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RESEARCH ARTICLE

Kinetic Study and Activation Parameters of Alkali Catalyzed Hydrolysis for Ethyl Caprylate in Aqueous Acetone Solvent Media

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Abstract

This study investigated the kinetics of alkaline hydrolysis of ethyl caprylate at different temperatures from 20 °C to 40 °C, differing by 5 °C and 40% to 80% acetone composition in aqueous acetone binary media. It evaluates specific reaction rate, iso-composition activation energy, thermodynamic activation parameters, and isokinetic temperature by using laboratory experimental design, volumetric method of analysis and titration technique. A controlled laboratory experimental design, volumetric method of analysis and titration technique were used to study the alkaline hydrolysis of ethyl caprylate. The specific reaction rate (k) measured by using second order kinetic equation was decreased gradually with increasing percentage of acetone in aqueous-acetone media. Iso-composition activation energy (Ec) required for alkaline hydrolysis of ethyl caprylate was found to be decreased with increasing composition of acetone in reaction media. The recorded value of free energy of activation (ΔG^*), known as solvolysis of ethyl caprylate, was decreased very slowly with increasing composition of acetone in reaction media. Evaluated values of enthalpy of activation (ΔH^*) for hydrolysis reaction were also decreased with increasing % of acetone in reaction. The observed value of entropy of activation ($\Delta S*$) for hydrolysis of ethyl caprylate in alkaline medium was reduced differently. Observed value of entropy of activation was decreased from positive values to zero and then negative values at 70% and 80% composition of acetone in water acetone binary solvent system. The value of enthalpy of activation (ΔH^*) was compensated by the value of entropy of activation ($\Delta S*$) in alkaline ester hydrolysis reaction. The recorded isokinetic temperature for alkaline hydrolysis of ethyl caprylate was found to be 291.7 Kelvin indicating slightly weak solvent-solute interaction in the binary reaction media. The study concluded that the values of specific reaction rate and thermodynamic activation parameters for alkaline hydrolysis of ethyl caprylate are influence by composition of organic cosolvent in aqueous-acetone media.

Keywords: activation parameters, hydrolysis, organic cosolvent, rate constant, titration technique.

Introduction

The studies related to the alkali catalyzed hydrolysis of esters of higher fatty acids in aqueous organic cosolvent media are applicable to increase the rate of formation of required products and to decrease the rate of formation of undesired byproducts in laboratory and industrial scale. The present research involving nontoxic chemicals and reagents is important from the view of industrial, medicinal, biochemical, and environmental aspects. The reactant ethyl caprylate ester can be extracted from natural fruit sources: strawberry, pineapple, guava,, and can be used as a flavoring agent in industries as well as an additive in beverages and dairy products. The major product, caprylic acid, of the hydrolysis reaction has health care and industrial benefits.

Although a large number of works have been reported on the acid or base catalyzed hydrolysis of alkyl or acyl halides, amides, and esters of carboxylic acids, only limited attention has been given to the hydrolysis of esters of higher fatty acids having longer carbon chains. Mabey and Mills (1978) examined the rate constants for hydrolysis of 12 classes of organic compounds in water under environmental conditions. The kinetic result of the hydrolysis of ethyl caprylate and propyl caprylate separately in different aqueous acetone media and temperatures has been investigated (Singh, 2021, 2025). Similarly, Bano and Singh (2017), Tiwari and Sigh (2020), Changi, Pinnarat, and Savage (2011), and Singh et al. (2002) had analyzed the hydrolysis of esters of fatty acid under different conditions.

The objectives of the research are to investigate the influence of dipolar aprotic organic cosolvent (acetone) composition on the specific reaction rate, iso-composition activation energy, and thermodynamic activation parameters from 40% to 80% composition of acetone at different temperatures ranging from 20 °C to 40 °C, differing by 5 °C. The study is theoretically based on the Arrhenius equation (Maron & Prutton, 1972), and the Wynne-Jones and Erying equation derived on the basis of Absolute Rate Theory.

Materials and Methods

Materials: Equipments, Apparatus and Chemicals

The main apparatus used to study the influence of the composition of organic cosolvent acetone on the rate and activation parameters for alkaline hydrolysis of ethyl caprylate in aqueous acetone media are: Baryta set of Borosil make, water thermostat, pipettes, volumetric flasks, conical flasks, stop watch. The important chemicals required for this study are: ethyl caprylate, acetone, hydrochloric acid, sodium hydroxide, baryta solution, phenolphthalein, and methyl orange indicators. All glass apparatus used during the experimental work were of Borosil brand grade, and all the chemicals were of analytical grade.

Methods

The alkali catalyzed hydrolysis of ethyl caprylate in aqueous acetone media at different temperatures from 20 ^{o}C to 40 ^{o}C differing by 5 °C, and different compositions of acetone from 40% to 80% was analyzed by using laboratory experimental design, volumetric method of analysis, and titration technique. In the reaction mixture, prepared by thermostated chemicals, the concentration of ethyl caprylate and alkali NaOH was 0.05 M and 0.1 M, respectively with the required percentage of acetone (40% to 80%) and water. Ester, ethyl caprylate, was added last, and stopwatch was started at half the mixing time. Immediately, 10 ml of the reaction mixture was withdrawn and allowed to run into the conical flask containing 20 ml of 0.1N HCl so that the reaction mixture could be quenched quickly. Excess of acid in the flask was titrated with standard (0.05N) baryta solution using phenolphthalein as an indicator. The titre values at 0 (T_0), after 5, 10, 20, 35, 55 minutes (T_t), at infinity time (T_∞) and at the end of reaction (T) were noted. 20 ml of 0.1N HCl in a conical flask was titrated with baryta solution and volume was noted as T.

Evaluation of Rate Constant

The values of the rate constant for the alkali catalyzed hydrolysis of ethyl caprylate were evaluated by using the second-order kinetic equation (Sharma & Gupta, 2018).

$$k = \frac{2.303}{t(a-b)} \log \frac{b}{a} \left(\frac{a-x}{b-x}\right)$$

Or $$k = \frac{2.303}{t\,(T-T_\infty)} \log \frac{(T_\infty - T_0)(T-T_t)}{(T-T_o)(T_\infty - T_t)}$$

$$\log \left(\frac{T-T_t}{T_\infty - T_t}\right) = \frac{k\,(T-T_\infty)t}{2.303} + \log \frac{(T-T_0)}{T_\infty - T_o}$$

Where, $a = T - T_o$ = Initial concentration of alkali in reaction mixture,

$b = (T_\infty - T_o)$ = Initial concentration of ester in reaction mixture,

$(a-x) = (T-T_t)$ = concentration of akali at time t,

$(b-x) = (T_\infty - T_t)$ = concentration of ester at time t,

Specific reaction rate for reaction was evaluated by measuring the slope of the $\log \frac{(T-T_t)}{(T\infty - T_t)}$ versus time (t) plot for each set of experiment.

$$Slope = \frac{k(T-T_\infty)}{2.303}, and\ k = 2.303 \times \frac{Slope}{(T-T_\infty)}.$$

The precision of rate constant was tested by average and standard deviation technique.

Calculation of Isocomposition Activation Energy

The values of isocomposition activation energy (E_c) were calculated with the help of Arrhenius equation (Maron & Prutton, 1972).

$$k = A\, e^{-Ec\,/RT}$$

$$\log k = \log A - \frac{Ec}{2.303} \frac{1}{T}$$

$$Slope = -\frac{Ec}{2.303\,R}, \text{ and } \qquad E_c = -2.303\ R \times Slope$$

Isocomposition activation energy (E_c) was calculated by measuring the slope of the straight

line obtained on plotting log k versus 1/T.

Calculation of thermodynamic activation parameters

To study the effect of solvent on thermodynamic activation parameters, free energy of activation (ΔG*), enthalpy of activation (ΔH*), and entropy of activation ((ΔS*), were evaluated by using a mathematical equation derived by Wynne-John and Eyring (Jones & Eyring, 1935) based on the Absolute Rate Theory (Glass Stone, Laidler & Eyring, 1941)

$$k = \frac{kT}{h}\, e^{\frac{-\Delta G*}{RT}}$$

$$\log k = \log \frac{kT}{h} - \frac{\Delta G^*}{RT}$$

$$\Delta G^* = 2.303\ RT(\log \frac{kT}{h} - \log k)$$

Where, k = Boltzmann constant, h = planck constant

The values of free energy of activation, also known as solvolysis of ethyl caprylate, were calculated from the known value of specific reaction rates at different temperature and % composition of acetone.

Rearranging Eyring equation,

$$\log \frac{k}{T} = \log \frac{kT}{h} + \frac{\Delta S^*}{2.303} - \left(\frac{\Delta H^*}{2.303R}\right) \frac{1}{T}$$

The values of enthalpy of activation (ΔH*) at different composition of acetone in aqueous

acetone media were evaluated by measuring the slope of the log $^{k/T}$ versus $^{1/T}$ of above straight line. ΔH* = -2.303 R × slope

The values of entropy of activation were calculated from the Gibbs-Helmholtz equation from the known values of ΔH* and ΔG*.

$$\Delta S^* = \frac{\Delta H^* - \Delta G^*}{T}$$

ΔS* may also be evaluated from the intercept of the rearranged Eyring equation.

Isokinetic Temperature

At isokinetic temperature, also known as solvent stabilizer operator (Loffler & Granwald, 1963), all similar reactions with different compositions of solvent have some specific reaction rate due to the enthalpy of activation and entropy of activation compensation in chemical reaction. At this temperature, the difference in enthalpy of activation (ΔH^*) are compensated by the difference in entropy of activation (ΔS^*). Chemical reactions with higher activation enthalpy have more favorable entropy and vice versa. Isokinetic temperature (β) was evaluated by measuring the slope of the ΔH^* versus ΔS^* plot straight line (Loffler, 1955).

$$\Delta H^* = \beta \Delta S^* + \text{constant}$$

$$\text{Isokinetic temperature, } \beta = \frac{\Delta H^*}{\Delta S^*}$$

β supports understanding the solvent-solute interactions in the reaction mixture (Barclay & Butler, 1938). For chemical reactions with considerable solvent-solute interactions, β the temperature falls between 300 K to 400 K.

Results and Discussion

Results

To evaluate the influence of solvent on alkali catalyzed hydrolysis of ethyl caprylate at different compositions of organic cosolvent acetone at different temperatures from 20 to 40 °C differing by 5 °C, experimentally recorded values of specific reaction rate (k) are given in Table1.

Table 1

Specific Reaction Rate Values for Alkali Catalyzed Hydrolysis of Ethyl Caprylate in Aqueous-Acetone Media

$k \times 10^3$ (dm)^3 / mol/ min

Temp (°C)	Percentage of Acetone (V/V)				
	40%	50%	60%	70%	80%
20	28.13	26.41	24.00	22.82	21.49
25	54.66	48.65	43.95	40.20	35.10
30	105.37	89.82	78.33	68.06	56.77
35	125.84	160.25	134.55	125.04	89.36
40	366.21	287.15	232.81	187.27	147.59

Table 1 presents the observed values of specific reaction rate for alkali catalyzed hydrolysis of ethyl caprylate from 40% to 80% acetone composition and at 20 °C to 40 °C temperature.

The values of (3+ logk) at different $10^3/T$ values and different % of acetone are given in Table 2.

Table 2

Variation of (3 + log k) Values with $10^3/T$ at different % of Acetone for Alkaline Hydrolysis of Ethyl Caprylate in Aqueous Acetone Media

(3+ logk) values

Temp (°C)	$10^3/T$	Percentage of Acetone (V/V)				
		40%	50%	60%	70%	80%
20	3.411	1.449	1.422	1.380	1.358	1.332
25	3.354	1.738	1.687	1.643	1.604	1.545
30	3.300	2.023	1.953	1,894	1.833	1.754
35	3.245	2.292	2.211	2.132	2.034	1.951
40	3.193	2.564	2.458	2.367	2.272	2.169

Table 2 Shows the change of recorded values of (3 + log k) with temperature and

percentage of acetone in aqueous acetone media for alkali catalyzed hydrolysis of ethyl caprylate.

Figure 1

Graphical Plot of (3+log k) Versus Values10³/T Values for Alkaline Hydrolysis of Ethyl Caprylate in Different % Composition of Acetone in Aqueous-Acetone Media

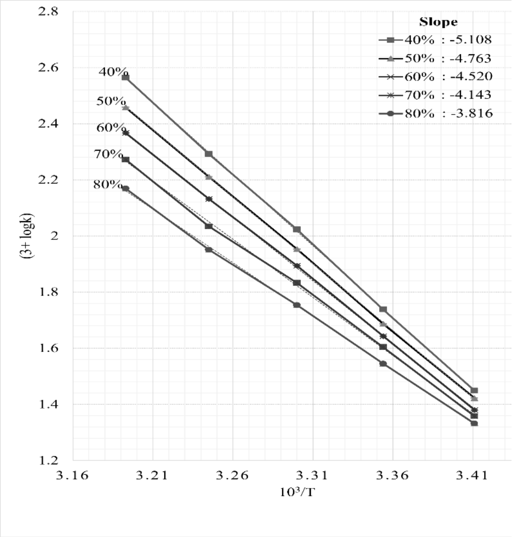


Figure 1 shows the variation of recorded values of the logarithm of specific reaction rate (3+ logk) with the reciprocal of temperature ($10^3/T$) in different % composition of acetone in aqueous-acetone media. From the slope of these straight lines, the isocomposition activation energy (E_c) is evaluated.

Calculated values of is-composition activation energy (E_c) obtained from the slope of (3 + logk) versus $10^3/T$ plot at different % of acetone from Figure 1 is given in Table 3.

Table 3

Calculated values of Isocomposition Activation Energy (Ec) at Different % of Acetone in Ethyl Caprylate Hydrolysis Reaction in Water Acetone Media

Percentage of Acetone (V/V)	40%	50%	60%	70%	80%
Ec (k J mol-1)	98.109	91.504	86.851	79.613	73.314

Table 3 represents the iso-composition activation energy decreases with increasing % amount of organic cosolvent acetone in the reaction mixture.

The free energy of activation (ΔG*) for alkali catalyzed hydrolysis reaction in aqueous - acetone media, calculated from Erying equation, is given below.

Table 4

Values of ΔG at Different Temperatures and Percentage Composition of Acetone in Aqueous Acetone Media for Alkaline Hydrolysis of Ethyl Caprylate*

ΔG (kJ/mol)*

Temp (^oC)	Percentage of Acetone				
	40%	50%	60%	70%	80%
20	80.478	80.630	80.865	80.989	81.135
25	80.237	80.526	80.777	80.937	81.337
30	79.967	80.367	80.716	81.070	81.528
35	79.238	79.717	80.138	80.191	81.245
40	79.445	80.081	80.626	81.190	81.807

The variation of free energy of activation with temperature and % amount of acetone for ester hydrolysis reaction are expressed in Table 4.

The evaluated values of enthalpy of activation according to Erying equation at different percentage of acetone the value of $(5 + \log {}^{k}/T)$at different 10^3 is given below:

Table 5

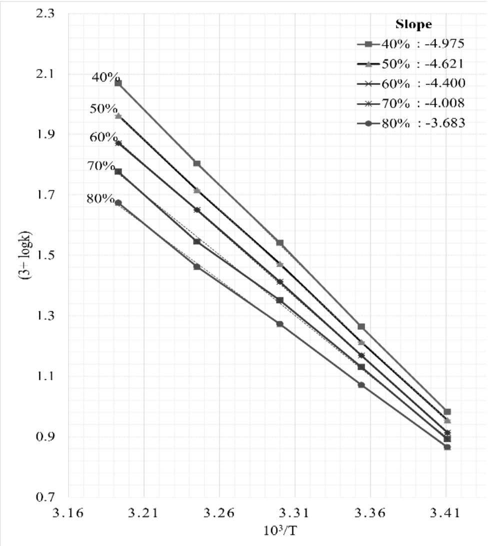
Values of $(5 + \log {}^{k}/\mathrm{T})$ *and* 10^3 *at Different Composition of Acetone for Alkaline Hydrolysis of Ethyl Caprylate in Aqueous Acetone Media*

Temp (°C)	10^3/T	Percentage of Acetone				
		40%	50%	60%	70%	80%
20	3.414	0.9820	0.9546	0.9131	0.8912	0.8651
25	3.354	1.2632	1.2126	1.1685	1.130	1.0708
30	3.300	1.541	1.4717	1.4122	1.3508	1.2725
35	3.245	1.8031	1.7160	1.6500	1.5448	1.4623
40	3.193	2.0679	1.9623	1.8712	1.776	1.6733

The values of $(5 + \log {}^{k}/_{\mathrm{T}})$ decreases with increasing % of acetone in aqueous-acetine media for alkaline hydrolysis of ethyl caprylate ester.

Figure 2

Graphical Plot of (5+Log k) Versus Values103/T Values for Alkaline Hydrolysis of Ethyl Caprylate in Different % Composition of Acetone in Aqueous Acetone Media



The variation of recorded values of the logarithm of specific reaction rate (5+ logk) with the reciprocal of temperature (10^3/T) in different % composition of acetone in aqueous-acetone media is presented in Figure 2. From the slope of these straight lines, the enthalpy of activation is evaluated activation (ΔH*) is evaluated.

The values of the enthalpy of activation for base-catalyzed hydrolysis of ethyl caprylate as evaluated by measuring the slope of a straight line obtained by plotting log ^{k} versus ^{10} from Figure 2 are listed below.

Table 6 presents the change (decrease) of ΔH* with the percentage composition of acetone in aqueous acetone media for the ester ethyl caprylate hydrolysis reaction.

According to Gibbs-Helmholtz equation, the evaluated values of free entropy of activation (ΔS*) from the known values of ΔH* and ΔS* at different temperatures and % of acetone is listed below.

Table 6

Value of ΔH at Different Percentage of Acetone in Aqueous-Acetone Media.*

% of Acetone	ΔH* (kJ /mol)
40	95.256
50	88.478
60	84.247
70	76.741
80	70.518

Figure 7 expresses the evaluated values of free entropy of activation (ΔS*), which decreases with increasing % composition of acetone.

Table 7

Variation of Values of ΔS at Different Temperature and Composition of Acetone in Aqueous-Acetone Media for Alkaline Hydrolysis of Ethyl Caprylate.*

ΔS +100 J/K/mol*

Temp (^oC)	Percentage of Acetone				
	40%	50%	60%	70%	80%
20	150.411	126.771	111.536	85.510	63.787
25	150.373	127.640	111.638	85.927	63.716
30	149.114	126.756	111.647	85.720	63.861
35	151.981	128.430	111.647	88.804	65.193
40	150.490	128.845	111.563	88.986	63.953

Table 8

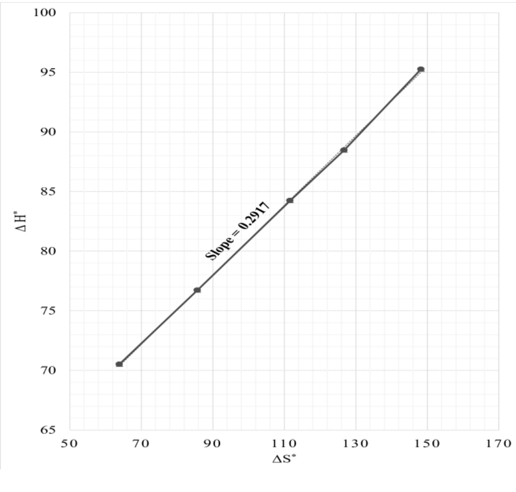
Values of ΔH and ΔS* (at 30 oC) Different Percentage of Acetone In Aqueous Acetone Media*

% of Acetone	ΔH* (kJ mol)	ΔS* (J/mol/K)	(ΔS* +100)(J/mol/K)
40	95.256	49.144	149.114
50	88.478	36.756	136.756
60	84.247	11.647	111.647
70	76.741	-14.28	85.720
80	70.518	-36.139	63.861

Evaluated values of ΔH* and ΔS* were decreased with increasing % composition of acetone, as presented in Table 8. ΔS* reduced to a negative value at a higher % of acetone in the reaction media.

Figure 3

*Graphical Plot of ΔH*Versus ΔS* Values for Alkaline Hydrolysis of Ethyl Caprylate in Aqueous-Acetone Media*



To evaluate the isokinetic temperature of reactions at different compositions of acetone for alkali catalyzed hydrolysis of ethyl caprylate, the evaluated values of ΔH* and ΔS* given in Table 8 were plotted.

Figure 3 presents the plot of evaluated values of ΔH* versus ΔS*is straight line. The slope of the line gives the isokinetic temperature for alkaline hydrolysis of ethyl caprylate at different temperature and composition of acetone in aqueous acetone media. From the figure, the isokinetic temperature was found to be 291.7 K.

Discussion

The observed specific reaction rate (k) values for alkali catalyzed hydrolysis of ethyl caprylate at different temperatures from 20 ^oC to 40 ^oC and different aqueous acetone compositions from 40% to 80%, calculated by using the second-order kinetic equation, are given in Table 1. At a particular composition of acetone in aqueous acetone media, the value of the specific reaction rate was increased with increasing temperature. It is due to increasing molecular energy, decreasing hydrogen bonding and solvation of reactants, and decreasing viscosity of the medium. At a given temperature, the observed value of specific reaction rate (k) was decreased slowly and gradually with increasing composition of acetone in aqueous acetone media. The rate constant value was decreased from 366.21×10^{-3} $(dm)^3$/mol/min at 20°C and 40% composition to 21.49×10^{-3} $(dm)^3$/mol/min at 40 °C and 80% composition of acetone in aqueous acetone media. With increasing acetone composition, the dielectric constant of the medium decreases, the solvation of the carbonyl part of the ester decreases, and the bonding of OH^- by solvent cluster increases; therefore, the rate of hydrolysis reaction decreases. Similar findings had been reported by Singh (2021) for the rate constant in the alkali catalyzed hydrolysis of ethyl caprylate in aqueous acetone media. Experimentally recorded isocomposition activation energy (E_c) values calculated by using Arrhenium equation for different compositions of acetone in binary media are listed in Table 3. With increasing the percentage of acetone in aqueous acetone media, the solubility of the hydrocarbon part of ethyl caprylate increases, the solvation of the ester carbonyl part decreases, enthalpy compensates entropy in the reaction and activation energy decreases.

The influence of solvent on thermodynamic activation parameters for the ester ethyl caprylate hydrolysis reaction in aqueous acetone media was evaluated by using the Eyring equation (Singh, 2025). Calculated values of free energy of activation (ΔG*), also known as solvolysis of ethyl caprylate, are presented in Table 4 at different temperatures and compositions of acetone in reaction media. Values of ΔG* were increased gradually from 40% composition of acetone to the 80% composition of acetone in water acetone reaction media. With an increasing amount of acetone, the increase in energy of the transition state is more than that of the reactants as well as the reactant state is moderately solvated, and the transition state is poorly solvated. The change of enthalpy of activation (ΔH*) is compensated by entropy of activation (ΔS*) in the reaction, and ΔG* increases only slowly. Enthalpy of activation (ΔH*) values

evaluated by measuring the slope of $\log^{k/T}$versus $^{1}/_{T}$ using Erying equation are given in the Table 6. The value of ΔH* was decreased on increasing the composition of acetone in aqueous- acetone media. With increasing % of acetone, the solubility of hydrophobic reactant ester increases, the solvation of the reactant hydroxyl ion decreases, the dispersion of the reactant increases, energy of the reactants increases. and ΔH* decreases. The analyzed values of entropy of activation (ΔS*) for base-catalyzed hydrolysis of ethyl caprylate in water acetone media using the Gibbs-Helmholtz equation at different temperatures and concentrations of acetone in aqueous acetone media are presented in Table 7. The entropy of activation values was decreased with increasing composition of acetone from 40% to 80% at each temperature and its value became negative -14.280 and -36.139 J/K/mol at 70% and 80% acetone respectively in aqueous acetone media. The decrease of experimental ΔS* values with an increase % of acetone in the reaction media is due to a relatively less oriented reactant state than that of the transition state. The dipolar aprotic organic co-solvent acetone stabilizes the ion-dipole activated complex more than the reactants. A similar result has been reported by Singh (2025).

Isokinetic temperature (β), also known as the solvent stabilization operator, was evaluated by measuring the slope of ΔH* versus ΔS* plot from the straight line $\Delta H^* = \beta\Delta S^* +$ constant. The value of β was found to be 291.7 K. For most of the organic reactions with considerable solvent solute interactions, the value of β falls between 300K to 400K.A smaller value of recorded isokinetic temperature (291.7 K) shows slightly weaker solvent-solute interaction in aqueous acetone media. At isokinetic temperature, all similar reactions in the series have same specific reaction rate and enthalpy change (ΔH^*) is completely balanced by entropy change (ΔS^*). The linear relation of ΔH^* versus ΔS^* plot shows the mechanism of S_N2 reaction remains same throughout at all solvent compositions, but the solvation may be changed.

Conclusion

A kinetic study on the hydrolysis of ethyl caprylate in alkaline medium at different temperatures from 20 ^oC to 40 ^oC differing by 5 ^oC and 40% to 80% organic co-solvent acetone composition, was investigated to evaluate specific reaction rate, isocomposition activation energy, thermodynamic activation parameters, and isokinetic temperature by using laboratory experimental design, volumetric method of analysis, and titration technique. The analyzed qualitative and quantitative data for rate constant, iso-composition activation energy, free energy of activation, enthaly of activation, entropy of activation, and isokinetic temperature are consistent with that from literature reviews. The composition of solvent acetone in aqueous acetone media for alkaline hydrolysis of an esyer of medium chain fatty acid, ethyl caprylate influences the specific reaction rate, isocomposition activation energy and thermodynamic activation parameters. This study contributes a deeper understanding of organic cosolvent effects in the field of chemical kinetics and provides a basis for further investigation into the alkaline hydrolysis of esters of higher chain fatty acid with different organic cosolvent compositions.

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Availability of Data and Materials

Data are safely stored. They will be made available in special request.

Conflict of Interest

The author declare that there is no conflict of interest in relation to this manuscript.

Ethical Compliance

"Not applicable"

Consent for Publication

"Not applicable"

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