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Kinetic modeling of the biodiesel production process using neem seed oil: An alternative to petroleum-diesel

Toyese Oyegoke * and Kazeem Ajadi Ibraheem

Chemical Engineering Department, Faculty of Engineering, Ahmadu Bello University, Zaria 234, Nigeria
oyegoketoyese@gmail.com (T.O.), kazeemolawale634@gmail.com (K.A.I.)

* *Corresponding author at: Chemical Engineering Department, Faculty of Engineering, Ahmadu Bello University, Zaria 234, Nigeria.*
e-mail: oyegoketoyese@gmail.com (T. Oyegoke).

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ABSTRACT

Promoting the green technology campaign that would actualize a biorefinery establishment and would promote cleaner fuel production and air in our environment. This study carried out kinetics studies of biodiesel production over a mixed oxide, Ca-Mg-O catalyst, providing relevant kinetics parameters. This study indicated that biodiesel production is a zero-order reaction, a process independent of the concentration. The results obtained from this study confirm the activation energy, E_a, of the reaction to be 406.53 J/mol, while the pre-exponential factor A was found to be 0.01618 1/min (or 0.9 1/h). Other are kinetics models that were developed for the prediction of the reaction kinetics for the production process is also reported in this study. The findings reported in this study would go a long way to facilitate the modeling, simulation, and design of the biodiesel production process.

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1. Introduction

Biodiesel, an example of biofuels [1-7] which is well known as an alternative petroleum diesel fuel [8], which is produced from the use of batteries, that is, renewable biological sources like algae, plant oils (neem seed oil, sunflower, soybeans, and many others) and animal fats [7,9,10]. Edible feedstocks are primarily discouraged in biofuel production, while nonedible oil like jatropha oil, neem seed oil, and others are promoted. This fuel category is well known for being non-toxic and biodegradable, which has been tested and confirmed to environmentally friendly due to its low emissions [7,11-15]. These features of biodiesel have attracted many attentions toward the promotion of fuels while discouraging the use of fossil fuels such as petroleum diesel, which is mainly used presently as a tool of combating the global warming effect [16,17] caused by the carbon monoxides [18-20] release from the incomplete combustion of fossil fuels and other health-related issues [20,21].

Research carried out so far has indicated that in biodiesel production, a heterogeneous catalyst has always been found to be highly preferred in comparison to homogeneous catalysts due to its effectiveness and easy separation steps for both products and catalysts, eliminate quenching process, and offer conditions for the continuous production system [7,9,22]. In recent studies, nanoparticles' use is significantly gaining greater attention in its use as a heterogeneous catalyst for biodiesel production, which has been observed to have been increasing rapidly. Some of the other studies include Zewude [8], obtained 92.8% yield for the use of neem oil and Na_2O-CaO; Abbah [23] obtained 94% yield from the use of neem seed oil and KOH (homogenous catalyst); Dianursanti *et al.* [24] obtained a yield of 36.8% for the use of CuO/Zeolite and Chlorella Vulgaris oil; Ribwar [25] obtained 83% for the use of waste cooking oil/chicken fat and KOH (homogenous catalyst); Jabiver *et al.* [26] work which obtained 73% yield for the use of soybean and CaO-ZnO; Ajala *et al.* [27] obtained 98.7% yield for the use of palm kernel oil and dolomite as the oil and catalyst (or reaction enhancer) respectively. Other works that employed biocatalysts like Istiningrum *et al.* [28] obtained 81.2% yield in biodiesel production via waste cooking oil and lipase. To further investigate the production of biodiesel, a recent report by Ibraheem *et al.* [19] unveiled the effect of reaction time and temperature on a biodiesel yield in presences of Ca-Mg-O, where the highest yield (96.4%) was obtained at 70 °C, 60 min, 500 rpm, 6:1 g methanol-to-oil ratio, and 1% *w*:*w* catalyst.

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Table 1. Several orders of reaction kinetics models [31].

Kinetic model	Rate constant, k_o (1/min)	Reaction order, n
$C_{ao}X = kt$	$\frac{k}{C_{ao}}$	Zeroth
$ln\left[\frac{1}{1-X}\right] = kt$	k	First
$\frac{1}{C_{ao}}\left[\frac{X}{1-X}\right] = kt$	$k(C_{ao})$	Second

A number of the previous studies have focused mainly on production optimization or effect/parametric studies, and only a few studies give attention to the study of reaction kinetics and the modeling of the kinetics involved in biodiesel production.

This understanding has motivated this present study to explore the use of reaction kinetics principles in providing a better insight into biodiesel production from neem-seed oil in the presence of mixed oxide (Ca-Mg-O) through the provision of relevant kinetic parameters and models. In this study, production was accounted for the yield obtainable across different time intervals (50 to 80 minutes) and different reaction temperatures (50 to 80 °C).

2. Experimental

2.1. Materials and reagents

Methanol (99.5%, analytical grade BDH), isopropyl alcohol (99.5%, analytical grade, BDH), hydrochloric acid (95%, analytical grade BDH), potassium hydroxide (analytical grade M&B), phenolphthalein (indicator), calcium carbonate (catalyst component) and magnesium carbonate (catalyst component) were the reagents and materials employed in this study.

2.2. Synthesis of biodiesel

2.2.1. Catalyst synthesis method

The mixed metal oxide catalyst was prepared by first mixing 75% (22.5 g) calcium carbonate with 25% (7.5 g) magnesium carbonate and then dissolved in 100 mL of water in a 250 mL beaker and stirred on a magnetic stirrer for 30 min without heating. The solution was filtered to remove the water and was dried in an oven at 120 °C overnight to constant weight, then calcined in a furnace at 900 °C for 90 min. The calcination temperature causes the carbonate compounds to decompose into an oxide compound, as presented in Equations (1) and (2).

$$CaCO_3 \rightarrow CaO + CO_2 \quad (1)$$

$$MgCO_3 \rightarrow MgO + CO_2 \quad (2)$$

The mixed metal oxide catalyst was synthesized in like way. Such in situ synthesis result in an intimate contact of the CaO with the MgO during the calcination, in agreement with Javier *et al.* [26] report for the synthesis for CaO-ZnO. The catalyst was placed in a desiccator to avoid moisture absorption. Details on the characterization of the prepared catalyst employed in this study can find in our previous report [19].

2.2.2. Transesterification

A two-step transesterification was adopted for this study where the oil was first pretreated with the use of sulphuric acid to have the free fatty acid (FFA) reduced to 1.6±0.05 %FFA in line with the report of Bello *et al.* [29], Yusuf [30], and Ibraheem *et al.* [19] which indicates that low-FFA oil would give a relative higher biodiesel yield in a later step. In the latter method, 12 g of the pretreated oil was heated to 50 °C, and 6:1 g methanol to oil ratio with 1% *w*:*w* of the catalyst was added. The oil was pretreated to reduce the FFA present in the oil to improve the yield, in agreement with our previous study [19], where the neem oil FFA and density were reported. The stirring speed was kept at 500 rpm. The process was repeated at 60-80 °C, and time varied from 50-70 min for each temperature, leaving other parameters constant. The maximum yield of biodiesel using the expression is presented in Equation (3).

$$X = \frac{Nb}{No} \quad (3)$$

Nb is the amount of biodiesel produced, and No is the amount of oil used. The results obtained were recorded and tabulated.

2.3. Kinetic modeling for the biodiesel production

In modeling the kinetics of the biodiesel reaction, the experimentally collected data were fitted into several set of kinetic models, which include the zeroth, first, and second-order models, which were adopted from literature reports about the different order of reaction kinetic models and the summary of the details regarding the kinetic models are presented in Table 1. With the use of computational tools, the experimental data were fitted using a linear regression analysis approach alongside the assessment of the models' fitness accuracy were investigated via the use of scattered plots and *R*-square values.

2.3.1. Reaction order and rate constant determination

The reaction order and rate constants were determined for different temperature ranges using the linear regression model (presented in Equation (4)) to fit the experimental data into the respective kinetic model to assess its fitness for the biodiesel production kinetics.

$$y = mx + c \quad (4)$$

To be specific, for the reaction studied, m denoted the slope or gradient, while c denotes the intercept that is constrained to be zero since the set of reaction kinetic models lacks intercepts. Summary of the respective parameters employed in the study for the regression analysis for the reaction kinetic models is presented in Table 2, where the axes (i.e., *y* and *x*) and slope (i.e., m) notations were presented.

2.3.2. Determination of activation energy and kinetic rate model

The set of slopes, temperature, and R-square values collected were used in the computation of the reaction activation, E_a energy, using the Arrhenius equation (in Equation (5)). The model was linearly (as presented in Equation (4)) fitted using regression analysis where the c intercept of the model was taken to be a nonzero value. According to the literature [32-34]:

$$k = A \exp\left(-\frac{E_a}{RT}\right) \quad (5)$$

Table 2. Regression model parameter relation with the reaction kinetics models.

Reaction order, n	Model	*y*	*x*	m	c
Zeroth	$X = \frac{k}{C_{ao}} t$	X	t	$\frac{k}{C_{ao}}$	0
First	$ln\left[\frac{1}{1-X}\right] = kt$	$ln\left[\frac{1}{1-X}\right]$	t	k	0
Second	$\left[\frac{X}{1-X}\right] = kt(C_{ao})$	$\left[\frac{X}{1-X}\right]$	t	$k(C_{ao})$	0

Table 3. Results of biodiesel yield obtained from the experiment.

t (min)	X (T = 50 °C)	X (T = 60 °C)	X (T = 70 °C)	X (T = 80 °C)
50	0.9180	0.8875	0.9250	0.9530
60	0.9380	0.9400	0.9640	0.9450
70	0.9380	0.9540	0.9150	0.9380
80	0.9170	0.9570	0.9420	0.9460

Table 4. Kinetic modeling via the use of a fitting model.

T (°C)	t (min)	1st Order, X	2nd Order, ln[1/(1-X)]	3rd Order, X/(1-X)
T_1 = 50	50	0.9180	2.5010	11.1951
T_1 = 50	60	0.9380	2.7806	15.1290
T_1 = 50	70	0.9380	2.7806	15.1290
T_1 = 50	80	0.9170	2.4889	11.0482
T_2 = 60	50	0.8875	2.1848	7.8889
T_2 = 60	60	0.9400	2.8134	15.6667
T_2 = 60	70	0.9540	3.0791	20.7391
T_2 = 60	80	0.9570	3.1466	22.2558
T_3 = 70	50	0.9250	2.5903	12.3333
T_3 = 70	60	0.9640	3.3242	26.7778
T_3 = 70	70	0.9150	2.4651	10.7647
T_3 = 70	80	0.9420	2.8473	16.2414
T_4 = 80	50	0.9530	3.0576	20.2766
T_4 = 80	60	0.9450	2.9004	17.1818
T_4 = 80	70	0.9380	2.7806	15.1290
T_4 = 80	80	0.9460	2.9188	17.5185

$$\ln k = \ln A - \frac{E_a}{R}\left(\frac{1}{T}\right) \qquad (6)$$

$$E_a = \frac{-d(\ln k)}{d\left(\frac{1}{RT}\right)} = \frac{-Rd(\ln k)}{d\left(\frac{1}{T}\right)} = -R(slope) \qquad (7)$$

$$A = \exp(\text{intercept}) \qquad (8)$$

where k = Rate constant, T = Reaction temperature, *A* = Pre-exponential factor, and R = Gas constant (8.3142 J/mol/K).

In the regression analysis, the ln *k* and 1/T were modeled as the vertical and horizontal axis, respectively, where the slope, m represents -E_a/R, while the intercept, c represents ln *A* in the analysis. The *R*-square values for the model were also assessed. A kinetic rate model is later modeled after using the results obtained for the rate constant model, k, and the reaction activation energy, E_a.

3. Results and discussions

3.1. Reaction order and rate constants analysis

The biodiesel yield results across different time and temperature ranges are presented in Table 3, while the other relevant expressions for the computation of the rate constants and the determination of reaction orders were presented in Table 4.

Table 4 presents the relevant parameters used to represent the scattered plot used to investigate the zeroth, first, and second-order reactions in the search for order that best fit the experiment phenomena. In all orders, the horizontal axis was presented as reaction time (t), while the vertical axis was presented as X, ln[1/(1-X)], and X/(1-X) for the zeroth, first and second-order models, respectively in line with the method presented in Table 2.

3.2. Rate constants for different temperature ranges

The rate constant for different temperatures from 50 to 80 °C using the scattered plot as presented in Table 5, where the slope (m) was used to compute the rate constant for each temperature, was collected alongside the *R*-square values. Findings from the results obtained for the rate constant at the different reaction temperatures ranging from 323 to 353 K indicated that all model orders showed a good *R*-square value, where the average *R*-square values were reported to be 97.3, 97.1, and 92.5 % for the zeroth, first, and second-order model indicating the zeroth-order displayed a better agreement with the experiment data employed in the modeling of the reaction.

Moreover, the rate constant (i.e., k_o = slope) was seen to generally increases as the reaction temperature rises, showing that there exists a direct relationship between the rate constant (i.e., k_o = slope) and reaction temperature for the zeroth reaction order, unlike other higher reaction order (first and second) which indicated a rise and fall at a specific temperature (333 and 343 K) across the range employed in this study evident in Tables 5 and 6.

In a nutshell, the analysis of temperature effects confirms the use of zeroth reaction order for the biodiesel production studied to use neem seed oil and Ca-Mg-O catalyst. The order obtained for this process was found to be similar to the report of Mamat [35] and Valenga *et al.* [36] for the use of Waste-Cooking-Oil/CaO and Moringa-Leave-Waste/KOH (homogeneous), respectively, following a zeroth-order reaction. However, the results were different from those reported in other literature [37,38] for the use of cottonseed oil/CaO and Aegyptiaca Oil/NaOH (homogeneous catalyst) as second order and first order, respectively.

3.3. Results for the computation of activation energy

The parameter employed in determining the reaction activation energy in Table 6, while the results obtained from the analysis were presented in Table 7 and Figure 1. Table 6 shows where k_1 = k/Cao, k_2 = k, and k_3 = kCao is zeroth, first, and second-order rate constants.

Table 5. The kinetic plot's *R*-square value, slopes, and intercepts at varying temperatures.

Temperature		**Reaction order, n**					
		Zeroth - ko = k/Cao = m, n = 0		**First - ko = k = m, n = 1**		**Second - k_3 = kCao = m, n = 2**	
T (°C)	**T (K)**	***R*-square**	**Slope m = k_1**	***R*-square**	**Slope m = k_2**	***R*-square**	**Slope m = k_3**
50.00	323.15	0.9710	0.0139	0.9679	0.0394	0.9479	0.1960
60.00	333.15	0.9793	0.0140	0.9954	0.0428	0.9719	0.2625
70.00	343.15	0.9710	0.0140	0.9569	0.0424	0.8415	0.2458
80.00	353.15	0.9701	0.0141	0.9633	0.0434	0.9391	0.2589
Average *R*-square		0.9729	-	0.9709	-	0.9251	-

Table 6. Activation energy and pre-exponential factor analysis where the rate constant, k_o in 1/min.

T (K)	**1/T (1/K)**	**k_1**	**k_2**	**k_3**	**ln k_1**	**ln k_2**	**ln k_3**
323.15	0.003095	0.0139	0.0394	0.1960	-4.27587	-3.23399	-1.62964
333.15	0.003002	0.0140	0.0428	0.2625	-4.26870	-3.15122	-1.33750
343.15	0.002914	0.0140	0.0424	0.2458	-4.26870	-3.16061	-1.40324
353.15	0.002832	0.0141	0.0434	0.2589	-4.26158	-3.13730	-1.35131

Table 7. Results of activation energy, E_a and preexponential factor, A.

Description	**Reaction order**		
	Zeroth, n=0	**First, n=1**	**Second, n=2**
Slope = (-E_a/R)	-48.896	-324.250	-893.910
Intercept = ln *A*	-4.1240	-2.2108	1.2160
R-square	0.8990	0.7207	0.5548
E_a (J/mol) = (-slope*R)	406.5311	2695.879	7432.147
A (1/min) = exp(intercept)	0.01618	0.109613	3.373666

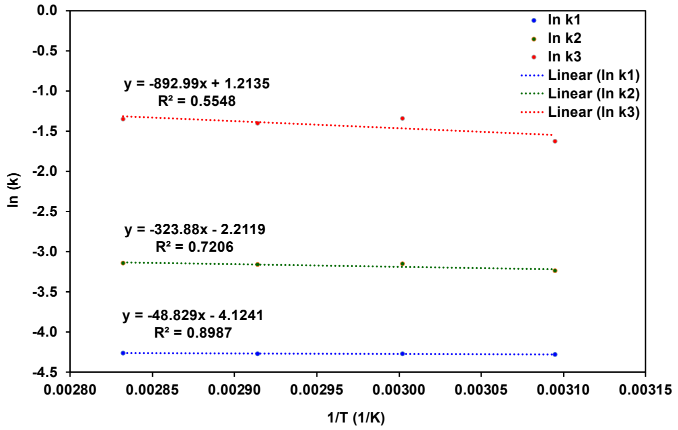

Figure 1. A plot for the computation of activation energy, E_a, and preexponential factor, *A*.

Moreover, the reaction's activation energy was therefore confirmed to be 406.53 J/mol, while the pre-exponential factor was found to be 0.01618 1/min (or 0.9 1/h). Findings from literature indicated that the results obtained for the activation energy in this study were smaller than that reported as 23.2 kJ/mol for Waste-Cooking-Oil/CaO [35]; 127.7 kJ/mol for cottonseed oil/CaO [37]; and 242.1 kJ/mol for Moringa-Leave-Waste/KOH [36]. Furthermore, the Arrhenius model for the rate constant prediction in line with Equation (5) can be written thus:

$$k_o = 0.01618\exp\left(-\frac{406.5311}{\mathrm{RT}}\right) \text{ in } 1/\text{min} \tag{9}$$

Recall that k_o (general form) is expressed in per second (Table 2), where *k* is the specific rate constant for the reaction whose relationship as presented in Table 2 were represented as:

$$k_o = \frac{k}{\mathrm{C_{ao}}} \qquad \text{(Zeroth order)} \tag{10}$$

$$k_o = k \qquad \text{(First order)} \tag{11}$$

$$k_o = k\mathrm{C_{ao}} \qquad \text{(Second order)} \tag{12}$$

Re-expressing the above expression in Equation (10) to (12) to make the specific rate constant subject of the kinetic rate constant expression result into:

$$k = k_o\mathrm{C_{ao}} \qquad \text{(Zeroth order)} \tag{13}$$

$$k = k_o \qquad \text{(First order)} \tag{14}$$

$$k = \frac{k_o}{\mathrm{C_{ao}}} \qquad \text{(Second order)} \tag{15}$$

Substituting Equation (9) into Equation (13) (since the zeroth-order fit best with the experimental output) to yield:

$$k = 0.01618\mathrm{C_{ao}}\exp\left(\frac{-406.5311}{\mathrm{RT}}\right) \text{ in mol/(m}^3\text{/min)} \tag{16}$$

Using C_{ao} = 1 mol/m^3, Equation (16) becomes:

$$k = 0.01618\exp\left(\frac{-406.5311}{\mathrm{RT}}\right) \text{ in mol/(m}^3\text{/min)} \tag{17}$$

In the rate constant expression, C_{ao} is the initial concentration of the feed (oil), R is 8.3142 J/mol/K, T is the reaction temperature in K, and *k* is the reaction specific rate constant (zero-order reaction) expressed in mol/(m^3.s), which were found to agree with the literature [32-34,36,37].

3.4. Kinetic rate model

In modeling the biodiesel production rate model, the rate of biodiesel production can be generally be modeled to be in the form:

$$\frac{\mathrm{dC_a}}{\mathrm{dt}} = k\mathrm{C_a^n} \tag{18}$$

Employing the results obtained for the reaction order, the reaction order (n = 0, $C_a^0 = 1$) can be substituted into Equation (18) to give us a zeroth-order reaction rate model:

$$\frac{\mathrm{dC_a}}{\mathrm{dt}} = k\mathrm{C_a^0} = k \tag{19}$$

The above model expression developed for the zeroth order-based reaction was found to be in line with relevant related works in the literature. The kinetic rate model can be represented by substituting the development rate constant expression in Equation (16) into (19) to give us:

$$\frac{\mathrm{dC_a}}{\mathrm{dt}} = 0.01618\mathrm{C}_{ao}\exp\left(-\frac{406.5311}{\mathrm{RT}}\right) \text{ in mol/(m}^3\text{/min)} \tag{20}$$

$$\frac{1}{\mathrm{C}_{ao}}\frac{\mathrm{dC}_a}{\mathrm{dt}} = 0.01618\exp\left(-\frac{406.5311}{\mathrm{RT}}\right) \text{ in mol/(m}^3\text{/min)} \tag{21}$$

$$\frac{\mathrm{dX}}{\mathrm{dt}} = 0.01618\exp\left(-\frac{406.5311}{\mathrm{RT}}\right) \text{ in mol/(m}^3\text{/min)} \tag{22}$$

where the initial concentration (i.e., $\mathrm{C_{ao}}$) is expressed in mol/m^3 in the form of Equation (20), while Equation (22) expresses the concentration as a normalized variable (i.e., $\mathrm{dC_a/C_{ao}} = \mathrm{dX}$) where all concentrations are expressed as a fraction of the initial concentration (i.e., $\mathrm{C_{ao}}$).

4. Conclusions

The exploration of reaction kinetics principles towards providing a better insight into biodiesel production from neem-seed oil in the presence of mixed oxide (Ca-Mg-O) through the provision of relevant kinetic parameters and models was successfully carried out.

Therefore, the study's findings indicate that the analysis of temperature effects confirms the use of zeroth reaction order for the biodiesel production studied with the use of neem seed oil and Ca-Mg-O catalyst. Moreover, the activation energy, E_a of the reaction, was therefore confirmed to be 406.53 J/mol, while the preexponential factor, A, was found to be 0.01618 1/min (or 0.9 1/h).

The kinetic parameters are computed together with the reaction kinetics models developed, which would fit nicely into prediction provisions and would go a long way to facilitate the modeling, simulation, and design of the biodiesel production process through the relevant kinetic data and model reported in this study.

Disclosure statement

Conflict of interests: The authors declare that they have no conflict of interest.
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ORCID

Toyese Oyegoke
https://orcid.org/0000-0002-2026-6864
Kazeem Ajadi Ibraheem
https://orcid.org/0000-0002-2474-881X

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