



GLOBAL JOURNAL OF SCIENCE FRONTIER RESEARCH: B
CHEMISTRY
Volume 18 Issue 3 Version 1.0 Year 2018
Type: Double Blind Peer Reviewed International Research Journal
Publisher: Global Journals
Online ISSN: 2249-4626 & Print ISSN: 0975-5896

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By Jibrin, M.S., Muhammad, C., Mukhtar, M., Baki, A.S., Bagudo, B.U. & Idris, M.O

Bayero University

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GJSFR-B Classification: FOR Code: 250603



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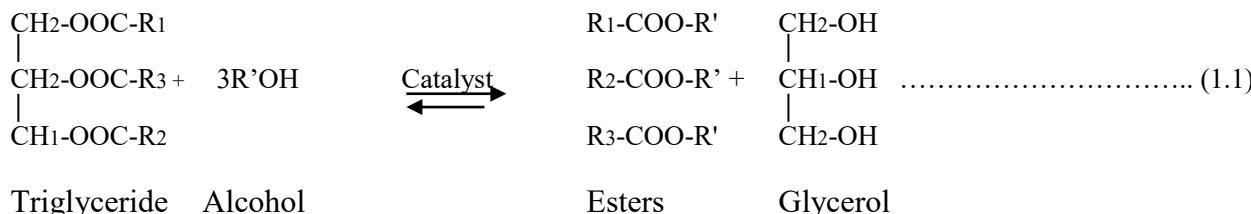
Abstract- Biodiesel from non-edible oil emerges as one of the most energy-efficient and environmentally friendly options in recent times to fulfill the sustainable future energy needs. Hence, there is a need to produce biodiesel on an industrial scale. In designing a reactor, for a large scale production, 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).



Author o p: Department of Pure and Applied Chemistry, Usmanu Danfodiyo University, P.M.B. 2346, Sokoto, Nigeria.
e-mail: muhammadsabiu44@gmail.com

Author Q: Department of Microbiology, Usmanu Danfodiyo University, P.M.B. 2346, Sokoto, Nigeria.

Author a: Department of pure and Industrial chemistry, Bayero University, Kano, Nigeria.

Author §: Department of Chemistry, Kogi State University, Anyigba, Kogi, Nigeria.

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 its 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 cm³ 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 cm³ 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 cm³) 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 cm³/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.

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

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⁻¹ 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	
2.3356	60	60
4.7344	120	
0.2867	30	
2.1876	60	55
3.3604	120	
0.3948	30	
1.6259	60	70
3.2015	120	

Table 4: Calculated rate constant

Rate constant k (min ⁻¹)	Correlation coefficients (R ²)
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)	lnk	Temperature (Kelvin)	$\frac{1}{T}$
6.228x10 ⁻⁴	-7.38123	333	0.00300
5.345x10 ⁻⁴	-7.53418	328	0.00305
5.080x10 ⁻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 (R²) 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 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 lnk and $\frac{1}{T}$ as the linear relationship with a slope of $-\frac{E_a}{R}$ and intercept value of lnA. Since the values of k at a different temperature were determined (as indicated in Table 4). The value of lnk and $\frac{1}{T}$ were calculated as presented in Table 5. By performing a linear regression of lnk 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.

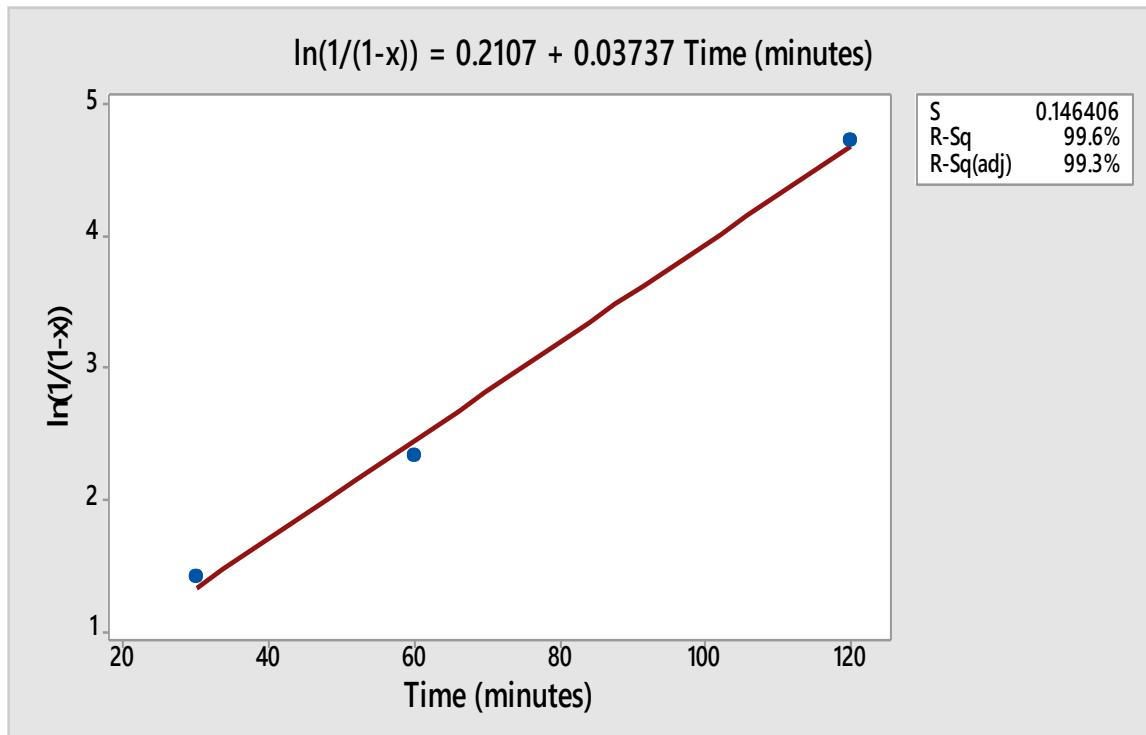


Fig. 1: Reaction rate constant at 60 °C

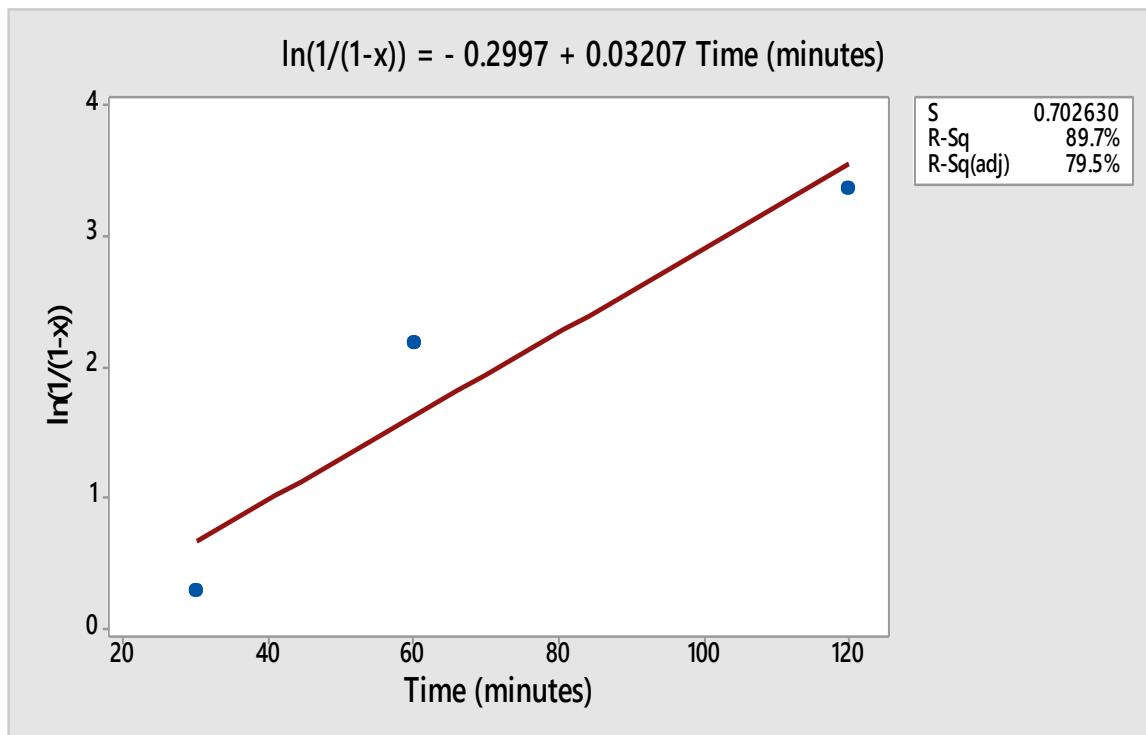


Fig. 2: Reaction rate constant at 55 °C

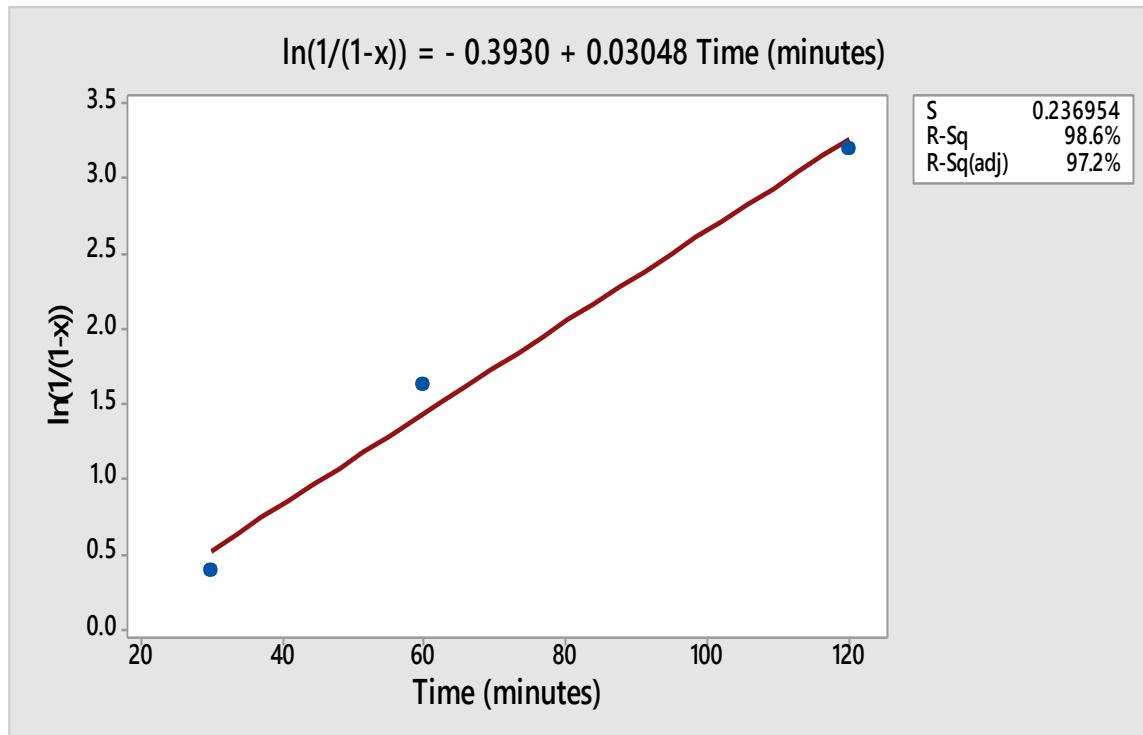


Fig. 3: Reaction rate constant at 70 °C

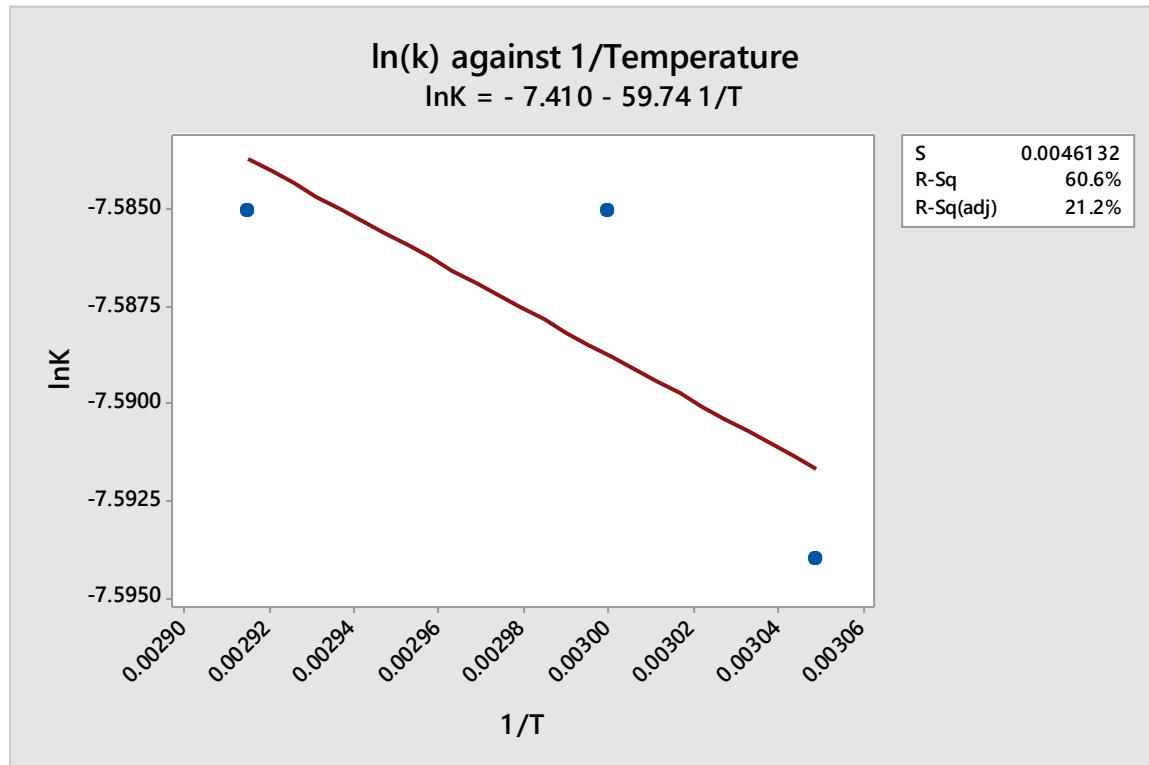


Fig. 4: Fitted line plot of $\ln K$ versus $1/T$

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

One of the authors, Muhammad Sabiu Jibrin (PTDF/ED/LSS/MSC/MSJ/389/17), acknowledged the Petroleum Technology Development Fund (PTDF) for sponsoring this research work.

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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 (%)
A1: Conversion at first 30 minutes of the reaction		
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
Total		75.5892
Non-methyl ester		24.4106
A2: Conversion at 60 minutes of the reaction		
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
Total		90.3251
Non-methyl ester		9.6749
A3: conversion at 120 minutes of the reaction		
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
Total		99.1212
Non-methyl ester		0.8788

FATTY ACID METHYL ESTER NAME	QUALITY	AREA (%)
B1: Conversion at first 30 minutes of the reaction		
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
Total		24.9229
Non-methyl ester		75.0771
B2: Conversion at 60 minutes of the reaction		
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
Total		88.7812
Non-methyl ester		11.2188
B3: conversion at 120 minutes of the reaction		
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
Total		96.5280
Non-methyl ester		3.4720
FATTY ACID METHYL ESTER NAME	QUALITY	AREA (%)
C1: Conversion at first 30 minutes of the reaction		
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
Total		32.6190
Non-methyl ester		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

