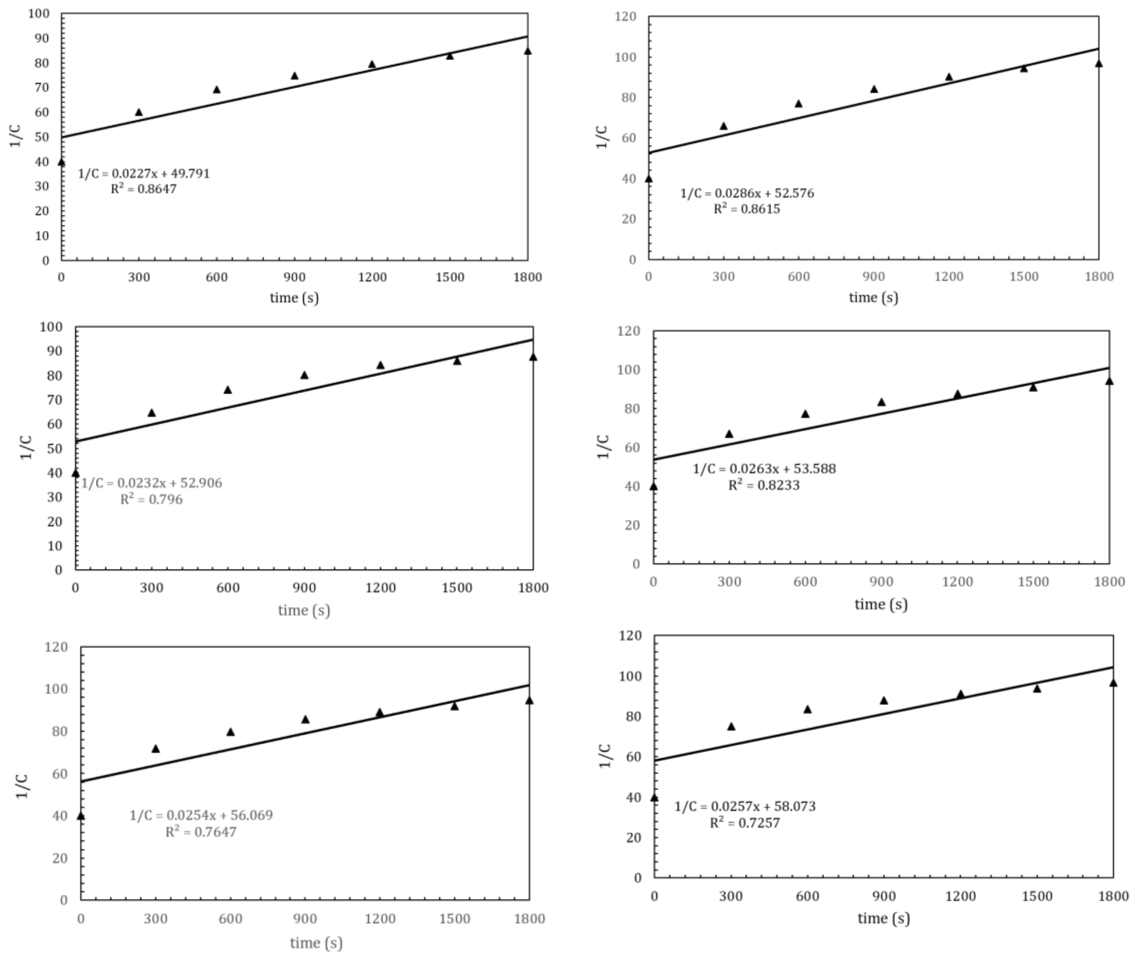

Figure 2. Fitting data of concentration versus time using conductometric method with initial concentration of sodium hydroxide 0.05M

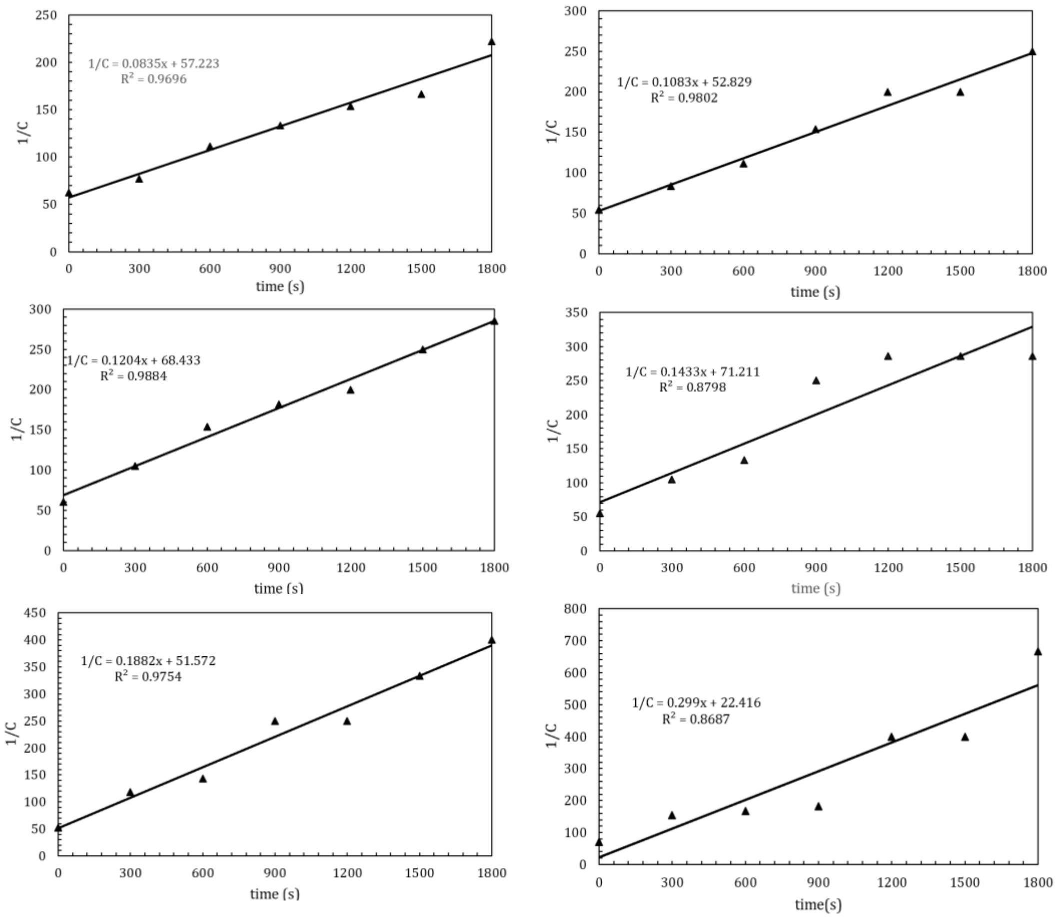

Figure 3. Fitting data of concentration versus time using titrimetric method with initial concentration of sodium hydroxide 0.05M

In the second-order reaction kinetics approach, one of the commonly used methods to determine the reaction rate constant (k) is by analyzing the relationship between the inverse of the reactant concentration (1/C) and time (t). Mathematically, this approach refers to the linear equation of the integrated form of the second-order reaction rate law as shown by equation (5). By plotting 1/C against time t, a linear graph is obtained as shown in **Fig. 2** and **Fig. 3** with a slope representing the reaction rate constant k. The validity of the kinetic model can be confirmed through the coefficient of determination (R^2) which shows the suitability of the experimental data to the second-order model.

The results of conductometric and titrimetric analyses indicate that the reaction is second-order, as evidenced by the linear plot of 1/C versus time using the integral method. The consistency of linearity in both methods confirms that the reaction rate is proportional to the square of the reactant concentration.

By analyzing the reaction rate constant at various temperatures, the activation energy and pre-exponential factor of the ethyl acetate saponification reaction can be determined through the Arrhenius equation approach, which describes the sensitivity of the reaction rate to temperature changes. Where k is the reaction rate constant which is thermodynamically described by the Arrhenius equation (**Eq. 7**).

$$k = Ae^{-\frac{E_a}{RT}} \qquad \text{(Equation 7)}$$

where,
A = frequency factor
Ea = activation energy (J/mol)
R = universal gas constant (8.314 J/mol·K)
T = absolute temperature (Kelvin)

The values of the reaction rate constant and activation energy can be obtained by solving the Arrhenius **Eq. 7**, shown in **Eq. 8-10**.

$$\ln k = \ln A + \ln\left(e^{\frac{-E_a}{RT}}\right) \qquad \text{(Equation 8)}$$

$$\ln k = \ln A - \frac{E_a}{RT} \qquad \text{(Equation 9)}$$

$$\ln k = -\frac{E_a}{R} \cdot \frac{1}{T} + \ln A \qquad \text{(Equation 10)}$$

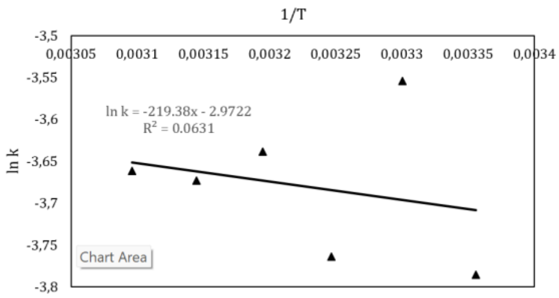

Figure 4. The Arrhenius equation is used to describe the dependence of the reaction rate coefficient on temperature for the conductometric method.

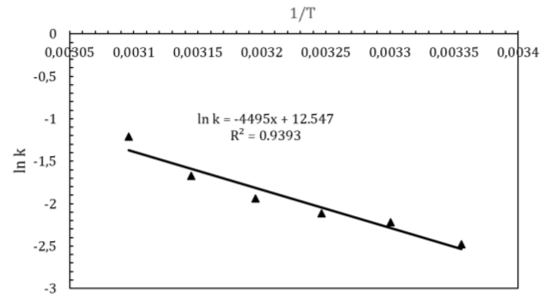

Figure 5. The Arrhenius equation is used to describe the dependence of the reaction rate coefficient on temperature for the titrimetric method.

A plot of ln k against 1/T yields a linear line as shown in **Fig. 4** and **Fig. 5**, where the slope is equal to $-Ea/\mathrm{R}$ and the intercept, when antilogated, gives the value of A.

The difference in the reaction rate constant values obtained has a significant effect on the activation energy estimation based on the Arrhenius equation as presented in **Table 7**. The data processing results show that the activation energy obtained using the conductometric method is 1.82 kJ/mol, while the titrimetric method produces a value of 37.40 kJ/mol. This difference is strongly suspected to be caused by inconsistencies in the readings of the NaOH solution concentration, as previously explained.

When compared with literature reference data [14,15], the activation energy values from the titrimetric method show relatively small deviations, indicating that the analytical procedures and results using this method have been carried out validly and are close to ideal conditions. In contrast, the values obtained from the conductometric method show quite significant deviations from the literature, indicating potential procedural or instrumental irregularities that require further evaluation. Thus, a review of the conductometric method is needed to improve the accuracy of the results and the consistency of the data produced.

Table 7. Rate constant and activation energy

Temp. (K)	**Conductometric Method**		**Titrimetric Method**	
	k	*Ea* (kJ/mol)	*k*	*Ea* (kJ/mol)
298	0.0227	1.82	0.0835	37.40
303	0.0286		0.1083	
308	0.0232		0.1204	
313	0.0263		0.1433	
318	0.0254		0.1882	
323	0.0257		0.2990	

This study also examined the effect of concentration variations on reaction rates through conductometric analysis. Observations showed that increasing feed concentration was directly proportional to the increase in the reaction rate constant, as shown in **Table 8**. Increasing the concentration of reactants accelerates the reaction rate because the number of effective collisions between molecules also increases.

Table 8. Comparison of reaction rate constants at a temperature of 25°C

Initial Conc. (M)	**Rate constant, *k* (L/mol.s)**
0.10	0.0674
0.05	0.0227
0.01	0.0074

4 Conclusion

This paper evaluated two analytical methods, conductometric and titrimetric, to study the kinetics of the ethyl acetate saponification reaction. The results showed that both methods indicated an overall reaction order of two, consistent with the literature for saponification reactions. However, significant differences were found in the conversion values and activation energies between the two methods. The titrimetric method produced more consistent and reliable data, with an activation energy value of 37.40 kJ/mol. Furthermore, the experimental results showed that increasing the reactant concentration contributed to an acceleration of the reaction rate, as the frequency of effective collisions between particles increased. Overall, the titrimetric method was deemed more accurate for characterizing the kinetics of the saponification reaction under the experimental conditions used in this study.

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