Original Research Article

Characterization of Biodiesel Produced from Neem Seed Oil

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ARTICLE INFORMATION	ABSTRACT
	The current challenges of fossil fuels have led to the need for alternative energy sources like biodiesel and biodiesel blends because they are renewable and environmentally friendly. The biodiesel production from neem seed involved, oil extraction, moisture/FFA reduction then base transesterification reaction using the extracted neem seed oil, methanol (6:1), 1.0% KOH at 60 °C, 400 rpm for 2 hrs. The physico-chemical and fuel properties of the neem seed oil biodiesel was analysed using standard analytical procedures, and the statistical data analysed using SPSS version 20.0. Most values were within the ASTM/EN standards, which can make a major contribution now and in the future by meeting the expected demand of petroleum diesel and providing improved properties of lower emission. As a result of this, it can be used in diesel engines and plants with little or no modification.

1. INTRODUCTION

Diesel engine is the most suitable one to run on biofuel but various engine types manufactured today are made to run on a wide variety of fuels, such as premium motor spirit (petrol), diesel, or gas as their primary fuel, but the diesel engine is the most suitable one to run on biodiesel. Biodiesels are very similar to petrodiesel in composition; therefore, there is no need for engine modification (Hansen, 2008). Biodiesel is a carbon-neutral fuel and is increasingly becoming popular, as a liquid fuel and is made through transesterification reaction that involves altering the chemical properties of vegetable oils and fats using alcohol (Silveira and Foster, 2008). This simple process yields high conversion with glycerin as the only byproduct. Biodiesel is very similar to petrodiesel, but are not identical. However, the difference is remarkably small when we compare the procedure for making biodiesel and petrodiesel (DOE, 2002).

In modern times, there is increase in the need to find and develop alternative energy sources which is largely due to a decline in the appeal of traditional energy sources such as fossil fuel that has been around for many

years. Fossil fuels have been linked to various environmental and ecological problems which have risen from their method of extraction, production, and eventually their intended point of use (Leadbeater and Stencel, 2006). The use of these energy sources over many years has resulted in rise in global temperature levels also known as global warming, which is due to high levels of carbon that are released as byproduct and exhaust gases (Hansen, 2008). Their methods of extraction have also led to serious cases of oil spills which tend to destroy the immediate or remote environment where such resources are found. This has also led to many political and socioeconomic problems over the world (Hansen, 2008). This unfortunate situation gave rise to the search for alternative sources of fuels that are renewable and more environmentally friendly.

In Africa, especially Nigeria, there is a huge potential for the development of biofuels as an alternative energy source, this is due to the fact that there is wide variety of plants in the country that could be used as biofuel sources (Thompson *et al.,* 1998). Oils from Jatropha, melon, palm fruit, soybean, sugar cane and used or waste oil can be used to make biodiesel. Among the crops grown in the tropics, considerable work has been done using vegetable oils such as soybean oil, palm oil, palm kernel oil, sunflower oil, rape seed oil, Jatropha oil etc (Best, 2006; Alamu *et al.,* 2007; Moser, 2009; Ogunwole, 2012).

Neem seed oil is obtained from neem plant *Azadirachta indica* derived from Persia, popularly known in Nigeria as *Dongonyaro*. The tree is a native of South East Asia and is a member of the Mahogany family *Meliaceae*. Although, other oils can also be used for the production, but that extracted from neem seeds is chosen because the seeds are readily available and not edible therefore, will not pose problem to humans in terms of food competition, consumption, security and also solve the challenge of pollution, management and treatment (Ogbuewu *et al.*, 2011). Also, large volumes of neem seeds are generated in Nigeria as well as other countries which could be a possible nuisance in our rural and urban areas. However, many researchers have analysed the nutritional and pharmaceutical values of the oil but search of the literature has shown that has little information exist about the oil as a feedstock for producing biodiesel.

2. MATERIALS AND METHODS

2.1. Sample Collection

Neem seed (*Azadirachta indica*) were collected from the botanical garden of the University of Ibadan, Ibadan in Oyo State, Nigeria. It was identified according to the flora of West Africa in the Herbarium Unit of University of Ibadan where vouchers of each specimen were deposited. Petroleum diesel was procured from the Nigerian National Petroleum Corporation (NNPC) filling station, Abeokuta in Nigeria.

2.2. Sample Preparation

The neem seeds were sun-dried and the shells were manually cracked to obtain the kernels. The kernels were then milled and stored at 4 °C until needed for analysis.

2.3. Oil Extraction

Oils from the milled seeds was exhaustively extracted with a Soxhlet apparatus using methanol as the solvent after which it was subjected to physico-chemical characterization using standard analytical methods on the appearance, water and sediment, viscosity, specific gravity, density, colour, pH, sulphur, acid and FFA values, total, free and bound glycerin, flash, pour and cloud points, cetane number, saponification, iodine and peroxide values, molecular weight as reported in AOAC (2000) and AOCS. (2003).

2.4. Biodiesel Production

The oil was heated in an oven at 100-120 ^{0}C for 20 min to reduce moisture/water concentration below 5 g/kg. The mechanism of reduction in free fatty acid was adopted for oils containing FFA up to 20%, by

lowering the acid value, making 300 cm^3 of oil used to contain FFA less than 1%. This was done by adding sufficient potassium hydroxide in 700 g/L aqueous solution, heating at 30-35 ^{0}C to precipitate the solids and allowing to settle overnight. The clear oil was decanted and the FFA determined according to ISO EN660, (1996).

The step by step approach used in the production of biodiesel during transesterification is:

i. Measurement and heating of 300 cm^3 of oil sample to a temperature of 60 °C
ii. Purification and measurement of 1800 cm^3 methanol to be used by re-distillation
iii. Preparation and addition of the 1.0% potassium methoxide solution into the warm oil sample
iv. Separation of the biodiesel from the lower layer (which comprises glycerol and soap)
v. Washing of the biodiesel to remove any excess glycerol and soap that may remain
vi. Drying of the washed biodiesel sample to remove excess methanol
vii. Measuring, analysing and recording of the quantity and quality of the biodiesel produced.

The oil to methanol ratio (1:6), catalyst concentration (1.0%), stirring time (2 hr) and rate (400 rpm), and the temperature (60 °C) were kept constant (Joshua, 2013).

2.5. Characterization of the Oil and Diesel

Standard procedures according to the American oil chemists' society (AOCS 2000, 2003, 2005), and American Society for Testing and Materials (ASTM) were adopted in the characterisation of the raw oil and the biodiesel produced.

2.5.1. Percentage yield

The percentage yield of the oil was calculated using Equation 1 (Ghadge and Raheman, 2006).

$$\text{Percentage yield} = \frac{Volume\,of\,samle\,extract}{Weight\,of\,sample\,taken} \times 100 \tag{1}$$

2.5.2. Appearance

A physical eye check was carried on the sample.

2.5.3. Water and sediment content

Sample of 5 g was thoroughly mixed by stirring and weighed into a previously dried and weighed crucible with lid. The crucible without the lid was heated in an oven set at 105±1 ^{0}C for 1 h. The crucible plus the sample was removed from the oven and covered with the lid and then cooled in a desiccator containing phosphorus pentoxide and weighed. It was later heated in the oven again for a further period of 1 h, cooled and weighed. This process was repeated five times until there was no change in weight between two successive observations (AOAC 2000).

$$\% \text{ Water and sediment content} = \frac{Sample\,loss\,of\,weight\,on\,drying}{Weight\,of\,sample\,taken} \times 100 \tag{2}$$

2.5.4. Viscosity

Viscosity of the samples was measured at room temperature (in centistokes) with the Oswald kinematic viscometer equipped with an attached water bath and a thermostat (AOCS 2003).

2.5.5. Specific gravity

The specific gravity of the oil extracted and biodiesel were determined by means of a pycnometer (AOAC 2005).

2.5.6. Density

This was determined from specific gravity using Equation 3.

$$\text{Density of sample} = \text{Specific gravity} \times \text{Density of water} \qquad (3)$$

Where density of water is 1 g/mol (AOAC 2000)

2.5.7. Colour

The colour of the oil extracted and the diesel were determined by comparison with Lovibond glasses of known colour characteristics, and the colour was expressed as the sum total of the yellow and red slides used to match the colour of the diesel in a cell of the specified size in the Lovibond Tintometer (AOAC 2000).

2.5.8. pH, sulphur and carbon content

These parameters of the extracted oil and diesel were determined according to the method described in the American Oil Chemists Society (AOCS 2003) official and tentative methods (Knothe, 2006; Oniya and Bamgboye, 2014).

2.5.9. Acid value

The acid value of the samples was determined by titrating the oils and the diesel in an alcoholic medium against standard NaOH solution (Knothe, 2006).

2.5.10. Free fatty acid

The free fatty acid in the samples was determined by titrating each sample against potassium hydroxide using phenolphthalein as indicator (Knothe, 2006).

2.5.11. Total, free and bound glycerin

A sample of 1 g was measured into a 250 cm^3 volumetric flask and 25 cm^3 chloroform-methanol mixtures were added to dissolve the sample. About 10 cm^3 of silisic acid and 10 cm^3 alcoholic KOH were added to precipitate the glycerin. The supernatant obtained was decanted and the precipitate was dissolved in 10 cm^3 of 0.2 M H_2SO_4 followed by the addition of 20 cm^3 0.05 M sodium metaperiodate and 5 cm^3 of sodium arsenite. The mixture was properly homogenised to get a uniform solution containing the total glycerin. Glycerin working standard of the range of 10 to 50 µg/cm^3 were prepared from 100 µg/cm^3 stock glycerin and treated similarly as sample above. The absorbance of the samples and working standards were taken on a UV Spectrophotometer at a wavelength of 560 nm (AOCS 2003).

$$\text{Total glycerin (GT)} = \frac{Absorbance \, x \, Gradient \, Factor \, x \, Df}{Weight \, of \, sample \, x \, 10,000} \qquad (4)$$

Free glycerin was obtained by titrating the dissolved precipitate against 0.0 M $Na_2S_2O_3$ using starch solution as indicator

$$\text{Free glycerin (GF)} = \frac{(Blank \, titre - Sample \, titre) \, x \, M \, of \, Na2S2O3 \, x \, 7.638}{Weight \, of \, sample \, used} \qquad (5)$$

Bound glycerin was calculated using Equation (6).

$$\text{Bound glycerin} = \text{Total glycerin} - \text{Bound glycerin} \qquad (6)$$

2.5.12. Flash point

This was done by determining the temperature at which the sample flashed when a test flame is applied under the conditions specified for the test (AOAC 2000).

2.5.13. Pour point

The pour point of the samples was determined by cooling the sample at a specified rate and the samples examined at 3 ºC intervals for flow characteristics. The lowest temperature at which samples movement was observed was then noted as the pour point (Oniya and Bamgboye, 2014).

2.5.14. Cloud point

The temperature at which a cloud is induced in the sample by cooling at a specified rate and examined at 3 ºC for first stage crystallization was determined according to AOCS (2000).

2.5.15. Cetane number

ASTM D613-18a (2008a). Standard test method for cetane number of diesel fuel oil was used to determine the cetane number of the samples.

2.5.16. Saponification value

The samples were saponified by refluxing with excess alcoholic KOH solution. The alkali required for the saponification was determined by titration of the excess KOH with standard HCl (AOAC 2000).

2.5.17. Iodine value

A sample of 0.25 g was taken into carbon tetrachloride and treated with excess iodine monochloride solution in glacial acetic acid (Wijs solution). The excess of the iodine monochloride was then treated with potassium iodide and the liberated iodine was estimated by titration with sodium thiosulphate solution (AOAC 2000).

2.5.18. Peroxide value

The peroxide present was determined by titrating the sample against sodium thiosulphate solution in the presence of KI using starch as indicator (Joshua, 2013).

2.3.19. Molecular weight

Saponification and acid values were first determined and then the molecular weight was calculated using the following equation.

$$\text{Mwt} = \frac{168300}{SV - Av} \tag{7}$$

Mwt is the molecular weight, SV is the saponification value, Av is the acid value and 168300 is the Constant (Haas, 2005).

2.3.20. Wet digestion

A sample of 1 g (oven dried at 60 ^{0}C) was weighed into a 125 cm^3 Erlenmeyer flask previously washed with acid and distilled water. Perchloric acid of 4 cm^3, 25 cm^3 concentrated HNO_3 and 2 cm^3 concentrated H_2SO_4 under a fume hood were added. The contents were mixed and heated gently at 120 ^{0}C on a hot plate under perchloric acid in a fume hood and heating continued until dense white fumes appear. The flask was allowed to cool and 1-2 cm^3 concentrated HNO_3 was added, digested again to the fuming stage and finally, heated strongly at 120 to 240 ºC for half a minute. It was allowed to cool, and 40-50 cm^3 distilled water was added

then boiled for half a minute on the same plate at 150 ^{0}C. The solution was cooled and filtered completely with Whatman No. 42 filter paper, then made up to 100 cm^3 mark in Pyrex volumetric flask with distilled water. The solution was analysed for Aluminium by Atomic Absorption Spectrometry and Potassium by Flame Emission Spectrometry at each wavelength of maximum absorption and emission.

2.6. Statistical Analysis

Results obtained were expressed as the mean of three separate observations. The data was statistically analyzed with a 2-way analysis of variance (ANOVA) using SAS software. The means were compared by Duncan's Multiple Range Test at 5% level of significance (p≤0.05).

3. RESULTS AND DISCUSSION

3.1. Physico-chemical Properties

Table 1 shows the physico-chemical properties of neem seed oil, neem seed oil biodiesel (B100) and petroleum diesel, with the appearance of the samples being similar. The appearance of neem seed oil was golden yellow liquid but Dangarembizi *et al.* (2015) had yellow green solid oil. The appearance of neem seed oil biodiesel and petroleum diesel was light golden yellow liquid but Ibikunle *et al.* (2019) had golden viscous liquid in both the oil and biodiesel produced. Appearance varies in ASTM/EN limit because the difference in chemical component of samples is responsible for these observations (Sustistyo *et al.*, 2006).

The yield of neem seed oil of 41.5% is much higher than that of Dangarembizi *et al.* (2015) who obtained 9.53%. neem seed oil biodiesel yield was 80.9% but varied compared with ASTM/EN standard**.** The oil yield is dependent on the physico-chemical composition, oil:solvent ratio and type, temperature and time while that of the biodiesel also includes these with catalyst type and concentration, stirring speed, transesterification type, separation and washing methods involved (Gonabad *et al.*, 2015). pH of neem seed oil was 6.10, which is slightly acidic and shows that the oil requires pre-treatment to reduce the acidity so as to make it suitable for biodiesel production. neem seed biodiesel pH was 6.69, tending neutral but differs in comparison with petroleum diesel and ASTM/EN standard. The viscosity of neem seed oil was 31.99±0.20 mm^2/s at 40°C, that of neem seed oil biodiesel was 4.99±0.10 mm^2/s at 40°C which is lower than that of the oil due to transesterification reaction, but higher than that of petroleum diesel of 4.05±0.00 mm^2/s at 40°C, but within ASTM, (2008b)/EN, (2009) standard of 1.90-6.00 mm^2/s at 40°C. The neem seed oil biodiesel value is lower than that of Aransiola *et al.* (2011), Taiwo *et al.* (2016) and De Lima da Silva *et al.* (2009). Less viscosity gives greater ease of fuel movement or better and faster fuel atomization and decrease in ignition delay.

Specific gravity and density of neem seed oil were 0.92±0.01 and 0.91±0.00 g/cm^3 respectively. The high values are because the weight is heavy due to some impurities that might be present which calls for pre-treatment of the oil to make them biodiesel production suitable (Moser, 2009). Those of neem seed oil biodiesel were 0.90±0.00 g/g and 0.89±0.00 g/cm^3 respectively which are lower than those of oil due to transesterification process but higher than that of petroleum diesel (0.85 and 0.84 g/cm^3 respectively) and ASTM, (2008b) standard of <0.88 and <0.86 g/cm^3 respectivel. The density value is also higher than that of Alli *et al.* (2013) (0.88 g/cm^3 and 0.61 g/ cm^3) for neem seed oil and biodiesel respectively. The specific gravity value of biodiesel is less than that of Aransiola *et al.* (2011). High values could be because the weight

is heavy due to some impurities that might be present (Moser, 2009). Density influences the air-fuel ratio due to mass of the oil and the biodiesel relative to the petroleum diesel.

Table 1: Physico-chemical properties of neem seed oil and neem seed oil Biodiesel (B100) and petroleum diesel

Parameters	Neem seed oil extract	Neem seed oil biodiesel	Petroleum diesel	PD/BD ASTM/EN standard
Appearance	Golden yellow viscous liquid	Light golden yellow viscous liquid	Light golden yellow viscous liquid	Varies
Yield (%)	41.5	80.9	Varies	Varies
pH	6.10	6.69	-	Varies
Viscosity (mm^2/s at 40 ^{o}C)	31.99±0.20	4.99±0.10	4.05±0.00	1.90-6.00
Specific gravity (g/g)	0.92±0.01	0.90±0.00	0.85±0.03	<0.88
Density (g/cm^3)	0.91±0.00	0.89±0.00	0.84±0.02	<0.86
Water and sediment (%)	1.82±0.20	0.95±0.01	3.48±0.10	0.05
Colour (Hz)	4.15±0.30	3.55±0.20	3.50±0.20	≤4.00
Flash point (^{o}C)	192.70± 2.00	157.52± 1.00	49.70± 0.20	>130
Pour point (^{o}C)	11.00±0.10	8.30±0.20	-	NA
Cloud point (^{o}C)	15.00±0.20	10.20±0.40	-	NA
Cetane Number	49.20	53.75	50.10	<51.00
Acid value (mgKOH/g)	20.90±0.30	9.60±0.02	0.30±0.01	<0.80
Free fatty acid (mgKOH/g)	10.45±0.20	4.80±0.01	0.15±0.01	<0.40
Total glycerin (%)	0.25±0.03	0.15±0.02	ND	0.25
Bound glycerin (%)	0.16±0.02	0.10±0.01	ND	0.25
Free glycerin (%)	0.09±0.01	0.05±0.00	ND	0.02
Iodine value (g/100 g)	76.50±0.30	60.93±0.20	54.35±0.30	<125
Saponification value (mgKOH/g)	196.89±0.04	178.45±0.30	-	NA
Peroxide value (mEq/kg)	8.15±0.10	5.75±0.02	-	10 (Oil)
Carbon residue (%)	0.22±0.02	0.16±0.01	0.26±0.02	0.05
Molecular weight (g/mole)	315.50	289.30	Varies	Varies
Sulphur (%)	0.73±0.30	0.48±0.02	1.16±0.20	0.05
K (mg/kg)	0.05±0.00	0.02±0.00	0.03±0.00	0.01-0.20
Al (mg/kg)	0.33±0.10	0.28±0.02	0.99±0.00	0.50

The water and sediment content was 1.82±0.20 % in neem seed oil, which is higher than 0.95±0.01 % in neem seed oil biodiesel thus it needs pre-treatment of heating, filtration to make it suitable for transesterification reaction. The biodiesel value was lower than that of petroleum diesel (3.48±0.10 %), but above 0.05 % ASTM, (2008b)/EN, (2009) standards. The value was lower than that of Taiwo *et al.* (2016) but higher than that of De Lima da Silva *et al.* (2009). Water and sediments are byproducts of storage due to ester oxidation, reactive glycerides and algae growth.

The colour value of the neem seed oil was 4.15±0.30 Hz which is higher than that of neem seed oil biodiesel (3.55±0.20 Hz) but close to that of petroleum diesel (3.50±0.20 Hz) and within permissible ASTM/EN standard of ≤ 4.00 Hz. Colour value of the biodiesel was less than that of Taiwo *et al.* (2016). The difference in chemical component of samples could be responsible for the variations observed (Sustistyo *et al.*, 2006).

The flash point of neem seed oil was 192.70±2.00 ºC which is higher than that of neem seed oil biodiesel (157.52±1.00 ºC). This is far higher than that of petroleum diesel (49.70±0.20 ºC), but within the permissible standard of > 130 ºC. Higher than values were reported by Tyson, (2001), De Lima da Silva et al., (2009) (93 ºC). Flash point is the safety requirements in handling and storage of fuel due to its hazardous nature and biodiesel is safer than petroleum diesel due to high values. Although pour point has no ASTM/EN standard, it is the lowest temperature which biodiesel can still move. neem seed oil value was 11.00±0.10 ºC, which is higher than that of neem seed oil biodiesel (8.30±0.20 ºC). This shows that the oil and the biodiesel will not perform better than petroleum diesel due to the presence of saturated fatty acid chains and monoglycerides (Arjun et al., 2008). The pour point value is greater than those of Aransiola et al. (2011) and Taiwo et al. (2016). Cloud point has no ASTM/EN standard but it is an important parameter for low temperature operation of fuel due to solidification of heavier components in biodiesel resulting in crystals within the body when cooled. neem seed oil value was 15.00±0.20 ºC, which is higher than that of neem seed oil biodiesel (10.20±0.40 ºC). This also shows that the oil and biodiesel will not perform better than petroleum diesel in low temperature. The cloud point values were greater than those of Aransiola et al. (2011) and Taiwo et al. (2016). Cetane number of neem seed oil was 49.20 which was lower than those of neem seed oil biodiesel (53.75) and higher than those of petroleum diesel (50.10) but within ASTM/EN standard and similar to that of De Lima da Silva et al. (2009) (50). Cetane number is critical in cold starting engine conditions because low values result in long ignition delay and high values in faster auto-ignition (Atabani et al., 2012). Free fatty acid and acid values of the neem seed oil was 10.45±0.20 mgKOH/g and 20.90±0.30 mgKOH/g which was more than double that of neem seed oil biodiesel (4.80±0.01 mgKOH/g and 9.60±0.02 mgKOH/g). This necessitate pre-treatment of the oil for reduction in acidity by addition of excess KOH to enhance biodiesel process and production. The biodiesel value was far higher than that of petroleum diesel (0.15±0.01 mgKOH/g and 0.30±0.01 mgKOH/g but are all above the ASTM, (2008b)/EN, (2009) standard of <0.40 to <0.80 mg/KOH/g. Also, the values are less than those of Aransiola et al. (2011) and Taiwo et al. (2016) but higher than that of Ibikunle et al. (2019). Acid value is the level of residual organic acid due to oxidation leading to deterioration. Total and bound glycerin values in neem seed oil was 0.25±0.03% to 0.16±0.02%, which was higher than that of neem seed oil biodiesel (0.15±0.02% to 0.10±0.01%), with all the values within the permissible standard of 0.25% which is an indication of more complete trans-esterification process in biodiesel production. The total and bound glycerin values are greater than those of Aransiola et al. (2011) and Taiwo et al. (2016). Free glycerin values in neem seed oil was 0.09±0.01% which is higher than that of neem seed oil biodiesel (0.05±0.00%) and was above the permissible standard of 0.02%. Iodine value of neem seed oil was 76.50±0.30 gI$_2$/100 g which is higher than that of Dangarembizi et al. (2015) and neem seed oil biodiesel of 60.93±0.20 gI$_2$/100 g and above petroleum diesel of 54.35±0.03 gI$_2$/100 g but all were within the ASTM/EN Standard of <125 gI$_2$/100 g. Iodine value is less than those of Aransiola et al. (2011), Taiwo et al. (2016) and De Lima da Silva et al. (2009)., but higher than that of Ibikunle et al. (2019). Low value could be as a result of the destruction of double bonds and vice versa, high values results in propensity for polymerization leading to deposit formation (Atabani et al., 2012).

Saponification value of neem seed oil was 196.89±0.04 mgKOH/g, which is lower than that obtained by Dangarembizi et al. (2015), but higher than that of neem seed oil biodiesel (178.45±0.30 mgKOH/g). The value was close to that of Aransiola et al. (2011), but lower than that of Reed et al. (1994) (155.50 to 159.70 mgKOH/g). The variation in the saponification value shows the differences in chain lengths of the free fatty acids present the samples. Long chain fatty acids found in fat and oil have low saponification value because they have a relatively fewer number of carboxylic functional groups per unit mass, therefore has high

molecular weight (Choe and Min, 2007). Standard saponification values for biodiesel are not stated but all values observed were within the standard value for oil of 192-198 mgKOH/g. The peroxide value of the neem seed oil was 8.15±0.10 mEq/kg which was higher than that of neem seed oil biodiesel (5.75±0.02 mEq/kg), showing good quality status in the biodiesel. Standard peroxide values for biodiesel are not stated but all values observed were within the standard value for oil samples of 10-20 mEq/kg but 30-40 mEq/kg are associated with a rancid taste (ISO, 17025, 2017). The variation in the values shows the extent deterioration has advanced in the samples and this is used for identifying the onset of oxidative change during which the oxygen (O_2) molecule penetrates the molecule in the form of a peroxide group (H_2O_2) (Choe and Min, 2007).This also the amount of unstable hydroperoxides (deteriorated biodiesel) when oxygen reacts with fatty esters. Carbon residue in neem seed oil was 0.22±0.02%, which is higher than that of in neem seed oil biodiesel (0.16±0.01%), with both lower than that of petroleum diesel (0.26±0.02%). Higher values indicate the presence of higher organic particles in the sample (Sprules and Price, 2010). Molecular weight of neem seed oil was 375.50 g/mole, which is higher than that of neem seed oil biodiesel of 289.30 g/mole but differs from that of petroleum diesel and ASTM/EN standards because of its dependence on the free fatty acid present coupled with the saponification value. Transesterification of vegetable oil reduces the molecular weight and viscosity of the oil, and improve its volatility to a suitable range for diesel engines (Aworanti *et al.*, 2012). Sulphur in neem seed oil was 0.73±0.30%, which was higher than that of neem seed oil biodiesel (0.48±0.02%) that is much lower than that of petroleum diesel (1.16±0.20%), with all above ASTM/EN permissible limit of 0.05%. Potassium in neem seed oil was 0.05±0.00 mg/kg which is lower than that of neem seed oil biodiesel (0.02±0.00 mg/kg and are lower than that of petroleum diesel (0.03±0.00 mg/kg, but all are within limit of 0.01-0.20 mg/kg. Aluminium in neem seed oil was 0.33±0.10 mg/kg which is higher than that of neem seed oil biodiesel (0.28±0.02 mg/kg) and much lower than that of petroleum diesel (0.99±0.00 mg/kg), but within ASTM, (2008b)/EN, (2009) standard of 0.50 mg/kg and less than that of Taiwo *et al.* (2016). This indicates the safety and rate of reduction in degradation because metals initiate spoilage that affects shelf-life stability of the samples and also their bio-accumulation in the body over time can lead to serious harmful effect and health risk.

4. CONCLUSION

The physico-chemical and fuel properties characterized have most values within the ASTM, (2008)/EN, (2009) standards except in the neem seed oil and petroleum diesel. The neem seed oil can be used for biodiesel production after pre-treatment to reduce the concentration of some of the parameters below permissible limits. neem seed oil biodiesel can be a major contributor now and thereafter in the future by meeting the demand expected of petroleum diesel.

5. ACKNOWLEDGEMENTS

6. CONFLICT OF INTEREST

There is no conflict of interest associated with this work.

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