Production And Investigation Of Physico - Chemical Properties Of Biodiesel Derived From Mahogany (Khaya Senegalensis) Seed Oil As Alternative Fuel For C.I. Engines

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Abstract—**This research presents Khaya senegalensis (mahogany) seeds. Oil was extracted by traditional method and base catalyst transesterification using methanol as solvent in the presence of a catalyst to produce biodiesel. The yield of the oil and the biodiesel produced was determined and found to be 251.5 ml and 545.6 ml respectively. The conventional diesel fuel (AGO) and ASTM standard procedures were used to determine the physicochemical properties of the biodiesel, which include the viscosity, density, pour point, cloud point and flash point. Predictions of four important chemical properties of biodiesel viz acid value, iodine value, saponification number and cetane number were performed based on fatty acid profiles of khaya senegalensis biodiesel. Acid value obtained was found to be 23.2 mg KOH/g and is higher compared to diesel fuel (AGO) and ASTM standard fuels. Iodine value of biodiesel was found to be 12.995 g of oil which is below than AGO with 18.4 g and saponification number of the biodiesel derived from kyaha senegalensis was determined to be 52.45. The cetane index of kyaha senegalensis biodiesel obtained is 156.5, which is higher than AGO and ASTM, The methyl esters and triglycerides values was 95.52% and 0.25% respectively, determined by high performance liquid chromatography (HPLC) used to monitor the process from the results obtained by base catalyzed transesterification used on khaya senegalensis oil to produce biodiesel, the triglycerides detected and by using free fatty acids as standard. It was determined that free fatty acids were absent after transesterification. After triglyceride and free fatty acids, these were converted to methyl ester that's make value about 95.52%.**

Keywords—*Mahogany, Transesterification, Biodiesel, Triglyceride, Methyl ester.*

1. INTRODUCTION

The society is continuously developing and evolving to enhance daily life. Petroleum based fuels have been used as one of the main sources of energy, however diesel or fossil fuel sources are projected to diminish as population increases. The petroleum reserves are not uniformly distributed and their increased use contributes to a variety of local and regional air pollution problems and potential climatic changes [1]. 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. The energy trend offers a challenge as well as an opportunity to look for substitutes of petrol-diesel fuels for both economic and environmental benefits. 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 commands crucial advantages such as technical feasibility of blending, superiority from the environment and emission reduction angle, its capacity to provide energy security to remote rural areas and employment generation [2, 3].

The petroleum base diesel is non-renewable and pollutes the environment by emitting greenhouse gases. In an attempt of finding solution to this predicament, researches into a renewable and less polluting form of fuel known as biodiesel becomes a necessity. 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.) [4].

The biodiesel from non-edible oil of mahogany is renewable and does not contribute to global warming due to its closed carbon cycle. Climate change due to global warming has its links with the effects of environmental degradation (for example, CO_2 emissions from petroleum based diesel fuels).

This mahogany seed oil or any form of oil from plants or animals cannot be used directly in diesel engines because of their properties. Transesterification method has been widely used to reduce the viscosities and improve the fuel properties of the mahogany seeds oils (*swietenia macrophylla* seeds) [5]. Transesterification is the chemical reaction between triglycerides and alcohol in the presence of catalyst to produce mono-esters. Most of the biodiesel produced worldwide is based on transesterification in homogeneous media with sodium methoxide as the reaction catalyst [5, 6].

Several researchers have reported on the production of biodiesel from different seed oil [6]. Recent research has concentrated on the study of a wide variety of feedstock for biodiesel [7]. For example, Kamal et al. [8] studied oil extraction from citrus seed and determined its chemical composition. Anwar et al. [9] investigated physico-chemical properties of citrus seeds and oils, and also analysed the fatty acid composition of the oil. Rashid et al. [4] obtained biodiesel from citrus seed oil and analysed the fatty acid composition of the fuel. Biodiesel is an alternative fuel which can be used by blending up to 20% with diesel without requiring any modification of diesel engines. Biodiesel production from seeds follows process steps of seed preparation, oil extraction, pre-treatment, and oil to biodiesel conversion.

2. MATERIALS AND METHODS

2.1 Materials and Equipment

The materials and Equipments used for the research are as follow:

Raw samples of seed, Potassium hydroxide, granular, Methanol, Isopropyl alcohol (99%), 0.01 M hydrochloric acid, 1% phenolphthalein in 95% ethanol, 1% bromophenol blue in water, 3 liter of distilled water, Potassium iodine solution, Sodium thiosulfate solution and Fire wood.

Some of the equipment used for the research are; Weighing machine, Pestle and mortar, grinder, Fine potash, Pan, Pot, plastic pipettes, half pint jars with sealing lids, disposable glass pipettes, burette setups with funnels, beakers, beakers, Erlenmeyer flasks, thermometer, digital scale, Stirring machine, Stop clock, viscometer, high performance liquid chromatography and tin layer chromatography, Bomb calorimeter and Pensky Martens Flash Point apparatus.

2.2 Methods

2.2.1 Collection Sampling

The *Khaya Senegalensis* (seeds) was obtained from Julvo town in Mubi Local Government area of Adamawa state, Nigeria. The period at which the seeds are considered matured is from February to March of every year. The pods are collected and sun dried for at least two day, the seeds were then collected after breaking the dried pods using either a hammer or stone. The collected seeds are further sun dried for at least three days to remove all the moisture content for processing as shown in figures 1 (a) and (b).

a. Mahogany fruit

b. Mahogany seeds from the fruit

Figure 1: Sample Fruit and Seeds

2.2.2 Oil Extraction Process

Pre-extraction process

A 900 ml pan was set on fire and heat the de-husked seeds for 20 minutes. Thereafter, it was air-roasted for 20 minutes. The roasted seeds were removed from the pan and kept under shade for 30 minutes. Finally, the seeds were crushed with pestle

and mortar and further grinded into fine powdery form by the use of a traditional grinding stone.

Maize stock was collected and burnt to ashes, the ashes were collected, weighed and mix together with certain quantity of water. The mixture was filtered and the solution was collected.

Extraction of oil

Traditional method was used in the extraction of the oil. During this, the fine grinded *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.

2.2.3 Base Catalyzed Transesterification

A Trans-esterification reaction was carried out in the 1500 ml reactor beaker placed upon a mechanical stirrer as shown in Figures 2 (a) – (e). A thermostat was used for maintaining constant reaction temperature. Thermometer and stop watch were used to monitor the reaction.

During the transesterification process, the 1500 ml reactor beaker was initially charged with 700 ml of *Khaya Senegalensis* oil and then heated to 65^0C, Potassium hydroxide (5.0 g) was dissolved in the 175 ml methanol and the solution was added to the reactor flask, The mixture (potassium hydroxide-methanol solution) was heated to 65^0C and maintained at constant temperature of 65^0C for one hour in the reactor. During this process, samples were taken every 15 minutes and placed in clean test tubes. A total of four samples were taken in order to determine the optimum time needed for the biodiesel to separate from glycerol. The samples were immediately quenched in water to stop the reaction, and two phases were formed for each. The biodiesel layer was then carefully separated using separation funnel.

The upper layer consist of methyl esters, the mid layer are biodiesel and traces of triglycerides and the lower layer contains glycerol, the exits methanol and the remains of catalyst together with the soap formed during the reaction.

a. Heating oil

b. weigh of KOH

c. mixes of catalyst

d. stir mixed oil catalyst

e. final product

Figure 2: Production of biodiesel and separation.

2.2.4 Biodiesel Washing and Drying

Washing of obtained biodiesel was done using a 500 ml separation funnel as shown in Figure 3. For every 200 ml of oil, 250 ml of distill water was used. The distill water was heat to 50^0C to avoid effect on the PH of the biodiesel. The warm water was pour gently into the separation funnel.

The separation funnel was shaken gently until milky mixture was formed. To allow the wash biodiesel to settle faster, the separation funnel was carefully placed into water bath and allowed to settle for 2 hours (the water bath was used to enable the tiny bubbles formed by the water in the biodiesel to clear faster and hence easy for separation).Washing was repeated until clear clean water separated was obtained. The cleaned washed biodiesel was transferred into a clean 2 liter pot and heated to the temperature of 180^0C to dry it. A clear golden yellow biodiesel was obtained.

Stages 1 – 3; Washing of Biodiesel,

Stage 4; Drying of biodiesel, Stage 5; final product.

Figure 3: Washing and Drying of Biodiesel

2.3 Determination of Biodiesel Physico chemical Properties

The physicochemical properties of mahogany biodiesel were investigated for alternative use in diesel engines.

2.3.1 Calorific Value (Heating)

The procedures to determine the calorific value is by using a bomb calorimeter as shown in Figure 4, the empty crucible was weighed using optical beam balance and the mass was recorded. The crucible was filled with fuel then it was re-weighed and the mass of the filled crucible was determined.

Then the crucible with the fuel was placed in the fire apparatus of the calorimeter. The bomb calorimeter was then assembled, charged with oxygen up to 25 atms and the stirrer turned on. The temperature read was recorded at intervals of one minute for five minutes to make sure that the temperature of the bomb was stable and then the bomb was fired. A temperature was recorded at an interval of half a minute for the entire range of the temperature raised and at one minute interval for five minutes after temperatures starts to drop.

Figure 4: Constant Volume Bomb Calorimeter

2.3.2 Acid value or free fatty acids (FFA)

This is the number of KOH required to neutralize the free fatty acid present in 1.0 gm of the sample. The acid value was calculated using the following equation [10].

$$AV = (V_{KOH} - B_{KOH})n\frac{56.1}{W_{oil}}$$

Where;

V_{KOH} = Amount of KOH (ml) consumed by the oil sample at the equivalent.

B_{KOH} = Amount of KOH consumed by the blank sample.

n = Concentration of KOH

W_{oil} = mass of the oil sample used

The molecular mass of KOH is 56.1

2.3.3 Pour point

The 5 ml oil Sample was poured into a 10 ml test tube and clamped to a retort stand. The set up was placed in a refrigerator and allowed to solidify. After it solidifies, the test tube was removed and a thermometer was used to read the temperature at which the solidified sample begins to melt and flow. This temperature was noted and recorded as the pour point of the oil sample.

2.3.4 Cloud point

The 5 ml oil Sample was poured into a 10ml test tube and placed in a test tube holder. The set up was placed in a refrigerator and allowed till it form cloud nature in the test tube. After it cloudiness the test tube was removed and a thermometer was used to read the temperature at which the cloudiness sample begins to start. This temperature was noted and recorded as a cloud point of the oil sample.

2.3.5 Iodine Value

The most important analytical determination of oil is the measurement of its unsaturation. The generally accepted parameter for expressing the degree of carbon to carbon unsaturation of oil or their derivatives is iodine value. Iodine value is defined as g of iodine absorbed by 100g of oil. It is useful

parameter in studying oxidative rancidity of triacylglycerols since, higher the unsaturation, greater is the possibility of rancidity [10].

The weight of 0.5 ml of the oil sample was weighed accurately into a 250 ml Erlenmeyer flask and 20 ml of carbon tetrachloride was added into the flask. The mixture of 25 ml of reagent was pipette into it. The flask contained the mixture was swirled and stored in dark and at room temperature for 30 minutes. At the end of 30 minutes, 10 ml of 30% potassium iodide solution was added to the sample solution followed by 100 ml of purifying water. The content was titrated with standard 0.1 M sodium thiosulfate solution until the yellow color almost disappears.

To continue the titration 1 ml of 1% starch indicator solution was added and swirled until the blue starch-iodine color disappears. The Iodine value was determined using the expression below.

$$Iodine\ Value = \frac{Blank\ Titer\ (ml) - Sample\ Titer\ (ml)\ x\ (1.269)}{Weight\ of\ Sample\ (g)}$$

2.3.6 Kinematic Viscosity

Kinematic viscosity of sample was measured with the help of Ostwald viscometer size D BS/U 790. Time of gravity flow of fixed value (50 ml) of sample was measured. The experiment was performed at temperature of 15 °C to 60 °C at increment of 5 °C. Kinematic viscosity was calculated using the following formula.

$$\frac{V_f}{V_w} = \frac{\rho_f t_f}{\rho_w t_w}$$

Where;

$\mathcal{V}_f$ = viscosity of the fuel sample at a given temperature

$\mathcal{V}_w$ = viscosity of water at a given temperature

ρ_f = density of the fuel sample at a given temperature

ρ_w = density of water at a given temperature

t_f = time taken for fuel sample to run

t_w = time taken for water to run

2.3.7 Flash point

Flash point of a fuel is defined as the temperature at which it will ignite when exposed to flame or spark. The flash point of sample was determined by Pensky Martens Flash Point apparatus. It was determined by heating the sample of the fuel in a stirred container and passing a flame over the surface of the liquid until the vapour ignites. The temperature at which the vapour ignites was noted and recorded as the flashpoint.

2.3.8 Saponification Value

Saponification value is the measure of the molecular weight of the fatty acid. A known quantity of oil is refluxed with an excess amount of alcoholic KOH. 2 g of the sample (*Khaya Senegalensis* oil) was weighed and transferred into a conical flask and 50 cm^3 of 0.5 N ethanolic KOH (that has stayed overnight was added to the sample. The mixture was heated to saponify the oil. The unreacted KOH was then being blank titrated with 0.5 M hydrochloric acid using 2 to 3 drops of phenolphthalein indicator The SVvalue of the samples analyzed was calculated thus:

$$SV = \frac{Blank\ titre\ value\ (dm^3) - Sample\ titre\ value\ (dm^3)(Mormalilty\ of\ NaOH)(56.1)}{Weight\ of\ sample\ (g)}$$
$$= \frac{mgKOH}{g}$$

2.3.9 Cetane Number

The calculated saponification value (SV) and iodine value (IV) were used to calculate the cetane number (CN), which is the ability of fatty acid methyl esters as a fuel to ignite quickly after being injected. Empirical formula as proposed by [11] was used in the work. The higher its value, the better is its ignition quality. This is one of the most important parameter which is considered during the selection of fatty acid methyl esters for use as a biodiesel.

$$Cetane\ Number = 46.3 + \frac{5458}{[SV - 0.225\ x\ IV]}$$

2.3.10 Ash Content

This is the alkaline catalyst residues remain after a fuel sample was carbonized and the residue subsequently treated with sulfuric acid and heated to the constant weight. It is the measured of the mineral ash residue when the fuel was burnt. It is an important test for biodiesel because it indicates the quantity of residue in the fuel that came from the catalyst used in the transesterification process.

The sulfated ashes for FAME are 0.010 % (mol/mol) and lower than the 0.05 ASTM maximum limits [12].

3. RESULTS AND DISCUSSIONS
3.1 Results

The results for the biodiesel yield from the mahogany seed oil (*Khaya Senegalensis*) at various catalyst concentration levels are presented in Table 1.

Table 1: Biodiesel yield from *Khaya Senegalensis* at various catalyst concentration levels

Catalyst amount KOH (g)	Concentration of catalyst KOH % w/v	Product volume (ml)	Biodiesel yield fraction (ml)
1.1	32.8	282.8	251.5
2.0	64.6	389.6	295.3
3.3	97.4	522.4	390.4
4.0	130.2	630.2	425.3
5.1	162.2	812.2	499.99
5.5	175	875	545.6

Table 2 Shows a comparison of the physicochemical properties of biodiesel (mahogany methyl ester (MME)) obtained in this research work, with product specification from petroleum based diesel or automotive gas oil (AGO) and American standard for testing of material (ASTM).

Table 2: Physico-chemical properties of biodiesel fuel compare with various standards.

Property	Unit	MME	AGO	ASTM D6751-06
Density (ρ) at 20 ^{0}C	g/cm^3	0.880	0.8393	0.20 – 0.90
Pour point	℃	-7.73	-16.1	-15 to -16
Viscosity at 40℃	Cps	4.86	2.9	1.9 - 6.0
Flash point	℃	81.2	16.5	<130
Acid value	mgKOH/g	0.232	-	0.5 max
Ash content	%	0.02	<0.01	-
Heating value	MJ	36.12	43.6	45.18
Moisture content	%	0.08	0.02	0.016
Saponification value	mgKOH/g	52.45	-	-
Iodine value	gI$_2$/100g	12.995	18.4	-
Cloud point	℃	- 4.1	-12.2	-3 to -12
Methyl ester	%	95.52	-	-
Triglycerides	%	0.25	-	-
Cetane number	-		47.4	49	47 - 69

3.2 Discussion of Results

From table 1, the results indicated that methyl ester and glycerin are influenced by the intervals (two hours, in this case) of reaction time. The table indicated that transesterification for 12 hours increased the yield of methyl ester from 251.5 ml to 545.6 ml. This represents an increase in the percentage of methyl ester during the base catalyst transesterification reaction.

3.2.1 Physicochemical Properties of Fuel

The Iodine value was determined to be 12.995 gI$_2$/100g. The result of iodine value of obtained biodiesel revealed a lower value compared to that of the conventional diesel fuel (AGO). This shows that conventional diesel is more stable compared to the obtained biodiesel. However, the double bonds in the biodiesel help to attract oxygen to the compound, and aid proper burning of biodiesel over pure diesel fuel. More so, Iodine value is used to calculate the cetane number of the biodiesel.

Acid value is defined as the number of milligrams of potassium hydroxide required to neutralize the free fatty acid (FFA) presents in 10ml of the sample (*khaya senegalensis* biodiesel). The acid value was determined to be 0.232 mgKOH/g. The test was done in typical titration method with 0.1M potassium hydroxide (KOH) solvent with 50 ml of isopropyl alcohol and the concentration wormed. As the concentration of biodiesel increases in the fuel

samples, the acid value increases. Temperature also has an effect on the acid value. If fuel is exposed to high temperature, oxidation occurs due to higher rate of reaction of fuel molecules with oxygen in the air, resulting in increase of acid value.

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. It was determined to be 52.45 mgKOH/g. It gives information concerning the character of the fatty acids of the oil- the longer the carbon chain; the less acid is liberated per gram of oil hydrolyzed. It is also considered as a measure of the average molecular weight of all the fatty acids present. The oil extracted is characterized by long chain fatty acids, hence low saponification value because they have a relatively fewer number of carboxylic functional groups per unit mass of the oil and therefore high molecular weight.

The cetane number of the produced biodiesel was determined to be 47.4 less than that of AGO. However, the standard value as specified by ASTM is within the range of 47 – 69. Increase in cetane number, increases the ignition quality of an engine and decreases the knocking tendency.

The density obtained as shown in table 2 is 0.880 g/cm^3 for the *khaya senegalensis* biodiesel at room temperature. This density was higher compared to that of conventional diesel which is 0.839 g/cm^3. However, is within the rate specified by ASTM D6751-06 standard. This is in agreement with what is obtained from literature as reported by [13], that Biodiesel fuels are characterized by higher density than conventional petroleum diesel, which means that volumetrically operating fuel pumps will inject greater mass of biodiesel than conventional diesel fuel.

The viscosity of *khaya senegalensis* biodiesel as seen from Table 2 was obtained to be 5.63 cP which is higher than the petro - diesel and within the range of specified ASTM standard. [14], reported that the viscosity of biodiesel is typically higher than that of petro-diesel often by factor two, the viscosity increases as the percentage of biodiesel increases. Viscosity is greatly affected by temperature; many of the problems resulting from high viscosity are most noticeable under low ambient temperature and cold start engine condition.

The flash point of the *khaya senegalensis* biodiesel was determined to be 81 ^{0}C as seen from Table 2, which is in agreement with the specified ASTM standard of <130^{0}C. The higher the flash point the safer the fuel and vice versa. The result obtained indicates that the produced biodiesel falls under the non-hazardous category of ASTM D6751-06.

The Cloud and Pour point of *khaya senegalensis* biodiesel produced are above the standard limit of biodiesel according to ASTM D standard. The Cloud and Pour points are -4.1^{0}C and -7.73 ^{o}C respectively, might give rise to low running problems in cold

season. This problem could be overcome by the addition of suitable cloud and pour point depressants or by blending with the conventional diesel fuel.

The methyl esters and triglycerides values was 95.52% and 0.25% respectively, which is determined by high performance liquid chromatography (HPLC) used to monitor the process from the results obtained by base catalyzed transesterification used on *khaya senegalensis* oil to produce biodiesel, the triglycerides detected and by using free fatty acids as standard, it was also determined that free fatty acids were also absent after transesterification. After triglyceride and free fatty acids, these were converted to methyl ester that's make value about 95.52%.

4. CONCLUSION

Based on the findings of this study the following conclusions are drawn:

1. It was found that the feed stocks with high FFA could not directly be trans-esterified with KOH catalyst. The reason for this is because KOH catalyst reacts with the FFA to form soap that prevents the separation of the glycerine and ester. The high FFA level of crude *Khaya Senegalensis* can be reduced to less than 2% in pretreatment process of esterification using acid catalyst at 65 °C temperature and 1 hour reaction time.

2. The biodiesel production by methanol of *Khaya Senegalensis* oil can be undertaken at ambient conditions. This is interesting due to the energy savings and reduction of the operating cost of production.

3. Under optimum operating conditions described in this study, a maximum biodiesel yield of 91% was obtained. Thus, this research work promotes production of biodiesel using non edible seeds.

4. The biodiesel products had comparable fuel properties with that of diesel fuel (AGO). The heating value of the biodiesel is less than that of diesel fuel. Meanwhile, the flash point of the biodiesel is less than the optimum value of Bureau standard of biodiesel but greater than that of diesel fuel (AGO).

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