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## Kinetic Empirical Modeling of Biodiesel Production from *Ricinus Communis* using Refluxed Calcined Snail Shell as Catalyst

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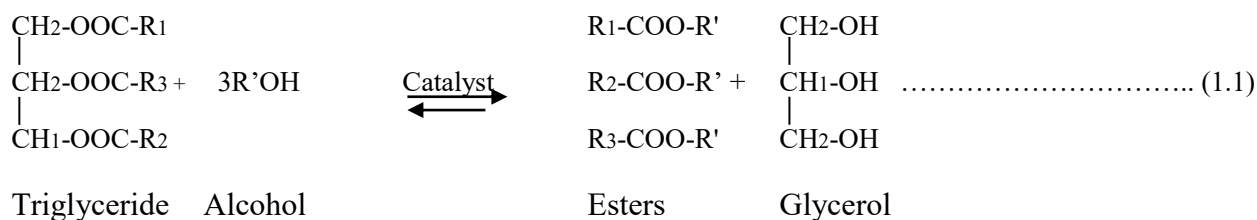
**Abstract-** Biodiesel from non-edible oil emerges as one of the most energy-efficient and environmentally friendly options in recent times to fulfill the sustainable future energy needs. Hence, there is a need to produce biodiesel on an industrial scale. In designing a reactor, for a large scale production, the thermodynamic, kinetics and optimization parameters must be studied. In this study, the kinetics of biodiesel production from refined castor seed oil using refluxed calcined snail shell as a catalyst is investigated. The transesterification variables and the level used in kinetics study are obtained from optimization studies carried out as preliminary studies, the experimental conditions of three different runs that gave the highest yield were selected. The kinetics studied of transesterification reaction shows pseudo-first-order kinetics. The rate constants of the reactions at three different runs are 0.03048, 0.03207 and 0.03737 with 496.678 J/Mol and 1652.4263 as activation energy and frequency factor. The lower activation energy may be due to the catalytic activity of refluxed calcined snail shell.

**Keywords:** kinetics modeling, castor seed oil, refluxed calcined snail shell and biodiesel.

## I. INTRODUCTION

The conventional energy sources are finite and associated with environmental pollution; there is a need to generate a sustainable alternative to support the civilization, i.e. transportation, agriculture, communication, etc. (Hribernik and Kegl, 2007; Ma *et al.*, 1999). Biodiesel emerges as one of the most energy-efficient and environmentally friendly options in recent times to fulfill the future energy needs (Nakarmi and Joshi, 2014; Owolabi *et al.*, 2012). Biodiesel is a renewable diesel substitute that can be obtained by reacting any natural oil or fat with alcohol. There are four

well-established liquid biofuels production methods; direct use and blending, micro-emulsions, thermal cracking (pyrolysis), and transesterification (Ma and Hanna 1999). Among these methods, transesterification is one of the most commonly used methods in the biodiesel production industry. Transesterification of vegetable oils and animal fats is the fundamental way to make biodiesel (Encinar *et al.*, 2010). Transesterification is a three-step reversible reaction of vegetable oil or animal fat with short-chain alcohol usually methanol to form fatty acid methyl esters (FAMES) and glycerol (Encinar *et al.*, 2010), in the presence of a catalyst. The stoichiometric ratio for transesterification, one mole of triglyceride (oil) requires three moles of alcohol as shown in the equation 1.1. Example of alcohols that could be used in the transesterification process are methanol, ethanol, propanol, butanol and amyl alcohol. Methanol is used most frequently because of its low cost, physical and chemical advantages (polar and shortest chain alcohol). However the molar ratio is associated with the type of catalyst used, and higher molar ratios result in greater ester conversion in a shorter time (Ma and Hanna, 1999). Base catalysts lead to the higher conversion of methyl esters at low temperature, atmospheric pressure and minimum response time, which reduces the cost of the process considerably (Ismail *et al.*, 2016).



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Transesterification reactions are basically heterogeneous because the nonpolar oil phase and the polar alcohol phase are immiscible with each other. Therefore, their overall reaction rates mainly depend on two important factors: the hydrodynamic effect between these two phases and the chemical reaction kinetics. To optimized biodiesel production process and design a high-performance reaction system, the hydrodynamic effect, and chemical reaction kinetics must be entirely understood. Previous kinetic studies on the transesterification reaction were mostly carried out in a pseudo-homogenous reaction system. Sufficient mixing is provided in these systems to eliminate the hydrodynamic effect on the overall reaction rate (Regina, 2013). There are many studies carried out in different approaches to investigate the kinetics of transesterification reaction (Regina, 2013). Among all the approach, it was observed that the transesterification reactions in most cases follow pseudo-first order kinetics (Labib *et al.*, 2013).

In this study, the kinetics of the transesterification reaction of refined castor seed oil using a heterogeneous (refluxed calcined snail shell (CaO)) catalyst was investigated.

## II. METHODOLOGY

### a) Preproduction Process

The castor seed was obtained from Yandodo, Kano State. The seed was sundried to reduce the moisture content. The castor seed was oven dried at 90 °C for 45 minutes. The dried seeds were grounded using mortar and pestle and weighed. Extraction of castor oil was carried out by soxhlet extraction method as reported by Edison *et al.* (2012). The extracted oil was

subjected to pretreatments such as water and acid degumming, and neutralization to upgrade it physicochemical properties for efficient transesterification.

The refluxed calcined snail shell (calcium oxide) catalyst that was used in this study was synthesized from snail shell through the hydrothermal method, as reported by Ismail *et al.* (2016).

### b) Transesterification Reactions for Kinetics Parameters Determination

The transesterification variables levels (indicated in Table 1) were determined after the optimization study was carried out using Taguchi orthogonal array design (Muhammad *et al.*, 2018). The transesterification reactions runs were conducted according to design through which three runs that gave highest yields; their experimental conditions were used in the kinetics study. The transesterification was carried out in 500 cm<sup>3</sup> two necks round bottom flask as reactor equipped with condenser, thermometer, and hotplate magnetic stirrer. During the transesterification reaction, samples were collected from the reaction mixture using 15 cm<sup>3</sup> syringe at a different time interval of 30, 60 and 120 minutes, transferred into test tubes, and then immersed in cold water at 4 °C to quench the reaction immediately. The equal volume (5 cm<sup>3</sup>) of water and n-hexane was immediately added to get clear separation, the top layer sample containing the methyl ester, non reacted triglyceride and some catalyst particle was collected. For better separation, the samples were centrifuged for 5 minutes at 2000 rpm, and, then the top layer sample was collected, washed with distilled water and then sent for GC/MS analysis (Regina, 2013).

Table 1: Transesterification variables and their levels used in kinetics study

S/N	Oil to Methanol Ratio	Catalyst Load (w/w %)	Reaction Temperature (°C)	Reaction Time (Hrs)
1	1: 06	3	60	2
2	1:08	1	70	2
3	1:18	0.5	55	2

### c) GC/ MS Analysis of Biodiesel Produced

The oil composition and methyl ester content were assayed using a GC/MS machine in the multi-purpose laboratory of Ahmadu Bello University (ABU), Zaria.

The GC/MS was equipped with an Econo-Cap EC-WAX Capillary Column (30.0 m in length × 250 µm in diameter × 0.25 µm in film thickness). The GC oven was maintained at 50 °C for 3 minutes, and then heated to 210 °C at a rate of 10 °C per minute and held at 210 °C for 9 minutes. The front inlet temperature of the oven

was 255 °C (split less-mode). The carrier gas was helium with a flow rate of 12 cm<sup>3</sup>/min. The analysis of refined castor oil composition and fatty acid methyl ester (FAME) of biodiesel was carried out by injecting 1.0 µL of a sample solution that was prepared by blending the biodiesel sample with a prepared internal standard of GC i.e., methyl heptadecanoate. The FAME content by weight was determined from equation 2.1.

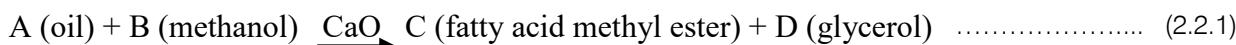
$$wt. \% = \left[ \frac{\sum(A_i - A_R)}{A_R} \right] \frac{C_R V_R}{w} \quad (2.1)$$

Where  $A_i$  = the peak area from chromatogram of FAME;  $A_R$  = the peak area from chromatogram of the internal standard;  $C_R$  = the concentration of the internal standard;  $V_R$  = the volume of the internal standard; and  $W$  = the total weight of the biodiesel sample (Regina, 2013).

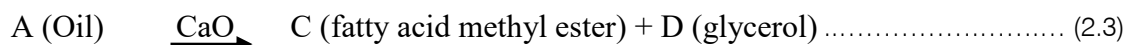


In developing the description of the overall kinetics, the following simplifying assumptions are made:

1. The presence of excess methanol reactant minimizes the reversibility of the transesterification reaction.
2. The reaction mixture is assumed homogeneous, the solid catalyst is in powder form and at a very low concentration in comparison to the whole volume of the reaction mixture (1- 4 % w/v).



The concentration of methanol, being in excess, does not influence the global reaction which is finally



Because of excess methanol was used to derive the reaction forward, the reverse reaction was ignored. The reaction rate  $r_A$  can be expressed in equation 2.4:

$$r_A = -\frac{d[A]}{dt} = k_1[A]_t^\alpha[B]_t^\beta \dots\dots\dots (2.4)$$

Where  $k_1$  = Observed rate of reaction

$[A]_t$  = Concentration of triglyceride (oil) at time  $t$ .

$[B]_t$  = concentration of methanol at time  $t$ .

While  $\alpha$  is the order of the oil and  $\beta$  is the order of methanol in the reaction.

Here,  $[B]_t^\beta$  could be considered as a constant since methanol is larger than the concentration of triglyceride. Then, the rate could be;

$$r = -\frac{d[A]}{dt} = k_2[A]_t^\alpha \dots\dots\dots (2.5)$$

Where  $k_2 = k_1[B]_t^\beta$

If we assumed reaction order  $\alpha = 1$ , then

$$-\frac{d[A]}{dt} = k_2[A]_t \dots\dots\dots (2.6)$$

The concentration of triglyceride at time  $t$  can be expressed using conversion rate ( $x$ ) of triglyceride at time  $t$  and initial concentration of triglyceride  $[A]_0$  as shown in equation 2.7:

#### d) Kinetic Modeling

The overall reaction of the transesterification was represented by the following equation 2.2.

3. Reaction mixture volume is assumed constant being put under reflux.

4. The reaction temperature is kept constant during the whole reaction time.

The overall reaction is assumed to be a single step, represented by equation 2.2, (neglecting intermediate steps) and is considered to develop kinetic rate equations (Pankaj, 2006).

The overall reaction in its simplified form is represented by equation 2.2.1.

represented by equation 2.3 which is in accordance with previous studies (Debk, 2003).

$$[A]_t = [A]_0(1 - x) \dots\dots\dots (2.7)$$

Thus, the reaction rate can be expressed using the initial concentration of triglyceride and conversion rate as shown in equation 2.8 (Gupta, 2003):

$$r = -\frac{d[A]_t}{dt} = \frac{d\{[A]_0(1-x)\}}{dt} = k_2 [A]_0 \frac{dx}{dt} \dots\dots (2.8)$$

By integrating equation 2.8:

$$\ln\left(\frac{1}{1-x}\right) = kt \dots\dots\dots (2.9)$$

Where,  $k$  is the reaction rate constant at specified time, temperature and catalyst concentration.

From the Arrhenius equation:

$$k = Ae^{-\frac{E_a}{RT}} \dots\dots\dots (2.10)$$

To calculate activation complex  $E_a$  and pre-exponential constant  $A$ , by taking the natural logarithm of both side to obtain equation (2.11) and then a plot of  $\ln k$  against  $1/T$ :

$$\ln k = \ln A - \frac{E_a}{RT} \dots\dots\dots (2.11)$$

### III. RESULTS AND DISCUSSION

#### a) Results

The kinetics study of biodiesel production from *Ricinus Communis* using refluxed calcined snail shell

was determined, Values in Table 2, show the conversion (x) of the triglycerides into fatty acid methyl esters at time interval of 30 minutes, 60 minutes and 120 minutes. GC/MS was used to accurately determine the fatty acid methyl esters formed at each stage (appendix I).

Tables 3 to 6, show the kinetic parameters obtained in the experiments. The rate constants (k) of three different reaction conditions are 0.0369, 0.0342 and 0.0312 min<sup>-1</sup> as indicated in Table 4. The slight

variability of the rate constants (k) might be due to the variability of the experimental conditions used. Their higher correlation coefficients i.e. 0.996, 0.897 and 0.996, validates the pseudo first-order kinetics of the reactions as assumed.

The pre-exponential factor which reflects the collision frequency and orientation of the reactant is 1652.4263 and the activation energy is 496.6784 J/mol (Table 6).

**Table 2:** Duration time and conversion rate at prescribed conditions

Reaction conditions	Conversion x (%)		
	30 mins	60 mins	120 mins
Oil to methanol ratio 1:6			
Catalyst load 3 w/w %			
Temperature 60 °C	75.5892	90.3251	99.1212
Oil to methanol ratio 1:18			
Catalyst load 0.5 w/w %			
Temperature 55 °C	24.9229	88.7812	96.5280
Oil to methanol ratio 1:8			
Catalyst load 1 w/w %			
Temperature 70 °C	32.6190	80.3271	95.9300

**Table 3:** Kinetics I

$\ln\left(\frac{1}{1-x}\right)$	Time (minutes)	Temperature (°C)
1.4101	30	60
2.3356	60	
4.7344	120	
0.2867	30	55
2.1876	60	
3.3604	120	
0.3948	30	70
1.6259	60	
3.2015	120	

**Table 4:** Calculated rate constant

Rate constant k (min <sup>-1</sup> )	Correlation coefficients (R <sup>2</sup> )
0.03737	0.996
0.03207	0.897
0.03048	0.996

Table 5: Rate constants and Temperature in Kelvin

Rate constant $k$ ( $s^{-1}$ )	$\ln k$	Temperature (Kelvin)	$\frac{1}{T}$
$6.228 \times 10^{-4}$	-7.38123	333	0.00300
$5.345 \times 10^{-4}$	-7.53418	328	0.00305
$5.080 \times 10^{-4}$	-7.58503	343	0.00292

Table 6: Calculated activation complex and frequency factor

Conditions	Pre-exponential factor A	Correlation coefficients ( $R^2$ )	Activation complex $E_a$ (J/Mol)
Oil to methanol ratio 1:6 to 1:18, Reaction time 2 hrs Catalyst load 0.5 to 7 wt% Temperature 55 to 70 °C	1652.4263	0.606	496.6784

#### IV. DISCUSSION

The kinetics of the transesterification of refined castor was discussed below:

##### a) Determination of Reaction Rate Constants and Activation Energy

The GC/MS analysis was used to determine the conversion of triglyceride to methyl ester. The percentage conversion ( $x$ ) was presented in Table 2 and Appendix I. Based on the previous discussion and results, the experimental setup was designed for studying the reaction kinetics of transesterification. The kinetics study was carried out under different experimental conditions which include catalyst concentrations, reaction temperature, reaction time and oil to methanol ratio as presented in Table 1. The agitation was kept constant at 600 rpm throughout the reaction. The rate constant and activation energy were estimated.

According to equation 2.9, the relationship between  $\ln\left(\frac{1}{1-x}\right)$  and time is linear, the value of the rate constant( $k$ ) equal to the slope of the linear regression fitted line plot. Thus,  $\ln\left(\frac{1}{1-x}\right)$  is plotted against time ( $t$ ) in 3 different experimental conditions as shown in Figure 1- 3. The data from Table 2 was used.

The resulting data fits pseudo- first-order kinetics behaviour. The high correlation coefficients ( $R^2$ ) of the linear equation as indicated in Table 4 shows that there is a first-order dependence of the transesterification reaction catalyzed by CaO (refluxed calcined snail shell). The rate constants obtained from

the fitted plot line are 0.03737, 0.03207 and 0.03048  $\text{min}^{-1}$ .

The activation energy and a frequency factor of the transesterification reaction were determined using equation 2.10, by taking the natural logarithm of both sides of equation 2.10 to get equation 3.1.

$$\ln k = \ln A - \frac{E_a}{RT} \dots\dots\dots (3.1)$$

Equation 3.1 shows the relationship between  $\ln k$  and  $\frac{1}{T}$  as the linear relationship with a slope of  $-\frac{E_a}{R}$  and intercept value of  $\ln A$ . Since the values of  $k$  at a different temperature were determined (as indicated in Table 4). The value of  $\ln k$  and  $\frac{1}{T}$  were calculated as presented in Table 5. By performing a linear regression of  $\ln k$  versus  $\frac{1}{T}$ , the activation energy and frequency factor determined from the slope and intercept of the regression trend line respectively as shown in Figure 4.

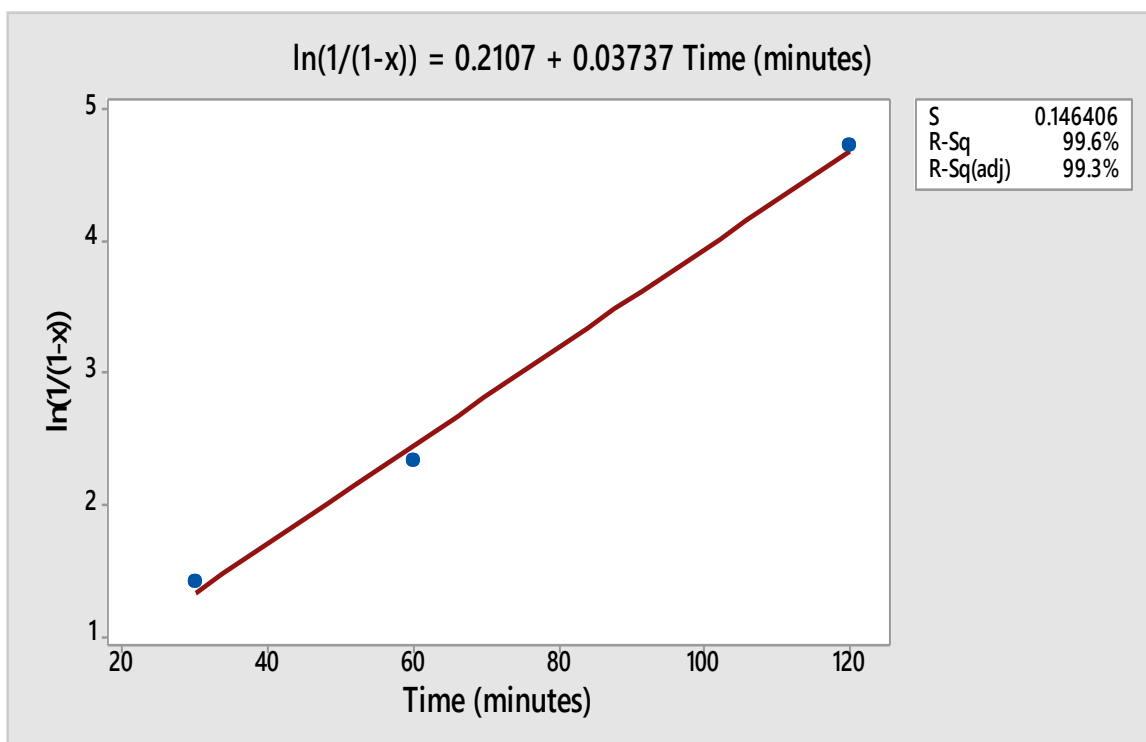


Fig. 1: Reaction rate constant at 60 °C

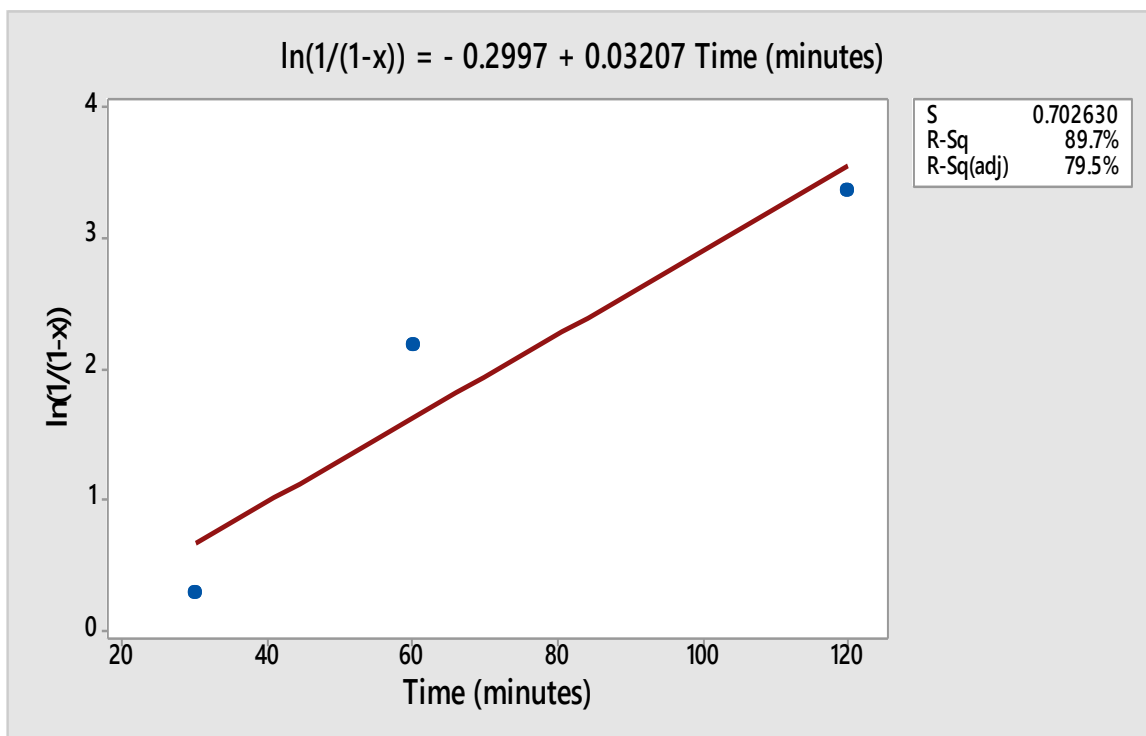


Fig. 2: Reaction rate constant at 55 °C



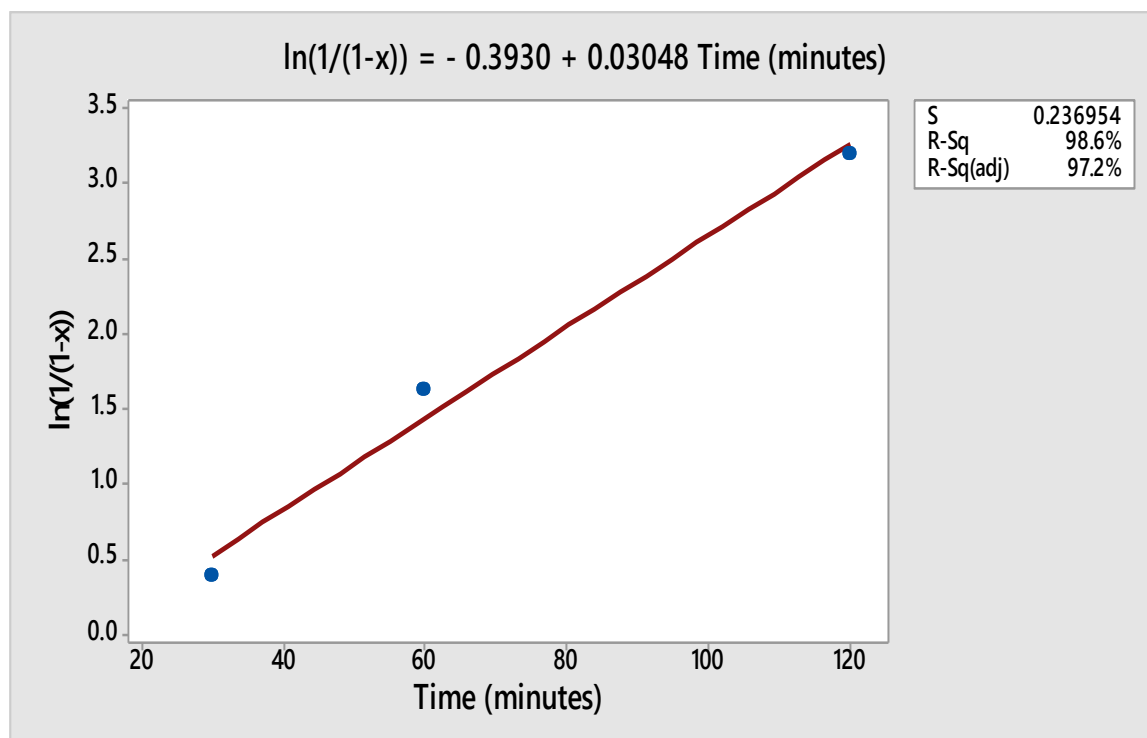


Fig. 3: Reaction rate constant at 70 °C

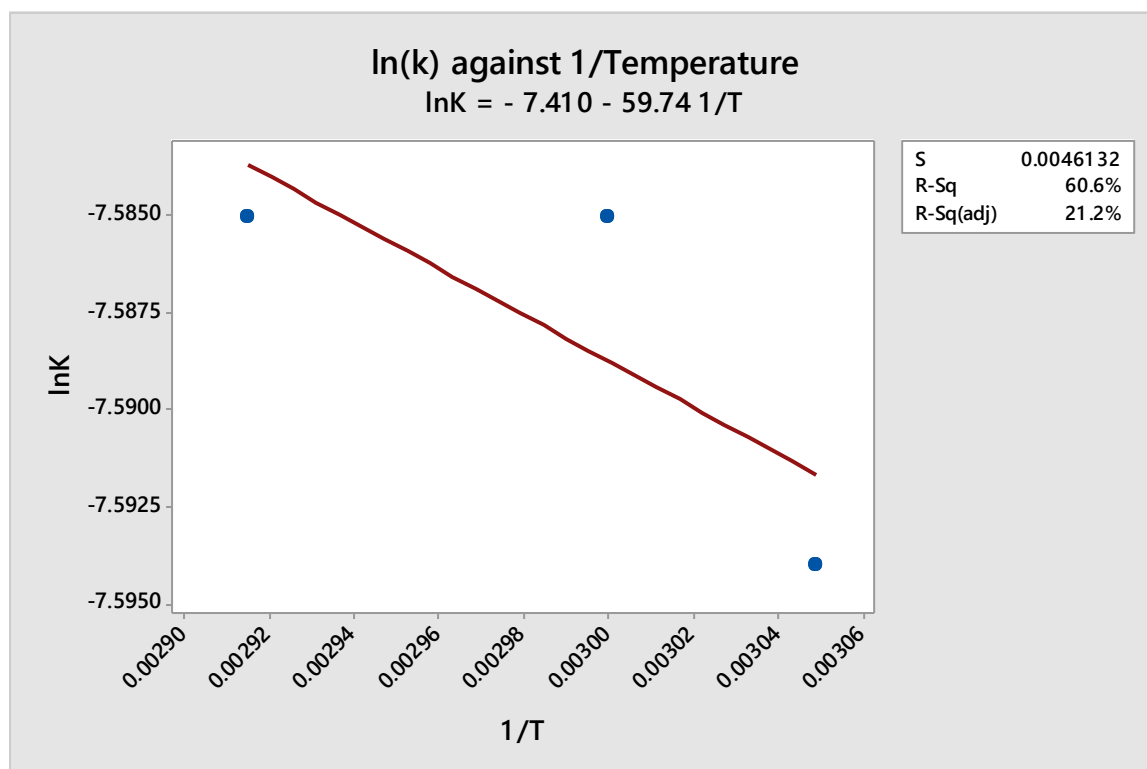


Fig. 4: Fitted line plot of  $\ln k$  versus  $1/T$



The results of the experimental condition, activation energy, frequency factor and correlation coefficient ( $R^2$ ) are shown in Table 6. The activation complex and pre-exponential factor (frequency factor) determined are 496.678 J/Mol and 1652.4263 respectively. Additionally, as the frequency factor increases, the catalyst concentration increases. The high the frequency factor, which is a measure of orientation and collision between reactants, indicates the transesterification reaction is more favoured at 3 wt% than 0.5 and 1 wt%.

Singh and Singh (2010) observed pseudo first-order kinetics for transesterification of palm oil with methanol using  $H_2SO_4$  as a catalyst and determined the activation energy of the reaction as 15.31 KJ/mol, which is high than the one observed in this experiment. Also, the high value of activation energy was determined by Makareviciene *et al.*, (2004) and Supardan (2008), where both observed pseudo- first-order kinetics of the transesterification reaction they conducted with activation energy 13.3 and 30.4 KJ/Mol respectively.

Thiruvengadaravi *et al.* (2009) were also observed pseudo- first-order kinetics for transesterification of *Pongamia* oil with methanol, where 280.1 J/Mol activation energy was determined, which is lower than the one obtained in this experiment.

The activation energy which is the minimum energy required for the reaction to take place, the lower activation energy obtained in this research indicates a significant high catalytic activity of the refluxed calcined snail shell (CaO).

## V. CONCLUSION

The kinetics modelling of transesterification reaction of refined castor seed oil using refluxed calcined snail shell as catalyst shows pseudo-first-order kinetics. The rate constants of the reactions obtained are 0.03048, 0.03207 and 0.03737 with 496.678 J/Mol and 1652.4263 as activation energy and frequency factor. The lower activation energy might be due to improve the catalytic activity of refluxed calcined snail shell or pretreatment of castor oil.

### Conflicts of Interest

There are no conflicts of interest to declare.

## ACKNOWLEDGEMENTS

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*Appendix I:* Fatty acid methyl ester of biodiesel produced at different stages during kinetics studies of the reactions detected by GC/ MS.

FATTY ACID METHYL ESTER NAME	QUALITY	AREA (%)
<b>A1: Conversion at first 30 minutes of the reaction</b>		
Hexadecanoic acid, methyl ester	90	1.8947
10,13- octadecadienoic acid methyl ester	97	3.0916
Methyl stearate	83	0.3495
9- octadecenoic acid, 12- hydroxyl-, methyl ester [R-(Z)]-	93	70.2534
<b>Total</b>		<b>75.5892</b>
<b>Non-methyl ester</b>		<b>24.4106</b>
<b>A2: Conversion at 60 minutes of the reaction</b>		
Hexadecanoic acid, methyl ester	93	1.9024
9- octadecenoic acid (Z)-, methyl ester	99	9.6731
Methyl stearate	99	1.1755
10,13- Octadecadienoic acid, methyl ester	70	3.1291
9- octadecenoic acid-12- hydroxyl-, methyl ester, [R-(Z)]-	94	74.4450
<b>Total</b>		<b>90.3251</b>
<b>Non-methyl ester</b>		<b>9.6749</b>
<b>A3: conversion at 120 minutes of the reaction</b>		
Hexadecanoic acid, methyl ester	99	1.1912
9- octadecenoic acid [Z]-, methyl ester	99	10.0618
Methyl stearate	99	1.1755
9- octadecenoic acid -12-hydroxy-, methyl ester, [R-(Z)]-	94	86.1838
Oxacyclotetradecane-2,11-dione,13-methyl ester	94	0.5089
<b>Total</b>		<b>99.1212</b>
<b>Non-methyl ester</b>		<b>0.8788</b>

FATTY ACID METHYL ESTER NAME	QUALITY	AREA (%)
<b>B1: Conversion at first 30 minutes of the reaction</b>		
2- Hexyne,4- methyl ester	43	0.055
2H- pyran-3,4- dihydro-6-methyl ester	18	0.1017
2- methyl-Z,Z-3,13- octadecadienoic, methyl ester	90	16.8552
Hexadecanoic acid- 2- hydroxyl-1-( hydroxymethyl), methyl ester	55	7.9110
<b>Total</b>		24.9229
<b>Non-methyl ester</b>		75.0771
<b>B2: Conversion at 60 minutes of the reaction</b>		
Methyl-7-methylhexadecanoate	51	0.1520
Hexadecanoic acid, methyl ester	90	17.1341
Methyl stearate	99	0.9555
2- methyl-Z,Z-3,13- octadecadienol, methyl ester	22	0.2013
9- octadecenoic acid-12- hydroxyl-, methyl ester, [R-(Z)]-	94	70.1682
<b>Total</b>		88.7812
<b>Non-methyl ester</b>		11.2188
<b>B3: conversion at 120 minutes of the reaction</b>		
Hexadecanoic acid, methyl ester	99	1.0701
Methyl-7-methylhexadecanoate	55	10.0618
Methyl stearate	99	0.9555
9- octadecenoic acid -12-hydroxy-, methyl ester, [R-(Z)]-	94	84.3821
9,12- octadecadienoic acid (Z, Z)-, methyl ester	99	4.9006
9- octadecenoic acid (Z)-, methyl ester	99	5.1742
<b>Total</b>		96.5280
<b>Non-methyl ester</b>		3.4720
FATTY ACID METHYL ESTER NAME	QUALITY	AREA (%)
<b>C1: Conversion at first 30 minutes of the reaction</b>		
Methyl-7-methylhexadecanoate	55	0.9821
Hexadecanoic acid methyl ester	67	4.8730
Methyl stearate	77	0.1782
9- octadecenoic acid, 12- hydroxyl-, methyl ester [R-(Z)]-	90	26.5857
<b>Total</b>		32.6190
<b>Non-methyl ester</b>		67.3810

### C2: Conversion at 60 minutes of the reaction

Hexadecanoic acid, methyl ester	80	1.2711
Methyl-7-methylhexadecanoate	30	0.6071
Methyl stearate	77	1.1728
10,13- Octadecadienoic acid, methyl ester	70	17.1079
9- octadecenoic acid-12- hydroxyl-, methyl ester, [R-(Z)]-	90	60.1682

**Total** 80.3271

**Non-methyl ester** 19.6729

### C3: conversion at 120 minutes of the reaction

Methyl-7-methylhexadecanoate	99	1.7210
9- octadecenoic acid [Z]-, methyl ester	99	10.7813
Methyl stearate	90	1.0671
9- octadecenoic acid -12-hydroxy-, methyl ester, [R-(Z)]-	95	78.3412
Hexadecanoic acid, methyl ester	88	4.0194

**Total** 95.9300

**Non-methyl ester** 4.0700