



Optimization and effect of varying catalyst concentration and trans-esterification temperature on the yield of biodiesel production from palm kernel oil and groundnut oil

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ABSTRACT

The negative environmental impact generated by fossil fuel has resulted in the demand to search for alternative routes of renewable sources of energy, such as biodiesel, that have unlimited duration while having little or no hazardous impact. In this study, trans-esterification of palm kernel oil and groundnut oil was carried out using sodium methoxide (CH_3ONa) as a catalyst. The effect of varying Sodium Methoxide (CH_3ONa) catalyst concentrations of (0.25, 0.5, 1.0, 1.5, and 2.0) % w/v at trans-esterification temperatures of (50, 55, and 60) °C on the yield of biodiesel from groundnut oil and palm kernel oil was determined. This was to identify the catalyst concentration and trans-esterification temperature with optimal process yield. The process gave optimum biodiesel yields of 98% and 84% by volume of groundnut oil and palm kernel oil at reaction conditions of 0.5%w/v CH_3ONa as catalyst, trans-esterification temperature of 55°C, 360 rpm mixing rate and a reaction time of 90 minutes. The biodiesel produced was analyzed for fuel properties using the American Society of Testing and Materials (ASTM) standard, and the results obtained were as follows; specific gravity (0.8835, 0.8815 at 15°C), flash point (98, 124) °C, viscosity (5.2, 7.6) mm^2S^{-1} at 40°C, pour point (9, -1)°C, iodine value (8.04, 17.11) /100, acid value (0.67, 0.48) mg/KOH/g, peroxide value (28, 60) mg Kg^{-1} , fire point (108,136)°C for palm kernel oil and groundnut oil respectively.

1. Introduction

Considering the rapid increase in the global population in the world today, the long-term strength of a complex environment is tested by the demand for a higher standard of living. This involves meeting the energy and food requirements of over 9 billion people [1]. A

recent statement by BP's Energy Outlook to 2035 proposed that the average world energy usage is expected to increase by 34% between 2014 and 2035 [2]. However, the use of petroleum is a suitable means of harnessing energy for global consumption. But its drastic increase in price, non-ecofriendly nature, and great addition to pollution of the atmosphere have led to the need to develop alternative routes of renewable sources of energy that have unlimited duration

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while having little or no hazardous impact on the environment [3]. Several alternative means, such as bio-ethanol from the ebullition of starch, biomass gasification, and biodiesel, have been harnessed over the years [4]. However, the use of biodiesel remains the best. It covers an estimated 82% of the total biofuel production as stated by the EU. It has also shown a substantial contribution to future energy demands of both domestic and industrial sectors [5,6]. In comparison with petroleum-based derived diesel, it is non-toxic, biodegradable, and a cleaner source of energy [2]. Vehicles using biodiesel emit less harmful greenhouse gases of carbon monoxide and sulfur dioxide [7]. Biodiesel could reduce the emission of particulate matter (PM) and act as a good lubricant for diesel engines, thus prolonging the shelf-life of the engine. In addition, biodiesel has a higher flash point, making it safer to handle than mineral diesel [8]. Other profitable characteristics of biodiesel that make it an effective alternative to mineral-derived-diesel oil are liquid nature portability, sustainability, ignition performance, and higher octane number [9]. Biodiesel, also known as fatty acids methyl esters (FAME) is a domestic and renewable biomass fuel for diesel engines obtained from vegetable oils or animal fats, designated B100. It must meet the requirements of ASTM D6751 [8]. Feedstocks used in biodiesel production are available and could always be re-planted or grown [8]. Biodiesel is produced through chemical processes such as transesterification or esterification reactions [10]. Trans-esterification is the reaction between an alcohol and an ester [11], while esterification is the reaction between a carboxylic acid and alcohol. During the process of trans-esterification, the alcohol functional group is deprotonated by the action of the base which compels it into a stronger nucleophile [12]. The most frequently employed alcohols in this process are ethanol or methanol. Under standard conditions, the trans-esterification reaction proceeds at a very slow rate or not at all, so, heats and catalysts (acid and/or base) are used to increase the reaction rate.

[13]. It is vital to know that catalysts are not absorbed during trans-esterification reactions [11]. Heterogeneous, homogeneous, Nano, and super-critical fluid catalysts have all been utilized to activate trans-esterification reactions [14]. But in this study, the homogenous catalyst is used for the trans-esterification process. This is because it permits a higher degree of interaction with the reaction mixture, and allows the complete conversion of feedstock to biodiesel [6]. More often in the presence of a base catalyst, an undesirable saponification reaction could occur if the feedstock contains free fatty acids. Therefore, feedstock containing less than 0.5wt% free fatty acid is employed during the trans-esterification process to avoid soap formation [15]. The feedstock composition controls the chemical pathway and dictates the type of catalyst to be utilized in the production of biodiesel [2]. The feedstock used for biodiesel production is Fats and oils from plants and animals; they comprise triglycerides which are esters that contain three fatty acids, trihydric alcohol, and glycerol. The feedstock includes a range of edible vegetable oil, non-edible oils, waste or recycled oils, and animal fats [7, 9 -10]. Edible oils are connected to edible biomass, examples are; soybean, rapeseed, sunflower, palm, coconut and linseed while the non-edible biofuels are biomass fuel, ranging from lignocellulose feedstock to municipal solid wastes [16]. From literature reviews various types of oil have been used, but in this study the use of edible oils like unrefined groundnut nut oil and palm kernel oil is selected owing to their unique properties. Groundnut oil is mild-tasting vegetable oil with a high smoke point compared to several cooking oils [17]. The oil is obtainable in purified, unrefined, cold pressed, and roasted variations have a strong peanut flavor and aroma [15]. Palm kernel oil is edible plant oil derived from the kernel of the oil palm [12]. Palm kernel oil is among one of the essential oils that contain saturated vegetable fats, this is because it is composed of 16-carbon saturated fatty acid and excessive palmitic acid [13]. Palm

Table 1. Literatures reviews of some studies conducted on the effect of varying catalyst concentration and Trans-esterification temperature on the yield of biodiesel in Nigeria

N	Feed Stock	Catalyst Type	T	Catalyst Concentration	RT	Biodiesel Yield	Ref.
1.	Palm kernel oil	Homogeneous	60°C	,1.5 ,1.25 ,1.0 ,0.75 ,0.5) and 2.0)%w/v of KOH 1.75	120	,85.2 ,95.8 ,95.0 ,90.5) % (71.3 ,71.1 ,73.3	[19]
2.	Milk Bush seed oil	Heterogeneous Homogeneous	65°C	wt. % of CSS and KOH 3.0	120	and 94.33% 81%	[20]
3.	False Shea seed oil	Homogeneous	50°C	0.3mol/dm ³ of NaOH	120	85.0%	[21]
4.	Water Melon Seed oil	Homogeneous	60°C	g of NaOH(0.18 ,0.15 ,0.13)	(150 ,120 ,90)	%(49 ,53 ,70)	[22]
5.	Jatropha curcas oil	Homogeneous	48°C	0.88M of KOH	240	84.70%	[23]
6.	Palm kernel oil	Homogeneous	60°C	w/v of KOH 1.0%	,90 ,75 ,60 ,45 ,30) (120 ,105	,94.2 ,92.5 ,90.1 ,87.4) % (96.0 ,96.0 ,96.0	[24]
7.	PKO-GO	Homogeneous	55°C	w/v of NaOH 0.7%	-----	91.98 ,90.53	[25]

RT: Reaction time (mins) **T:**Temperature **PKO-GO:** Palm kernel oil and groundnut oil

kernel oil is semi-solid at room temperature, stable at high cooking temperatures and has extended storage capacity [18]. Several studies have conducted experiments on biodiesel production using different catalyst types and feedstock to varying temperatures as summarized in Table 1. In this study, biodiesel will be produced from palm kernel oil and groundnut oil through different trans-esterification temperatures by varying catalyst concentrations of sodium methoxide. The result from this study will be a basis for determining the optimal reaction conditions for the production of biodiesel production.

2. Experimental

The data generated from the experimental results were modelled using linear, interaction, pure quadratic, quadratic, 3rd order polynomial, and 4th order polynomial. From the result obtained, the 4th-order polynomial showed a good correlation with the experimental results; demonstrating that the model was useful for optimization. Newton Raphson's multivariable optimization technique and Response Surface Methodology (RSM) were further used to enhance the process parameters of the trans-esterification reaction. Newton Raphson's

multivariable optimization technique gave an optimal yield of 100.5 mL and 90.7 mL for groundnut oil and PKO FAME with a corresponding catalyst concentration and trans-esterification temperatures of (0.25%, 0.48%) and (51.3 °C, 50 °C). Whereas the surface plots gave optimal yields of 104.8 mL and 89.8 mL with the catalyst concentration and trans-esterification temperatures of (0.6%, 0.425%) and (58°C, 50°C) for groundnut oil and Palm kernel Oil (PKO) based Fatty Acid Methyl Esters (FAME). The findings from this study were in good correlation with ASTM standards for fuel. Therefore, it can be used as an excellent alternative fuel for diesel engines.

2.1. Materials

The reagents used were distilled H₂O, Concentrated Sulphuric acid (H₂SO₄), Methanol (CH₃OH), Sodium hydroxide (NaOH), and two different oils, namely groundnut and palm kernel. Reagent like NaOH was properly reserved in an airtight plastic container to prevent them from absorbing moisture from the atmosphere since it is deliquescent in nature. Methanol was reserved in an airtight brown bottle to prevent evaporation as methanol is a volatile liquid.



Fig. 1a. Low FFA oil after acid-catalyzed esterification

2.2. Sampling

Groundnut oil and Palm kernel oil were procured from a commercial shop in Ogbete main market Enugu state, Nigeria. The experiment was conducted in laboratory 3 of the materials and Energy Technology (MET) department of the Project Development Institute (PRODA), Enugu state Nigeria.

2.3. Acid-catalyzed esterification

Delving directly into base-catalyzed transesterification may result in soap production instead of biodiesel due to the high FFA content of the unrefined groundnut oil and palm kernel oil. In order to eradicate the possibility of this side reaction (i.e., saponification), the FFA content of the unrefined sample is reduced to the barest minimum by acid-catalyzed esterification reaction using conc. Sulfuric acid (conc. H_2SO_4) as catalyst [8]. The diagrammatic setup is shown in Figure 1a and 1b.

2.3.1. Experimental procedure of the acid-catalyzed esterification

Unrefined groundnut oil and palm kernel oil were poured into a conical flask and heated to a temperature of $60^\circ C$ for 10 minutes. The temperature was monitored using mercury in a glass thermometer fitted with a ca lamp in the retort stand. Methanol (60% weight of the sample) was introduced into the beakers containing the preheated



Fig. 1b. High FFA oil before acid catalyzed esterification

oil samples. Concentrated sulfuric acid (H_2SO_4) of 1.2% weight of the sample was added to the mixture. The mixture was stirred using a magnetic hot plate at $50^\circ C$ in an open system for an hour. The mixture was transferred into a separating funnel and allowed to separate overnight. The mixture is divided into three phases: the lower phases (impurities), the middle phase (the preheated sample) and the upper layer (the methanol-water phase).

2.4. Base catalyzed transesterification experimental procedure

9.65g of NaOH pellets were weighed and introduced into a round bottom flask containing 200mL of $CH_3OH(aq)$. It was stirred and allowed to dissolve completely by shaking vigorously until a solution of sodium-methoxide (CH_3ONa) was formed in the process. The CH_3ONa solution was added to 100mL of groundnut oil and palm kernel oil from the acid catalyzed esterification process into the different conical flasks. The mixture was then heated to a preferred trans-esterification temperature of $60^\circ C$ using the magnetic hot plate. At this point, the stirrer was introduced into the solution. Stirring was done at a constant speed (e.g., 360 revolutions per minute). It was continued until a given time of 90 minutes was attained. While heating and stirring simultaneously, the solution was made air-tight using a masking foil to prevent CH_3ONa from evaporating. After

the given time, the solution was removed from heat and poured into a separating funnel. It was left overnight for the separation to take place. Glycerin settled below, while the biodiesel (ethyl esters) which was the supernatant settled above. The glycerin was discarded and the biodiesel was washed with distilled water until the impurities were completely removed. These impurities were in the form of a foamy solution that settled. The biodiesel was again washed with hot water to remove further impurities. Measurements were also taken before and after the washing of biodiesel. The waste product removed with water was tested using phenolphthalein, which turned pink on the addition of phenolphthalein, confirming that sodium hydroxide was still present. So, in order to get purer biodiesel, continued washing with water was done until the product was removed as waste does not turn pink using the phenolphthalein Indicator. To neutralize the presence of NaOH(aq) ultimately, 1mL H₂SO₄(aq) was added after every negative phenolphthalein test because acids have no negative effect on biodiesel. The already washed biodiesel was collected and heated gradually at about 100 to give off the leftover water after washing, then was allowed to cool. A viscous solution with pale gold color was obtained and that was the biodiesel. The procedure was repeated using the same catalyst concentrations of 0.25%w/v at the trans-esterification temperature of 55°C and 50°C, respectively.

2.5. Physicochemical Characterization of Biodiesel

2.5.1. Determination of specific gravity at 55°C and viscosity at 40°C

empty S.G. bottle was weighed and filled with distilled water, and the reading was noted. An S.G bottle was filled with biodiesel and weighed again. The S.G. was calculated using Equation 1.

$$SG = \frac{\text{weight of the biodiesel}}{\text{weight of distilled water}} \quad (\text{Eq.1})$$

The viscosity of the biodiesel was determined using “Ostwald’s Viscometer”. This was done by filling the viscometer to the mark; sucking it up into the other side of the fuse, and setting a stop-clock or stop-watch to time when the oil flows back to the first tube with which the oil was first filled. The viscosity was then calculated as Equation 2.

$$\text{Viscosity} = \frac{4.39 \times t}{8} \quad (\text{Eq.2})$$

Where; 4.39 = centistokes constant, 8 = sugar or glucose constant, t = time taken to move in the viscometer.

2.5.2. Free fatty acid (FFA) or acid value

The acid number test was conducted using ASTM D-664 Test Method. 5g of the biodiesel sample was measured into a conical flask, and three drops of phenolphthalein indicator and 20 ml of ethanol were added. It was titrated with 0.1 M NaOH solutions and a pink coloration was observed. The FFA was calculated by Equation 3.

$$FFA = \frac{T.V \times N \times 5.61}{W} \quad (\text{Eq.3})$$

Where; T.V = Titre value, N = Normality of titrate, 5.61 = Acid constant, W = Weight of the sample

2.5.3. Saponification Value (SV)

The saponification value test for biodiesel in this present study was conducted in accordance to ASTM D5558 standard testing method. 5g of the biodiesel was measured into a conical flask, 0.5M of ethanolic KOH was added and refluxed (heat) in a round bottom flask, then allowed to stand for 3 minutes. The essence of refluxing was to get a perfect dissolution of biodiesel in the ethanolic potassium hydroxide. Three drops of phenolphthalein indicator were added and titrated with 0.5M hydrochloric acid. A blank

titration was also run; the saponification value was calculated using Equation 4.

$$SV = \frac{56.1 \times 0.5 \times (v_2 - v_1)}{\text{weight of biodiesel used}} \quad (\text{Eq.4})$$

Where; V_2 = Titer of blank, V_1 = Titer of sample, 56.1 = MW of KOH, 0.5 = Normality of KOH.

2.5.4. Determination of iodine value using EN 14112 test method

5.0 g of biodiesel was measured into a conical flask; 15mL of chloroform and 25mL of Wijis (iodine monochloride) solution were added and mixed together. The mixture was tightly covered and placed in the dark for 30 minutes. 20mL of 10% KI (Potassium iodide) and 50mL of distilled water were added and the resulting solution turned to red. The reddish solution was titrated with 0.1M Sodium Thiosulphate, 5.0 mL of 1% starch indicator was added and the color turned blue-black. It was later titrated with 0.1M Sodium Thiosulphate and turned colorless. Blank was also titrated and the iodine value was calculated by Equation 5.

$$IV = \frac{12.69 \times (v_2 - v_1) \times N}{\text{weight of biodiesel used}} \quad (\text{Eq.5})$$

Where; 12.69 = Constant for iodine value, N = Normality of Titrant, V_2 = Titer of blank, V_1 = Titer of sample.

2.5.5. Determination of peroxide value using ASTM D37031-13 methodology

5g of biodiesel was measured into a 100 mL beaker, 25 mL of acetic acid and chloroform solution in the ratio of 2:1 was added. 1mL of 10% Potassium Iodide was later added and shaken vigorously. The mixture was covered and kept in the dark place for 1 minute. 35 mL of the starch indicator was added and Titrated with 0.02M Sodium Thiosulphate $\text{Na}_2\text{S}_2\text{O}_3$ and a white color

was observed. A blank titration was also prepared in the same way as described excluding the step of addition of biodiesel. The peroxide value was calculated as Equation 6.

$$PV = \frac{100(v_1 - v_2) \times N}{\text{volume of biodiesel used}} \quad (\text{Eq.6})$$

Where; N = Normality $\text{Na}_2\text{S}_2\text{O}_3$, V_1 = Titer of sample, V_2 = Titer of blank, 100 = Peroxide value constant.

2.5.6. Determination of pour and flash point

This is the minimum temperature at which the oil can pour down. This test was done in accordance to the ASTM D97 Test method. The biodiesel was brought out at room temperature, it was allowed to melt gradually and the temperature at which the biodiesel became a complete liquid was recorded as the pour point.

The flash point of an oil is the lowest temperature at which vapour from biodiesel will ignite when a small flame is applied under standard test conditions. The test was carried out using the D93 test method. A source of the fire was placed at a distance away from the smoking biodiesel in a closed cup and the temperature at which the biodiesel catches fire was noted.

2.6. Model methodology

The yields of biodiesel obtained from both samples (i.e., groundnut oil and palm kernel oil) in this study were modeled concerning two independent variables (catalyst concentration and trans-esterification temperature) using Several models such as linear, interaction, pure quadratic, quadratic, 3rd order polynomial and 4th order polynomials.

2.7. Optimization methodology

Data obtained were optimized using MATLAB optimization tool box and Response Surface Methodology (RSM) as described by [40].

3. Results and discussion

3.1. Experimental result on the yield of biodiesel obtained from groundnut oil and palm kernel oil by varying catalyst concentration and transesterification temperature

The results obtained by varying transesterification temperature from 50-60°C at catalysts concentration of 0.25-2.0% w/v of CH₃ONa is presented in Table 2 and 3. From the result it can be deduce that biodiesel yield increases gradually with an increase in catalysts concentrations and trans-esterification temperature, but with an additional increase in catalyst concentration of 1%w/v resulted to a decrease in biodiesel yield. Hence, the maximum output of biodiesel was at 0.5 % w/v of CH₃ONa catalyst and a trans-esterification temperature of 55°C. This implies that, at that temperature and concentrations equilibrium is attained and this can be further explained by trans-esterification reversible reaction. The discovery from this research was in close proximity to the works of [26] who reported that catalysts concentrations above 1% w/v favored backward reaction, thereby shifting the equilibrium to the left as well as resulting in the loss of sodium methoxide and a reduction in the yield of biodiesel.

3.2. Physicochemical characterization of the biodiesel

The result for the physicochemical characterization of the biodiesel is presented in Table 4. The physiochemical characterization of biodiesel obtained from PKO and groundnut oil in this present study showed a good conformity to that of ASTM standard values for mineral diesel. Hence, it can be utilized as a better alternative for petroleum diesel. The density of the oil is a very vital factor to be considered because the fuel injection system works with volume metering approach. The density of biodiesel provides required details on the weight of the oil at specific temperatures. From this present study the specific gravities of biodiesel harnessed from palm kernel oil and groundnut oil were 0.8835 and 0.8815 respectively. These values are within the limits of 0.8833 of biodiesel specified by ASTM [27] and

were also in close proximity with various scientific studies carried out by [28] who reported specific gravities of 0.881, 0.865, and 0.887 for biodiesel from Mango seed oil, Palm kernel oil and Shea butter oil. Viscosity is one of the basic criteria to be considered when evaluating the quality of biodiesel. It is a key property which measures the resistance flow of fluids under the effect of gravity [29]. The viscosity value obtained from biodiesel of PKO and groundnut oil conducted in this experiment are 5.2 mm² S⁻¹ and 7.6 mm² S⁻¹ respectively. These values were within the limits of 4.0 - 6.0 mm² S⁻¹ specified by ASTM, but the biodiesel from groundnut oil was a bit higher than ASTM required limit. The viscosity of biodiesel explains the effective lubricity of fuel; it shows that the biodiesel analyzed in this present study may protect diesel fuel pumps and engines from wear and seepage. Thus enhances the atomicity and combustion as well as reducing emissions of fumes from exhaust engines. The values obtained also showed good correlation with biodiesel values of 7.65 and 5.92 mm² S⁻¹ from palm kernel oil and groundnut oil [30]. It was said to be somewhat higher than the values of 3.62 mm² S⁻¹ reported in shear butter oil [28] and mango seed oil (5.82 mm²/S) [26]. Flash point refers to the lowest temperature at which the biodiesel produces enough vapour to ignite when exposed to thermal sources; it is also a measure of degree of flammability [31]. It is a basic criterion to consider when handling, storing and transporting fuel. The flash point of the biodiesel obtained from PKO and groundnut oil in this research was 98°C and 124°C. It was within the range of (100 – 170) °C set by ASTM. Therefore, the flash point value of the biodiesel from this research shows that it is safe, non-hazardous, and can hardly ignite at higher temperatures. The values obtained from this research for biodiesel from PKO was slightly lower than flash points of 120°C, 132°C, and 167°C reported by [32-33]. The biodiesel from this study is less volatile and free from basic impurities like methanol which could reduce the flash point of biodiesel.

Table 2. Palm Kernel Oil base FAME experimental results

Trans-esterification Temperature (°C)	Catalyst Concentration (Yield %)				
	0.25	0.5	1.0	1.5	2.0
50	76	82	54	0	0
55	50	84	68	62	40
60	66	66	52	62	48

Table 3. Groundnut Oil base FAME experimental results

Trans-esterification temperature (°C)	Catalyst Concentration (Yield %)				
	0.25	0.5	1.0	1.5	2.0
50	96	92	74	74	16
55	95	98	89	72	22
60	50	95	89	38	0

The fire point is the temperature at which the biodiesel may like burn for a few seconds after ignition in an open flame. The fire point of biodiesel of PKO and Groundnut oil obtained in this experiment are 108°C and 136°C, it is higher than ASTM values of 68°C for diesel [32]. This shows that biodiesel is very suitable for use as it can hardly burn even at higher temperatures after ignition. The pour point is a necessary criterion when evaluating the low-temperature Performance of fuel. It is considered as the operational capacity of the fuel under given weather, it shows how effective biodiesels can be utilized even in cold climatic regions. The ASTM specified value for the pour point of biodiesel is -5 to 10 and -35 to 15 for mineral diesel [27]. In this research the biodiesel pours point values obtained from palm kernel oil and groundnut oil were 9 and -1, respectively. These values are within the ASTM standard values and are in close range of 0.0°C, 2°C, 5°C, and 2°C pour point values [32-34]. The biodiesel produced from the palm kernel oil in this experiment has a high pour point value of 9 due to the degree of unsaturation of carbon to carbon (C-C) bond formed; this implies that it can improve the performance of an engine. Saponification value of biodiesel plays a vital role in assessing adulteration [35]. Saponification of

biodiesel can be defined as the mass in milligram of potassium hydroxide needed to saponify 1g of oil, and it is relatively dependent on the average molecular weight of fatty acids present in the primary oil [28]. Saponification value is a measure of degree on how the biodiesel oxidizes during storage, an increase in saponification value increase volatility of biodiesel. The saponification values of biodiesel produced from PKO and groundnut oil in this experimental study are 423.55 and 227.66 mg KOH g⁻¹ respectively. It is above the ASTM value of 120 mg KOH g⁻¹. The high saponification value is an indication that the primary oil had high amount of soap content, this might result to uneven combustion and increase emissions of thick fumes from exhaust engine. However, it also has an the advantage of purifying the internal component of the engine, and as such reduces friction between the surface parts of the engine [32]. Considering the yield of biodiesel, high saponification values should be reduced to the barest minimum as it would likely prevent the separation of biodiesel from glycerin. In comparison with other findings, the values in this study were obtained within the same range of saponification values of 229.9 mg KOH g⁻¹ and 226 mg KOH g⁻¹ obtained in biodiesel produced from oil and palm kernel oil reported by [32,34].

Table 4. Properties of biodiesel and mineral diesel compared to biodiesel produced from Palm kernel oil and Groundnut oil in this study.

Fuel properties	Mineral-diesel ASTM D975 Limits	Biodiesel ASTM D6751	Palm kernel	oil biodiesel Groundnut oil biodiesel
Kinematic viscosity (mm ² S ⁻¹) at 40°C	1.3 - 4.1	4.0-6.0	5.2	7.6
Specific gravity at 15°C.	0.85	0.88	0.8835	0.8815
Flash point (°C)	60-80	100 – 170 (ASTM D93)	98	124
Pour point (°C)	-35 to -15	-5 to -10 (ASTM D97)	9	-1
Acid value (mKOH/g)	-	0.5 (ASTM D-664)	0.673	0.48
Peroxide value (meq/kg)	-	-ASTM D37031-13	28	60
Iodine value (g/100)	-	7.5-8.6 (EN 14112)	8.04	17.11
Saponification value (mgKOH/g)	-	95 – 370 (ASTM D5558)	423.5	227.66
Fire point (°C)	-	68	108	136

Source: Biodiesel Handling and Use Guide (Fifth Edition). November 2016 for the standard properties for biodiesel and diesel fuels.

The acid content plays an important role when evaluating the quality of biodiesel. It determines how stable the biodiesel can stay over a long period of time. The acid value is defined by the amount of KOH in mg needed to neutralize 1.0 g of free fatty acids [36]. The acid value of biodiesel produced from palm kernel oil and groundnut oil in this present study are 0.673 and 0.48 mg KOH g⁻¹ which was in good correlation with ASTM D 664 value of 0.5 mg KOH g⁻¹. The acid values of biodiesel from this experiment was also in a close range with values of 0.37 mg KOH g⁻¹, 1.2 mg KOH g⁻¹, and 0.8 mg KOH g⁻¹ of biodiesel produced from Shea butter, mango seed oil, and palm kernel oil respectively [26,28,32]. The values obtained in this study shows that the biodiesel is not corrosive. Iodine value is a measure of degree of unsaturation resulting from the formation of carbon to carbon bonds. It is defined as the mass of iodine that is added to 100.0 g of oil [28]. Low iodine value signifies presence of saturation and vice versa, saturated oil has resistance against oxidation and deterioration. The iodine value for every biodiesel set by EN 14112 is (7.5-8.6) g per100. In this present study, the iodine value obtained from the biodiesel produced from palm kernel oil and groundnut oil were 8.04 and 17.11. The palm kernel oil biodiesel was within the specified range but that of the groundnut oil

was slightly above the range. This shows that PKO biodiesel is a suitable alternative fuel for diesel engines based specifically on the measure of iodine value. The PKO biodiesel possess low oxidative resistance unlike that of groundnut oil, although iodine value of biodiesel from groundnut oil is technically on a good range when compared with results of 65.09 g per 100, 34.24 g per100, 36.00g per100 of biodiesel from PKO, Shea butter oil, and palm oil [32,28,33]. Peroxide value plays a vital role on stability of biodiesel during storage. It is defined by the amount of peroxide oxygen per 1.0 kg of biodiesel. Peroxide value is directly proportional to the rate of oxidation which is greatly influenced by the level of saturation of the biodiesel. In other words, biodiesel with high peroxide value will easily oxidize, thus increasing the rate of biodegradation as well reducing its stability [37]. The rate at which biodiesel undergo oxidation is controlled by certain factors like heat, amount of Oxygen, light, water content, and temperature. Excessive heat, light and high temperature enhances the rate of oxidation. In this present study 28.0 meq kg⁻¹ and 60.0 meq kg⁻¹ was recorded for biodiesel produced from palm kernel oil and groundnut oil, this shows that biodiesel produced from groundnut oil will easily degrade compare to that from PKO.

3.3. Modeling of Data generated from Trans-esterification experimental result

Several models were used to fit the experimental data from the groundnut oil and PKO biodiesel. They are linear, interaction, pure quadratic, quadratic, 3rd order polynomial and 4th order polynomials with their respective regression coefficients of 0.6694, 0.6695, 0.8981, 0.8982, 0.9213, and 0.9926 for groundnut oil, while PKO was 0.4914, 0.7162, 0.5042, 0.7712, 0.8666, and 0.9773 as shown in (Table 5). The F test was carried out by comparing the variances of each model with the experimental

results, it shows that each of the models is actually adequate because their calculated F values are less than the F critical based on 14 degrees of freedom for both the numerator and denominator. However, regression coefficient of a model should be more than or approximately equal to 0.95 [38-39]. Hence, the 4th order polynomial is obviously the most suitable for both oils. The equation of the 4th order polynomial for groundnut oil is at Equation 7. Using the 4th order polynomial for both groundnut and PKO, were generated at a constant temperature (Figure 2(a) to Figure 2 (d))

$$y_{model} = a_0 + a_1x_1 + a_2x_2 + a_3x_1^2 + a_4x_1x_2 + a_5x_2^2 + a_6x_1^3 + a_7x_1^2x_2 + a_8x_1x_2^2 + a_9x_2^3 + a_{10}x_1^4 + a_{11}x_1^3x_2 + a_{12}x_1^2x_2^2 + a_{13}x_1x_2^3 + a_{14}x_2^4 \quad (\text{Eq.7})$$

$$\text{where } a_0 = -1702; a_1 = 35.48; a_2 = 79.3; a_3 = 1292; a_4 = -47.97; a_5 = -0.8628; a_6 = -581.6; a_7 = -7.308; a_8 = 0.9442; a_9 = 0; a_{10} = -49.42; a_{11} = 14.72; a_{12} = -0.4235; a_{13} = 0; a_{14} = 0$$

While for PKO;

$$y_{model} = a_0 + a_1x_1 + a_2x_2 + a_3x_1^2 + a_4x_1x_2 + a_5x_2^2 + a_6x_1^3 + a_7x_1^2x_2 + a_8x_1x_2^2 + a_9x_2^3 + a_{10}x_1^4 + a_{11}x_1^3x_2 + a_{12}x_1^2x_2^2 + a_{13}x_1x_2^3 + a_{14}x_2^4 \quad (\text{Eq.7})$$

Table 5. Data generated for different models used

0	X1	X2	Y _{expt}	Y _{model} (1)	Y _{model} (2)	Y _{model} (3)	Y _{model} (4)	Y _{model} (5)	Y _{model} (6)
Groundnut oil									
1	0.25	50	96	106.3	106.9	102.2	87.43	92.51	98.79
2	0.25	55	95	98.3	98.3	94.4	91.65	90.49	91.22
3	0.25	60	50	90.3	89.7	85.9	70.26	62.91	50.98
4	0.5	50	92	96.4	96.8	98.7	94.15	94.91	87.37
5	0.5	55	98	88.4	88.4	90.9	98.54	99.95	103.3
6	0.5	60	95	80.4	80.0	82.3	77.34	79.41	94.35
7	1.0	50	74	76.6	76.7	84.4	90.58	83.92	75.63
8	1.0	55	89	68.6	68.6	76.6	95.34	96.11	89.70
9	1.0	60	89	60.6	60.6	68.1	74.51	82.71	86.66
10	1.5	50	74	56.9	56.5	60.7	64.36	57.64	74.81
11	1.5	55	72	48.9	48.9	52.9	69.48	67.73	67.99
12	1.5	60	38	40.9	41.2	44.4	49.02	52.19	41.19
13	2.0	50	16	37.1	36.4	27.6	15.48	23.02	15.38
14	2.0	55	22	29.1	29.1	19.7	20.98	21.73	23.81
15	2.0	60	0	21.1	21.8	11.2	0.874	-5.21	-1.19
R ²				0.6694	0.6695	0.8981	0.8982	0.9213	0.9926
F				1.4939	1.4939	1.2561	1.1133	1.0854	1.0075

F CRITICAL = 2.4837

PKO									
1	0.25	50	76	65.38	83.02	61.91	76.30	77.40	72.593
2	0.25	55	50	73.58	73.58	69.59	77.06	62.36	53.121
3	0.25	60	66	81.78	64.14	77.99	57.42	57.89	66.286
4	0.5	50	82	59.26	71.39	59.86	68.33	77.25	89.138
5	0.5	55	84	67.46	67.46	67.54	74.61	72.91	77.801
6	0.5	60	66	75.66	63.53	75.94	60.48	69.46	65.061
7	1.0	50	54	47.02	48.13	51.68	48.69	51.58	47.491
8	1.0	55	68	55.22	55.22	59.35	65.99	68.28	72.941
9	1.0	60	52	63.42	62.32	67.76	62.88	66.52	53.368
10	1.5	50	0	34.79	24.87	38.03	24.10	14.18	3.5282
11	1.5	55	62	42.99	42.99	45.71	52.43	51.47	59.789
12	1.5	60	62	51.99	61.11	54.11	60.35	50.92	60.682
13	2.0	50	0	22.55	1.61	18.93	-5.43	-8.41	-0.7502
14	2.0	55	40	30.75	30.75	26.60	33.92	48.99	40.347
15	2.0	60	48	38.95	59.89	35.01	52.87	49.19	48.407
R ²				0.4914	0.7162	0.5042	0.7712	0.8666	0.9773
F				2.0352	1.3962	1.9834	1.2966	1.1539	1.0232

F CRITICAL = 2.4837

(1)=linear i.e. $a_0+a_1x_1+a_2x_2$
 (2)=interaction i.e. $a_0+a_1x_1+a_2x_2+a_3x_1x_2$
 (3)=pure quadratic i.e. $a_0+a_1x_1^2+a_2x_2^2$
 (4)=quadratic i.e. $a_0+a_1x_1+a_2x_2+a_3x_1^2+a_4x_1x_2+a_5x_2^2$
 (5)=3rd order polynomial i.e. $a_0+a_1x_1+a_2x_2+a_3x_1^2+a_4x_1x_2+a_5x_2^2+a_6x_1^3+a_7x_1^2x_2+a_8x_1x_2^2+a_9x_2^3$
 (6)= 4th order polynomial i.e. $a_0+...$
 X1=catalyst concentration as a percentage of weight of sample
 X2=transesterification temperature(°C)
 Y_EXPT=Yield from experiment (ml)

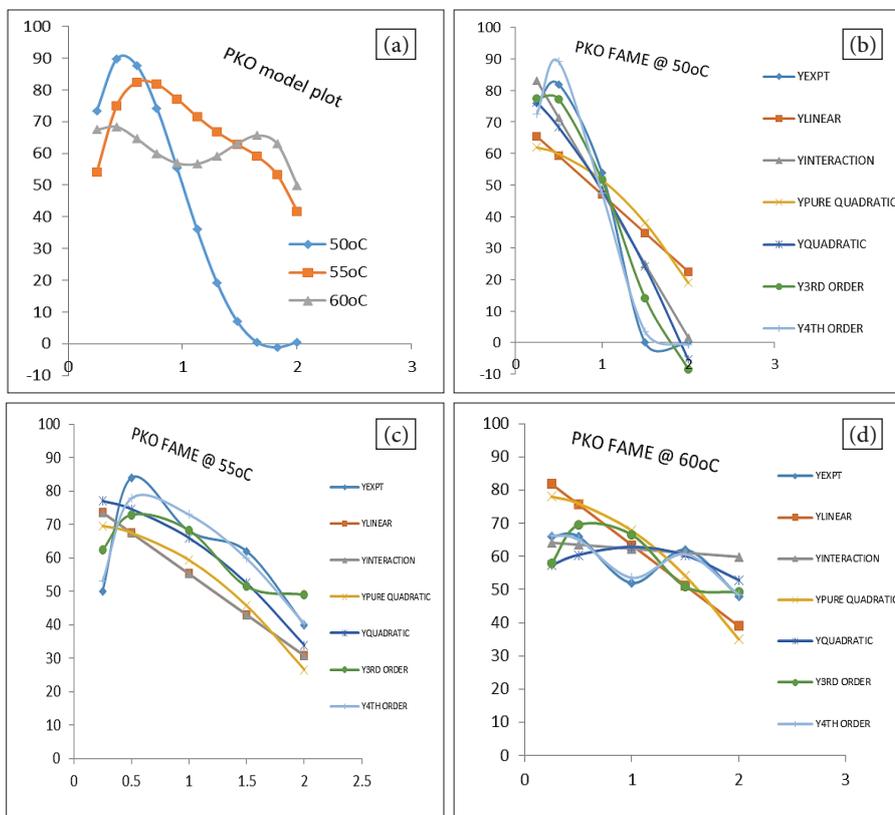


Fig. 2. (a) Varying catalyst concentration at constant temperature for PKO base FAME and comparison of the different groundnut oil base FAME models with the experimental data at (b) 50°C (c) 55°C (d) 60°C

3.4. Optimization of parameters

The main aim of optimization is to obtain the process parameter which gives the maximum FAME yield. This was done using different techniques namely: MATLAB optimization toolbox and response surface methodology (RSM)

3.4.1. MATLAB optimization toolbox

This optimization toolbox uses the principle of NEWTON RAPHSON's method of multivariable optimization technique. Firstly, a function was created in a function M-file as given below for groundnut oil and PKO base FAMES respectively.

Function f = projopt1(x)

$$f = -(-1702 + 35.48 * x(1) + 79.3 * x(2) + 1292 * x(1).^2 - 47.97 * x(1) .* x(2) - 0.8628 * x(2).^2 - 581.6 * x(1).^3 + 7.308 * x(1).^2 .* x(2) + 0.9442 * x(1) .* x(2).^2 - 49.42 * x(1).^4 + 14.72 * x(1).^3 .* x(2) + 0.4235 * x(1).^2 .* x(2).^2);$$

Function f = pkoopt(x)

$$f = -(4261 - 9215 * x(1) - 160.1 * x(2) + 1465 * x(1).^2 + 378.5 * x(1) .* x(2) + 1.498 * x(2).^2 + 874.3 * x(1).^3 - 108.5 * x(1).^2 .* x(2) - 3.709 * x(1) .* x(2).^2 - 56.99 * x(1).^4 - 10.67 * x(1).^3 .* x(2) + 1.315 * x(1).^2$$

$$.* x(2).^2);$$

Notice however that negative of the function was minimized, as this gives the negative of the maximum value of the function. Next, a program was written to minimize the functions respectively using the “fmincon” command as below table.

<code>x0 = [0.25 50]; A = [1 1]; B = 62; lb = [0.25 50]; ub = [2 60];</code>
<code>[x fval] = fmincon (@projopt1, x0, A, B, lb, ub)</code>
<code>x0 = [2 60]; A = [1 1]; B = 62; lb = [0.25 50]; ub = [2 60];</code>
<code>[x fval] = fmincon (@pkoopt, x0, A, B, lb, ub)</code>

The optimum/maximum value for groundnut oil FAME yield was obtained as 100.5141 with the corresponding independent variables (X_1 , X_2) of (0.25, 51.3463). On the other hand, the optimum/maximum value for PKO FAME yield was gotten as 90.7254 with the corresponding independent variables (X_1 , X_2) of (0.4843, 50).

3.4.2. MATLAB response surface methodology

The surface plots with the contour of the groundnut oil and PKO FAME model were plotted as indicated in Figure 3.

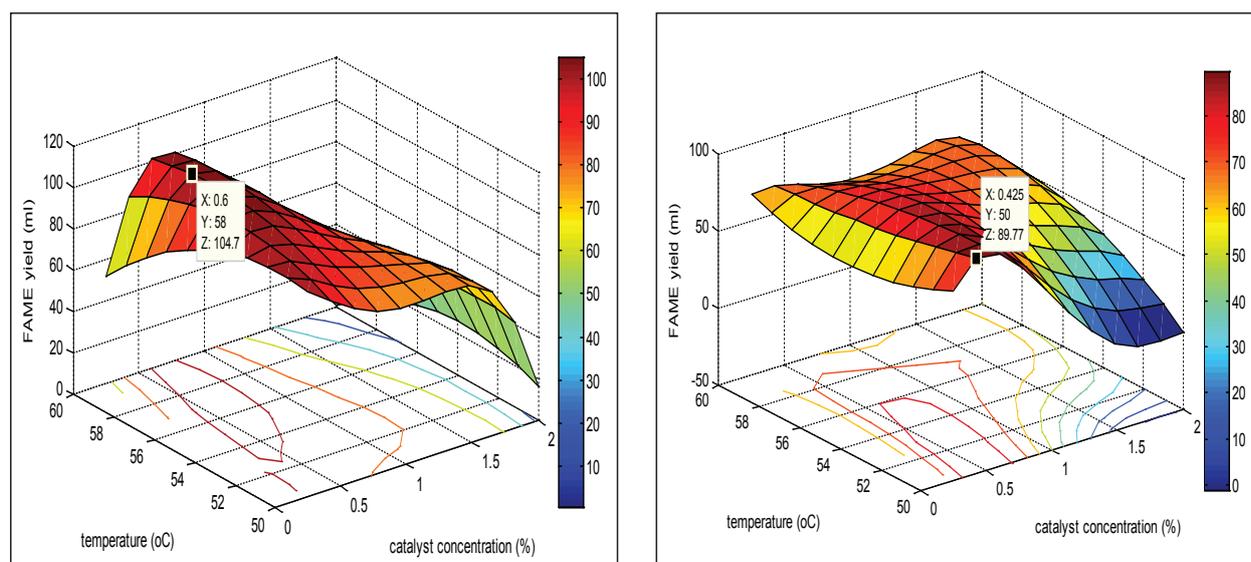


Fig. 3. Surface plot of the groundnut oil and PKO base FAME showing the optimal points

4. Conclusion

The results obtained from this present study showed that the optimum reaction conditions for the production of biodiesel from groundnut oil and palm kernel oil was obtained at a trans-esterification temperature of 55°C, 0.5 % w/v of CH₃ONa catalyst, mixing rate of 360 rpm and a reaction time of 90 minutes. At these conditions, an optimum yield of 98% and 84% by volume of FAME from groundnut and palm kernel oil was obtained. The biodiesel produced in this present study was characterized for fuel properties, and it gave good promising results; except for the pour points of biodiesel produced from palm kernel oil was found to be somewhat higher, which may point to potential difficulties in cold starts and filter plugging trouble. But however, the biodiesel from this experiment would be a better means in harnessing the supply of energy to the global economy as compared to mineral diesel. The 4th order polynomial model showed a good agreement with the experimental results, demonstrating that these methodologies were useful for modelling. Newton Raphson's multivariable optimization technique gave an optimum yield of 100.5 mL and 90.7 mL for groundnut oil and PKO FAME with a corresponding catalyst concentration and trans-esterification temperatures of (0.25%, 0.48%) and (51.3°C, 50°C). While the surface plots gave optimum yields of 104.8 mL and 89.8 mL with the catalyst concentration and trans-esterification temperatures of (0.6%, 0.425%) and (58°C, 50°C) for groundnut oil and Palm Kernel Oil (PKO) based Fatty Acid Methyl Esters (FAME). The findings obtained from this study showed that the Newton Raphson's multivariable optimization technique and Response Surface Methodology (RSM) were useful in enhancing the process parameters of the trans-esterification reaction.

5. Conflict of interest and Abbreviations

No conflict of interest to declare.

FAME: *Fatty Acid Methyl Ester*; PKO: *Palm Kernel Oil*; ASTM: *American Society of Testing and Materials*; KOH: *Potassium Hydroxide*; NaOH: *Sodium Hydroxide*; CH₃OH: *Methanol*; H₂SO₄: *Sulphuric Acid*; RSM: *Response Surface*

Methodology; FFA: *Free Fatty Acid*; CH₃ONa: *Sodium Methoxide*.

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