



KINETICS AND THERMODYNAMICS STUDY OF BIODIESEL PRODUCTION FROM NEEM OIL USING ALUMINA AS A CATALYST

¹Suleiman, K. K., ²Isah, M., ¹Abdulfatai, A. S., ³Danyaro, Z.

¹Department of Chemistry, Faculty of Physical Science, Federal University Dutsin – Ma, Katsina State, Nigeria.

²Department of Chemistry, Faculty of Applied Science, Sule Lamido University, Kafin Hausa, Jigawa State, Nigeria.

³Department of Chemistry, College of Remedial and Advanced Studies, Babura, Jigawa State.

*Corresponding authors' email: kskabo@fudutsinma.edu.ng

ABSTRACT

Biodiesel was produced from the transesterification of neem oil with methanol using alumina catalyst. The transesterification was carried out under the optimal condition of methanol to oil molar ratio of 6:1. The reaction for the production of biodiesel using alumina catalyst conforms to a pseudo – first order rate law with reaction rate constants of 0.0028, 0.0044, and 0.009 min⁻¹ at 308, 318, and 328 K, respectively. Activation energy for the reaction was 48.45 kJmol⁻¹ and pre – exponential factor (A) of 13.003. The thermodynamics parameters for the reaction was calculated as 46.02 kJmol⁻¹ and -0.1449 kJmol⁻¹ for enthalpy and entropy respectively. The Gibbs free energy was calculated to be 90.68, 92.13, and 93.58 kJ mol⁻¹ at 308, 318, and 328 K, respectively. In this research, kinetics and thermodynamics study of biodiesel production from neem oil was carried out. This shows that kinetics study was carried out to determine the rate constants of the reaction, the reaction rate constants also increases concerning the temperature of the reaction. The thermodynamics study was carried out, the positive value of ΔH , negative ΔS and positive ΔG indicate this reaction is endothermic and non – spontaneous.

Keywords: Activation energy, Biodiesel, Catalyst, Kinetics, Thermodynamics

INTRODUCTION

Ongoing trends focus on the search for other sources of fuel, which biodiesel can be used for replacing fossil fuels due to the increase in the price and environmental concerns about pollution (Azam *et al.*, 2005). Fossil fuels are non – renewable, depleting, add to global carbon emission (global warming), and petrochemicals also pollute environment. The major challenge now a days is production of economical and less toxic viable biodiesel (Gumahin *et al.*, 2019). Quick depletion of global petroleum reserves and increasing ecological concerns has created a great demand for environmentally benign renewable energy resources (Ashish *et al.*, 2010). Biodiesel is a substitute to fossil fuels due to its bio – renewable nature, biodegradability, less toxic emission and good transport and storage properties. Biodiesel can be referred to as a fuel comprises of mono – alkyl esters of long chain fatty acids obtained from renewable sources, such as vegetable oils and animal fats (Math *et al.*, 2010). Transesterification involves the conversion of vegetable oils or animal fats into biodiesel using catalysts. The most commonly used raw materials in production of biodiesel are vegetable oils, and it's composed of triglycerides and small amount of diglycerides (Haernandez *et al.*, 2009).

Biodiesel is produced via transesterification reaction of vegetable oils or animal fats with an alcohol (VanGerpan *et al.*, 2004; Fukuda *et al.*, 2001). This chemical reaction converts vegetable oils or animal fats into biodiesel. Transesterification reaction is carry out using catalysts to accelerate the rate of the reaction (Vicente *et al.*, 2004). Due to the increasing in energy demand and the pollution problems caused by the use of fossil fuels, the used of vegetable oils and their derivatives as alternative for biodiesel is the best solution in current situation (Lawan and Serder, 2019). Production of biodiesel is a recent area for researchers and has a positive impacts (Ahmia *et al.*, 2014).

Alumina is an important industrial mineral, which can be used as catalyst, abrasive and as adsorbent (Salahudeen *et al.*, 2015). Alumina exists as stable form (α -alumina) and in a

variety of meta-stable forms including γ -, η -, δ -, θ -, κ - and χ - alumina (Wang *et al.*, 2009). Alumina occurs naturally pure inform of the mineral corundum, although the most important natural ore for alumina is bauxite. Bauxites have been widely used in industry to produce alumina via the Bayer process (Hosseini *et al.*, 2011).

Some of the works that gave attention to the study of biodiesel production include Sivamani *et al.* (2022) obtained 85.6 % yield for the use of sunflower oil; ChukwuemekeUlakpa *et al.* (2019) obtained 92.0 % yield for the use of neem seed oil (NSO); Abdulhakim *et al.* (2022) obtained 96.35 % yield for the use of candlenut oil; Nandi *et al.* (2019) obtained 93.24 % yield for the use of soybean oil.

Different researchers produced biodiesel using different kinds of oil. Sanjay *et al.* (2012) examined composition of biodiesel produced from *Gmelina arborea* seed oil; Krishnan and Mahendira (2012) produced biodiesel via transesterification of waste cooking palm oil; Jibril *et al.* (2012) explained that the transesterification of two oil samples “Jatropha and Neem oils”; Aransiola *et al.* (2011) produced biodiesel using crude neem oil; Kofi and Ketoka (2019) investigated the kinetics and thermodynamics of Moringa oleifera oil biodiesel; Dang *et al.* (2013) investigated the application of kaolin-based catalysts in biodiesel production via transesterification of vegetable oils in excess methanol.

Therefore, this research is aimed at kinetics and thermodynamics study of biodiesel production from Neem oil.

MATERIALS AND METHODS

Production of Biodiesel

The reactants used were methanol and neem oil, with alumina as a catalyst. The transesterification reaction is carried out in two steps. Firstly, to choose the optimum catalyst amount (between 1, 2, and 3wt %) at a constant molar ratio (methanol: oil) of 6 to 1 at a reaction temperature and time of 318 K and 90 min, respectively. 1, 2, and 3wt % (weight of catalyst / weight of oil) of catalyst was measured into a separate 250 ml

beaker, 0.6841 mol of methanol was measured. The catalyst was mixed with the methanol. The mixture was left in a water bath at a temperature of 318 K, and after that the methoxide mixture was poured into conical flask containing 0.1140 mol of neem oil. The mixture were left to react in the water bath at the reaction temperature and specified reaction time (90 minutes). When the reaction reached the specified reaction time (90 minutes), the heating and stirring were then stopped. The resulting mixture was transferred to a separating funnel to allow the glycerol to settle down. The settled glycerol was tapped out and upper layer was the biodiesel. The catalyst amount with the highest yield was determined and considered as the optimum catalyst. At the second step or kinetic study, experiment was conducted with the optimum catalyst amount obtained in step1, methanol to oil molar ratio 6:1, and reaction temperatures of 308 K, 318 K, and 328 K. The yield of methyl ester production was measured after the reaction times of 30 min, 60 min, 90 min, 120 min and 150 min (Elgharabawy, 2017; Jaber et al., 2015).

GC – MS Analysis

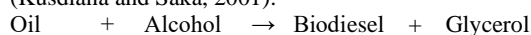
The Gas chromatography/ Mass Spectroscopy (GC/MS) was carried out using GC-MS (Shimadzu, Japan, Model/Serial Number GCMS-QP2010 PLUS), to determine the percentage of methyl esters present in the biodiesel obtained from the neem oil. These analyses were carried out at the National Research Institute for Chemical Technology, NARICT, Zaria, Kaduna State, Nigeria.

FTIR Analysis

Fourier Transform Infra – red Spectroscopy was done using an FTIR spectrophotometer, which comprised a detection device with a spectral range of 4000 – 680 cm^{-1} with a resolution of 8 cm^{-1} . FTIR spectra were analyzed and the analysis was carried out at Department of Biochemistry, Bayero University Kano.

Kinetics study

Based on the assumption that transesterification reaction of neem oil with methanol to obtained biodiesel and glycerol is a pseudo first order reaction. The assumption is based on the reason that the reaction was carried out with excess methanol (Kusdiana and Saka, 2001):



$$\text{Rate of reaction} = \frac{-d[O]}{dt} = k[O] \quad (1)$$

Integrating equation (2)

$$\int_0^t \frac{d[O]}{[O]} = -k \int_0^t dt$$

$$\ln[O] \Big|_0^t = -kt \Big|_0^t = -kt$$

$$[O]_t - [O]_0 = e^{-kt}$$

$$\ln \frac{[O]_t}{[O]_0} = -kt$$

$$\frac{[O]_t}{[O]_0} = e^{-kt} \dots \quad (2)$$

$[O]_0$ = Oil concentration at $t = 0$ and at different t is $[O]_t$ where $[O]_0 > [O]_t$

Generally; oil conversion to biodiesel is equal to biodiesel yield

$$\text{So, we have; Yield} = 1 - \frac{[O]_t}{[O]_0} \quad (3)$$

$$\text{Therefore; } \frac{[O]_t}{[O]_0} = 1 - \text{Yield} \quad (4)$$

Substitute $\frac{[O]_t}{[O]_0}$ with $1 - \text{Yield}$ in equation (2)

$$1 - \text{Yield} = e^{-kt} \quad (5)$$

Taking natural logarithm of equation (5)

$$\ln(1 - \text{Yield}) = -kt \quad (6)$$

Plot a graph of $\ln(1 - \text{Yield})$ versus t

Thermodynamics study

Standard Gibbs free energy, enthalpy, and entropy are the thermodynamics parameters. The thermodynamics parameters were estimated to determine feasibility and endothermic nature of the transesterification reaction process. There are two equations that describe the dependence of the reaction rate constant with the temperature (Arrhenius and Eyring equations), both derived from statistical thermodynamics in kinetic theory of gas.

$$k = k^\ddagger \frac{k_B T}{h} e^{-\frac{\Delta G}{RT}} \quad (7)$$

Where: k = rate constant

k^\ddagger = transmission coefficient usually take value of 1

k_B = Boltzmann constant = $1.38 \times 10^{-23} \text{ JK}^{-1}$

h = Planck constant = $6.63 \times 10^{-34} \text{ Js}$

$$\text{Recall that: } \Delta G = \Delta H - T\Delta S \quad (8)$$

Where: ΔG is the Gibbs free change, ΔH is the enthalpy change, and ΔS is the entropy change.

Putting equation (8) into equation (7)

$$k = k^\ddagger \frac{k_B T}{h} e^{-\frac{T\Delta S - \Delta H}{RT}} \quad (9)$$

Taking natural logarithm of equation (9)

$$\ln \frac{k}{T} = \frac{\Delta S}{R} - \frac{\Delta H}{RT} + \ln k^\ddagger + \ln \frac{k_B}{h} \quad (10)$$

$$\ln \frac{k}{T} = -\frac{\Delta H}{R} \left(\frac{1}{T} \right) + \left[\ln k^\ddagger + \ln \frac{k_B}{h} + \frac{\Delta S}{R} \right] \quad (11)$$

Plot a linear graph of $\ln \frac{k}{T}$ versus $\frac{1}{T}$

RESULTS AND DISCUSSION

Table 1: Yield of the biodiesel produced to determine the optimum catalyst.

Catalyst amount (wt %)	Reaction time (minutes)	Yield (%)
1	90	60
2	90	61
3	90	52

Table 1 showed that catalyst amount (2 wt %) with the highest yield (61 %) was considered as the optimum catalyst.

Fourier Transform Infra – red Spectroscopy of biodiesel produced

Fourier Transform Infra – red (FTIR) Spectroscopic analysis was performed for monitoring the progress of transesterification reactions of neem oil. The FTIR result was shown in Figure 1.

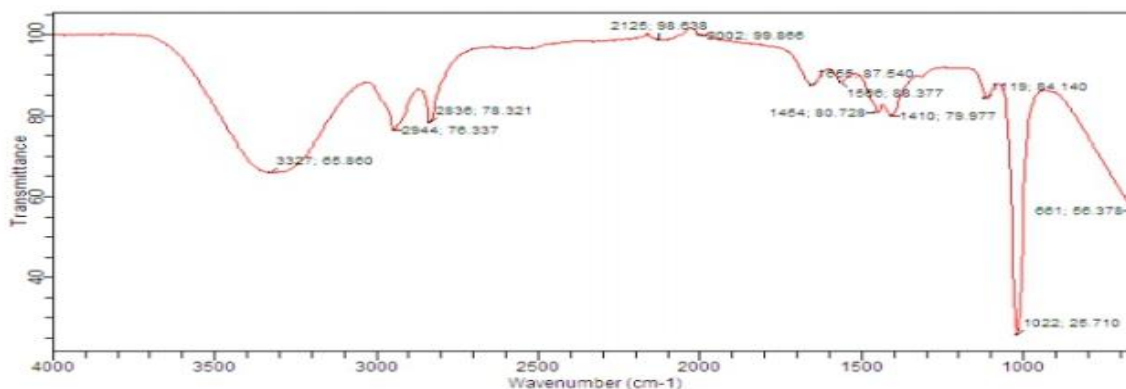


Figure 1: FTIR spectrum for the neem oil biodiesel.

The FTIR spectra of neem oil biodiesel was shown in figure 1. The peak observed at 1655 cm⁻¹ is assigned to C=O, which typically belongs to esters. The peaks located at 2944 and 2836 cm⁻¹ confirmed the presence of =C-H asymmetric stretching vibrations and -CH₂ symmetric stretching

vibrations, respectively, in neem oil biodiesel. The peaks located at 1454 and 1410 cm⁻¹ indicate bending vibrations of -CH₂. The peak at 1022 cm⁻¹ indicates the C - O - C anti - symmetric stretching vibration.

Gas Chromatography / Mass Spectroscopy of the produced biodiesel

Table 2: Composition of neem oil biodiesel Produced

Peak Number	Compound (Methyl Ester)	Area (%)	Retention Time (RT)
1	Dodecanoic acid, methyl ester	1.98	9.427
3	Hexadecanoic acid, methyl ester	1.87	13.968
4	Octadecadienoic acid, methyl ester	3.16	15.565
5	Octadecenoic acid, methyl ester	6.43	15.644
6	Heptadecanoic acid, methyl ester	2.40	15.933
10	Hexadecadienoic acid, methyl ester.	2.44	17.468

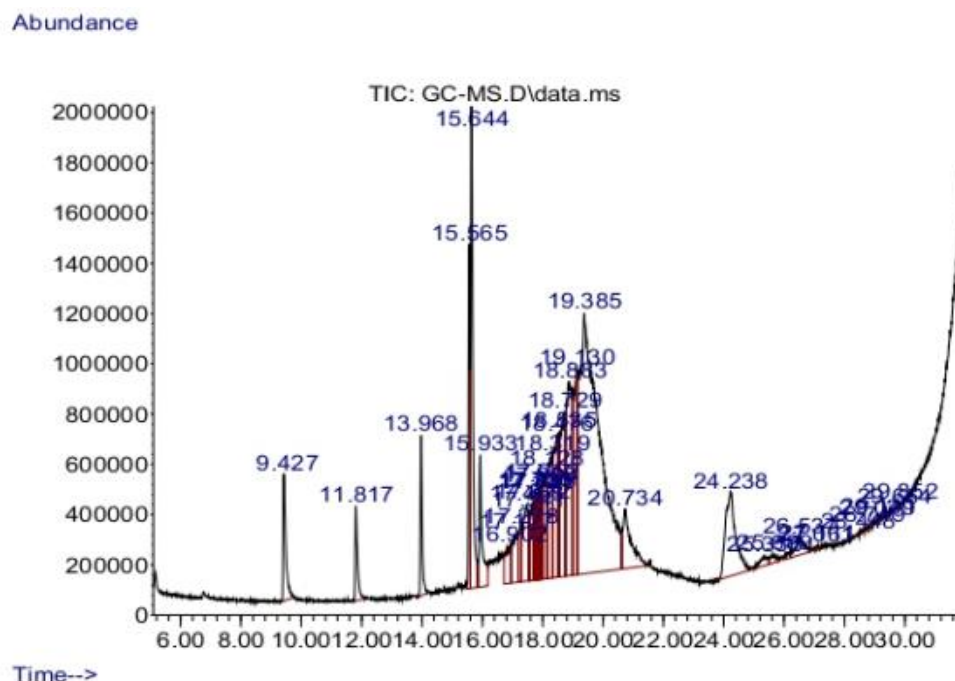


Figure 2: GCMS Result of Neem Biodiesel

The percentages of methyl esters present in the neem oil biodiesel products as determined using Gas Chromatography / Mass Spectroscopy are presented in Figure 2. The methyl esters' composition in biodiesel produced from neem oil are shown in Table 2. The methyl esters are depicted by the

following peaks: Peak 1- Dodecanoic acid methyl ester; Peak 3- Hexadecanoic acid methyl ester; Peak 4- Octadecadienoic acid methyl ester; Peak 5- Octadecenoic acid methyl ester; Peak 6- Heptadecanoic acid methyl ester Peak 10- Hexadecadienoic acid methyl ester.

Kinetics study

The data for the kinetics study are presented in Table 3.

Table 3: Data for the kinetics study, the rate constants and R² values.

Temperature (K)	Time (min)	Yield (%)	-ln(1-yield)	k	R ²
308	30	60	0.91	0.0028	0.7169
	60	48	0.65		
	90	61	0.94		
	120	64	1.02		
	150	68	1.14		
318	30	70	1.20	0.0044	0.6213
	60	59	0.89		
	90	73	1.31		
	120	75	1.40		
	150	79	1.60		
328	30	82	1.71	0.0091	0.5256
	60	71	1.24		
	90	85	1.90		
	120	88	2.20		
	150	93	2.60		

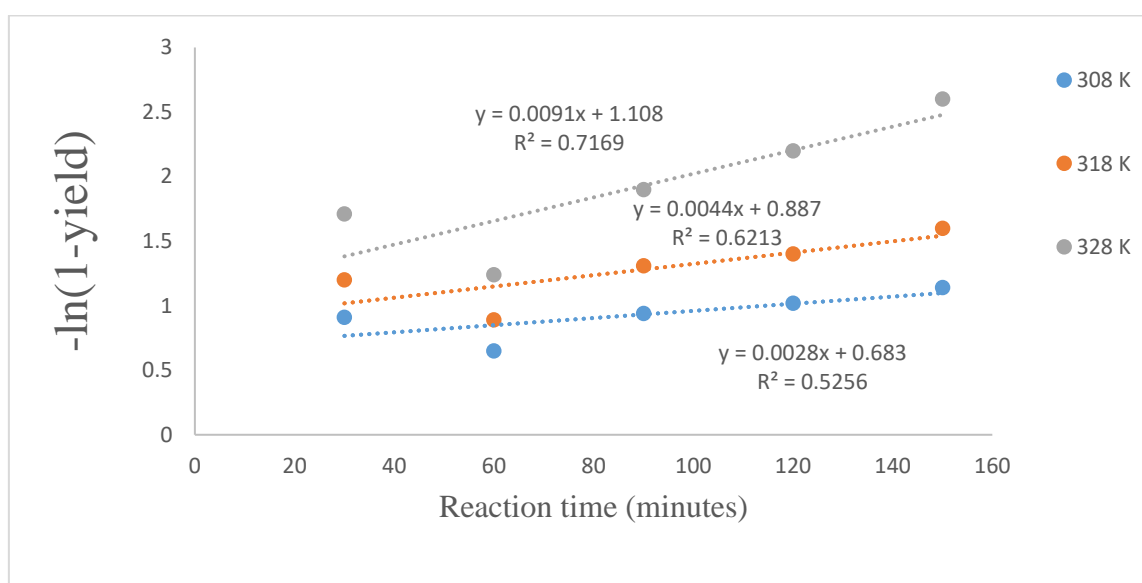


Figure 3: Plot of $-\ln(1 - \text{Yield})$ against reaction time at 308, 318, and 328 K.

Figure 3 shows the relationships of $-\ln(1-\text{yield})$ to reaction time at various temperature (308, 318, and 328 K). From the slope of the plot of $-\ln(1-\text{yield})$ versus time in Figure 3, the rate constants (k) at different temperatures are obtained. The linear nature of these plots signified that the reaction followed pseudo first – order rate law. From figure 3, the slope is the rate constant. It can be seen that at 308 K, the rate constant

value was 0.0028 (min^{-1}) and correlation coefficient was 0.5256, at 318 K, the rate constant value was 0.0044 (min^{-1}) and correlation coefficient was 0.6213, and at 328 K, the rate constant value was 0.0091 (min^{-1}) and R^2 was 0.7169. There is an increase in k value at higher temperature. The obtained results revealed that increasing the reaction temperature improved the rate constant of the reaction.

Activation energy**Table 4: Data for determination of Arrhenius parameter.**

Rate constant (min^{-1})	Temperature (K)	$\ln k$	$1/T$ (K^{-1})
0.0028	308	-5.8781	0.00325
0.0044	318	-5.4262	0.00314
0.0091	328	-4.6995	0.00305

Use the data given in Table 4 to plot a graph of natural logarithm of rate constant against reciprocal temperature to obtain the activation energy of the reaction. The activation

energy is the minimum amount of energy required for the reaction to take place.

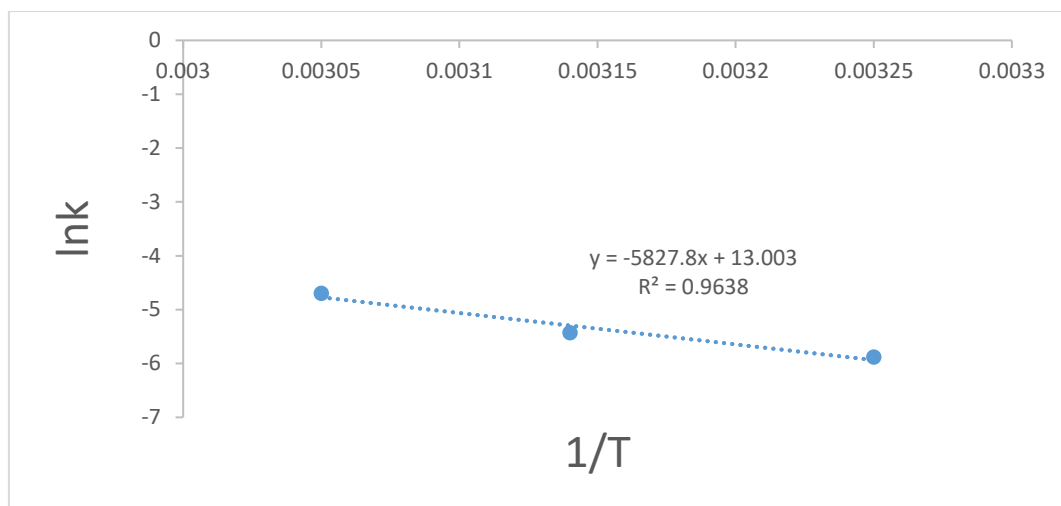


Figure 4: Arrhenius plot of natural logarithm of reaction rate constant with reciprocal temperature.

From the Arrhenius equation

$$k = Ae^{-\frac{E_a}{RT}}$$

Where; A = Frequency factor

E_a = Activation energy

R = Gas constant, and T = Temperature

The figure 4 shows activation energy values which can be expressed as follow:

$$\frac{E_a}{R} = 5827.8$$

$$\text{But: } R = 8.314 \text{ Jmol}^{-1}\text{K}^{-1} = 0.008314 \text{ kJmol}^{-1}\text{K}^{-1}$$

$$E_a = 5827.8 \times R$$

$$E_a = 0.008314 \times 5827.8$$

$$E_a = 48.45 \text{ kJmol}^{-1}$$

Therefore, activation energy values calculated from Arrhenius equation is 48.45 kJmol⁻¹.

The oil adsorption reaction has activation energy, 48.45 kJmol⁻¹. The activation energy (E_a) of the rate determining step and the pre – exponential factor, A (13.003).

Figure 4 shows the relationships between the absolute temperature and pseudo first – order rate constant for calculating the activation energy. The calculated activation energy for biodiesel production from neem oil was 48.45 kJmol⁻¹. The positive activation energy reveals that the transesterification process is a temperature – dependent process, indicating the production of biodiesel increased with increasing temperature. The activation energy is inversely proportional to the rate constant, the high value of the activation energy (48.45 kJmol⁻¹) implies a slow rate of biodiesel formation.

Thermodynamics study

Data for determination of thermodynamics parameters are presented in Table 5.

Table 5: Data for determination of thermodynamics parameters using alumina catalyst.

Rate constant (min ⁻¹)	Temperature (K)	k/T × 10 ⁻⁵	ln (k/T)	1/T (K ⁻¹)
0.0028	308	0.91	-11.61	0.00325
0.0044	318	1.38	-11.19	0.00314
0.0091	328	2.77	-10.49	0.00305

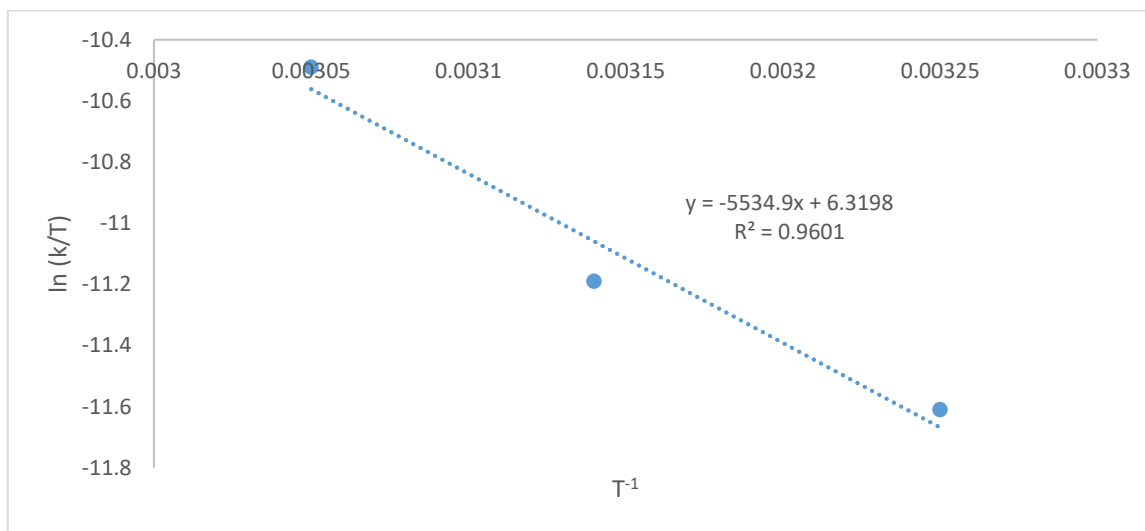


Figure 5: Plot of ln (k/T) versus T⁻¹

From Figure 5, the plot of $\ln(k/T)$ versus T^{-1} , ΔH and ΔS can be calculated

$$\ln \frac{k}{T} = -\frac{\Delta H}{R} \left(\frac{1}{T}\right) + \left[\ln k^\ddagger + \ln \frac{k_B}{h} + \frac{\Delta S}{R}\right]$$

Relating equation above with Figure 5, we have:

$$-\frac{\Delta H}{R} = -5534.9$$

$$\Delta H = 5534.9 \times 0.008314$$

$$\Delta H = 46.02 \text{ kJmol}^{-1}$$

$$\ln k^\ddagger + \ln \frac{k_B}{h} + \frac{\Delta S}{R} = 6.3198$$

Substituting the values, we have:

$$\ln(1) + \ln(1.38 \times 10^{-23} / 6.63 \times 10^{-34}) + \frac{\Delta S}{R} = 6.3198$$

$$0 + 23.76 + \frac{\Delta S}{R} = 6.3198$$

$$\frac{\Delta S}{R} = 6.3198 - 23.76$$

$$\frac{\Delta S}{R} = -17.4402$$

$$\Delta S = -17.4402 \times 0.008314$$

$$\Delta S = -0.14499 \text{ kJmol}^{-1}$$

Using the relation: $\Delta G = \Delta H - T\Delta S$

At 308 K

$$\Delta G = 46.02 - 308(-0.14499)$$

$$\Delta G = 46.02 + 44.66$$

$$\Delta G = 90.68 \text{ kJmol}^{-1}$$

At 318

$$\Delta G = 46.02 + 46.11$$

$$\Delta G = 92.13 \text{ kJmol}^{-1}$$

At 328

$$\Delta G = 46.02 + 47.56$$

$$\Delta G = 93.58 \text{ kJmol}^{-1}$$

The calculated ΔH and ΔS values were 46.02 kJmol^{-1} and $-0.14499 \text{ kJmol}^{-1}$, respectively. The positive ΔH value indicated that biodiesel production from neem oil was endothermic. While, the negative value for ΔS proposes an associative mechanism. The calculated positive ΔG values (90.68 , 92.13 , and 93.58 kJmol^{-1}) revealed that biodiesel production from neem oil was non-spontaneous.

Table 6: Transesterification kinetics and thermodynamics result

Catalyst	Temp. (K)	Rate constant (kJ/mol)	Gibbs free energy, ΔG (kJ/mol)	Enthalpy, ΔH (kJ/mol)	Entropy, ΔS (kJ/mol)	Pre-exponential factor	Activation energy (kJ/mol)
	308	0.0028	90.68				
Alumina	318	0.0044	92.13	46.02	-0.1449	13.003	48.45
	328	0.0091	93.58				

CONCLUSION

Biodiesel was produced from the transesterification of neem oil with methanol using alumina as a catalyst. Kinetics and thermodynamics parameters of biodiesel produced were calculated. The positive activation energy (46.02 kJmol^{-1}) reveals that the transesterification process is a temperature-dependent process, indicating the production of biodiesel increased with increasing temperature. The rate constants obtained (0.0028 , 0.0044 , and 0.0091 min^{-1}) increased with the increase in temperature used (308, 318, and 328 K). The positive ΔG values (90.68 , 92.13 , and 93.58 kJmol^{-1}) revealed that biodiesel production from neem oil was non-spontaneous. The IR bands in biodiesel IR spectra and the percentages of methyl esters present in the neem oil biodiesel products as determined using Gas Chromatography / Mass Spectroscopy, clearly demonstrated the transesterification of neem oil into biodiesel.

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