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OPTIMIZATION, KINETICS AND THERMODYNAMICS STUDY OF TRANSESTIRIFICATION OF BAOBAB OIL USING HOMOGENEOUS CATALYSTS AS BIODIESEL

Hassan Saidu Baba, Shamsuddeen Mukhtar Ladan, Usman ishaq Shehu, Bishir Usman

Department of Pure and Industrial Chemistry, Faculty of Physical Science, College of Natural and Pharmaceutical Science, Bayero University Kano

Corresponding Author: hassansaidbaba2019@gmail.com, busman.chm@buk.edu.ng

ABSTRACT

Response surface methodology (RSM), based on central composite design (CCD) was used to determine the optimum condition for the production of biodiesel using Baobab seed oil. the process variable were conducted and evaluated at two level factorial (2^4 experimental design) .the parameters are methanol to oil molar ratio(3:1 – 12:1), the catalyst concentration (0.5%w/w -25w/w), reaction time (30min – 120min) and reaction temperature(37 -87.5 ^{0}C). The physicochemical analysis result indicated that baobab oil has acid value 2.14mg NaoH/g, free fatty acid 1.07mg NaoH/g, density 0.916g/cm^3, specific gravity 0.91g/cm^3 saponification 209 KOH/g of oil. The optimum biodiesel yield obtained was 93.3% with, methanol to oil ratio 9:1, catalyst dosage 0.5, time 120 And temperature 75 ^{0}c used. The fatty acid methyl ester composition of the baobab biodiesel was determined using Gas Chromatography couple with Mass Spectrophotometer (GC-MS). The FAME profile indicate that the dominants compound in biodiesel are ester compound. The kinetics and thermodynamics of the process was studied. The activation energy, free exponential factor, enthalpy and entropy for transestirification were found to be 44.2 kJ/mol^{-1}, 5.02*10^5 dm^3 min^{-1},42.12 kJ/mol^{-1}, -140.7 jmol^{-1} respectively. . the Gibbs free energy value was calculated as 85.74 kJ/mol, 87.56 kJ/mol, 89.32 kj/mol, 91.08 kJ/mol at 37°C ,50°C, 62.5^{o}C, and 75°C which show the reaction was non spontaneous and endergonic in nature. The results showed that homogeneous base catalysis (NaoH) of Boabab seed oil (Adansonia digitata) under optimum reaction conditions provides high yield of biodiesel.

Keywords: Biodiesel, Baobab, transesterification, homogeneous catalyst.

1.0 Introduction

During the last century, the consumption of energy has increased a lot due to the change in the life style and the significant growth of population. This increase of energy demand has been supplied by the use of fossil resources, which caused the crises of the fossil fuel depletion, the increase in its price and the serious environmental impacts as global warming, acidification, deforestation, ozone depletion, Eutrophication and photochemical smog. As fossil fuels are limited sources of energy, this increasing demand for energy has led to a search for alternative sources of energy that would be economically efficient, socially equitable, and environmentally sound. Two of the main contributors of this increase of energy demand have been the transportation and the basic industry sectors, being the largest energy consumers. The transport sector is a major consumer of petroleum fuels such as diesel, gasoline, liquefied petroleum gas and compressed natural gas (Mekonnen *et al.* 2021). Demand for transport fuels has risen significantly during the past few decades. (Zahan *et al*. 2020). The demand for transport fuel has been increasing and expectations are that this trend will stay unchanged for the coming decades. In fact, with a worldwide increasing number of vehicles and a rising demand of emerging economies, demand will probably rise even harder. Transport fuel demand is traditionally satisfied by fossil fuel demand. However, resources of these fuels are running out, prices of fossil fuels are expected to rise and the combustion of fossil fuels has detrimental effects on the climate. The expected scarcity of petroleum supplies and the negative

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environmental consequences of fossil fuels have spurred the search for renewable transportation biofuels' (Bello *et al*. 2022).

Biodiesel is a methyl or ethyl ester of fatty acid made from vegetable oil or animal fat. Biodiesel contains monoalkyl esters of long chain fatty acids derived from vegetable oil. As a fuel, biodiesel is biogradable and non-toxic and can be used in any diesel engine as a pure fuel or mixed with petrol, without many adjustments because biodiesel and diesel have similar structures. It has a favourable combustion compared to petroleum diesel and has low CO emission and sulfur content. CO_2 produced can be recycled by photosynthesis, fact that favours the decreasing of greenhouse effect.

Due to its clean emissions profile, ease of use, and many other benefits, biodiesel is quickly becoming one of the fastest growing alternative fuels in the world. With minimal subsidy, it will be cost competitive with petroleum diesel, and millions of users have found and enjoyed the benefits of the fuel. The future of biodiesel lies in the world's ability to produce renewable feedstock such as vegetable oils and fats to keep its cost competitive with petroleum, without supplanting land necessary for food production or destroying natural ecosystems in the process. Creating biodiesel in a sustainable manner will allow this clean, renewable, and cost effective fuel to help ease the world through increasing shortages of petroleum, while providing economic and environmental benefits well into the 21st century (biodiesel.com).

Biodiesel can be prepared from a variety of sources including vegetable oils such as oilseeds (rapeseed, soya bean, canola oil, sunflower, Baobab oil), animal fats, waste oils and greases (Patil *et al*., 2023) and most recently potentially from algae which have much higher yields and therefore poses less of a threat to food and water security (El-Dalatony *et al*. 2024). There are five primary ways for making biodiesel: direct use and blending of vegetable oil (Adeyemi *et al*., 2022),use of micro emulsions with short chain alcohols (Rahman *et al*, 2023), (iii) thermal cracking (pyrolysis) of vegetable oils (iv) transesterification of triglycerides catalysed by bases or esterification of fatty acids with alcohols using acid catalysts(H2SO4) or solid acid catalysts (Kim *et al*. 2004; Bournay *et al*., 2005; Ondrey, 2004) and use of enzymes (Hsu *et al*., 2004; Saka and Kusdiana, 2001) (v) catalyst free processes

Different types of catalysts such as base, acid or lipase are used in transesterification for biodiesel synthesis but the base-catalyzed reaction is the most common in the industry due to easier, faster and cheaper processing (Hariram *et al*, 2025). The conventional catalysts used for transesterification are homogeneous and heterogeneous catalysts. The most preferred catalyst used for the production of biodiesel is the homogeneous catalyst, as they are simple to use and require less time to achieve a complete reaction. Both acidic and basic catalysts come under this category. Homogeneous catalysts are usually dissolved in a solvent that is in the same phase with all re actants. Furthermore, alkali homogeneous catalysts such as NaOH and KOH are more beneficial over others because the process is carried out under atmospheric pressure with short time (30− 90 min) at low temperature (40−60 °C) and reduced cost and result in high conversion, this makes them the preferred catalysts to be used in industries. In this regard, the NaOH catalyst was selected to transform the oil obtained from Baobab seed into the corresponding alkyl ester, biodiesel. Baobab (Adansonia digitata) is a promising bio resource distributed across African. Baobab is the most widely spread of the Adansonia species on the Africa which belongs to Bombacaceae family the sub family of the Malvaceae. Adansonia species comprises of 8 different species. One of these species is the A. digitata L., it occurs throughout the drier parts of Africa, Baobab also known as dead rat tree, Ethiopian sour gourd, Judas's bag, lemonade tree, monkey bread tree,

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monkey tamarind, Senegal calabash, and upside down tree, there are 8 species of Adansonia genus. Adansonia digitata a curious shaped, medium sized, deciduous tree (Mekonnen *et al*. 2021).many studies have been carry out on the production of biodiesel using different seed oil but the use of Baobab seed oil gain little attention. In this research, biodiesel will be produced using Baobab seed oil and optimized different parameters such as methanol to oil molar ratio, catalyst concentration, reaction time and reaction temperature.

2.0 Material and Method

Baobab seed oil was collected from Technology Incubation center located within Kano metropolitan. The oil is 100% pure. All reagents used were analytical grade without any further purification, methanol, ethanol, diethylether, phenolphthalein powder, distilled water, sodium hydroxide pellets.

2.1 Transesterification of the Oil

30 grams of baobab oil were weighed and poured into a conical flask (250). Proper proportion of methanol to oil ratio and catalyst were added to the oil, the mixture was then placed in a water bath and heated at given temperature as stipulated in the design matrix. The four Transesterification process parameters (Methanol to oil molar ratio, catalysts, reaction time and reaction temperature) were varied to attain maximum methyl ester conversion. After the Transesterification process, the catalyst was separated from the bottom layer by centrifugation at 600rpm for 10 min. The mixture were cooled to room temperature and transferred in to separating funnel, to allow the glycerol to settle down overnight. The mixture was washed with distilled water ($10cm^3$) and n-hexene ($10cm^3$) were added to enhance the separation into an upper phase (Biodiesel) and lower phase (glycerol). The settled glycerol was discarded and the upper biodiesel was collected. The yield was calculated using the equation 6. The fatty acid methyl ester was analyzed using GC-MS to identify the FAME
(Zahan & Kano, 2020).

$$\%\text{Yield of Biodiesel} = \frac{\text{mass of biodiesel produced (g)}}{\text{mass of oil taken for reaction(g)}} \times 100\%$$

2.2 Design of Experiment

Design of experiment for the process parameters of biodiesel production using baobab seed oil was carried out by apply Response Suface Methodology with central composite design (CCD).
Design expert software (version 7.0) was used to design the experiment and optimized difference condition. The 2- level 4 factorial experimental design was employed requiring 30 experiments consisting of 12 axial points and 6 center points. The level of each was chosen based on the importance to the experiment. The following four parameters (methanol to oil molar ratio, catalyst concentration, reaction time and reaction temperature.) were varied in order to maximize the biodiesel yield. The experimental range and level of independent variables of the central composite design for the optimization of the transesterification process are shown in Table 1

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Parameters	Minimum value	Maximum value
Methanol/oil	1 :3	1:12
Catalyst w/w	0.5	2
Time (min)	30	120
Temperature (^{0}C)	37.5	87

3.0 **Results and Discussion**

Physicochemical Properties of Baobab Seed Oil. The quality of crude baobab seed oil samples was determined by evaluating the physicochemical properties. The parameters analysed were free fatty acids (FFA), Density and specific gravity. The oil extract has a yellow colour. The acid value of baobab oil was 2.14 KOH/g, which need no pretreatment before transestirification. Free fatty acid were 1.02, the density was 0.905 g/cm3 more than that reported by Alia (2016) and Hagir (2017) which were 0.881 g/cm3 and 0.768 g/cm3 respectively. The specific gravity obtained was 0.91%.

Table 2 show the actual and the predicted yield of biodiesel obtained after conducting 30 experiment.it was observed that the highest yield obtained was 93.33 with oil/alcohol 1:9, catalyst dosage 0.5%, time 120 min and temperature 75^{0}C.similarly, the lowest yield was observed at 1:3 oil/ alcohol, catalyst dosage 0.5, time 120 min and temperature of 50^{0}c.

Table 2 Response design for the experimental values and predicted values for biodiesel production from Baobab seed oil.

S/N	Methanol/oil	Catalyst w/w	Time(min)	Temperature(^{0}C)	Actual yield	Predicted yield
1	3	0.5	60	50	54	50.2
2	9	0.5	60	50	72.8	71.48
3	3	1.5	60	50	56.6	63.86
4	9	1.5	60	50	68.2	69..54
5	3	0.5	120	50	39.33	43.32
6	9	0.5	120	50	70.26	76.02
7	3	1.5	120	50	59.66	61.49
8	9	1.5	120	50	72.40	78.59
9	3	0.5	60	75	52.5	49.49
10	9	0.5	60	75	74.5	77.86
11	3	1.5	60	75	56.20	55.63
12	9	1.5	60	75	69.2	68.39
13	3	1.5	120	75	45.6	49.45
14	9	0.5	120	75	93.33	89.23
15	3	0.5	120	75	55.6	60.10
16	9	1.5	120	75	75.3	84.29
17	0	1	90	62.5	40.35	37.5
18	12	1	90	62.5	88.5	82.97
19	6	0	90	62.5	66.5	68.3

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20	6	2	90	62.5	87.2	77.02
21	6	1	120	75	62.5	65.46
22	6	1	30	63.5	85.8	74.47
23	6	1	90	37	63.33	56.89
24	6	1	90	87.5	63.8	61.87
25	6	1	90	62.5	86.8	86.66
26	6	1	90	62.5	87.2	87.5
27	6	1	90	62.5	86.66	86.66
28	6	1	90	62.5	86.66	86.66
29	6	1	90	62.5	86.66	86.66
30	6	1	90	62.5	86.66	86.66

Second order quadratic model was best fitted for the 30 experiment as indicated by the Model F –test (ANOVA). The model F- value of 10.54 implies the model is significant. Values of P value less than 0.0500 indicate model terms are significant. In this case A, AB, A2, B2, C2, D2 are significant model terms. Lack of fit F-value of 64.3 implies that the lack of fit is insignificant relative to the pure error. Insignificant lack of fit shows good agreement between experimental data and the model.

Table 3 Anova for Response Surface Quadratic Model

SOURCE	SUM OF SQUARE	df SQUARE	MEAN	F VALUE	P- VALUE
MODEL	6330.26	14	452.16	10.54	<0.0001
A(M/O)	3100.14	1	3100.14	72.30	
B(CATALYST)	113.84	1	113.84	2.65	0.1241
C(Time)	121.73	1	121.73	2.84	0.1127
D(Temperature)	37.23	1	37.23	0.87	0.3662
AB	243.28	1	243.28	5.67	0.0307
AC	130.36	1	130.36	3.04	0.1017
AD	50.16	1	50.16	1.17	0.2915
BC	20.41	1	20.41	0.48	0.5008
BD	56.5	1	56.5	1.32	0.2689
CD	46.75	1	46.75	1.09	0.3129
A2	1196.86	1	1196.86	27.91	<0.0001
B2	335.90	1	335.90	7.83	0.0135
C2	477.98	1	477.98	11	0.0045
D2	1276.04	1	1276.04	29.76	<0.0001
Residual	643.21	15	643.21		
Lack of fit	643.21	10	643.21		
Pure	0.0000	5	0.0000		
Cor Total	6973.47	29	6973.47		

Final Equation in Terms of Coded Factor
The regression equation as a function of the selected variables for FAME yield (%) is given by

Yield = +86.66+11.37*A+2.18*B+2.25*c+1.25*D-3.90*A*B+2.85*A*C+1.77*A*D+1.13*B*C-1.88*B*D+1.71*C*D-6.61A2-3.50*B2-4.17*C2-6.82*D*

equation 1

where “Y” is the response, that is the conversion to biodiesel, A is Methanol/oil, B

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is Catalyst, C is Reaction time D is Temperature respectively. The Positive sign in front of the terms indicates synergistic effect in increase FAME yield.while negative sign indicates antagonistic effect of the factor (Bello & Alamu, 2022). The model regression equation (equation 1) indicated that the positive coefficients indicated linear to increase the FAME yield.Although,the quadractic termshad negative effects that decrease the FAME yield.

3.1 Response surface plot

The interactive effects of the process variables on the percent yield of FAME were studied by plotting three-dimensional surface curves against any two independent variables while keeping other variables at their central (0) level. The 3D curves of the response (Yield of methyl ester) from the interactions between the variables are shown in the figures 1,2and 3. The process variables were found to have significant interaction effects

The response surface plot in Fig 4.1 illustrate that the biodiesel yield is sensitive to the interaction between molar ration and the catalyst dose, the result indicate that at low catalyst concentration from 0.5-1 high methyl ester yield was obtained with increased methanol to –oil molar ratio (9:1). However at high catalyst dosage (1.5wt) and low methanol to oil molar ratio 3:1 low yield was observed (45.6%).according to this result an addition beyond a certain value of excess methanol to oil ratio and catalyst amount resulted in poor mixing of the reaction hence aggregation and dilution of catalyst. Mekonen et al,. (2021) reported that a sufficient amount of alcohol reactant with the optimum catalyst amount is desirable for high ester yield hence, the catalyst is a very important model term having a very significant effect on FAME yield.

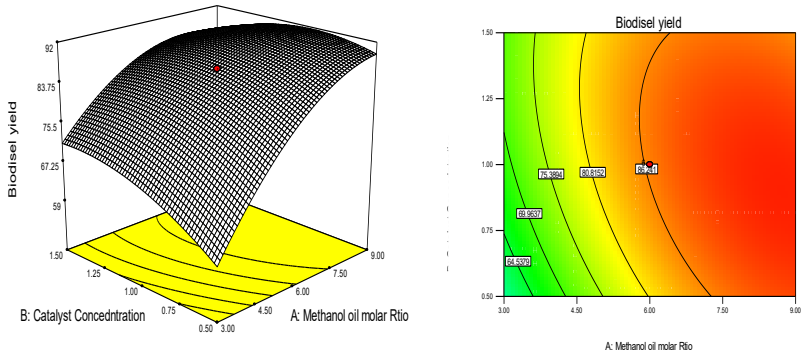

Fig 1. 3D interaction between catalyst and Mthanol to oil ratio

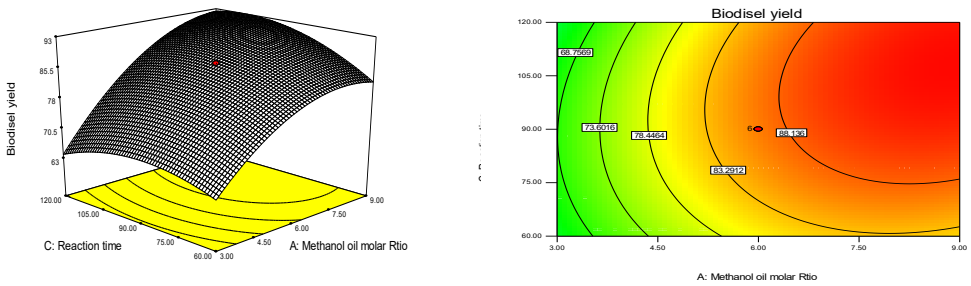

Fig 2. 3D interaction between Time and Methanol to oil ratio

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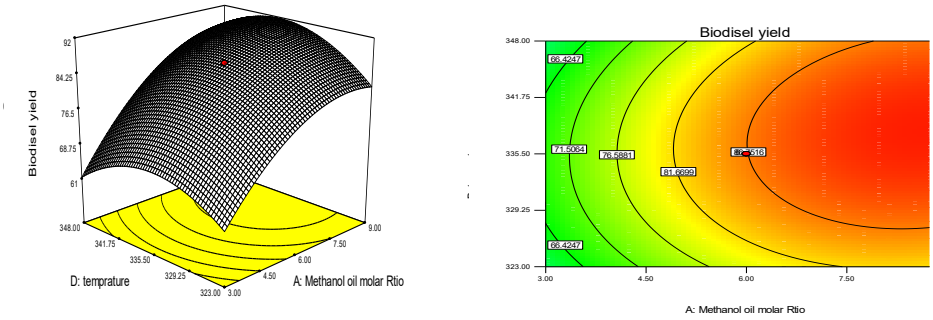

Fig 3. 3D interaction between Temperature and Methanol to oil ratio

The contour plot in Fig 4.2 revealed significant interaction effect between time and methanol to oil molar ratio. At high reaction time and methanol to oil molar ration an increase of biodiesel yield was observed. Similarly at lower reaction time and methanol to oil molar ratio biodiesel yield decrease. Excess methanol is advantageous in the trans esterification reaction for a higher ester yield as an increase in the reactant concentration promotes a forward reaction in reversible reactions. However, an extreme increase in molar ratios results in the reduction of FAME yield due to the solubility of glycerol, which makes the separation of methyl esters and glycerin difficult because of the formation of emulsion.

The interaction between temperature and methanol to oil molar ratio are present in Fig 4.3. The result showed that at high temperature and methanol to oil molar ratio, an increase in biodiesel yield was observed. However it showed a negative effect at high temperature (>75^{0}c) and methanol to oil molar ratio (>9:1).the yield decreased at high temperature, causing excessive methanol loss due to evaporation, and the reaction is reversed.

Table 4 GC-MS Showing composition of Baobab seed oil

Peak No	Retention Time	Area Ratio	Identified compound	Quality
1	13.8332	0.4648	Hexadecanoic acid, methyl ester	97
2	15.2005	-0.0294	1-Octadecyne	46
3	15.4113	0.735	9,12-Octadecadienoic acid (Z,Z)-, methyl ester	99
4	15.4971	1.321	10-Octadecenoic acid, methyl ester	99
5	15.7893	0.8466	n-Hexadecanoic acid	80
6	17.2074	54.7016	Carbonic acid, decyl 2,2,2-trichloroethyl ester	25
7	18.6724	1.9084	9-Oxabicyclo[6.1.0]nonane	90
8	18.9095	0.1488	9-Octadecenoic acid (Z)-, 2,3-dihydroxypropyl ester	46
9	20.4844	0.555	9-Octadecenoic acid (Z)-, 2,3-dihydroxypropyl ester	46
10	26.5664	38.9865	9-Octadecenoic acid (Z)-, 2,3-dihydroxypropyl ester	68
11	28.6526	0.3618	cis-Vaccenic acid	70

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The chemical composition detected by GC-MS of Baobab oil show eleven component as Shown in Table 4. The GC-MS shows the main constituents of baobab oil which are 9,12-Octadecadienoic acid (Z,Z)-, methyl ester with a RT 15.4113, 10-Octadecenoic acid, methyl ester with a RT 15.49, and Hexadecanoic acid, methyl ester with RT 13.83 respectively.

3.2 Kinetics

The kinetics for the transesterifications reaction of triglyceride and methanol in the presence of CaO catalyst to yield fatty acid methyl ester and glycerol has been reported as (pseudo) first order or second order sometimes zero order kinetics depending on the nature of the relationship.

The steps of the reaction occur below.

$$TG + CH_3OH \underset{}{\overset{catalyst}{\rightleftharpoons}} DG + FAME \quad ------ \quad 1$$

$$DG + CH_3OH \underset{}{\overset{catalyst}{\rightleftharpoons}} MG + FAME \quad ------ \quad 2$$

$$MG + CH_3OH \underset{}{\overset{catalyst}{\rightleftharpoons}} GL + FAME \quad ------ \quad 3$$

the reaction overall is follows as

$$TG + 3CH_3OH \underset{}{\overset{catalyst}{\rightleftharpoons}} GL + FAME$$

Where M, TG, DG, MG and GL are methanol, Triglyceride, diglyceride, monoglyceride, and glycerol. Where MG and DG are the intermediate product formed from the reaction.

The overall fourth order rate law can be express in equation (4) below.

$$-r = \frac{-d[TG]}{dt} = k[TG][M]^3 \quad - - - - - - - - (4)$$

Therefore the reaction to continuous more methanol is needed, so the methanol is assumed to be at constant concentration and the rate is expressed as function of TG shown in equation (5)

$$-r = k[TG] \quad - - - - - - - - - - ---------- - - - - - - (5)$$

Accordingly, the order is regarded as (pseudo) first order kinetics (Patil et al., 2023).

3.3 Reaction Rate Constant

From the slope of the plot of –ln (1 - x) vs. t in Fig.4. The linear nature of the plot supports the pseudo first order reaction rate law. (Kaur, and Ali, 2015; Bello and Yacob 2021).The data for the kinetics study in this research, the rate constant, and R^2–value presented in the Table 5 Shows that the rate constant (k) with the highest value of R^2 is obtain at 75 ^oC, thus the kinetics parameter are computed at this temperature. The K value at this temperature was 0.0191, the highest value of K obtained from this research correspond with rate constant for transesterifications reaction.

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Table 5. Raw data for the kinetics study, k and R^2 value

Temp ^{0}C	M/O	catalyst	time	Yield (%)	X	-ln (1-X)	K	R2
37	9:1	0.5	60	39.5	0.395	0.501	0.007	0.946
	9:1	0.5	80	43.5	0.435	0.571		
	9:1	0.5	100	50.5	0.505	0.703		
	9:1	0.5	120	60.2	0.602	0.921		
50 ^{0}C	9:1	0.5	60	51.2	0.512	0.717	0.0083	0.907
	9:1	0.5	80	55.3	0.553	0.755		
	9:1	0.5	100	60.8	0.608	0.936		
	9:1	0.5	120	70.2	0.702	1.211		
62.5 ^{0}C	9:1	0.5	60	62.2	0.622	0.973	0.015	0.929
	9:1	0.5	80	65.5	0.655	1.064		
	9:1	0.5	100	75.6	0.756	1.411		
	9:1	0.5	120	84.6	0.846	1.871		
75 ^{0}C	9:1	0.5	60	75.5	0.755	1.410	0.0191	0.957
	9:1	0.5	80	82.8	0.828	1.760		
	9:1	0.5	100	86.2	0.862	1.981		
	9:1	0.5	120	92.6	0.916	2.603		

The activation energy Ea. was obtained from the slope of the plot of lnK versus 1/T (k), while the pre-exponential factor (A) was found from the intercept of the pot. Values of activation energy (Ea) and pre-exponential factor (A) are computed with respective values of 44.2 kJ/mol and 5.0*105dm3mol-1 min-. The Ea is within the range of 26-84 kJ mol-1 reported for heterogeneous catalysts. The activation energy greater than 25 kJ mol-1 implies the methanolysis reaction is chemically controlled (El-Dalatony et al., 2024).

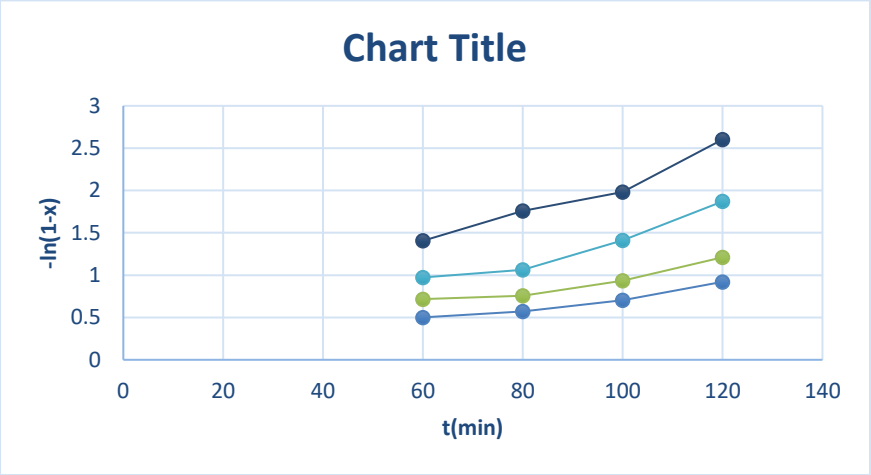

Fig 4. –ln(1-x) vs t at 37, 50, 62.8, 75^{0}C

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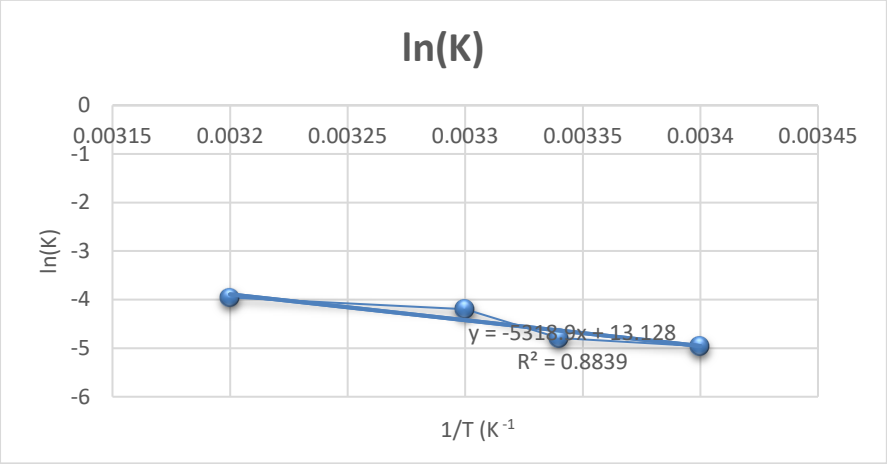

Fig 5 Arrhenius plot of lnK versus 1/T

3.4 thermodynamic parameters

The standard enthalpy (ΔH◦), entropy change (ΔS◦) and free energy (ΔG◦), was estimated to evaluate the feasibility and endothermic nature of the transesterification process. From the plot of lnK/T vs 1/T the enthalpy of the reaction was found to be 42.12 kjmol-1. The positive value of the enthalpy shows that the methanolysis reaction is endothermic, meaning the external heat energy is required for the process to take place. (Adeyemi et al., 2022).The entropy of the reaction was also found from the graph of Fig 6. using the intercept of the plot. The entropy found to be (ΔS) = - 140.7j mol-1. The negative value of the entropy signifies an associative mechanism for the methanolysis reaction .this suggest that the transition state has an ordered structure than the reactant in the ground state (Rahman & Kalam, 2023). In addition, the negative value signifies a reversible reaction. Gibbs free energy (ΔG) shows the degree and spontaneity of a chemical reaction,The ΔG value was calculated as 85.74 kJ/mol, 87.56 kJmol, 89.32 kjmol, 91.08 kJmol at 37oC ,50oC, 62.5oC, and 75oC. the positive value of ΔG proposed a non – spontaneous reaction. And also indicated that the reaction species are at lower energy level than the transition state, so the external energy is highly needed for the reaction to proceed.(Ong, and Kurniawan, 2013; Bello and Yacob, 2021).

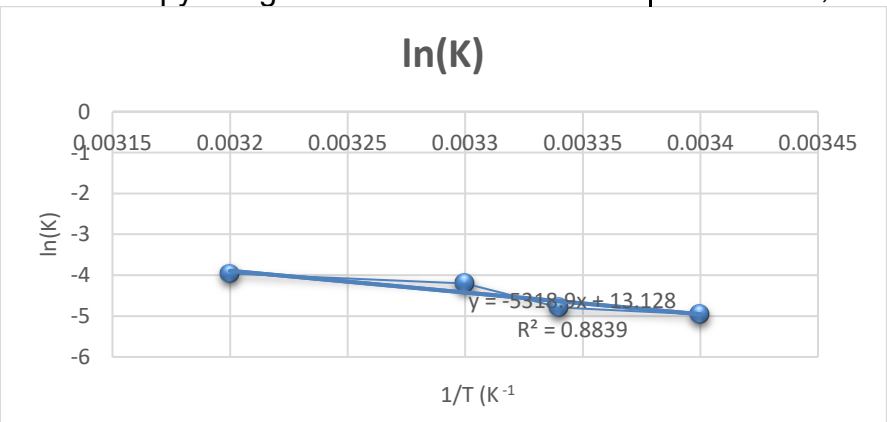

Fig 6 Eyring plot of ln(K/T) VS 1/T

4.0 CONCLUSION

Baobab oil methyl ester was successfully prepared from low free fatty acid baobab oil via trans esterification process using sodium hydroxide as catalyst. The effect of methanol to oil molar ratio, catalyst, reaction time and reaction temperature was studied. base on the central composite design it was observed

that the highest yield obtained was 93.33 when methanol to oil molar ratio of 9:1, catalyst dose of 0.5 %w/w, reaction time of 120 min and 75 ^{0}C was used. The kinetics and thermodynamics of the process was studied. The activation energy, free exponential factor, enthalpy, entropy, for transesterification were found to be 44.2 kj/mol, 5.02*10^5 dm^3 min^{-1} ,42.4 kj/mol -140.7J/mol respectively. the Gibbs free energy ΔG value was calculated as 85.74 kJ/mol, 87.56 kJmol, 89.32 kjmol, 91.08 kJmol at 37°C ,50°C, 62.5°C, and 75°C respectively which show the reaction was non spontaneous and endergonic in nature.

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