

Kinetics of the Saponification of Mixed Fats Consisting of Olein and Stearin

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ABSTRACT

This research presents the kinetics of the saponification reaction using mixed fats of olein and stearin [in the ratio (3:1)] with NaOH solution. In this reaction, excess solution of NaOH was used to ensure the reaction being irreversible. Three parameters were varied to show their effects on the reaction rate. They are: percentage excess of NaOH solution (10% - 100%), temperature (100-150)°C, and stirring speed (400-1100) rpm. It was noticed that increasing the percentage excess of NaOH solution enhances the rate of reaction while increasing temperature decreases the reaction rate since it is exothermic reaction. Increasing stirring speed also improves the reaction rate because it is mass transfer controlled. Calculations of the activation energy and the frequency factor were also performed.

A new mathematical model for calculation of the reaction – rate constant was derived. It is shown that a good approximation was obtained between the experimental and calculated values of the reaction – rate constant k .

Key words: kinetics, saponification reaction, mixed fats, olein and stearin, mathematical model.

حركية تفاعل الصوبنة لمزيج من زيتي الاولين والستيارين

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مدرس

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الخلاصة

يقدم هذا البحث حركية تفاعل الصوبنة باستخدام خليط من زيتي الاولين والستيارين [بنسبة (1:3)] مع محلول هيدروكسيد الصوديوم. يستخدم في هذا التفاعل نسبة زائدة من محلول هيدروكسيد الصوديوم لضمان كون التفاعل غير عكسي. تم تغيير ثلاثة عوامل لبيان تأثيرهم على سرعة التفاعل وهم نسبة الزيادة المئوية لمحلول هيدروكسيد الصوديوم (10% - 100%)، درجة الحرارة (100-150)°م، وسرعة الخلط (400-1100) دورة/دقيقة. وقد لوحظ ان زيادة نسبة الزيادة المئوية لمحلول هيدروكسيد الصوديوم تؤدي الى زيادة سرعة التفاعل بينما زيادة درجة الحرارة تقلل سرعة التفاعل لأنه تفاعل باعث للحرارة. أيضا زيادة سرعة الخلط تؤدي الى زيادة سرعة التفاعل لأنه من التفاعلات التي يسيطر عليها انتقال المادة. كذلك تم احتساب طاقة التنشيط وعامل الذبذبة. تم اشتقاق نموذج رياضي جديد لحساب ثابت سرعة التفاعل. لقد تبين وجود تقارب جيد بين القيم العملية والقيم المحسوبة لثابت سرعة التفاعل.

الكلمات الرئيسية: الحركية, تفاعل الصوبنة, مزيج من الزيوت, الاولين والستيارين, نموذج رياضي.



1. INTRODUCTION

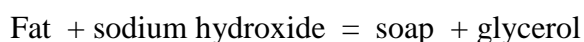
It has been said that the use of soap is a gauge of the civilization of a nation . Many literature were published in the field of saponification of various fats and fatty acids. **Frost and Schwemer, 1952**. had studied the differential rate equations for the kinetics of competitive consecutive second – order reactions . Measurements of the rate of saponification of ethyl adipate and ethyl succinate were used to verify the theoretical approach . **Newberger and Kadlec , 1973** had studied the kinetics of the saponification of diethyl adipate between 302K and 358K , for both stirred and tubular reactors . They had suggested numerical search method to verify the proposed model. **Vicente, et al., 2006**. Studied the kinetics of Brassica Carinata oil methanolysis . They found that the methanolysis reaction can be described as a pseudo – homogeneous catalyzed reaction , following a second – order mechanism for the forward and reverse reactions . They also found that higher temperature and catalyst concentrations increased the reaction rates . Appleton **Appleton, 2007**, had published a handbook of soap manufacture . He had made a review of oils and fats and their saponification . He had presented the steps of soap – making even soap perfuming and molding . **Lab. Report , 2012**, was presented to determine the order, rate constant , activation energy , and pre – exponential factor for the reaction of ethyl acetate with base using conductance measurements. An experimental study of the reaction kinetics of a simple homogenous liquid – phase system was published , **Chem. Eng. Lab. , 2013**. The reaction kinetics of the saponification of isopropyl acetate with sodium hydroxide was presented.

There are some aspects of interest still need to be studied . Determination the order , rate constant and many other parameters in the saponification kinetics are of great importance . The objective of this research is to study the parameters that affect saponification reaction. These parameters can be used to model the reaction to a large – scale applications , enhance an existed production and adopt an optimum set of parameters for a new production .

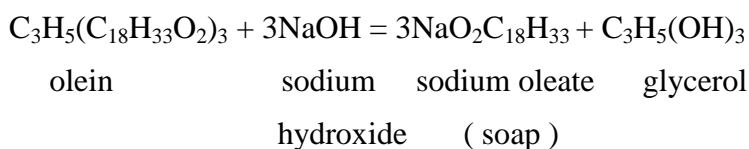
2. THEORITICAL ASPECTS

2.1 Saponification Reaction

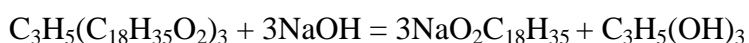
The saponification reaction is of the form



The fat used is a mixture of olein and stearin with a ratio (3:1) . These fats are the most important ones from a soap –maker's point of view , **Appleton , 2007** . Using (3:1) ratio ensures the product (soap) to be soft enough (by using olein) and solid enough (by using stearin) for toilet use. Appleton had published various types of fats and fatty acids . Olein (derived from olive and cotton – seed oil) is saponified according to the following reaction :



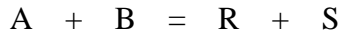
Stearin (derived from tallow) is saponified according to the following reaction :





stearin sodium sodium stearate glycerol
hydroxide (soap)

Therefore; the chemical equation for saponification reaction can be represented by :



Where A , B refer to the reactants (fat and caustic soda) and R , S refer to the products (soap and glycerol). The reaction rate is , **Smith ,1981**.

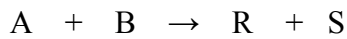
$$r_A = k C_A C_B - k' C_R C_S$$

where : k is the forward reaction – rate constant

k' is the backward reaction – rate constant

C represents the molar concentration of the specified substance in (mol / l)

If an excess of NaOH solution is added , the reaction will be considered as irreversible , such as **Smith , 1981**.



and the rate equation will be :

$$r_A = k C_A C_B$$

Since the reaction is in liquid phase (constant volume),**Smith , 1981**.

$$r_A = - dC_A / dt = k C_A C_B \tag{1}$$

2.2 Kinetics of Saponification Reaction

The kinetics study of this work consists of the following:

- (i) Determination of the order of the reaction
- (ii) Determination of the rate constant
- (iii) Determination of the activation energy
- (iv) Determination of the frequency factor or (pre - exponential) factor

under the variation of three parameters : the percentage excess of NaOH , the temperature and the stirring speed.

The basic relation in kinetics study is the concentration –time relation of the reactants. The concentration – time data can be detected using titration of samples and refractive indices determination.

2.3 Modeling the Rate Equation

Previous works that studied the kinetics of saponification reaction had either simplified the solution of the differential equation by approximation, **Lab. Report, 2012** or solved it by iteration , **Newberger, and Kadlec , 1973**.



In this work, the differential equation will be dealt as it is and solved with somewhat tedious integration method , as follows:

$$r_A = - dC_A / dt = k C_A C_B \quad (1)$$

The fractional conversion , x_A , is defined as :

$$x_A = (C_{A0} - C_A) / C_{A0}$$

Differentiation:

$$- dC_A / dt = C_{A0} dx_A / dt \quad (2)$$

C_A and C_B are defined through x_A as:

$$C_A = C_{A0} (1 - x_A) \quad (3)$$

$$C_B = C_{B0} - 3 (C_{A0} - C_A) \quad (4)$$

Where Eq. (3) is the stoichiometric factor and C_{A0} , C_{B0} are the initial concentrations of reactants A and B , respectively. Dividing Eq. (4) by C_{A0} yields:

$$C_B / C_{A0} = C_{B0} / C_{A0} - 3 (C_{A0} - C_A) / C_{A0}$$

$$C_B / C_{A0} = C_{B0} / C_{A0} - 3 x_A$$

$$\text{or } C_B = C_{B0} - 3 x_A C_{A0} \quad (5)$$

Substituting Eqs. (2) , (3) , and (5) into Eq. (1) , yields :

$$C_{A0} dx_A / dt = k C_{A0} (1 - x_A) (C_{B0} - 3x_A C_{A0})$$

Dividing by C_{A0} leads to:

$$dx_A / dt = k (1 - x_A) (C_{B0} - 3x_A C_{A0}) \quad (6)$$

Separating the variables , yields :

$$dx_A / (1 - x_A)(C_{B0} - 3x_A C_{A0}) = k dt \quad (7)$$

Integrating from $t = 0$, $x_A = 0$ to $t = t$ and $x_A = x_A$ yields :

$$\int_0^{x_A} \frac{dx_A}{(1 - x_A)(C_{B0} - 3x_A C_{A0})} = \int_0^t k dt \quad (8)$$

Using fractional integration :

$$\frac{1}{(1 - x_A)(C_{B0} - 3x_A C_{A0})} = \frac{E}{1 - x_A} + \frac{F}{C_{B0} - 3x_A C_{A0}} \quad (9)$$

Multiplying by $(1 - x_A) (C_{B0} - 3 x_A C_{A0})$, yields :

$$1 = E (C_{B0} - 3 x_A C_{A0}) + F (1 - x_A) \quad (10)$$

$$1 = E C_{B0} - 3 E x_A C_{A0} + F - F x_A$$

Equating the coefficients of x_A^0 :

$$1 = E C_{B0} + F \quad (11)$$

Equating the coefficients of x_A^1 :



$$0 = -3 E C_{A_0} - F \quad (12)$$

Therefore; $F = -3 E C_{A_0}$

Substituting into Eq. (11) , yields:

$$1 = E C_{B_0} - 3 E C_{A_0} \quad (13)$$

$$\text{or } E = 1 / (C_{B_0} - 3C_{A_0}) \quad (14)$$

$$\text{and } F = -3 C_{A_0} / (C_{B_0} - 3C_{A_0}) \quad (15)$$

Substituting Eqs. (14) and (15) into Eq. (9) , yields :

$$\frac{1}{(1-x_A)(C_{B_0}-3x_A C_{A_0})} = \frac{1}{(1-x_A)(C_{B_0}-3C_{A_0})} - \frac{3C_{A_0}}{(C_{B_0}-3C_{A_0})(C_{B_0}-3x_A C_{A_0})}$$

and

$$\int_0^{x_A} \frac{dx_A}{(1-x_A)(C_{B_0}-3x_A C_{A_0})} = \frac{1}{(C_{B_0}-3C_{A_0})} \int_0^{x_A} \frac{dx_A}{(1-x_A)} - \frac{3C_{A_0}}{(C_{B_0}-3C_{A_0})} \int_0^{x_A} \frac{dx_A}{(C_{B_0}-3x_A C_{A_0})}$$

Substituting into Eq. (8) , yields :

$$\frac{1}{(C_{B_0}-3C_{A_0})} \left[\int_0^{x_A} \frac{dx_A}{(1-x_A)} - 3C_{A_0} \int_0^{x_A} \frac{dx_A}{(C_{B_0}-3x_A C_{A_0})} \right] = \int_0^t k dt$$

Integrating , yields :

$$\frac{1}{(C_{B_0}-3C_{A_0})} \left[-\ln(1-x_A) + \ln(C_{B_0}-3x_A C_{A_0}) \right] = k t$$

$$\text{or } \frac{1}{(C_{B_0}-3C_{A_0})} \left[\ln \frac{C_{B_0}-3x_A C_{A_0}}{1-x_A} \right] = k t$$

$$\text{so } \ln \left[\frac{C_{B_0}-3x_A C_{A_0}}{1-x_A} \right] \frac{1}{(C_{B_0}-3C_{A_0})} = k t$$

is the proposed model . This model can be verified through the experimental work .

3. EXPERIMENTAL WORK

3.1 Apparatus

The apparatus used in this work consists of 1000 ml- beaker (modified with lower tapped glass tube to ensure the separation of the lower glycerol layer from the upper soap layer) , hot plate stirrer (RLABINCO , Model L-81, The Netherlands) . It's provided with two nobs ; one for controlling the temperature (100 - 150) °C and the other for controlling the stirring speed (400 – 1100) rpm . The apparatus also consists of thermometer (for measuring the actual temperature of the reacting mixture) , various glassware for preparation of solutions and titration of samples ,



Refractometer (Model Optika , no. 2WAJ SN 281006 , Italy) . The refractometer is used to measure the refractive index of the samples .

3.2 Materials

Materials used are : olein and stearin fats (source: Republic Company of Vegetable Oils) , sodium hydroxide (NaOH) solution (Aldrich mark) standardized against standard hydrochloric acid (HCl) solution (commercial grade) , **Vogel , 1961.** , saturated solution of brine , distilled water .

3.3 Procedure

The procedure consists of the following steps:

- 1-Weighing the required amounts of fats then transferring them to the beaker.
- 2- Switch on the heater and stirrer and fixing them on certain values.
- 3-Adding an excess amount of sodium hydroxide solution with changeable percentage excess (10% - 100%).
- 4-Beginning the reaction for specified C_{A0} and C_{B0} .
- 5-Taking a sample every 5-10 minutes till the end of the reaction (soap formation). Samples were titrated against standard HCl solution using methyl orange as an indicator ,**Vogel , 1961.** also the refractive indices were measured for these samples.
- 6-Adding distilled water to cool the reaction mixture and to suppress the froth of the soap when necessary .
- 7-Adding saturated brine solution to facilitate the separation of layers.
- 8-The soap was then molded, dried, and used.

4. RESULTS AND DISCUSSION

Experiments were performed at different levels of the three parameters (percentage excess of NaOH solution (10%-100%) , temperature (100-150) °C , and stirring speed (400-1100) rpm . Each experiment depicted a concentration – time curve . To calculate the order of the reaction and the rate constant , the differential method was adopted . This was achieved by calculating different slopes at different concentrations of tangent points of the concentration – time curve . These slopes represented dC_A/dt (reaction rate with respect to the limiting reactant A) . Taking the natural logarithms for each dC_A/dt and C_A , then drawing data gives a straight line of slope equal n_A (the order of the reaction with respect to A) and of intercept equals $\ln k$. The inverse of $\ln k$ gives the experimental value of k (reaction – rate constant).**Table 1** shows a sample of concentration –time data for both reactant A and reactant B , with the refractive indices and conversions for run no.(4).**Fig.1** shows the concentration - time curve of reactant A. The curve shows decreasing of C_A with time until complete consumption. This is because reactant A is the limiting reactant . **Table 2** shows data for the slopes determined at the tangent points of **Fig. 1**. The data illustrate decreasing of slopes' magnitudes for the points . This means decreasing the rate of reaction with the concentration decrease, which indicates the progress of reaction. **Table 3** shows the logarithm of the rate of reaction $\ln (-dC_A/dt)$ with the logarithm of the concentration $\ln C_A$. **Fig. 2** depicted these data. It was shown that the order of reaction with respect to A is approximately 1. The value of the rate constant was also determined. These calculations were also done for the excess reactant B (NaOH solution) to find the order of the reaction with

respect to it (n_B). **Fig. 3** traces the concentration - time profile of reactant B. It is clear that reactant B dose not tend to complete consumption , since it is the excess reactant. **Table 4** shows $(-dC_B / dt)$ versus C_B data . Similarly , the rate of reaction decreased with decrease in concentration .**Table 5** shows the variation of $\ln (-dC_B/dt)$ with $\ln C_B$.**Fig. 4** traces $\ln(-dC_B/dt)$ against $\ln C_B$. The relation is linear with slope equals n_B . Again the value of n_B is about 1 . Experimental results showed that the order of reaction for each reactant $n_A , n_B \approx 1$, so the overall order of reaction was second order . This result agreed with the fact that most of esterification reactions are second order .Saponification reaction is an example of these reactions. It was also noticed that the values of the refractive index RI decreases as the conversion increases. After approximately 50% conversion, the values of RI increases again. To explain this behavior, the relationship between RI and the density must be noticed. RI is a physical property that inversely proportional to density. As NaOH solution was added to the fat , the density of the fat is increased (RI is decreased).When the soap was produced the density again decreased (RI is increased). The concentration - time data were substituted in the mathematical model using conversions instead of concentrations.

Fig. 5 shows the relation between the expression $\ln D \left(\left[\frac{C_{B_0} - 3 x_A C_{A_0}}{1 - x_A} \right]^{C_{B_0} - 3 C_{A_0}} \right)$ and time. The relation is linear with slope equal k_{calc} . The calculated values of the reaction-rate constant were somewhat deviate from the experimental ones. It was noticed that k values obtained from the mathematical model were overestimated when the percentage excess of NaOH solution were too low or too high. At medium levels of % excess of NaOH solution the calculated values of k were underestimated. This can be discussed as follows: at low values of % excess of NaOH solution , the reaction rate decreases because of the decreased concentration of NaOH giving reduced values of reaction –rate constant k_{exp} .This makes the calculated values be higher . High values of % excess of NaOH solution reduce the number of reacting molecules , as a result of hindering their movement , causing reduced values of k_{exp} again. This also makes k_{calc} be higher. The average error between experimental and calculated values of k was calculated by the following equation:

$$Av. Error = \frac{\sum_{i=1}^N \left(\frac{k_{exp} - k_{calc}}{k_{exp}} \right)}{N}$$

Where N is the number of runs . The average error was approximately 20%. This figure may be accepted because the experimental errors were thought to be lumped within it . Fortunately; the underestimated values of k_{calc} resulted from the mathematical model allow safe design and simulation calculations.

Fig. 6 shows the effect of increasing the percentage excess of NaOH solution on reaction – rate constants (k_{exp} and k_{calc}) . The calculated values trace lower curve than experimental ones except at approximately 100% excess . Evidently, increasing NaOH solution enhances the rate of reaction because it suppresses the backward reaction according to Le Chatelier rule .

The effect of temperature is presented in **Fig. 7** . The natural logarithms of k is plotted against the reciprocal of the absolute temperature . This figure shows a linear relation between $\ln k$ and $1/T$ as it was predicted by Arrhenius.

$$\ln k = -\frac{E_a}{R_g} \left(\frac{1}{T}\right) + \ln A^\circ$$

where the slope $(-\frac{E_a}{R_g})$ represents the negative value of the activation energy divided by the universal perfect-gas law constant . The intercept of the plot $(\ln A^\circ)$ determines the frequency factor . The values of the activation energy and the frequency factor were found to be as follows:

$$E_{a \text{ exp}} = -20.44 \text{ J.mol}^{-1} \quad \text{and}$$

$$A^\circ_{\text{exp}} = 9.8358 \times 10^{-5} \text{ l.mol}^{-1}.\text{min}^{-1}$$

$$\text{While } E_{a \text{ calc}} = -4.67 \text{ J.mol}^{-1} \quad \text{and}$$

$$A^\circ_{\text{calc}} = 6.2 \times 10^{-3} \text{ l.mol}^{-1}.\text{min}^{-1}$$

The experimental and calculated values are different by an order of magnitude which is attributed to the experimental error .The value of the activation energy is rather low. This indicates that the reaction is fast since the activation energy represents the energy barrier that molecules must gain to reach the final product .

Fig. 8 shows the effect of stirring speed upon the rate constants. It is clear that the effect of stirring is considerable on the reaction rate. This leads to the conclusion that the reaction is mass - transfer controlled. This viewpoint agrees well with the notation of underestimated values of k_{exp} at high values of percentage excess of NaOH solution. Therefore; increasing stirring speed enhances the reaction rate. However, there is a constraint limited by the soap - froth rising .If the values of k_{exp} were normalized (i.e. making the total increase in k_{exp} corresponds 100%) as shown in **Table 6**. This was done for the runs that have only the excess ratio varied , i.e. the temperature and stirring speed were fixed . The normalization shows that percentage excess over 50% has less effect on the value of k_{exp} than the percentage excess below 50%. This may be attributed to the hindrance of reacting molecules by the NaOH molecules which confirms the mass- transfer control on the reaction.

5. CONCLUSIONS

- The % excess of NaOH solution plays an important role in the saponification reaction. 88% of increased value of the rate constant k can be achieved at 50% excess . Adding more solution of NaOH seems to be unnecessary and costing .
- The saponification reaction is mass – transfer controlled and stirring speed appears as an effective parameter , although excessive stirring may raise the froth of the soap .
- The derived mathematical model is adequately described the system especially at moderate values of % excess of NaOH solution .
- The mathematical model can be applied to scale – up the laboratory batch reactor to a large – scale continuously stirred tank reactor (CSTR).



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NOMENCLATURE

- A limiting reactant (fat)
- A° frequency factor ($l \cdot mol^{-1} \cdot min^{-1}$)
- B excess reactant (NaOH solution)
- C concentration ($mol \cdot l^{-1}$)
- D expression ($\left[\frac{C_{B_0} - 3 x_A C_{A_0}}{1 - x_A} \right]^{C_{B_0} - 3 C_{A_0}}$)
- E arbitrary constant
- E_a activation energy ($J \cdot mol^{-1}$)
- F arbitrary constant
- k forward reaction-rate constant ($l \cdot mol^{-1} \cdot min^{-1}$)
- k' backward reaction-rate constant($l \cdot mol^{-1} \cdot min^{-1}$)
- N number of runs
- n order of reaction
- R product 1 (soap)
- R_g universal gas-law constant ($8.314 J \cdot mol^{-1} \cdot K^{-1}$)
- RI refractive index
- r reaction rate ($mol \cdot l^{-1} \cdot min^{-1}$)

rpm revolution per minute

S product 2 (glycerol)

T absolute temperature (K)

t time (min)

x fractional conversion (-)

Subscripts

o initial (C_{Ao} initial conc. of reactant A)

Table 1. Concentration – time data of reactant A (fat) with refractive indices and conversions for run no.(4) [%excess=50 , Temp.=100°C , rpm=400].

t (min)	C_A (mol . l ⁻¹)	RI (-)	x_A (-)	C_B (mol . l ⁻¹)
0	0.3119	1.468	0	2.844
10	0.0815	1.42	0.7387	2.1528
20	0.0789	1.412	0.7471	2.1449
30	0.0577	1.428	0.8151	2.0813
35	0.0471	1.472	0.8490	2.0496
40	0.0259	1.52	0.917	1.986

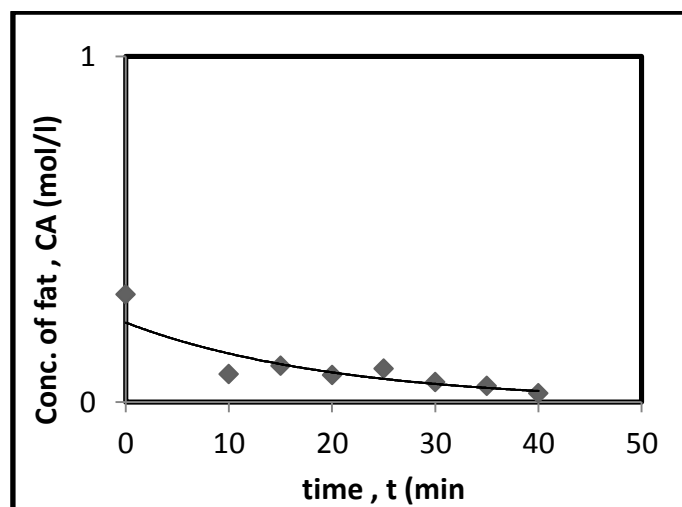


Figure 1. Concentration-time profile with respect to reactant A (fat) of run no.(4) [% excess=50 , Temp.=100°C , rpm=400].



Table 2.Rate of reaction with respect to reactant A(slope) versus the concentration of A of run no.(4) [% excess=50 , Temp.=100°C , rpm=400].

Slope (dC _A /dt)	C _A
-0.0115	0.22
- 0.0086	0.20
-0.0063	0.16
-0.0043	0.11

Table 3.Logarithm of reaction rate versus logarithm of concentration of reactant A (fat) of run no.(4) [% excess=50 , Temp.=100°C , rpm=400].

ln(- dC _A /dt)	ln C _A
-4.4654	-1.5141
-4.7560	-1.6094
-5.0672	-1.8326
-5.4491	-2.2073

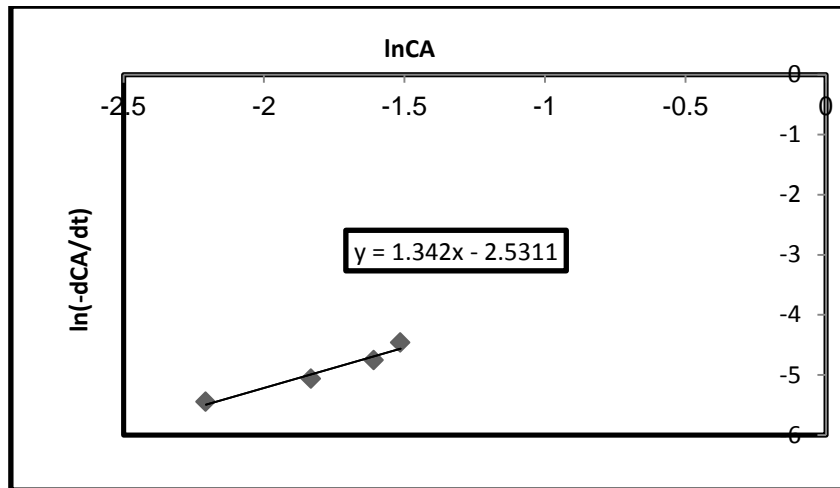


Figure 2.The linear relation between ln(-dC_A/dt) and ln C_A gives the order of reaction with respect to reactant A. n_A(slope) and ln k_{exp} (intercept) of run no. (4) [% excess=50 , Temp.=100°C, rpm=400].

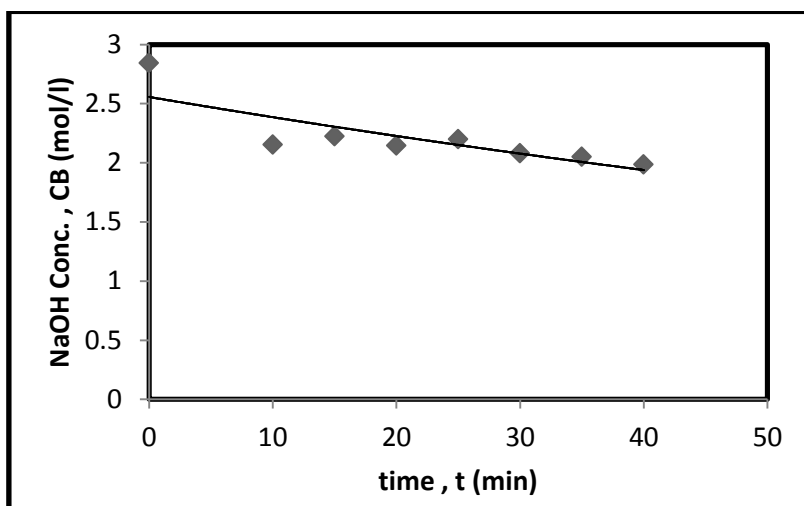


Figure 3.Concentration-time profile with respect to reactant B (NaOH solution) of run no. (4) [% excess=50 , Temp.=100°C , rpm=400].

Table 4. Rate of reaction with respect to reactant B(slope) versus the concentration of B of run no.(4) [% excess=50 , Temp.=100°C , rpm=400].

Slope (dC_B / dt)	C_B
-0.1556	2.5
-0.1183	2.0
-0.0942	1.7
-0.0740	1.2
-0.0663	1.1



Table 5.Logarithm of reaction rate versus logarithm of concentration of reactant B(NaOH solution) of run no. (4) [% excess=50, Temp.=100°C , rpm=400]

$\ln(-dC_B/dt)$	$\ln C_B$
-1.86	0.92
-2.13	0.69
-2.36	0.53
-2.60	0.18
-2.71	0.10

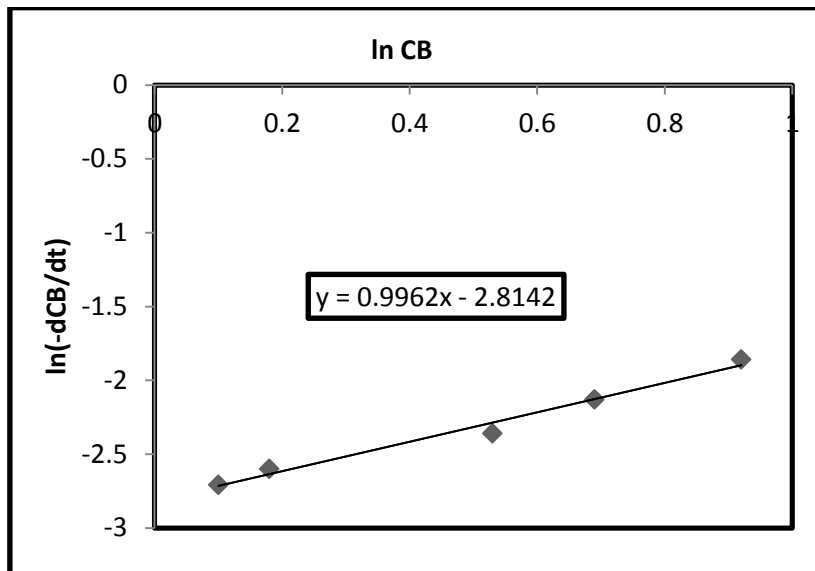


Figure 4 .The linear relation between $\ln (-dC_B/dt)$ and $\ln C_B$ gives the order of reaction with respect to reactant B n_B (slope).

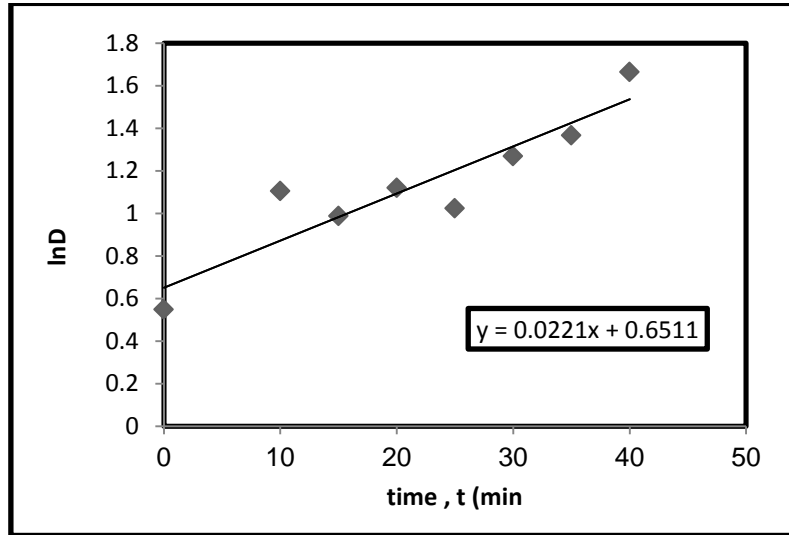


Figure 5. The proposed model shows linearity between $\ln D \left(\left[\frac{C_{B_0} - 3 x_A C_{A_0}}{1 - x_A} \right]^{C_{B_0} - 3 C_{A_0}} \right)$ and time with C_{A_0} and C_{B_0} of run no. (4) the slope determines k_{calc} .

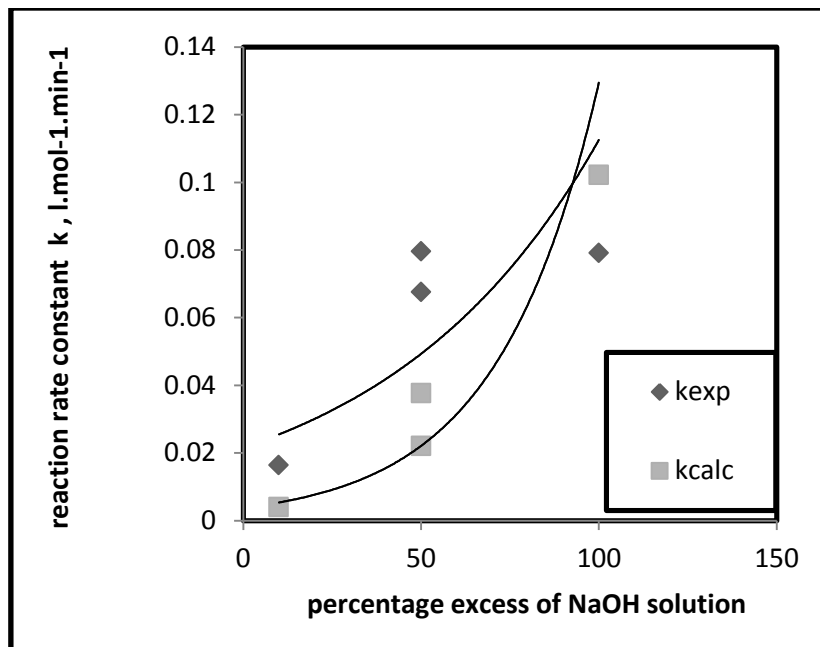


Figure 6. The effect of % excess of NaOH solution on the experimental and calculated reaction - rate constants k_{exp} and k_{calc} .

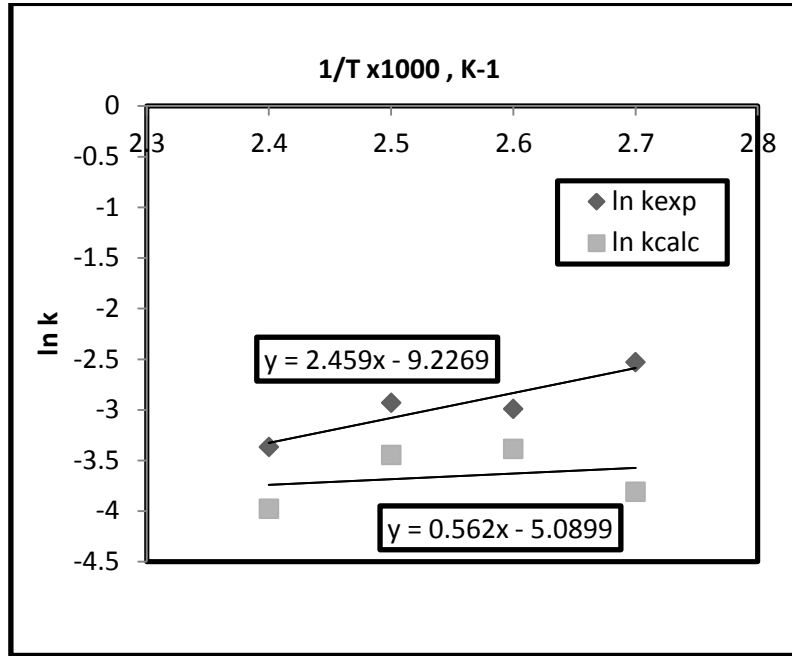


Figure 7. Plot of Arrhenius equation for saponification of mixed olein and stearin with NaOH solution. The slope gives $(-E_a/R_g)$ and the intercept gives $\ln A^0$.

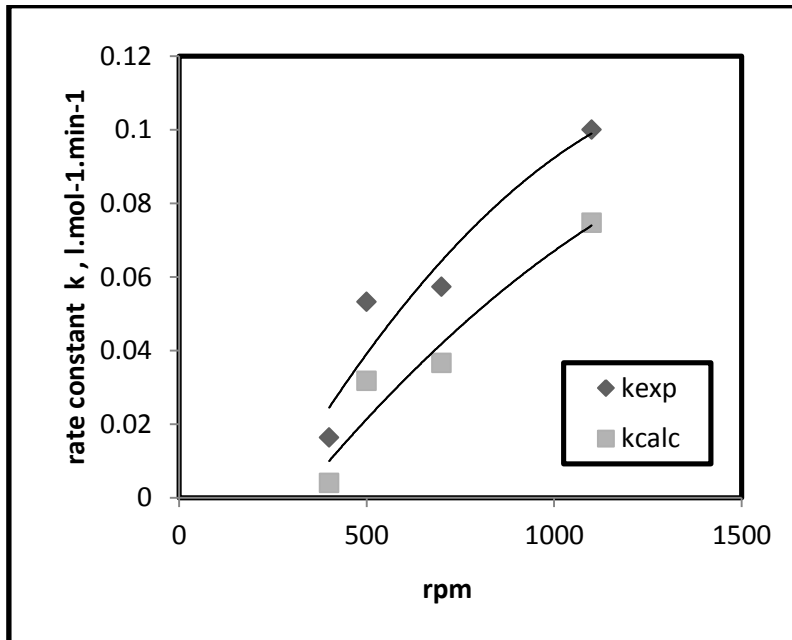


Figure 8. The effect of stirring speed on the experimental and calculated reaction- rate constants.

**Table 6.** The effect of % excess of NaOH solution on the value of k_{exp}

%excess	Temp. (°C)	rpm	k_{exp} ($l.mol^{-1}.min^{-1}$)	% increase in k_{exp}
10	100	400	0.0164	0
50	100	400	0.0716	88
100	100	400	0.0791	100