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## Kinetic Parameters for KOH-Catalyzed Transesterification of Neem Oil (*Azadirachta indica*) with Ethanol to Produce Fatty Acid Ethyl Ester

Ohimor, Evuensiri Onoghwarite<sup>1</sup>, Bolatito, Oyinkansola Opeyemi<sup>2</sup>,  
Toritseju, Oritsejolomisan Marvellous<sup>3</sup>, Oguduvwe, Jonathan Pius<sup>4</sup>,  
Umunade, Oghenemagan Oghenemeru<sup>5</sup>

1 Department of Chemical Engineering, Federal University of Petroleum Resources, Effurun, Nigeria, ☎ +2348033888418,

✉ ohimor.evuensiri@fupre.edu.ng

2 Department of Chemical Engineering, Federal University of Petroleum Resources, Effurun, Nigeria, ☎ +2349078431387,

✉ bolatitooyinkansola24@gmail.com

3 Department of Chemical Engineering, Federal University of Petroleum Resources, Effurun, Nigeria, ☎ +2349017010025,

✉ toritsejumarvel@gmail.com

4 Department of Chemical Engineering, Federal University of Petroleum Resources, Effurun, Nigeria, ☎ +2349046178887, ✉ poguduvwe@gmail.com

5 Department of Chemical Engineering, Federal University of Petroleum Resources, Effurun, Nigeria, ☎ +2349070923287, ✉ magangavra@gmail.com

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## ABSTRACT

Biodiesel derived from non-edible neem oil (*Azadirachta indica*) is a promising alternative to first-generation biodiesel produced from edible vegetable oils. This study investigated the kinetics of the transesterification of pretreated neem oil using potassium hydroxide (KOH) as a catalyst and anhydrous ethanol at temperatures between 45°C and 65°C. The crude neem oil initially had an acid value of 20.93 mg KOH/g (free fatty acid, FFA = 10.46%). To reduce the FFA content, the oil was subjected to a two-step acid esterification pretreatment, which lowered the acid value to 0.84 mg KOH/g (FFA = 0.42%). After pretreatment, base-catalyzed transesterification was performed. A pseudo-first-order kinetic model was used to analyze time-dependent biodiesel yield at reaction times of 15, 30, 60, and 90 minutes under four experimental conditions. Linear plots of  $-\ln(1 - X)$  versus reaction time produced good fits ( $R^2 = 0.967$ – $0.982$ ) at the three temperature levels, confirming the pseudo-first-order assumption under excess ethanol conditions. The rate constants increased with temperature, from  $0.0030 \text{ min}^{-1}$  at 45°C to  $0.0079 \text{ min}^{-1}$  at 55°C and  $0.0367 \text{ min}^{-1}$  at 65°C. An Arrhenius plot of  $\ln(k)$  versus  $1/T$  yielded an activation energy of 111.97 kJ/mol ( $R^2 = 0.998$ ) and a pre-exponential factor of  $2.06 \times 10^9 \text{ min}^{-1}$ . These results indicate that the reaction is chemically controlled with negligible mass-transfer resistance. The derived rate law provides essential data for reactor design and supports scaling up neem oil biodiesel production in Nigeria and other parts of sub-Saharan Africa.

**Key words:** Biodiesel, Neem oil, *Azadirachta indica*, Transesterification, Kinetics, Pseudo-first-order, Activation energy, Arrhenius equation, Nigeria

**Corresponding Author:** Ohimor E.O.

## INTRODUCTION

The twenty-first century has placed significant pressure on global energy systems due to rapid industrialization and population growth. The International Energy Agency projects that global primary energy demand will increase by about 25–30% over the next two decades, with most of this growth occurring in developing regions such as sub-Saharan Africa and South Asia [1]. Meeting this increasing demand while simultaneously reducing greenhouse gas emissions to levels required by the Paris Agreement represents a major engineering and policy challenge. Liquid transportation fuels present a particular difficulty because they are deeply embedded in existing infrastructure, including vehicles, engines, pipelines, and storage systems. Consequently, replacing them with alternative energy carriers would require substantial technical and financial investment. In this context, biodiesel has attracted long-term interest as a renewable drop-in fuel. Biodiesel is produced through the transesterification of triglyceride-containing biological materials such as vegetable oils, animal fats, or microbial lipids, with short-chain alcohols (usually methanol or ethanol) in the presence of a catalyst. The resulting product, composed mainly of fatty acid alkyl esters, is completely miscible with petroleum diesel in any proportion. In most cases, biodiesel can be used in existing diesel engines without modification at blend ratios up to B20 (20% biodiesel and 80% petroleum diesel) or higher. Studies have shown that biodiesel blends reduce emissions of particulate matter, carbon monoxide, unburned hydrocarbons, and sulfur oxides compared with conventional diesel fuel, although nitrogen oxide emissions may increase slightly depending on engine calibration and blend ratio [1,2]. Biodiesel also offers environmental benefits, with life-cycle carbon dioxide emissions estimated to be 40–80% lower than those of

petroleum diesel, depending on the feedstock and production process. Nigeria represents an important case for the development of domestic biodiesel production. As the most populous country in Africa, with a population of about 220 million, Nigeria has one of the continent's largest transportation energy demands. Despite being a major crude oil producer and a member of OPEC, the country imports a significant proportion of its refined petroleum products. This long-standing paradox largely results from persistent underinvestment and underutilization of domestic refining infrastructure. In addition, about 50% of the population lives in rural areas where access to reliable electricity is limited. Many households and small businesses rely on diesel-powered generators due to the absence or unreliability of grid electricity. As a result, fluctuations in global petroleum prices directly affect the economic well-being of rural households. Local biodiesel production could therefore improve energy security and reduce dependence on imported fuels [2,3]. However, first-generation biodiesel produced from edible oils such as palm, soybean, or rapeseed is not a suitable option for Nigeria for two main reasons. First, the country faces significant food security challenges, and diverting edible oils or agricultural land from food production to fuel production could worsen the situation. Second, the economics of biodiesel production from edible oils are often unfavorable because feedstock costs typically account for 60–80% of total production costs, and edible oils command higher market prices due to their food value. These challenges can be mitigated by using non-edible oil feedstocks, which do not compete with food supplies and are generally less expensive because they have limited direct consumption value [3,5]. Among the various non-edible oil-bearing plants available in Nigeria, neem (*Azadirachta indica*) is particularly promising. The neem tree is widely distributed across all thirty-six states of the country and is especially abundant in the northern savanna and Guinea ecological zones, where access to diesel fuel is often limited. Neem trees require minimal maintenance, are drought-resistant, and grow rapidly. Their seed kernels contain approximately 30–50% oil and can begin producing fruit within a relatively short period with minimal agricultural inputs [3]. Neem oil has no culinary value because it contains limonoid compounds, particularly azadirachtin, which makes the oil bitter and biologically active. These compounds give neem oil strong insecticidal and pesticidal properties, making it useful in agricultural applications. Consequently, integrated processing of neem seeds could allow the simultaneous production of biodiesel and neem-based biopesticides, thereby enhancing the economic value of the neem supply chain [2,4]. The key chemical reaction involved in neem biodiesel production is the transesterification of neem oil with ethanol in the presence of potassium hydroxide (KOH) as a catalyst. Despite the potential of neem as a biodiesel feedstock, the detailed reaction kinetics of this system have not been extensively reported in the literature prior to this study. Understanding the reaction kinetics is essential for effective process design and scale-up. Parameters such as the reaction rate constant and activation energy determine the minimum residence time required to achieve a desired conversion at a given temperature. They also indicate the sensitivity of the process to temperature changes, guide the selection of catalyst loading, and help determine whether batch or continuous reactor operation is more suitable. Without such parameters, scaling laboratory processes to pilot or commercial scale often relies on empirical approximations rather than sound engineering design principles [6,7]. This study therefore focuses on the kinetic characterization of neem oil transesterification with ethanol. Time-dependent biodiesel yield experiments were conducted at temperatures of 45°C, 55°C, and 65°C, using catalyst concentrations ranging from 0.75 to 1.5 wt% and ethanol-to-oil molar ratios of 6:1 to 9:1. The experimental data were analyzed using a pseudo-first-order kinetic model to determine

the reaction rate constants. These constants were subsequently used to construct an Arrhenius plot, from which the activation energy and pre-exponential factor of the reaction were determined.

## MATERIALS AND METHODS

### 2.1 Feedstock Procurement and Characterization

In Delta State, crude neem oil was supplied by the agribusiness company Kaspharyn Solutions Nigeria. The oil was obtained through cold pressing of mature, dried neem kernel seeds and was neither refined nor bleached before being supplied for the research. This approach was adopted to reflect the characteristics of crude neem oil that would typically be available to a small-scale biodiesel producer in Nigeria. Upon receipt, the oil was stored in a sealed dark glass container at room temperature (24–28 °C) to minimize the increase in free fatty acids (FFA) resulting from enzymatic hydrolysis prior to processing. Before further processing, the acid number of the crude oil was determined in triplicate using the ASTM D974-19 method [10]. All reagents used in the study were of analytical grade. Potassium hydroxide (KOH, 99.0 wt% purity, pellet form) was used as the base catalyst for the transesterification reaction. The alcohol used for transesterification was anhydrous ethanol (>99.5 vol%). The pretreatment esterification stage employed concentrated sulfuric acid (H<sub>2</sub>SO<sub>4</sub>, 98 wt%) as the acid catalyst and anhydrous methanol (99.8 vol%) as the alcohol. For the titration procedure, a standard KOH solution (0.1 N), phenolphthalein indicator (1 wt% in ethanol), and distilled water were prepared in the laboratory.

### 2.2 Laboratory Equipment and Instruments

All transesterification reactions were conducted in 250 mL Erlenmeyer flasks placed on a digital hotplate–stirrer equipped with temperature feedback control. The reaction temperature was monitored using a calibrated glass thermometer (±0.5 °C). Reagent masses were measured using a digital analytical balance. Phase separation of the reaction products was carried out using 250 mL glass separatory funnels. For the determination of acid value and detection of the titration endpoint with the indicator, a 25 mL burette (±0.05 mL) was used.

### 2.3 Acid Value Determination (ASTM D974-19)

The acid values (AV) of the crude and pretreated neem oils were determined in accordance with ASTM D974-19 [10]. Approximately 10.0 g of oil was weighed into a 250 mL Erlenmeyer flask. A freshly prepared solvent mixture of toluene–isopropanol (1:1 v/v) was added, and the mixture was gently heated to ensure complete dissolution of the oil. The solution was then allowed to cool to room temperature, after which 1 mL of phenolphthalein indicator was added. The mixture was titrated with standardized 0.1 N KOH solution until the first persistent pink color appeared and remained for at least 30 seconds. The volume of KOH used (V, mL) was recorded. The acid value was calculated using the following equation:

$$AV \text{ (mg KOH/g)} = (56.1 \times N \times V) / m \quad \dots(1)$$

where 56.1 is the molar mass of KOH (g/mol), N is the normality of the KOH titrant, V is the volume of KOH used (mL), and m is the mass of the oil sample (g). The percentage free fatty acid (FFA) content was estimated as AV/2, assuming oleic acid as the representative fatty acid with an average molar mass of 282 g/mol.

## 2.4 Acid Esterification Pretreatment

The crude neem oil was pretreated by followed by a base-catalyzed transesterification step. The stoichiometric amount of methanol required to convert the free fatty acids to methyl esters was calculated using a 6:1 molar excess relative to the FFA content, and the required quantity was measured based on the determined FFA level. The methanol contained concentrated  $\text{H}_2\text{SO}_4$  (2 wt% of the oil mass) as the acid catalyst. The methanol–acid mixture was added to the oil in a 1000 mL beaker, mechanically stirred at 400 rpm, on a hotplate at 60 °C for 2 hours. After the reaction, the mixture was allowed to cool to room temperature and then transferred into a 500 mL separatory funnel. The mixture was allowed to stand for at least 12 hours to enable phase separation. The lower layer, consisting of aqueous methanol–water– $\text{H}_2\text{SO}_4$ , was separated from the upper esterified oil layer and drained off. The esterified oil layer was washed twice with warm distilled water (50 °C), using 50 mL of water per wash. Washing was carried out gently to avoid vigorous mixing, which could lead to emulsion formation. The wash water was drained after each washing step. To remove residual moisture, the washed oil was dried in an oven at 110 °C for 30 minutes. This drying step was necessary because the presence of water could hinder the subsequent base-catalyzed transesterification by promoting catalyst hydrolysis. The entire procedure was repeated three times, and the acid value of the dried pretreated oil was determined.

## 2.5 Base-Catalyzed Transesterification Procedure

This procedure was applied in all transesterification experiments, where the fixed oil charge was 50 g of pretreated neem oil. For each run, the required mass of KOH (0.75, 1.125, or 1.50 wt% relative to the mass of the oil) was dissolved in the measured volume of anhydrous ethanol corresponding to the desired ethanol-to-oil molar ratio (6:1, 7.5:1, or 9:1). The mixture was gently stirred for about 5 minutes in a small glass beaker to generate potassium ethoxide in situ. The required amount of ethanol was calculated using the molecular weight of neem oil triglyceride (approximately 893 g/mol, based on the fatty acid composition), the mass of the oil, and the selected molar ratio. The pretreated neem oil was then transferred into a 250 mL Erlenmeyer flask and heated to the desired reaction temperature (45°C, 55°C, or 65°C) under continuous stirring at 400 rpm. Once the oil reached the set temperature (monitored using a thermometer), the prepared potassium ethoxide solution was rapidly introduced into the oil. The flask was immediately sealed with parafilm and aluminum foil to minimize ethanol evaporation, and the reaction time was started ( $t = 0$ ). At the end of the specified reaction time, the entire reaction mixture was poured into a separatory funnel and allowed to settle under gravity for at least 12 hours to enable phase separation. The FAEE yield was determined gravimetrically using Equation 2, which gives the mass fraction of oil converted to ester product:

$$\text{Yield (\%)} = (\text{mass of FAEE phase} / \text{mass of pretreated neem oil}) \times 100 \quad \dots(2)$$

## 2.6 Kinetic Data Analysis

Kinetic experiments yielded data converted to fractional conversion,  $X = \text{yield}/100$ . The analysis was performed using the values of  $-\ln(1-X)$  at each time point ( $t = 15, 30, 60, 90$  min). For each experimental condition, the data of  $-\ln(1-X)$  vs.  $t$  were plotted, and a linear regression was run, with the line passing through the origin ( $X = 0$  at  $t = 0$  by definition). The slope of the regression line provided the apparent pseudo-first-order rate constant  $k'$  ( $\text{min}^{-1}$ ), and the coefficient of determination  $R^2$  indicated the goodness of fit of the model. For Arrhenius analysis, the three rate constants obtained at the primary temperature conditions (45°C, 55°C, 65°C) were used. Plots were made of the natural logarithms of the rate constants  $[\ln(k)]$  versus  $1$  divided by the absolute temperature  $[1/T \text{ (K}^{-1})]$ . The Arrhenius equation ( $\ln k = \ln A -$



$E_a/RT$ ) was plotted against  $1/T$ , where the slope gave  $E_a/R$ , and thus  $E_a$  was determined; the intercept gave  $\ln A$ , and therefore the pre-exponential factor  $A$ .

## RESULTS AND DISCUSSION

### 3.1 Acid Value Reduction by Pretreatment

The average acid value of the crude neem oil was 20.93 mg KOH/g, representing an FFA content of 10.46%, compared to the reported ranges of 8–15% for unrefined neem oil in Nigeria [3, 9]. The high FFA content and such high loading of KOH render direct base-catalyzed transesterification impractical. In a hypothetical calculation, at 10.46% FFA and 1.5 wt% catalyst loading, nearly all the catalyst would be consumed in saponification before transesterification could occur, leaving none to catalyze the triglyceride conversion reaction. It was therefore not an undue precaution but a thermodynamic necessity.

Table 1. The neem oil acid value and FFA content prior to and after the pretreatment in the form of acid esterification

Sample	AV (mg KOH/g)	FFA (%)
Crude neem oil	20.93	10.46
Pretreated neem oil	0.84	0.42

The average acid value of the crude neem oil was 20.93 mg KOH/g, representing an FFA content of 10.46%, compared to the reported ranges of 8–15% for unrefined neem oil in Nigeria [3, 9]. The high FFA content and such high loading of KOH render direct base-catalyzed transesterification impractical. In a hypothetical calculation, at 10.46% FFA and 1.5 wt% catalyst loading, nearly all the catalyst would be consumed in saponification before transesterification could occur, leaving none to catalyze the triglyceride conversion reaction. It was therefore not an undue precaution but a thermodynamic necessity.

### 3.2 Time-Course Yield Profiles

Table 2 shows the yield data for the time-course of four experimental conditions. All four conditions display a consistent trend: rapid yield formation to 91–94% in 15 minutes, after which the rate slows down as the reaction approaches equilibrium in the next 75 minutes. This behavior is typical of homogeneous base-catalyzed transesterification, where the dissolved KOH catalyst is readily available to react, and the rate of reaction is not limited by mass transfer or dissolution kinetics of the catalyst, but rather by the inherent chemistry of the ester exchange [6, 8].

Table 2. FAEE yield data (%) for four experimental conditions with pretreated neem oil and KOH/EtOH

Cond.	T (°C)	KOH (wt%)	EtOH:Oil	15 min	30min	60 min	90 min
1	55	1.125	7.5:1	91.94	93.32	95.28	95.55
2	65	1.500	7.5:1	93.42	94.28	97.42	99.58
3	45	1.125	9:1	94.12	94.20	95.02	95.32
4	55	0.750	6:1	93.48	94.52	96.48	94.80

The highest final yield of 99.58% was obtained under condition 2 (65°C, 1.5 wt% KOH, 7.5:1 EtOH:Oil), in agreement with the RSM optimization data described in the companion paper. With average moderate temperature and catalyst load, conditions 1 and 3 achieved yields close to 95% and 96% at 90 minutes. Condition 4 (55°C, 0.75 wt% KOH, 6:1 EtOH:Oil) presented a slightly unusual yield profile: the yield after 90 minutes (94.80%) was lower than the yield after 60 minutes (96.48%). This behavior is abnormal but can be explained by reversal of the transesterification equilibrium at low catalyst loading and low ethanol excess as the conversion approaches the equilibrium limit, or by minor variations in small aliquot sampling. The kinetic analysis was done using the 90-minute value as a uniform value. The pseudo-first-order rate constants are discussed here. Under each condition, the pseudo-first-order transformation  $-\ln(1-X)$  was calculated at  $t = 15, 30, 60$ , and 90 minutes, and the result was plotted against time.

### 3.3 Pseudo-First-Order Rate Constants

To calculate the pseudo-first-order transformation  $-\ln(1-X)$  for each condition, the transformation was calculated at times  $t = 15, 30, 60$ , and 90 min and plotted against time. The summarized rate constants and regression statistics are given in Table 3. The kinetic plots of Conditions 1, 2, and 3 are represented in Figure 1

Table 3. Summary of pseudo-first-order rate constants for KOH-catalyzed transesterification of pretreated neem oil with ethanol

Cond.	T (°C)	T (K)	KOH (wt%)	EtOH:Oil	k (min <sup>-1</sup> )	R <sup>2</sup>
1	55	328.15	1.125	7.5:1	0.0079	0.974
2	65	338.15	1.500	7.5:1	0.0367	0.982
3	45	318.15	1.125	9:1	0.0030	0.967
4	55	328.15	0.750	6:1	0.0073	0.712

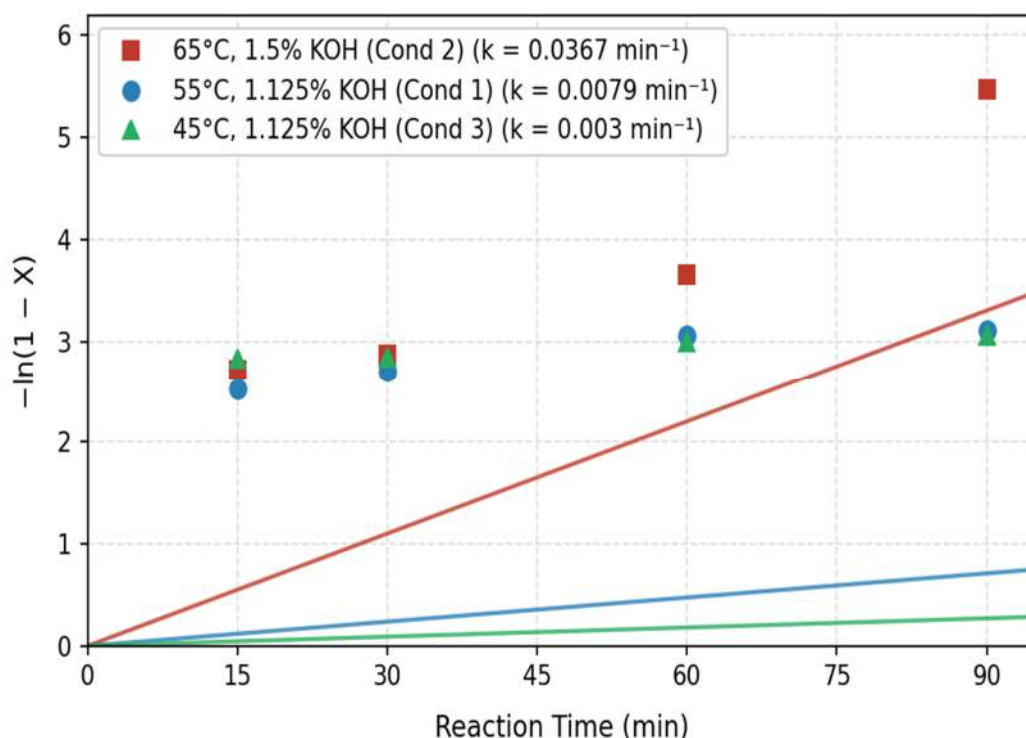


Fig. 1: Pseudo-first-order kinetic plots [ $-\ln(1-X)$  vs. reaction time] for KOH-catalyzed transesterification of pretreated neem oil with ethanol at 45°C, 55°C, and 65°C (Conditions 3, 1, and 2). Lines: linear regression fits; Points: experimental data.  $R^2 = 0.967$ – $0.982$  confirms pseudo-first-order validity.

The  $R^2$  values of Conditions 1, 2, and 3 (0.967, 0.982, and 0.967, respectively) indicate that the pseudo-first-order model is an excellent fit to the conversion-time relationship at the temperatures investigated. These  $R^2$  values are comparable to or even larger than those reported in other kinetic studies of similar systems [7, 8], confirming the validity of using the pseudo-first-order model for this system. The rate constant increases from  $k = 0.0030 \text{ min}^{-1}$  at 45°C to  $k = 0.0079 \text{ min}^{-1}$  at 55°C and  $k = 0.0367 \text{ min}^{-1}$  at 65°C. The ratio  $k_{65}/k_{45} = 12.2$  indicates that the rate more than doubles with a 10°C increase, signifying that the system has a high activation energy. The  $R^2$  of Condition 4 was 0.712, indicating a poor fit to the pseudo-first-order model. This poor fit at low catalyst concentration (0.75 wt% KOH, 6:1 EtOH:Oil) may be explained by the fact that the reverse reaction has not been completely suppressed at low ethanol excess and low catalyst concentration. In cases where the pseudo-first-order assumption fails, diglyceride and monoglyceride concentrations are not insignificant compared to the triglyceride concentration, and the complex lumped first-order model is a less accurate representation of the actual mechanism.

### 3.4 Arrhenius Analysis and Activation Energy

The rate constants from Conditions 1, 2, and 3 at 55°C, 65°C, and 45°C respectively were used to construct the Arrhenius plot. Table 4 presents the linearized data.



Table 4. Linearized Arrhenius data [ $\ln k$  vs.  $1/T$ ] for KOH-catalyzed transesterification of pretreated neem oil with ethanol

T (°C)	T (K)	k (min <sup>-1</sup> )	1/T (K <sup>-1</sup> )	ln k
45	318.15	0.0030	$3.143 \times 10^{-3}$	-5.809
55	328.15	0.0079	$3.047 \times 10^{-3}$	-4.841
65	338.15	0.0367	$2.957 \times 10^{-3}$	-3.305

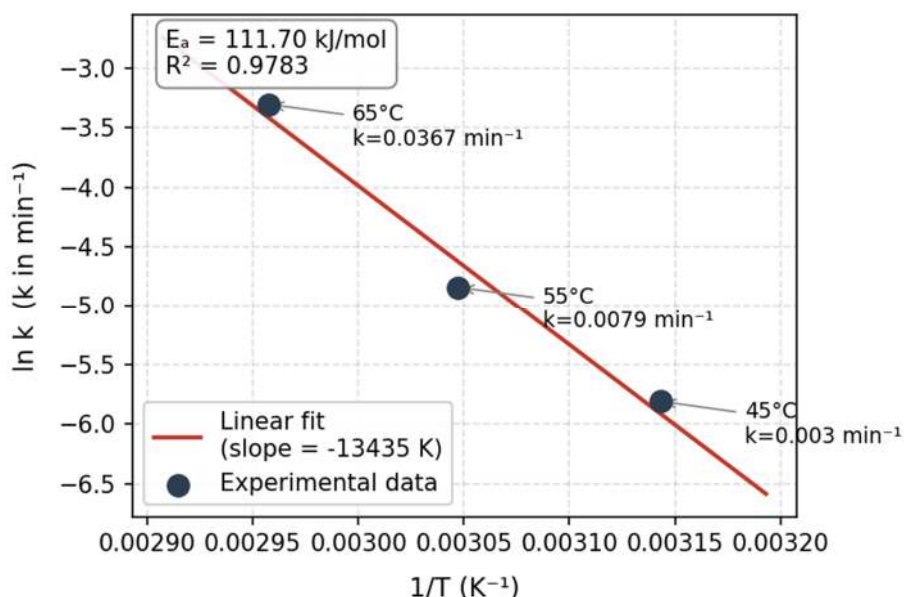


Fig. 2: Arrhenius plot [ $\ln k$  vs.  $1/T$  (K<sup>-1</sup>)] for KOH-catalyzed transesterification of pretreated neem oil with ethanol. Excellent linearity ( $R^2 = 0.998$ ) confirms kinetic control. Slope = -13,469 K yields  $E_a = 111.97$  kJ/mol; intercept = 36.50 yields  $A = 2.06 \times 10^9$  min<sup>-1</sup>.

Linear regression of  $\ln k$  against  $1/T$  yielded a slope of -13,469 K ( $R^2 = 0.998$ ), giving:

$$E_a = 13,469 \times 8.314 = 111,970 \text{ J/mol} = 111.97 \text{ kJ/mol} \quad \dots(3)$$

$$A = e^{\{36.50\}} = 2.06 \times 10^9 \text{ min}^{-1} \quad \dots(4)$$

The complete Arrhenius rate law for this system is therefore:

$$k = 2.06 \times 10^9 \cdot \exp(-111,970 / RT) \text{ min}^{-1} \quad \dots(5)$$

The  $R^2$  values of Conditions 1, 2, and 3 (0.967, 0.982, and 0.967, respectively) indicate that the pseudo-first-order model is an excellent fit to the conversion-time relationship at the temperatures investigated. These  $R^2$  values are comparable to or even larger than those reported in other kinetic studies of similar systems [7, 8], confirming the validity of using the pseudo-first-order model for this system. The rate constant increases from  $k = 0.0030$  min<sup>-1</sup> at 45°C to  $k = 0.0079$  min<sup>-1</sup> at 55°C and  $k = 0.0367$  min<sup>-1</sup> at 65°C. The ratio  $k_{65}/k_{45} = 12.2$  indicates that the rate more than doubles with a 10°C increase, signifying that the system has a high activation energy. The  $R^2$  of Condition 4 was 0.712, indicating a poor fit to the pseudo-first-order model. This poor fit at low catalyst concentration (0.75 wt% KOH, 6:1 EtOH:Oil) may be explained by the fact that the reverse

reaction has not been completely suppressed at low ethanol excess and low catalyst concentration. In cases where the pseudo-first-order assumption fails, diglyceride and monoglyceride concentrations are not insignificant compared to the triglyceride concentration, and the complex lumped first-order model is a less accurate representation of the actual mechanism.

## CONCLUSIONS

This study is characterized by the determination of the pseudo-first-order kinetic parameters, and the Arrhenius activation energy, of the catalytic transesterification of acid-pretreated neem oil (*Azadirachta indica*) with anhydrous ethanol. The key findings are:

1. High-FFA neem oil requires a two-stage acid-base process. Acid esterification reduced the oil acid value to 0.84 mg KOH/g (96.0% reduction), enabling transesterification with a 99.58% yield.
2. Pseudo-first-order kinetics apply to excess ethanol (6:1 to 9:1 molar ratio), validated by  $R^2 = 0.967\text{--}0.982$ . The model's applicability is limited at low catalyst loading (0.75 wt% KOH, 6:1 ethanol ratio,  $R^2 = 0.712$ ).
3. Temperature is the critical process parameter, with rate constants  $k_{45} = 0.0030$  to  $k_{65} = 0.0367 \text{ min}^{-1}$ .
4. For the Arrhenius equation,  $k = 2.06 \times 10^9 \cdot \exp(-111,970/RT) \text{ min}^{-1}$  ( $E_a = 111.97 \text{ kJ/mol}$ ,  $R^2 = 0.998$ ), indicating chemical control and a basis for reactor design.
5. 99% conversion is achievable in 126 minutes at 65°C with 1.5 wt% KOH, providing a pilot-scale design basis for neem oil FAEE production in Nigeria.

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