Journal of Agricultural Engineering and Technology (JAET) Volume 27 No 1 (2022)

PHYSICOCHEMICAL PROPERTIES OF BIODIESEL BLENDS DERIVED FROM MAHOGANY (*KHAYA SENEGALENSIS*) SEED OIL FOR USE AS ALTERNATIVE FUEL

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ABSTRACT

Plants from different regions produce oils with characteristics that vary by wide margins and depend more on the regions in which the plants are grown than the species from which the oils are derived, most probably as a result of the environmental conditions to which they are exposed. This study aims at producing biodiesel by transesterification of Khaya Senegalensis seed oil (KSO) and the determination of physicochemical properties of the biodiesel blends, compared with ASTM D6751 and EN14214 standards. Properties analyzed include: density, viscosity at 25°C, iodine value, saponification value, cloud point, pour point, flash point, acid value, free fatty acid, higher heating value and sulphur content. The transesterification reactions were maintained at the optimum process conditions of methanol/oil ratio 6:1, at 70˚C for 60 minutes at 0.84% (w/v) of catalyst concentration. A biodiesel yield of 89.50% was obtained. The results for density (844 – 837 kg/m^3), kinematic viscosity (4.44 – 4.07 mm^2/s), iodine value (92 - 127 mgI$_2$/100g), saponification value (11.22 – 36.47 Mg/KOHg), cloud points (9 - 14˚C), pour points (11 - 19 °C), flash points (110 - 190 °C), acid value (0.21 – 0.78 Mg/KOHg) and the sulphur contents (2.15 – 2.86 mg/kg) of the biodiesel blends (B10, B20, B30, B40, B50 and B100) conformed with both the ASTM D6751and EN14214 standard, with higher heating value of the blends slightly above both standards. It was concluded that with the high yield and quality properties of the biodiesel blends, the potential of using KSO biodiesel blends as an alternative fuel on a diesel engine.

KEYWORDS: Alternative fuel, biodiesel, khaya senegalensis, physicochemical properties, transesterification.

1. INTRODUCTION

The recent witnessed fuel crises and the world oil price fluctuations have generated the need to develop renewable fuels to decrease the dependence on petrol-based diesel fuel. The need to reduce the growth of the consumption of petrol-based diesel fuels, for economic and environmental reasons can best be accomplished by using renewable substitutes (Dasin *et al.*, 2019). Renewable resources offer the opportunity to tap local resources and reduce dependency on fossil energy resources. Most biodiesel oils, particularly of the nonedible type can be used as fuel in diesel engines. They contain a significant amount of oxygen (Srivastava and Prasad, 2000). Diesel engines have provided power units for road transportation systems, ships, railway locomotives, equipment used for farming, construction, and in almost every type of industry due to their fuel efficiency and durability (Pandhare and Padalkar, 2013). A diesel engine is widely used in agriculture, industry, transportation and other fields. Due to the global warming threat, governments have strict rules regarding emissions to the environment. Therefore, efforts to increase the quality of combustion and reduce environmental pollutants along with the use of alternative fuels as well as conducting research in this field are crucial (Samadi and Heidarbeigi, 2020; Mesri *et al*., 2021).

Khaya senegalensis is a dry zone mahogany plant which is widely found in sub-Saharan Africa. In northern Nigeria, African mahogany is often planted by the road sides to provide shade. Furthermore, African mahogany is exported to the United States, where it substitutes expensive American mahogany for America's wood industry (Rogers, 2004). The fruit of Khaya Senegalensis is spherical woody capsule (4-6cm in diameter), each capsule contains at least six seeds. Mahogany seeds have about 53% by weight oil which does not contain essential fatty acids; hence, it does not have nutritional value and the seed oil is inedible (Auwal, *et al.,* 2012).
Investigation and development of biodiesel as an alternative and renewable source of energy for transportation has become a major target in the effort towards energy self-reliance. Biofuel has crucial advantages such as technical feasibility of blending, superiority from the environment and emission reduction perspective, its capacity to provide energy security to remote rural areas and employment generation (Pramanik, 2003). Biodiesels are renewable eco-fuels mainly produced from biological resources such as vegetable oil (from jatropha, karanja, beauty leaf, soybean etc.), animal fats (from lard, tallow, etc.), agricultural and factory waste (from waste oil, grease, etc.), as asserted by Rashid *et al*. (2013). Biodiesel has recently attracted much attention all over the world as an alternative renewable oxygenated fuel because of its availability, similarity to diesel in terms of fuel properties, superior lubricating properties, low/zero sulphur content, non-toxicity and clean emission profile. In view of the several advantages, non-edible-based biodiesel has potential to replace petroleum-based fuels in the long run (Ramadhas *et al.,* 2005).

Many researchers also have observed different characteristics in biodiesels produced in the same way from similar plants from different regions. Emil *et al*. (2010) compared jatropha seed oil from Malaysia, Indonesia and Thailand which was produced in the same way using the same facilities, and found that the physicochemical characteristics of the oil produced were very different; the free fatty acid content, iodine value, saponification value, oil content and density were different in oils from different regions. Lalas and Tsaknis (2002) compared the characteristics of oils from *Moringa oleifera* of Indian origin and Kenyan (Mbololo) origin and found that, even though the oil extraction and processing procedure were identical, the properties of the oils differed according to the regions from which they were sourced.

Quality is a prerequisite for the long-term success (successful use, without technical problems) of a biofuel. Biodiesel quality depends on several factors that reflect its chemical and physical characteristics. The quality of biodiesel can be influenced by a number of factors: the quality of the feedstock; the fatty acid composition of the parent vegetable oil or animal fat; the production process and the other materials used in the process; the postproduction parameters; and the handling and storage (Barabas and Todoruţ, 2011). Given the fact that most current diesel engines are designed to be powered by diesel fuel, the physicochemical properties of biodiesel should be similar to those of diesel oil. The physicochemical properties of biodiesels are strongly influenced by the nature and the composition of the feedstocks used in their production. Therefore, quality requirements for the marketing of biodiesel vary from region to region and the largest differences are found in cetane number, oxidation stability, iodine value, density and viscosity (Barabas and Todoruţ, 2011).

In order to assess the quality of biodiesel substantial standards were formulated and biodiesel fuels must meet the specifications of biodiesel prescribed by the American Society for Testing and Materials (ASTM 6751) and European Standard (EN 14214) (Sakthivel *et al.*, 2018). These standards set guidelines for testing biodiesel fuels and prescribe the suitable ranges for various physical and chemical properties of the fuel in order to use it in the engine. Table 1 shows the ASTM 6751 and EN 14214 standards of biodiesel. The current standards for regulating the quality of biodiesel on the market are based on a variety of factors which vary from region to region, including characteristics of the existing diesel fuel standards, the predominance of the types of diesel engines most common in the region, the emissions regulations governing those engines, the development stage and the climatic properties of the region/country where it is produced and/or used, and not least, the purpose and motivation for the use of biodiesel (European Commission, 2007).

Table 1: ASTM D 6751 and EN 14214 Biodiesel Standards

Property	ASTM D6751	EN 14214	Unit
Density	860 – 890	860 - 900	Kg/m^3
Kinematic Viscosity @ 40^{o}C	3.5 – 5	1.9 - 6	mm^2/s
Flash Point	130 min.	120 max.	^{o}C
Sulphur Content	10 max.	10 max.	mg/Kg
Acid Value	0.8 max.	0.5 max	mg KOH/g
Iodine Value	120 max.	130 max.	g I/100 g
Water Content	500 max.	500 max.	mg/Kg
Oxidative Stability, 100^{o}C	6	6	Hours
Pour Point	-3 to 16	-15 to 16	^{o}C
Caloric Value (Calculated)	45.18	35	MJ/Kg
Saponification Value	<500	-	mg KOH/g
Cetane number	47	51	-
Cloud Point	-3 to 12	-3 to 10	^{o}C
FFA	<0.45	-	%

Sources: ASTM (2003), Knothe (2005), Knothe (2006), Demirbas (2009), ACEA (2009)

Therefore, this study was aimed at investigating the quality properties of the crude *Khaya Senegalensis* seed oil and the biodiesel blends in a view to ascertain the potential suitability and operation of KSO biodiesel originating from Nigeria for use as an alternative fuel.

2. MATERIAL AND METHODS

The raw Mahogany (*Khaya Senegalensis*) seed oil (5L) used for this study was obtained from Michika market in Adamawa State, Northeastern Nigeria, where it is produced locally and readily available, while the Diesel fuel (AGO) was purchased from NNPC petrol station. All chemicals such as Methanol (CH_3OH), potassium hydroxide (KOH), anhydrous calcium chloride ($CaCl_2$), sulphuric acid (H_2SO_4), sodium hydroxide pellet (NaOH) and potassium hydroxide (KOH) were all of analytical grade and sourced from reputable suppliers. Laboratory experiments were conducted at the Chemistry Department of Abubakar Tafawa Balewa University, Bauchi.

2.1 Extraction of Khaya Senegalensis (mahogany) oil

The traditional method was used in the extraction of the oil as described by Dasin *et al*., (2019), were the fine grind *Khaya Senegalensis* (mahogany) seeds were mixed with the potash solution, a pot of water was set on fire and allowed to boil, the mixture of the fine grinded *Khaya Senegalensis* (mahogany) seeds and potash were added to the boiling water and stirred continuously until the whole mixture agglomerated, thereafter, a layer of oil appeared on the top surface which was then drained from the pot. The drained oil was allowed to cool at room temperature and was then filtered out.

2.2 Acid-Base Catalyzed Transesterification reaction of Khaya Senegalensis oil

A two-step transesterification reaction was used. This was due to the fact that KSO has a high free fatty acid value; hence, an adopted method by Awolu and Layokun (2013) was used. The method includes acid transesterification followed by base transesterification. The acid-catalyzed transesterification was carried out using 50 ml of methanol and 0.2 ml of concentrated H_2SO_4 mixed together inside a 250-ml conical flask. The conical flask was inserted into a water bath at 50 °C. The mixture was later added to 200 ml of warmed (preheated) KSO inside a 500-ml round bottom flask and placed on a hotplate stirrer (HS 131 model), continuously stirred, for 1 h for the acid transesterification to take place. For the base-catalyzed transesterification, the product of the reaction in acid transesterification was used for the base transesterification. 600 ml of methanol and 0.84% (w/w) of catalyst was mixed in a 250 ml flask and heated for 10 min. on a hot plate stirrer. The mixture was then added to 100ml of the esterified oil and continuously heated at 70 ^{o}C for 60 minutes for the transesterification to complete.

2.3 Removal of Impurities and Biodiesel Yield Determination

After the base transesterification process, the biodiesel was allowed to settle for at least 24 h inside a separating funnel to allow clear separation of the biodiesel from glycerin. The layer on the top is biodiesel, while the bottom layer is glycerin. The biodiesel separation was carried out by decantation as the glycerin was drained off while the biodiesel remained. Warm distilled water at 50 °C was added to the separated biodiesel, and the mixture was shaken vigorously. The water was allowed to drain through the bottom of the separating funnel (Awolu and Layokun, 2013). This was carried out five times until clear biodiesel was obtained. Anhydrous $CaCl_2$ was added to the biodiesel and heated gently at 50 °C on a HS 131 hotplate stirrer. The anhydrous $CaCl_2$ was later separated from the biodiesel to obtain a clean dry KSO biodiesel. The volume of the biodiesel obtained from each sample was determined, while the percentage of biodiesel yield was calculated from Equation 1 (Yesilyurt *et al*., 2018).

$$Y\,(\%) = \frac{V_{bd}}{V_{kso}} x\,100 \qquad (1)$$

Where Y is Yield of KSO Biodiesel (%), V_{bd} is volume of KSO biodiesel produced (ml.) and V_{kso} is volume of KSO used in the reaction (ml.).

2.4 Biodiesel–diesel blending

Khaya *Senegalensis* oil (KSO) biodiesel was blended with diesel at 10, 20, 30, 40, 50 and 100% (B10, B20, B30, B40, B50 and B100) by volume using a magnetic stirrer (HS 131 hotplate stirrer) at 2000 rpm for 30 mins. Where;

B10 = 10% biodiesel and 90% fossil diesel

B20 = 20% biodiesel and 805 fossil diesel

B30 = 30% biodiesel and 70% fossil diesel

B40 = 40% biodiesel and 605 fossil diesel

B50 = 50% biodiesel and 50% fossil diesel

B100 = 100% biodiesel.

2.5 Experimental layout

Randomized complete design (RCD) was employed as the design layout in the collation and analysis of data from the experimental test. The RCD will result in a smaller error variance. The fuel samples used include raw KSO, 6 biodiesel/diesel blends (B10, B20, B30, B40, B50 and B100) and 11 parameters respectively. This will give a total of 7 x 11 = 77 tests. Each test was conducted in three replications, which gave a total of 77 × 3 = 231 experimental tests. All the data generated was subjected to statistical analysis to evaluate the mean and standard deviation using SAS version 9.4 software.

2.6 Physiochemical characterization of crude KSO and biodiesel blends

The physicochemical analyses of the KSO and the biodiesel blends were carried out using the Association of Official Analytical Chemists methods (AOAC, 2000). The analyses carried out include; density, kinematic viscosity (using NDJ – 5S Digital Viscometer), iodine value by Wiji's method (AOAC, 2000), saponification value (AOAC, 2000), cloud point determination (ASTM, 2002), pour point determination (ASTM, 2002), flash point determination using an improvised method by Giwa *et al.* (2016), acid value (AOAC, 2000), free fatty acid (FFA), higher heating value (HHV) using calculated model developed by Demirbas and Kara (2006) and sulphur content using Eschka's method by Mott and Wilkinson (2007).

2.6.1 Determination of density

To determine the density of the oil extracted, the weight of a small beaker was determined using an electronic weighing balance. 1 ml of the oil was poured into it and the weight was noted. The mass of the 1 ml oil poured into the beaker was determined by taking the difference in the mass measurements. Then, the density of the oil was calculated using Equation 2 (Giwa *et al*., 2016). The same procedure was repeated for the biodiesel blends.

$$\text{Density} = \frac{\text{Mass of sample oil}}{\text{Volume of sample oil weighed}} \qquad (2)$$

2.6.2 Determination of kinematic viscosity

In order to determine the viscosity, which is a measure of resistance to flow of the sample fuel, a calibrated digital electronic viscometer (NDJ – 5S Digital Viscometer) as described by Giwa *et al*., (2016) was employed. The electronic viscometer normally measures fluid viscosity at a given shear rate (15 – 1000 mPa.S). The principle of operation of the digital electronic viscometer used was to rotate a spindle (which was immersed in the test fuel samples) through a calibrated spring and, then, measuring the viscous drag of the fluid against the spindle deflection by the spring deflection. Spring deflection was measured with a rotary transducer, which provided a torque signal. The measurement range of the digital electronic viscometer was determined by the rotational speed of the spindle. The procedure was repeated thrice for all other samples and the mean was recorded.

2.6.3 Determination of iodine value (IV)

1 g of each sample (KSO and biodiesel blends) was dissolved in 15 ml of carbon tetrachloride. The solution was mixed with 25 ml of Wiji's solution. The flask with the content was stoppered and allowed to stand in the dark for thirty minutes at room temperature to enable oxidation to take place. Then 100 ml of distilled water and 20 ml of 10% potassium iodide solution were added to the content of the flask. The resultant mixture was titrated with 0.1 M sodium thiosulphate using 10% starch solution (weight by volume). A blank determination was carried out in the same manner under similar conditions. The iodine value was calculated using Equation 3 (Onukwuli *et al*., 2017):

$$IV \ = 12.69\ X\ M\ X\ (B - V)/W \qquad (3)$$

Where, IV is iodine value in gI_2/100 g of oil, M is the morality/strength of standard thiosulphate solution, B is the volume of $Na_2S_20_4$ in ml used in the blank titration, V is the volume of $Na_2S_20_4$ in ml used in test titration, and W is the weight in a gram of the test sample.

2.6.4 Determination of saponification value (SV)

1 g of each sample (KSO and biodiesel blends) was dissolved in 25 ml of 0.5 M ethanoic potassium hydroxide solution, using 250 ml quick fit flask. The flask was heated in a steam bath under reflux for 30 min with occasional swirling. The resultant solution was heated with 0.5 M HCl using phenolphthalein indicator. A blank determination was carried out under similar conditions. The difference in the values above 2 liters give the number in milliliters of HC1 acid required to neutralize the alkali after saponification. The saponification value was calculated with Equation 4 (Onukwuli *et al*., 2017) as follows:

$$SV\ (mgKOH) = 56.1\ X\ M\ X\ (B - V)/W \qquad (4)$$

Where, M is the morality of standard HCl (0.5 M), B is the volume of HCl in ml used in the blank titration, V is the volume of HCl in ml used for KSO and biodiesel blends titration respectively, 56.1 is the molar mass of potassium hydroxide, and W is the weight in gram of the test sample.

2.6.5 Determination of acid value (AV)

1 g of each sample (KSO and biodiesel blends) was dissolved in a 25 ml neutral mixture of solvent (equal volume of diethy1 ether and absolute ethanol). The resultant test solution was titrated with 0.1 M potassium hydroxide (KOH) solution, using phenolphthalein indicator. The titration continued, until a pink coloured solution was obtained, indicating the end point. The acid value was calculated using the Equation 5 (Onukwuli *et al*., 2017) below:

$$AV\ (mgKOH/g)\ = 56.1\ X\ M\ X\ V/W \qquad (5)$$

M is the morality of standard KOH (0.l M), V is the volume of KOH in ml and 56.1 is the molar mass of potassium hydroxide. W is the weight in gram of the oil sample.

2.6.6 Determination of free fatty acid (FFA)

1 g each of sample (KSO and biodiesel blends) was dissolved in a 25 ml neutral mixture (equal volume of diethyl ether and absolute ethanol). The resultant test solution was titrated with 0.1 M potassium hydroxide solution with a phenolphthalein indicator added in 3 drops. The titration continued until the end point was reached. The end point was recorded as the appearance of a permanent pink colour. The free fatty acid was expressed in terms of oleic acid and computed using the Equation 6 (Onukwuli *et al*., 2017.) below:

$$FFA\ (\%) = TVX\ 0.0282\ X\ 100/W \qquad (6)$$

Where, TV is the titre value, 0.0282 is constant (weight of oleic acid neutralized by 1 mg of KOH). W is the weight in gram of the sample.

2.6.7 Determination flash point (FP)

Flash point of the sample (KSO and biodiesel blends) was determined by the method reported by Giwa *et al*., (2016). A 50 ml conical flask was filled with 1 ml of the oil and heated at a low constant rate on a hot plate. The flash point was obtained when the application of a test flame caused the vapour above the oil to ignite. The process was replicated and the average calculated.

2.6.8 Determination cloud point (CP)

The cloud point of KSO and biodiesel blend samples was determined by pouring 20cm^3 of each sample into a test tube and held vertically in a beaker containing ice and sodium chloride (Danbature *et al*., 2015). The temperature at which the appearance of cloud was noticed was recorded as the cloud point. The process was replicated and the average calculated.

2.6.9 Determination of pour point (PP)

Sample of KSO and biodiesel blends (20 cm^3 each) was allowed to super cool in a deep freezer (reading up to -20 °C). They were then removed and placed in test tube rackets under room temperature (Danbature *et al.*, 2015). The temperature at which each sample began to melt was recorded as the pour point. The process was replicated thrice and the mean taken.

2.6.10 Determination of higher heating value (HHV)

Higher heating value (HHV) of the KSO and biodiesel blends were calculated by using the following empirical Equation 7 (Yesilyurt *et al.*, 2020):

$$\mathrm{HHV} = 49.43 - [0.041(\mathrm{SV}) + 0.015\,(\mathrm{IV})] \tag{7}$$

Where SV is saponification value and IV is iodine value.

2.6.11 Determination of percentage Sulphur

10 g of the Eschka's mixture (mixture of magnesium oxide and calcium carbonate) and 2 g each of the KSO and biodiesel blends to be analyzed for Sulphur content was added to a nickel crucible. The crucible was placed in a furnace at 800 °C for 4 hours and then allowed to cool. The contents of the crucible are transferred to a beaker containing water, and 75 mL of 6 M HCl was added to neutralize all the oxides and carbonates. 1 milliliter of bromine water (bromine liquid dissolved in water) was added to the mixture to convert all of the sulfur compounds into sulfate. The solution was then heated to drive off any excess bromine. Finally, 10 mL of 0.1 M $BaCl_2$ was added to precipitate the sulfate as barium sulfate. A clean porcelain crucible was heated for an hour at 800 °C to remove any water. It was cooled in a desiccator and weighed. The barium sulfate precipitated by the barium chloride was collected by passing the solution through very fine filter paper. The filter paper and barium sulfate precipitate was placed in the crucible and the crucible slowly heated so that the filter paper does not catch on fire and spatter the barium sulfate out of the crucible. The crucible was heated at 800 °C for an hour, cooled in the desiccator, and weighed. Same procedure was repeated for the biodiesel blends. Hence, Sulphur content for each sample was calculated using Equation 8 (Mott and Wilkinson, 2007) below

$$S = \frac{\text{wt. of BaSo4 formed x 32}}{\text{wt. of oil sample taken x 233}} \tag{8}$$

Where the mass of $BaSO_4$ is the mass (g) of the crucible and barium sulfate weighed after being heated minus the mass (g) of the empty crucible weighed before the barium sulfate was added.

3 RESULTS AND DISCUSSIONS

3.1 Khaya Senegalensis Oil Biodiesel yield

The mean maximum yield of KSO biodiesel was obtained from the two step acid – alkaline transesterification and presented in table 2 below. 89.50% was obtain at a reaction conditions of

6:1 molar ratio, 0.84% (w/w) catalyst concentration, 70 °C reaction temperature and 60 minutes reaction time respectively.

Table 2: Mean Khaya Senegalensis Oil Biodiesel yield

Experiment number	Methanol/oil ratio	Catalyst conc. (% w/w)	Reaction temp. (°C)	Reaction time (min)	Biodiesel Yield (%)
1	6:01	0.84	70	60	89.67
2	6:01	0.84	70	60	89.49
3	6:01	0.84	70	60	89.33
Mean					**89.50**

3.2 Physicochemical Properties of Khaya Senegalensis Seed oil Biodiesel Blends

The physicochemical properties of the raw KSO and its biodiesel blends (B10, B20, B30, B40, B50 and B100) produced via transesterification are presented in Table 3 and compared with ASTM D6751 and EN 14214 standards for biodiesel fuel.

Table 3: Physicochemical Properties of Khaya Senegalensis Seed oil Biodiesel Blends

Properties	**B10**	**B20**	**B30**	**B40**	**B50**	**B100**	**KSO**
Density, Kg/m^3	844 (2.0)	851 (1.0)	848.33 (4.93)	852 (2.0)	851 (1.0)	837 (1.0)	908.7(2.52)
Kinematic Viscosity, mm^2/s	4.44 (0.01)	4.47(0.01)	4.51 (0.02)	4.53(0.02)	4.58(0.02)	4.07(0.59)	19.61(0.01)
Iodine Value, gI$_2$/100g	95 (2.0)	127 (0.2)	102 (2.0)	83.17(0.29)	70 (0.5)	92 (1.0)	296.8 (0.3)
Saponification Value, mg/KOHg	27.49(0.01)	33.66(0.03)	31.98 (0.02)	36.47(0.02)	29.17(0.02)	11.22(0.02)	23 (2.0)
Cloud Point, °C	14 (0)	12(0.1)	9.6 (0.1)	9.4(0.1)	9.5(0.1)	9 (0.1)	8.5(0.5)
Pour Point, °C	19(1.0)	17.5(0.02)	17 (0.5)	16 (1.0)	15 (1.0)	11(1.0)	12 (1.0)
Flash Point, °C	110(1.0)	115 (1.0)	112.33 (0.58)	121 (1.0)	118 (0.2)	190 (0.2)	295 (1.0)
Acid Value, mg/KOHg	0.71 (0.02)	0.35(0.006)	0.65 (0.03)	0.42 (0.02)	0.21(0.015)	0.78 (0.02)	2.19 (0.01)
FFA, %	0.36 (0.02)	0.17 (0.01)	0.33 (0.02)	0.21 (0.01)	0.11 (0.01)	0.39 (0.01)	1.12 (0.03)
HHV, MJ/Kg	46.87(0.02)	46.15(0.02)	46.75 (0.3)	46.69(0.02)	47.18(0.03)	47.59(0.02)	44.51(0.01)
Sulphur, mg/Kg	2.15 (0.02)	2.31 (0.01)	1.79 (0.02)	2.86 (0.02)	2.19 (0.02)	2.83 (0.02)	4.76 (0.02)

Values in bracket are standard deviation of the means.

3.2.1 Density

Biodiesel produced from *Khaya Senegalensis* (B100) has a density value of 837 Kg/m^3 at a temperature of 40 °C.The default value of 40°C density specified in ASTMD6751 and EN 14214 are 860 to 890, and 860 to 900 Kg/m^3 respectively. Density is one determinant of the quality of biodiesel as it pertains to the value of the generated heat and power diesel engines (Raymond et al., 2014). Density value obtained for KSO was obtained as 908.7 kg/m^3, which is slightly higher than both the ASTM D6751 and EN 14214 standards. However, the densities of the biodiesel blends decrease as shown in Table 3, with B10, B20, B30, B40 and B50 having values of 844, 851, 848.33, 852 and 851kg/m^3 respectively. When compared to the standards, the biodiesel from the *Khaya Senegalensis oil* can be included in the range of density values that have been set by the standards.

3.2.2 Kinematic Viscosity

Kinematic viscosity results obtained in this research work for KSO was 19.61mm^2/s, where the value which is higher than the standard value of kinematic viscosity range recommended in ASTM D6751 and EN 14214 are 3.5 to 5 and 1.9 to 6 mm^2/s respectively. While the viscosity values for biodiesel blends (B10, B20, B30, B40, B50 and B100) obtained are 4.44, 4.47, 4.51, 4.53, 4.58 and 4.07 mm^2/s respectively, and are within the range of specified standards. One of the causes of high and low kinetic viscosity is varying the catalyst concentration and temperature. If concentration of catalyst is high, so the viscosity will decrease, this is because the concentration of excess catalyst will accelerate the breakdown of fatty esters triglyceride into three grades which will reduce the viscosity by 5–10% (Raymond *et al*., 2014).

3.2.3 Iodine Value

The iodine value (IV) is a useful parameter in studying oxidative rancidity and chemical stability properties of different oil and biodiesel fuels. Oils with iodine value above 125 g I_2/100 g are classified as drying oils; those with iodine value of 110 – 140 g I_2/100 g are classified as semi - drying oils and those with iodine value less than 110 g I_2/100 g as considered as non-drying oil (Onyezeka *et al*., 2020). Biodiesel with high iodine numbers will produce esters with the flow and solidification at low temperature. Biodiesel which has a higher degree of unsaturation is not suitable for use as biodiesel because unsaturated molecules will react with oxygen from the atmosphere, be converted into peroxide crosslinking, result in the unsaturated, and cause biodiesel polymerized to form a similar plastic material, especially if the temperature increases. As a result, the diesel engine will not work properly and be damaged (Azam *et al*., 2005). From Table 3, it was observed that the iodine value of KSO was 296.8 g I_2/100 g, which is greater than the maximum standard value of iodine value of 120 and 130 g I_2/100 g recommended in ASTM D6751 and EN 14214 respectively. However, iodine values for B10, B20, B30, B40, B50 and B100 are 95, 127, 102, 83.17, 70 and 92 g I_2/100 g respectively. These values met the quality standards iodine number in ASTM D6751 and EN 14214 for biodiesel (Table 1).

3.2.4 Saponification Value (SV)

The Saponification value is the number of milligrams of potassium hydroxide required to neutralize the fatty acids resulting from the complete hydrolysis of 1g of oil. Table 3 present the saponification value of KSO as 23 mg/KOHg. The lower the molecular weight, the higher the saponification number and vice versa (Nirwana, 2012). The values for biodiesel blends B10, B20, B30, B40, B50 and B100 as presented are 27.49, 33.66, 31.98, 36.47, 29.17 and 11.22 mg/KOHg respectively. These values which are smaller than the standard values of saponification value in ASTM D6751 and EN 14214 standards for biodiesel of less than 500mgKOH/g. Based on these results biodiesel from *Khaya Senegalensis* has a low saponification value and enters the biodiesel quality control set by the standards.

3.2.5 Cloud Point (CP)

The cloud point (CP) is the temperature at which crystals first start to form in the fuel. The cloud point is reached when the temperature of the biodiesel is low enough to cause wax crystals to precipitate. The CP is the most commonly used measure of low-temperature operability of the fuel. Table 1 show the cloud point of KSO as 8.5 ^{o}C and KSO biodiesel blends B10, B20, B30, B40, B50 and B100 are 14, 12, 9.6, 9.4, 9.5 and 9 ^{o}C respectively. These values are within the standard value ranged of -3 to 10 ^{o}C and -3 to 12 ^{o}C for ASTM D6751 and EN 14214 standards for biodiesel (Table 1). This property is used to specify cold temperature usability of a fuel. The variation in cloud point of KSO biodiesel blends may be as a result of the ratio of fossil diesel in the blend, with B10 having the highest value and decreases as the percentage of diesel increases.

3.2.6 Pour Point (PP)

The pour point (PP) is the temperature at which the fuel contains so many agglomerated crystal that it is essentially a gel and will no longer flow. This occurs if the temperature of the biodiesel drops below CP, when the microcrystals merge and form large clusters, which may disrupt the flow of the biodiesel through the pipes of the engine's fuel system (Barabás and Todoruţ. 2011). Table 3 presents the pour points of KSO and biodiesel blends of KSO. The pour point of KSO was obtain as 12 ^{o}C, with a variation in the values of its blends B10, B20, B30, B40, B50 and B100 as 19, 17.5, 17, 16, 15 and 11 ^{o}C respectively. B40, B50 and B100 values are within the ASTM D6751 and EN 14214 standards of -3 to 16 ^{o}C and -15 to 16 ^{o}C, while, the values for B10, B20 and B30 are slightly above the quality Standards for biodiesels. Although CP and PP are relatively easily determined, they only provide indicative values for the minimum temperature at which the fuel can be used.

3.2.7 Flash Point (FP)

Flash point is one of the most important properties of biodiesel fuel. It indicates the minimum ignition temperature of the fuel. Higher flash point makes fuel safer for handling and storage and prevent unexpected ignition of fuel during combustion (Arbab *et al.*, 2013). It is used to classify fuels for transport, storage and distribution according to hazard level. The flash point does not affect the combustion directly; higher values make fuels safer with regard to storage, fuel handling and transportation. Table 3 shows the flash points of KSO, B10, B20, B30, B40, B50 and B100 as 295, 110, 115, 112, 120, 118 and 190 ^{o}C respectively. The flash point values for KSO and B100 are higher than maximum recommended values of 130 and 120 ^{o}C respectively for ASTM D6751 and EN 14214 standards for biodiesel. Flash point limitation in biodiesel is imposed to ensure that no methanol is left in the fuel. Low flashpoint can cause engine ignition problems (Tsoutsos *et al*., 2018).

3.2.8 Acid Value (AV)

Acid value is defined as the number of milligrams of potassium hydroxide required to neutralize the free fatty acid (FFA) presents in 10 ml of the sample oil. Acid value is a pointer of the presence of free fatty acids or acids formed as a result of the degradation that may occur during storage (Anguebes-Franseschi *et al*., 2019). The standards ASTM D6751 and EN 14214 (Table 1) establishes a maximum value of 0.8 and 0.5 mg KOH/g respectively. The acid value of KSO obtained is 2.19 mgKOH/g which is higher than the maximum standards (Table 3). The acid values for KSO biodiesel blends B10, B20, B30 B40, B50 and B100 reported are 0.71, 0.35, 0.65, 0.42, 0.21 and 0.78 mgKOH/ g respectively. Acid value exceeded the ASTM D6751 0.5 mg KOH/ g limit in B10, B30 and B100 blends, which is probably because of either the presence of free fatty acids in the final product due to incomplete reaction or because of the insufficient washing/separation in the neutralization phase of the catalyst (Tsoutsos *et al*., 2018).

3.2.9 Free Fatty Acid (FFA)

The free fatty acid of KSO of 1.12% is presented in Table 1, which is higher than 0.45% recommended in ASTM D6751 biodiesel standards (Table 1). The higher the free fatty acids, the lower the quality of diesel fuel. High free fatty acids may also reduce the life of the pump and filter (Raymond *et al.*, 2014). Free fatty acid value of KSO biodiesel blends B10, B20, B30 B40, B50 and B100 results of this research in which the value of 0.36, 0.18, 0.33, 0.21, 0.11 and 0.39% respectively, met the standard levels in ASTM D6751.

3.2.10 Higher Heating Value (HHV)

The suitability of biodiesel as alternative fuel is signified by calorific value as it specifies energy density of fuel (Nithyananda *et al.*, 2019). Table 3 presents the HHV for KSO as 44.51 MJ/kg which is less than the established ASTM D6751 45.18 MJ/Kg, but higher than 35 MJ/Kg of EN 14214 quality standards. A Higher heating value for fuel is desired because it facilitates the heat release during combustion and improves engine performance. However, the HHV values for KSO biodiesel blends B10, B20, B30, B40, B50 and B100 varies as the blend ratio increases, with mean values as 46.87, 46.15, 46.75, 46.69,47.18 and 47.59 MJ/Kg respectively. The higher heating values of biodiesel blends (46.15 – 47.59 MJ/kg) are higher than that for ASTM D6751 and EN 14214 standards for biodiesel (Table 1).

3.2.11 Sulphur Content

The engine combustion of fuels that contain sulphur leads to the formation of Sulphur dioxide emissions and particulate matter. Sulphur limits are generally imposed for environmental reasons. Table 3 shows the sulphur content for KSO and KSO biodiesel Blends (B10, B20, B30, B40, B50 and B100) were 0.047, 0.0215, 0.0231, 0.0179, 0.0286, 0.0219 and 0.0283 mg/kg respectively, a very low value compared with the maximum value of 10 mg/kg established by the quality standards ASTM D6751 and EN 14214. High sulphur levels in the diesel may give rise to the production of H_2SO_4 and sulphates compounds in the engine. H_2SO_4 causes corrosion in the engine, while sulphates lead to increased particulate matter emissions (Tsoutsos *et al.*, 2018).

4 CONCLUSION

From the study carried out, it was found that KSO has a high acid value which necessitate the use of two step acid – alkali transesterification process in the production of biodiesel. The maximum yield obtain was 89.49% when sodium hydroxide (KOH) catalyst was used. The biodiesel was blended at 10, 20, 30, 40, 50 and 100% and the physical and chemical properties (density, kinematic viscosity, iodine value (IV), saponification value (SV), cloud point (CP), pour point (PP), flash point (FP), acid value (AV), free fatty acid (FFA), Higher heating value (HHV) and Sulphur content) of both the KSO and its biodiesel blends were determine using standard methods. Though, most of the properties were within the ASTM D6751 and EN 14214 standards for biodiesel, some blends had a slightly higher or lower values than the established ASTM D6751 or EN 14214 standards. The higher heating values for the biodiesel blends ranged between 46.52 – 47.59 MJ/kg above the reported standards. On the other hand, sulphur contents for both the KSO (0.0470 mg/kg) and its biodiesel blends (0.0215 – 0.0283 mg/kg) were all lower with respect to values established by the standards. Hence, it is recommended that performance characteristics of the biodiesel blends be investigated on a diesel engine to evaluate which of the blends is suitable and will be adopted as an alternative energy fuel.

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

Acknowledgements: Authors acknowledges Agricultural Mechanization and Sustainable Environment Programme and Centre of Excellence in Agricultural Development and Sustainable Environment (CEADESE), Federal University of Agriculture, Abeokuta, Mr. Emmanuel Joseph and Abubakar Habib Idris of the Department of Chemistry, Abubakar Tafawa Balewa University Bauchi, for assistance in carrying out some laboratory analyses.

REFERENCES

ACEA, (2009). “Biodiesel Guidelines”, European Automobile Manufacturers Association, Brussels, Belgium.

Anguebes-Franseschi, F., Bassam, A., Abatal, M., May Tzuc, M., Aguilar-Uc´an, C., Wakida-Kusunoki, A. T., Diaz-Mendez, S. E. and San Pedro, L. C. (2019). Physical and Chemical Properties of Biodiesel Obtained from Amazon Sailfin Catfish (Pterygoplichthys pardalis) Biomass Oil. *Hindawi Journal of Chemistry*, Article ID 7829630, 12 pages.

Anwar, F., Naseer, R., Bhanger, M., Ashraf, S., Talpur, F. N. and Aladedunye, F. A. (2008). Physico-chemical Characteristics of Citrus seeds and seed oils from Pakistan. *J. Am. Oil Chem. Soc*., 85, 321–330.

AOAC (2000). Official Methods of Analysis, 17th edn. Association of Official Analytical Chemists, Inc, Maryland USA.

Arbab, M. I., Masjuki, H. H.., Varman, M., Kalam, M. A., Imtenan, S. and Sajjad, H. (2013). Fuel properties, Engine Performance and Emission Characteristic of Common Biodiesels as a Renewable and Sustainable source of Fuel. *Renewable and Sustainable Energy Reviews* 22, 133-147.

Rogers, A. (2004). *Khaya senegalensis* – current use from its natural range and it’s potential in Sri-Lanka and elsewhere in Asia Working Paper presented at a Workshop on "Prospects for high-value hardwood timber plantations in the 'dry' tropics of northern Australia", Mareeba, Queensland: 1 - 9**.**

American Society for Testing and Materials (ASTM) (2002) Water and Environmental Technology. Annual Book of ASTM Standards, Sec., 11: 11.01 and 11.02, West Conshohocken.

ASTM (2003) American Standards for Testing of Materials. D 189-01, D 240-02, D 4052-96, D 445-03, D 482-74, D 5555-95, D 6751-02, D 93-02a, D 95-990, D 97-02.

Auwal A., Elisha, L. and Abdulhamid, H. (2012). Production of Biodiesel via NaOH Catalyzed Transesterification of Mahogany seed oil. *Advances in Applied Science Research,* 2012, 3 (1):615-618.

Awolu, O. O. and Layokun, S. K. (2013). Optimization of Two-step Transesterification Production of Biodiesel from Neem (Azadirachta indica) oil. *International Journal of Energy and Environmental Engineering*, 4(39): 1 – 9.

Azam, M. M., Waris, A. and Nahar, N. M. (2005). "Prospects and potential of fatty acid methyl esters of some non-traditional seed oils for use as biodiesel in India," *Biomass and Bioenergy*, 29(4): 293 – 302.

Babakura, M., Jibrin, M. Y., Abubakar, I., Bashir, M. A., Jibrin, Y. Y., Jamilu, B. U, Musa, M. A. and Alexender, O. O. (2019). Synthesis and Characterization of Biodiesel from Khaya senegalensis Seed Oil using Heterogeneous Catalyst. *International Journal of Science and Management Studies (IJSMS)*, 2(4): 101 – 107.

Barabás, I. and Todoruţ, I. (2011). Biodiesel Quality, Standards and Properties, Biodiesel-Quality, Emissions and By-Products, Dr. Gisela Montero (Ed.), *InTech*: 3 – 28. Available from: by-products/biodiesel-quality standards-and-properties.

Berthiaume, D. and Tremblay, A. (2006). Study of the Rancimat Test Method in measuring the Oxidation Stability of Biodiesel Ester and Blends. NRC a project no. CO414 CETC-327, OLEOTEK Inc., Québec, Canada.

Benjumea, P., Agudelo J. and Agudelo, A. (2008). Basic properties of palm oil biodiesel–diesel blends. *Fuel,* 87: 2069–2075.

Danbature, W. B., Jauro, A. and H. M. Adamu (2015). Production of Methyl ester from Mahogany (*Khaya senegalensis*) seed oil. *Journal of Chemistry and Chemical Sciences.* 5(9): 489-499.

Dasin, D. Y., Felix, R. and Yahuza, I. (2019). Production and Investigation of Physico – Chemical Properties of Biodiesel Derived from Mahogany (Khaya Senegalensis) Seed oil as Alternative Fuel for C.I. Engines. *Journal of Multidisciplinary Engineering Science and Technology (JMEST)*, 6(5): 10068 – 10074.

Demirbas, A. and Kara, H (2006). New options for Conversion of Vegetable oils to Alternative Fuels. *Energ. Sour*. Part A 28, 619-626.

Demirbas, A. (2009). Characterization of Biodiesel Fuels. Energy Sources, Part A, 31:889–896.

Drapcho C. M., Nhuan N. P. and Walker T. H. (2008). Biofuels Engineering Processing Technology, New York: McGraw – Hill. Pp. 197-262

Giwa S. O., Sulaiman Y., Maryam I. and Giwa, A. (2016). Extraction of Oil from Jatropha Seed Kernels: Optimization and Characterization. *International Journal of ChemTech Research*, 9(05): 758 – 770.

Ibrahim, H., Fasanya, O. O., Aminu, H. and Evidence, O. O. (2018). Fatty Acid Composition of Mahogany Seed Oil and its Suitability for Biodiesel Production. *Nigerian Journal of Technological Research,* 13(1): 45-49.

Knothe, G., Van Gerpen, J. and Krahl, J. (2005). *The Biodiesel Handbook*, (eds.), AOCS Press, Champaign, Illinois, 302 pp.

Knothe, G. (2006). Analyzing Biodiesel: Standards and Other Methods. Review, *JAOCS*, 83 (10): 823 – 833.

Lalas, S. and Tsaknis, J. (2002). Characterization of *Moringa oleifera* seed oil variety "Periyakulam 1". *J. Food Compos Anal.*15(1): 65–77.

Maina, P. (2014). Engine emissions and combustion analysis of biodiesel from East African countries. *S. Afr. J. Sci.* 110(3/4): 1 – 8.

Mesri Gundoshmian, T., Heidari-Maleni, A. and Jahanbakhshi, A. (2021). Evaluation of performance and emission characteristics of a CI engine using functional multi-walled carbon nanotubes (MWCNTs-COOH) additives in biodiesel-diesel blends. *Fuel*, 287:119525.

Mott, R. A. and Wilkinson, H. C (2007). The use of Eschka's method for the determination of high sulphur contents. *Journal of applied chemistry* 3(5): 218 – 223.

Nirwana, I. H. S. (2012). Effect of Stirring Speed for Biodiesel Production from Jatropha Oil (Jatropha Curca L) by Using Catalysts Abu Tandan Sawit Research Institue, Riau University, Riau, Indonesia.

Nithyananda, B. S., Anand, A., Prakash, G. V. N., Vinay, K. B. and Naveen, A. (2019). Effect of Simarouba Biodiesel on Performance and Emissions of CI Engine. *International Journal of Recent Technology and Engineering (IJRTE),* 8(4S5): 170- 175.

Onukwuli, D. O., Lovet N. E., Callistus N. U., Sandra O. A. and Mathew C. M. (2017). Optimization of Biodiesel Production from Refined Cotton seed oil and its Characterization. *Egyptian Journal of Petroleum. 26, 103-110.*

Onyezeka, E.G., Nnaji, J. C., Omotugba, S. K. and Garba, M. H. (2020). Physicochemical Properties of Biodiesel Produced by Transeterification of Various Esterified Waste Frying Oils. *Nigerian Journal of Chemical Research*, 35(1): 44 – 56.

Pramanik, K., (2003). Properties and use of Jatropha curcas oil and Diesel Fuel Blends in Compression Ignition Engine, *Renewable Energy*, 28: 239–248.

Ramadhas, A. S., Jayaraj, S. and Muraleedharan, C. (2005). "Biodiesel production from high FFA rubber seed oil", *Fuel*, 84:335 – 340.

Rashid, U., Ibrahim, M., Yasin, S., Yunus, R., Taufiq-Yap, Y. and Knothe, G. (2013). Biodiesel from *Citrus reticulate* (Mandarin orange) seed oil, a Potential non-food feedstock. *Ind. Crops Prod.* 45: 355–359.

Raymond, K., Indah, R. and Muhammad, Z. (2014). Production of Biodiesel from Lipid of Phytoplankton *Chaetoceros calcitrans* through Ultrasonic Method. *The Scientific World Journal*, Article ID 231361, 1 – 5.

Samadi. S, and Heidarbeigi, K. (2020). Acoustic analysis of a single-cylinder diesel engine using magnetized biodiesel-diesel fuel blends. Heliyon, 6(9):e05113.

Sakthivela, R., Ramesh, K., Purnachandran, R. and Mohamed Shameer, P. (2018). A review on the properties, performance and emission aspects of the third generation biodiesels. *Renewable and Sustainable Energy Reviews,* 82: 2970–2992.

Srivastava, A., and Prasad, R. (2000). Triglycerides-based diesel fuels. *Renew. Sust. Energy Rev*. 4: 111–133.

Tsoutsos, T., Stavroula T., Zacharias, G., Orlando, P., Filippo, G., Pablo, Q. G., João, B., Haris, A. and Monica, F. (2018). Quality Characteristics of Biodiesel Produced from used Cooking Oil in Southern Europe. *Chem Engineering,* 3(19): 1 – 13.

Yesilyurt, M. K., Zeki, Y. and Mustafa, A. (2020). The Performance, Emissions and Combustion Characteristics of an Unmodified Diesel Engine running on the Ternary Blends of Pentanol/safflower oil Biodiesel/diesel Fuel. *Journal of Thermal Analysis and Calorimetry* 140:2903-2942.

Yesilyurt, M. K., Mevlüt, A. and Tanzer, E. (2018). Application of response surface methodology for the optimization of biodiesel production from yellow mustard (*Sinapis alba* L.) seed oil, *International Journal of Green Energy,* 16(1):1 - 12.