Production and Characterization of Fatty Acid Methyl Esters from Palm Oil

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Abstract: *Diesel production through biological source is better alternative to conventional diesel as they are environmentally friendly, renewable, non toxic, sustainable, cheap, and cleaner fuel options amongst available biofuels. This study is aimed at trans-esterified Palm oil to Fatty Acid Methyl Ester and characterization of the diesel produced. Trans esterification of palm oil to biodiesel was performed using Sulphuric acid as a catalyst. The biodiesel was analyzed by Gas Chromatography Mass Spectroscopic (GC-MS). Physicochemical properties of biodiesel were determined using predictive models based on fatty acid composition and American Society for Testing for Materials (ASTM) protocols. Data obtained were subjected to appropriate statistical analysis. The fatty acid content of biodiesel had 45.80% saturated, 35.90% monounsaturated and 18300% polyunsaturated fatty acids. The physicochemical properties of the methyl ester produced were : Colour (Colorless), specific gravity (0.861), kinematic viscosity(1.99 mm^2/s at 40°C), flash point (133°C), cloud point (-5°C), pour point (-5°C), refractive index (1.41), heat of combustion (36.50MJ/kg), density (0.8610 g/cm^3), ash content (0.01%) , acid value (0.38mgKOH/g), saponification value (221 mgKOH/g), peroxide value (1.9 meq/kg), iodine value (46. 81mgI$_2$/g), free fatty acid (1.24 %), cetane number (73), oxidative stability (6.80 hrs), long chain saturated factor (24.2 ^{o}C), cold-filter plugging point (69.30^{o}C), degree of unsaturation (64.52), high heating value (41.25^{o}C) and water content (0.04). Palm oil biodiesel was discovered to be a good starting material for biodiesel production while results characterization conducted shows conformity with standard as required by regulatory authority*

Keywords: Palm oil, Transesterification, Characterization, GCMS, Methyl esters, diesel engine

1. INTRODUCTION

The leading energy source prior to 19th century was biomass, all countries rely on biomass-energy source for industrialization growth and development and technology advancement is the reason behind continues search for more efficient and reliable energy sources. Over many centuries, the transitions of energy regimes from wood to coal and from coal to fossil fuel was engendered by industrialization energy demands (Mosarof et al., 2015). Eventually, fossil fuels gained dominance as a source of energy across the universe, covering about 48% of global energy demand (Emodi, 2016).

Human existence and activities depends majorly on availability of energy; presently most of energy used for several activities ranging from homes, industrial sector, electricity and transportation are from non renewable source, this source that contributes over 86% of the global energy supply is depleting (Atadashi *et al.*, 2011). Non-renewable energy sources are energy obtained from fossil sources. They are energy from coal, oil shale's, bitumen, tar sands, various petroleum products such diesel, oil, gas, petrol, liquid fuels.

Over reliance on petroleum as the energy source comes at high cost, while its burning is detrimental to human health through releases of carbon-dioxide, carbon monoxide, sulphur oxide and others poisonous gas. The aforementioned poisonous gases are the principal causes of global warming. Also, the world petroleum production has perhaps reached its peak and supplies will soon be depleted (Adekanmi *et al.*, 2020). The search for a cheap, an eco-friendly, a new alternative and renewable energy source such as biodiesel was as a result of shortage of resources, high crude oil prices, depletion of ozone layers and environmental hazards attributed to its discharge (Hossain and Mekhled, 2010).

Today, above stated problems together with limitations of intensive burning of fossils energy such as depleting global reserves, continuous environmental pollution and market unpredictability had justified the basis for rapid increase and continuous research and development in replacement of current non-renewable energy usage in all over the countries (Mamudu and Olukanmi, 2019; Olatunji et al., 2019a,b).

Biofuel fits in as alternative replacement to widely known fossil energy source and presently is speedily gaining applications in the modern age through usage in various energy sectors, automobile and Industrial processes. They are either raw or extracted products from plant biomass that can release their embedded energy (Guo et al., 2015). Biodiesels are considered more sustainable, renewable, cheap, cleaner and fuel options amongst available biofuels (Thushari and Babel, 2018).

The first compression ignition (C.I) engine was invented around 1890s by Sir Rudolph Diesel and in 1990 the engine was operated on extracted raw oil in raw at the World's Exhibition in Paris (Zahan and Kano, 2018). The use of vegetable oil as automotive fuel continued up to 1920s when fossil fuel completely took over the function of powering vehicles (Guo et al., 2015). As a result of higher capacity to produce energy in comparison to other engines, fossil fuelled diesel engines were increasingly used in

agricultural and mechanical engineering applications (Kaisan et al., 2017; Venkatesan et al., 2019). However, based on its higher combustion temperature, inappropriate fuel combustion and, sulphur and carbon contents, diesel engines significantly contribute to environmental pollutants (Gopidesi et al., 2018).

Biodiesel is proven fuel with desirable qualities with success in powering C.I. engines as both stand-alone fuels and diesel blend (Mukhtar et al., 2019). Biodiesel is feasible alternative being actively researched because is a cleaner fuel usable in current diesel engines with/without engine alterations (Gashaw and Getachew, 2015). Increased profitability, biodegradability and low aromatics as well as sulphur contents are some of advantages of vegetable oil-based biodiesel over conventional diesel (Monisha *et al*., 2013). Biodiesel is one of the safest and non-toxic fuels option compared to petroleum based diesel because of higher flashpoint (Aransiola *et al*., 2013).

The application and use of biodiesel within a diesel internal combustion system improved the lubricity of the system and extended the catalytic converters life when compared to the base system (ChabishaP Makgaba and Daramola, 2015). The emission from engine powered with biodiesel is drastically cuts down to about seventy-eight percent when compared to petroleum-derived diesel as a results of the system production process and general utilization (Devarajan et al., 2017).

The first type of crop used to produce biodiesel are edible vegetable oils such as soybean oil, palm oil and sunflower oil and they are known as the first-generation biodiesel starting materials (Bowyer et al., 2018). The second generation biodiesel feedstock are non-edible vegetable oils such as jatropha, mahua, jojoba oil, salmon oil and sea mango, animal fats, used cooking oils, agricultural and solid municipal wastes (Aditiya *et al*., 2016; Olatunji *et al*., 2019a,b). Microalgae, Cyanobacteria, fungi and other single-celled oleginous microorganisms are the third-generation biodiesel feedstock (Oyedepo et al., 2019). Recently, research has discovered genetically engineered plants that consume more CO2 from the atmosphere than they may emit as fuel later during combustion and this new discovery is regarded as fourth-generation biofuels feedstock (Shokravi, 2019). Pyrolysis, gasification, refining, solar-to-fuel, conversion of vegetable oil and biodiesel to bioethanol and biogas using advanced technologies, as well as genetic manipulation are also categorized as fourth-generation technologies.

The oil palm (*Elaeis guinensis*, family – palmae) is the most important economic crop in the tropics. It is the most important source of oil and produces more oil per acre than many of the oil producing crops. It has two types of oils – palm oil and palm kernel oil. Palm oil is extracted from the stone part called kernel. The two oils have distinct properties and are used for differentpurposes. Palm oil is used for the manufacture of soap, production of margarine lubricating oils, candle and in tinplate and sheet industries. Palm kernel oil is used for soap, margarine manufacture and vegetable oil production while the cake left after production is used for livestock feed manufacture.

Palm oil is obtained from the flesh of the palm fruit. Each palm tree produces approximately one fruit bunch, containing as many as 3000 fruit lets, per month. Also, each palm tree continues producing fruit economically for up to 25 years. This ensures a constant stable supply, as compared with other annual crops (Mamilla *et al*., 2012). Palm oil is extracted from the mesocarp of the fruit of the palm Elaeis guineensis. There are a few varieties of this plant but Tenera, which is a hybrid of the Dura and the Pisifera, present abundantly throughout the whole Peninsular. The mesocarp comprises about 70 - 80% by weight of the fruit and about 45 -50% of this mesocarp is oil. The rest of the fruit comprises the shell, kernel, moisture and other non fatty fiber.The extracted oil is known as crude palm oil (CPO) which until quite recently was known as the golden commodity(Mamilla *et al*., 2012)..

Palm oil is one of the most widely consumed edible oils in the world today. Beside, it contains more monounsaturated fatty acids than many other vegetable oils. In addition, compared with other vegetable oils, palm oil is a rich source of the anti-oxidant vitamin E containing about 360 – 600 ppm in its refined form Palm oil like all natural fats and oils comprises mainly Triglyceries, mono and diglycerides. Free fatty acids, moisture, dirt and minor components of non oil fatty matter referred to collectively as unsaponifiable matter. Palm oil is composed of fatty acids, esterified with glycerol just like any ordinary fat. It is high in saturated fatty acids. Palm oil gives its name to the 16-carbon saturated fatty acid palmitic acid. Monounsaturated oleic acid is also a constituent of palm oil.

The quest for the current study is part of synergy for complete takeover of renewable diesel from conventional hazardous diesel through continuous research on various starting materials that can enhance and improve the most known feedstock biodiesel production. This work focuses on transesterification of palm oil into biodiesel. The objective of this works includes production of biodiesel from palm oil; Characterization of biodiesel produced from palm oil.

2. METHODOLOGY

2.1 Sample collection

Palm oil was purchased from market at Esa-oke, Osun state, Nigeria. The purchased palm oil was taken to Biology Laboratory for transesterification and characterization at Osun State College of Technology, Esa Oke

2.2Trans-esterification of Palm oil to Fatty Methyl Ester (FAME)

This was achieved using the Lepage and Roy (1994) method, with slight modifications by Anitha and Sriman (2012). The crude palm oils (about 10 mg) was dissolved using 2 mL of a freshly prepared mixture of chloroform-methanol (2:1, v/v) in a 10 mL Pyrex tube with a Teflon-sealed screw cap. 1 mL of methanol as reagent and 0.3 mL of sulfuric acid 95-97 % (ScharlauChemie, reagent grade) as catalyst was added, for the trans-esterification of about 10 mg of oils. After screwing the lid, the tube with the mixture was weighed and vigorously shaken for 5 min. Finally, the tube was incubated at 100 °C for 10 min and cooled to room temperature. Followed by addition of 1 mL of distilled water for phase separation (by adding water two distinct phases are formed, the upper layer rich in water, methanol, glycerol, and sulphuric acid, and the lower layer rich in chloroform and esters). Then, 1 mL of distilled water was added for a gentle water washing of esters with chloroform layer (more dense than the water layer) followed by discard of the water rich upper layer (less dense). This step was repeated twice more. The esters rich layer was filtered using a disposable Nylon syringe filter (0.2 μm pore, 13 mm diameter, Cronus, UK). The chloroform was then evaporated to dryness from the esters in a laboratory hood, at room temperature (of about 25 ºC).

2.3 Gas Chromatography and Mass spectroscopic of Palm oil Fatty Acid Methyl Ester

The FAME obtained from the palm oil was analyzed according to the method described by Medina *et al*. (1998) using a Shimadzu 2010 gas chromatography (Shimadzu Scientific Instruments, Columbia, MD, USA) equipped with a flame Ionization detector and a diethylene glycol succinate capillary column (30m × 0.25 ×0.25 µm). Both initial column temperature and injection port temperature were 180°C. Detector temperature is 230°C, and was increased to 300°C at a temperature gradient of 15°C/min. Biodiesel sample (100 µL) was placed into capped test-tubes, saponified with 1 ml of saturated KOH-CH_3OH solution at 75°C for 10min, and then it was subjected to methanolysis (Schreiner, 2006) with 5% HCL in methanol at 75°C for another 10 min. Thereafter, the Phase containing the fatty acid methyl esters was separated by adding 2 mL of distilled water, and methanol was recovered. The components were identified by comparing their retention times and fragmentation patterns with those of the standards. Six fatty acid methyl esters (C16:1, C17:0, C18:1, C18:2 and C18:3) were used as the standard materials.

2.4 Physicochemical Characterization of the Palm oil Methyl Esters Produced

2.4.1 Specific Gravity

A 3ml aliquot of the methyl ester was weighed and its density calculated using the equation below

$$\text{Density (\%)} = \frac{\text{Mass of Methyl ester(g)}}{\text{Volume of water(ml))}} \text{ x}100$$

Then, the specific gravity of the methyl ester was calculated using the formula in equation below

$$\text{Specific gravity (\%)} = \frac{\text{Density of Methyl Ester (g)}}{\text{Density of water(g)}} \text{ x}100$$

2.4.2 Relative density

This was carried out using the method described by ASTM D6751 (2009). The mass was obtained by weighing the empty and filled (Palm oil methyl ester) Erlenmeyer flask. The difference was the mass of the Palm oil methyl ester. With these two values, the density was obtained, which is mass divided by volume.

2.4.3 Moisture content

This was determined by method described by AOCS. Cd 3-25 (1998) An empty flask was weighed with and without the amount of methyl ester and dried in an oven at 105°C for 7h, weighing each 2 hours till a constant weight was obtained, and finally the weight was taken and compared with the initial recorded weight. The percentage moisture content was calculated using below following equation.

$$\text{Moisture content (\%)} = \frac{\text{(W1}-\text{W2) (g)}}{\text{W2(g))}} \text{ x}100$$

Where,

W_1 = Original weight of the sample before drying

W_2 = Weight of the sample after drying

2.4.4 Viscosity

This was done using the method of AOAC (1998). Methyl ester was gradually poured into the viscometer until its lobe was almost filled and then it was placed in a water bath and allowed to heat up to an equilibrium temperature of 40°C. The methyl ester on the broad arm was sucked through the narrow arm until it reached the upper mark above the lower lobe of this narrow arm. The methyl ester was then allowed to flow back to the lower mark just below the lower lobe. The time taken for the flow (flow time, t) was recorded. Then, the viscosity was calculated using below equation:

Viscosity (%) $= \frac{Vp_1t_1}{p_2t_2}$ x100

Where,

n = Viscosity of the methyl ester mm^2/s

v = Viscosity of water mm^2/s

p_1 = Density of the methyl ester, Kg/m^3

p_2 = Density of water, Kg/m^3

t_1= Time taken for the Palm oil methyl ester to flow back

t_2 = Time taken for the water to flow back

2.4.5 Flash Point

The flash point was determined according to open cup method described by ASTM D6751, (2009). The cup was filled with a sample of the methyl ester up to the mark (75ml) and the cup was heated with a Bunsen burner maintaining a small open flame from an external supply of natural gas. Periodically, the flame was passed over the surface of the methyl ester. When the flash temperature was reached, the surface of the methyl ester caught fire. The temperature (at the moment) was noted and recorded as the flash point temperature.

2.4.6 Cloud Point

This was carried out based on the ASTM D6751, (2009) method. A test tube with a thermometer inserted in it, was filled with a sample of the methyl ester. The methyl ester was cooled at 2°C/min rate and continuously monitored until a white cloud appeared on the bulb of thermometer. The temperature that corresponds to the first formation of a cloud in the methyl ester was recorded.

2.4.7 Pour Point

This was carried out using method described by ASTM D 97-96a (1998). A sample of the methyl ester in a capillary tube was solidified; thereafter, it was attached to a thermometer and inserted into a gradually heating beaker of water. The temperature at which the sample started moving in the capillary tube was recorded as pour point.

2.4.8 Percentage ash content

The ASTM D874,(2007) method was used for the determination of percentage ash content. To a washed porcelain crucible, One gram of palm oil methyl ester was added. It was weighed after drying and cooling in an oven at 100°C and a desiccator respectively. Then, heated for 4 hours at 600°C inside a muffle furnace, after which it was removed and subsequently cooled in a dessicator and weighed again. The percentage ash content was calculated using below equation:

Ash content (%) $= \frac{A-B}{C}$ x100

Where,

A = Weight of crucible + ash,

B = Weight of crucible

C = Weight of original sample

2.4.9 Refractive Index

This was determined according to the method of Alamu *et al*. (2008) using a refractometer (Abbe refractometer bench). The power switch was turned on; the illuminating lamp came up and the display showed 0000. A drop of the methyl ester was introduced on the working surface of the lower refracting prism. The rotating arm and the collecting lens cone of the light gathering illuminating units were rotated so as to make the light-intake surface of the upper light-intake prism to be illuminated evenly. The field of view was observed through the eye piece and the adjustable hand wheel was rotated so as to make the line dividing the dark and light areas fall in the cross line. The dispersion correction hand wheel was rotated so as to get a good contrast between the light and dark area and minimum dispersion. The read button was pressed and the refractive index was displayed on the screen

2.4.10 Conductivity

This was determined using a conductivity meter. The conductivity meter was standardized with 0.01M KCl solution. The electrode was rinsed with deionized water, wiped and dipped into a sample of the ethyl ester and left for some time to stabilize the reading. The reading was displayed on the screen and then recorded in micro Siemens per centimeter (μS/cm).

2.4.11 Heat of Combustion

This was carried out using a bomb calorimeter by method of AOAC (1998). The calorimeter was standardized using benzoic acid. To a crucible of the calorimeter, a weighed amount of the methyl ester (1.058g) was added and the fuse wire was attached between the electrodes. Thereafter, it was placed in the bomb, which was pressurized to 18atm of oxygen. The bomb was placed in a vessel containing a measured quantity of water (2000g). The ignition circuit was connected and the water temperature was noted. After ignition, the temperature rise was monitored every minute till a constant temperature was reached and recorded. The pressure was released, the length of unburned fuse wire was measured and the residue titrated with 0.7M of sodium carbonate solution using phenolphthalein as indicator. The heat of combustion was calculated using below equation:

Heat of Combustion $= \frac{E\Delta T - 2.3V}{g(KJ/Kg)}$ x100

Where,

E = Energy equivalent of the calorimeter using benzoic acid

ΔT = Temperature rise

L = Length of burnt wire

V = Titration volume

g = Weight of sample

2.4.12 Acid Value

This was determined by method of ASTM D6751 (2009). A standard alcoholic KOH solution (0.5M) was prepared by dissolving KOH (pellet) with ethanol. The solution was then filtered and stored in brown bottle for Five days. Furthermore, a mixture of 95% ethanol and diethyl ether in a ratio of 1:1 (v/v) was prepared. 5 grams of the methyl ester was dissolved in 25ml of 1:1 (v/v) mixture of ethanol and diethyl ether. The solution was titrated with 0.5M ethanolic KOH solution in the presence of 5 drops of phenolphthalein as indicator until the end point (colorless to pink) was recognized. The total acidity (acid number) in KOH/g was calculated using below equation.

Acid value $= \frac{V \times N \times 56.1}{M}$

Where,

V= Volume expressed in milliter of 0.5M solution of ethanolic KOH,

M= Weight of the sample in gram

N= Concentration of ethanolic KOH

2.4.13 Peroxide Value

The method of AOAC (1998) was used for the determination of peroxide value. To a 30 ml of mixture of glacial acetic acid and chloroform (3:2, v/v), 2.206g of the methyl ester was dissolved in the mixture, then, 20% of potassium iodide (0.5 ml) was added and the solution swirled in the dark for one minute after which 75ml of distilled water was added. The mixture was titrated with 0.1M sodium thiosulphate with vigorous shaking until the yellow colour of the iodine had disappeared. Starch indicator (0.5ml) was added then to obtain a blue colour and titration continued until all the blue colour had disappeared. The peroxide value was calculated using below equation:

$$\text{Peroxide Value} = \frac{(S-B) \times M \times 100}{\text{Sample weight(g)}}$$

Where,

S = Sample titre value in ml

B = Blank titre in m

M = Molarity of Sodium thiosulphate

2.4.14 Iodine Value

This was determined using the method of AOAC (1998). An aliquot of the methyl ester (0.8825g) was weighed into a conical flask, tetrachloromethane (15ml) and 25 ml of Wig's solution was added. This mixture was placed in a stopper conical flask, swirled gently and placed in a dark cupboard for one hour after which 20 ml of 20% potassium iodide solution and 100ml of distilled water were added. After gentle shaking, liberated iodine was titrated with 0.1M sodium thiosulphate solution until the yellow colour of the iodine had appeared. Starch indicator (1ml) was added then to obtain a blue colour and titration continued until all the blue colour had disappeared. The iodine value was calculated using below equation

$$\text{Iodine Value} = \frac{(B-S) \times M \times 100}{\text{Sample weight(g)}}$$

Where,

S = Sample titre value in ml

B = Blank titre in ml

M = Molarity of Sodium thiosulphate

2.4.15 Percentage Free Fatty Acid

This was carried out using the method of AOAC (1998). Two grams of the methyl ester was weighed into a conical flask and 10 ml of 95% ethanol was added. This was then titrated with 0.1 M sodium hydroxide using phenolphthalein as an indicator. The conical flask was shaken constantly until a pink colour that persisted for 30 seconds was obtained. The percentage free fatty acid was obtained from the equation below

$$\text{Free Fatty Acid} = \frac{V \times M \times 2.92}{\text{Sample weight(g)}} \times 100$$

 Where,

V= Volume of 0.1M Sodium hydroxide used in ml

M= Molarity of NaOH.

2.5 Determination of other Properties Based on Fatty Acid Methyl Esther (FAME) Profiles

This was achieved using predictive models of Fatty Acid composition. Saponification value (SV), iodine value (IV), Cetane number (CN) degree of unsaturation (DU), long chain saturated factor (LCSF) and cold filter plugging point (CFPP) was determined by equations proposed by Islam $et\ al$. (2015):

$SV = \sum (560 \times A_i) / M_{wi}$

$$IV = \sum (254 \times A_i \times D) / M_{wi}$$

$$CN = 46.3 + (5458/SV) - (0.225 \times IV)$$

$$DU = \sum MUFA + (2 \times PUFA)$$

$$LCSF = 0.1 \times (C16{:}0, wt\%) + 0.5 \times (C18{:}0, wt \%) + 1 \times (C20{:}0, wt \%) + 1.5 \times (C22{:}0, wt\%) + 2.0 \times (C24{:}0, wt\%)$$

$$CFPP = (3.1417 \times LCSF) - 16.477$$

Where, D is the number of double bonds, M_W is the fatty acid (FA) molecular mass, and A_i is the percentage of each fatty acid (FA) component of the palm oil.

The kinematic viscosity (υ), density (ρ) and higher heating value (HHV) of each Fatty Acid Methyl Ester (FAME) were determined by equation proposed by Ramirez-Verduzco*et al*. (2012):

$$In (vi) = -12.503 + 2.496 + In (Mi) -0.178 \times N$$

$$\rho = 0.8463 + 4.9/Mi + 0.0118 \times N$$

$$HHVi = 46.19 - 1794/Mi - 0.21 \times N$$

Where,

(*vi* is the kinematic viscosity of at 40 °C in mm2/s; *ρi*is the density at 20 °C in g/cm^3; *HHVi* is the higher heating value in MJ/kg of *i*th FAME, N is the number of double bonds, M_i is the molecular mass of each fatty acid component.

Predictive oxidative stability was calculated based on C18:2 and C18:3 content of Fatty Acid composition as suggested by Park *et al*.(2008):

$$OS= 117.9295/ (wt\% \; C_{18.2} + wt\% \; C_{18.3} + 2.5905 \; (0{<}100)$$

Where,

wt% $C_{18.2}$is the content of linoleic, wt% $C_{18.3}$ is the content linolenic acids (wt%) (0 <*X* < 100); and *OS* is the oxidation stability in hours.

3.RESULT AND DISCUSSION

Chromatogram of biodiesel obtained from palm oi was shown in Figure 1 . Gas chromatography and mass spectroscopy of diesel obtained from palm oil showing fatty acids composition in percentage of total fatty acid methyl esters components (Table 1). The composition, quality and quantity of fatty acids in biodiesel of palm oil were examined through gas Chromatography and Mass spectroscopy analysis. Ten fatty acids were obtained from characterized biodiesel extracted from coconut oil namely: Palmitic, Palmitoleic, Stearic, Oleic, Linoleic, Arachidic and Behenic acid

The biodiesel extracted from palm oil had eight fatty acids, namely Palmitic acid: 29.50%, Palmitoleic acid: 7.40%, Stearic acid: 11.60%, Oleic acid: 28.50%, Linoleic acid: 18.30%, Arachidic acid: 3.4% Behenic acid: 0.7% and Linoceric acid 0.6%..The highest carbon carbon chain $C_{16} - C_{18}$ of 95.3% was recorded in palm oil dieselwhile percentage composition of saturated, monounsaturated and polyunsaturated fatty acids in the biodiesel extracted from lipids were 45.3%, 35.90% and 18.30% respectively (Table 1).
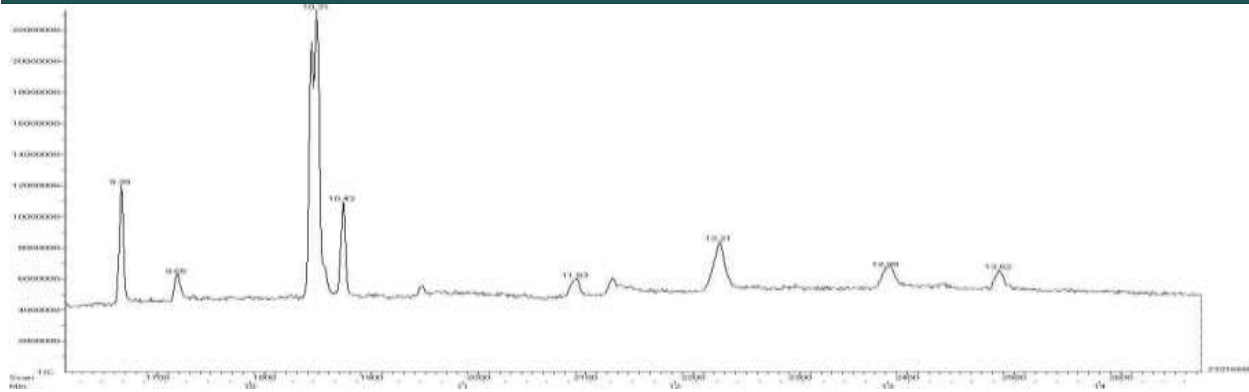
Figure 1: Chromatogram of diesel obtained from Palm oil

Table 1: Gas chromatography and mass spectroscopy of palm oil biodiesel showing fatty acids composition in percentage of total fatty acid methyl esters components

Structure	Fatty Acids	Molecular Formular	Palm oil diesel
			Percentage Composition of each Fatty Acids
$C_{16:0}$	Palmitic Acid	$C_{16}H_{32}O_2$	29.50
$C_{16:1}$	Palmitoleic Acid	$C_{16}H_{30}O_2$	7.40
$C_{18:0}$	Stearic Acid	$C_{18}H_{36}O_2$	11.60
$C_{18:1}$	Oleic Acid	$C_{18}H_{34}O_2$	28.50
$C_{18:2}$	Linoleic Acid	$C_{18}H_{32}O_2$	18.30
$C_{18:3}$	Linolenic Acid	$C_{18}H_{30}O_2$	-
$C_{20:0}$	Arachidic Acid	$C_{20}H_{40}O_2$	3.4
$C_{20:1}$	Gondoic Acid	$C_{20}H_{38}O_2$	-
$C_{22:0}$	Behenic Acid	$C_{22}H_{44}O_2$	0.7
$C_{24:0}$	Linoceric Acid	$C_{24}H_{48}O_2$	0.6

$C_{16} - C_{18}$ (%)		95.3
Saturated Fatty Acid (%)		45.80
Monounsaturated Fatty Acid (%)		35.9
Polyunsaturated Fatty Acid (%)		18.30

The fuel properties considered for examine the capacity of biodiesel as replacement of diesel fuel are specific gravity, Kinematic viscosity, flash point, cloud point, pour point volatile matter, refractive index, heat of combustion, Conductivity, Density, Ash content, Acid value, Saponification value, Peroxide value, Iodine value, Free fatty acid, Cetane number, Oxidative stability, Long

chain saturated factor, Cold filter plugging point, Moisture content, Degree of Unsaturation, High Heating value and water content (Table 2).

The specific gravity of the palm oil biodiesel is 0.8610. The specific gravity obtained conform satisfactorily with stipulated standard by US (ASTM D6751-08) AND EUROPE (EN14214) Table 2. The specific gravity obtained was found to be within the limits of EN 14214 (0.86 – 0.90) biodiesel fuel standard. The specific gravity was found to be lower than those of palm kernel oil ethyl esters (0.883), rapeseed oil ethyl esters (0.876) (Alamu *et al.,* 2008), and sunflower oil ethyl esters (0.876) (Lang *et al*., 2001), but was higher than that of petrol diesel (0.853) (Alamu *et al*., 2008) and almost the same with that of canola oil ethyl esters (0.869) (Lang *et al*., 2001).

The viscosity of the palm oil ethyl esters at 40 °C was 1.99 mm2/s; this value was found to be within the limits of ASTM D6751 (1.9 – 6.0 mm^2 /s) biodiesel fuel standard (Table 2). This is similar to work of Rashid *et al*.(2012) that reported that viscosity of the palm oil ethyl ester was found to be lower than those of Jatropha oil biodiesel (4.80 mm^2/s), canola oil ethyl esters (4.892 mm^2/s) (Alamu *et al*., 2008), olive oil ethyl esters (4.00 mm^2/s), (Anastopoulos *et al*., 2009) and that of rapeseed ethyl esters (6.170 mm^2/s) (Alamu *et al*., 2008).

The flash point of the palm oil ethyl ester was 133°C. The value was found to be above the minimum value (120 °C) of the EN 14214 and (130°C) of the ASTM D6751 biodiesel fuel standard (Table 2). This was in line with work of Alamu *et al*. (2008) and Anastopoulos *et al*. (2009) that reported that flash point of the palm oil ethyl ester was lower than that of palm kernel oil biodiesel (167°C), Canola ethyl esters (177°C), sunflower ethyl esters (178°C), olive ethyl ester (182°C), and rapeseed ethyl ester (181°C). In the same vein, Alamu *et al*. (2008) found out that the flash point of palm oil was extremely higher than that of petrol diesel with a value of 74°C. This showed is the presence of little or no residual alcohol in palm oil biodiesel and is of importance in both prevention of fire outbreak in the compressor engine when used as fuel and also for storage and transportation of the fuel (Moser, 2009).

The cloud point of the palm oil ethyl ester was -5°C and was within the limits of ASTM D6751 (-3 to 12°C) biodiesel fuel standard. The present study is similar to work of Alamu *et al*. (2008) that reported that the cloud point of palm oil ethyl esters was higher compared to that of canola ethyl esters (-6 ^{o}C), rapeseed ethyl esters (-10°C) and extremely higher than that of petrol diesel (-12°C) but lower to those of palm kernel oil biodiesel (6°C).

The pour point of (-5) palm oil biodiesel satisfied required standard range (Table 2). The lowered pour point and cloud point implies that the palm oil ethyl ester can be used as fuel in regions where the temperature is within the range of -5 to -10°C.

The volatile matter for the palm oil biodiesel is 99.60.. The density of palm oil biodiesel is 0.8610 and it fall within the standard range (0.86 – 0.90g/cm^3) (Table 2).

The standard value for heat of combustion as stipulated by US (ASTM D6751- 08) and EN 14214 is min 35, the values recorded for palm oil diesel was 36.50 MJ/kg and it meets the set standard by regulatory authority (Table 2). The value reported in this work is lower than that of petrol diesel (45MJ/kg) as reported by Lang *et al*. (2001). The above result is related to the work of Anastopoulos *et al*. (2009) reported that the heat of combustion of the palm oil ethyl ester was comparable to those of sunflower (38.6 MJ/kg), olive oil ethyl ester (39.2 MJ/kg)

Bello *et al*. (2011) pointed out that Cetane number is one of the primary indicators of a good diesel fuel quality and is related to the ignition delay time a fuel experiences once injected into a diesel engine combustion chamber. The palm oil biodiesel had empirically calculated cetane number of 73 which is above the minimum value of the ASTM D6751 (40 minimum) and EN 14214 (51 minimum) international biodiesel fuel standards. The cetane number of the palm oil biodiesel (ethyl ester) was found to be higher than some conventional biodiesels such as jatropha oil biodiesel (55) as reported by Reddy and Ramesh (2005), waste cooking oil biodiesel (10.96) by work of Owolabi *et al*. (2011) and Kumar *et al*.(2001) that reported 51 for coconut biodiesel. Thus, the higher cetane number of palm oil ethyl ester indicates a shorter ignition delay time.

The biodiesel of tested palm oil is within the standard value (US (ASTM D6751- 08) of 0.01 set for Ash content (Table 2). The ash content is a measure of the amount of residue left when the fuel is heated to 600°C (Sanford *et al*., 2009); this was found to be 0.02% for the coconut oil ethyl ester. The ash content of the palm oil ethyl ester was found to be within the limit of ASTM D 6751 (0.01%) and EN 14214 (0.02%) biodiesel fuel standard. The ash content of the palm oil ethyl ester was also comparable to that of jatropha (0.016%) as reported by Rashid *et al*. (2010). The slight increase of the ash content of the palm oil ethyl ester of the above the biodiesel fuel standard could be as a result of the presences of little amount of metal contaminants.

The values of refractive index obtained for palm oil biodiesel is (1.41) and it conform satisfactorily with required biodiesel standard (US (ASTM D6751- 08). The values recorded in this result are similar to values of 1.430 and 1.431 reported for palm oil methyl ester and ghee methyl ester by Deshpande and Kulkarni (2012). Conductivity is a measure of the ability of water to pass an electrical current, it is indicative of the presence of water in the biodiesel (Sanford *et al*., 2009). The conductivity of the palm oil ethyl ester was 0.00 µS/cm, this could be due to proper drying and short storage time before the test was carried out.

The acid values of the palm oil ethyl ester (0.35 mgKOH/g) was found to be within the limits of the ASTM D6751 (0.5 mgKOH/g maximum) and EN 14214 (0.5 mgKOH/g maximum) biodiesel fuel standards. Similarly, Lang *et al*. (2001) reported acid values of 0.265 mgKOH/g for canola oil ethyl ester which is similar to acid value found in the above work. In related to the above study, Lang *et al*. (2001) and Anastopoulos *et al*. (2009) reported low acid value of the palm oil ethyl ester compare to 0.610 and 1.02 mgKOH/g for sunflower oil ethyl ester and rapeseed oil ethyl ester. The low acid value of the palm oil ethyl ester indicates that the fuel contains relatively little or no water which could hydrolyze the biodiesel to free fatty acids.

The saponification value (MgKOH/g) of 221 was obtained for Palm oil biodiesel (Table 2). The saponification value of the palm oil ethyl ester was found to be higher than that of sunflower oil ethyl ester (192.1 mgKOH/g), rapeseed oil ethyl ester (170.4 mgKOH/g), olive oil ethyl ester (196.2 mgKOH/g) and used frying oil ethyl ester (193.2 mgKOH/g) (Anastopoulos *et al*., 2009).

The iodine value of the palm oil ethyl ester was found to be 46.81mgI $_2$/g; this implies lower degree of unsaturation and better oxidative stability of the palm oil ethyl ester. The iodine value of the palm oil ethyl ester was found to be within the limits of EN 14214(120 mgI$_2$ /g maximum) biodiesel fuel standard, but was lower than that of Jatropha methyl ester (104 mgI$_2$/g) (Singh and Padhi, 2009).

The value recorded for Peroxide (meq/kg) is 1.9 (Table 2).The peroxide value of the palm oil ethyl ester was 1.90 meq/Kg. This value was lower when compared to that of the coconut oil and groundnut oil methyl ester (3.23 meq/kg) as reported by Ibeto *et al*. (2011). The low peroxide value of the palm oil ethyl ester could be as a result of low level of unsaturated fatty acids (as indicated by the iodine value of the ethyl ester). Also, proper storage and handling of the coconut oil ethyl ester to avoid contaminants and factors that enhance autoxidation could have contributed to the low peroxide value.

Oxidative stability (hr) of 6.80 observed in biodiesel obtained from palm oil diesel conforms satisfactorily within ASTM D6751-08 and EN 14214 biodiesel standard (Table 2). The biodiesel obtained from oil extracted from palm oil had 27.41 long chain saturated factor (LCSF) while 69.30 was recorded in biodiesel of palm oil for Cold filter plugging point. The degree of unsaturation in palm oil biodiesel is 64.52, the higher heating value 41.25 while water content is 0.04 (Table 2).

Table 2: Physico-chemical properties of the diesel obtained from palm oil in comparism with US (ASTM) D6751-08 and Europe (EN 14214) standard

S/N	Physicochemical parameters	Palm Oil	USA (ASTM) D6751-08	EUROPE (EN 14214)
1	Specific Gravity	0.861	0.88	
2	Kinematic Viscosity (mm^2/s) at 40^{0}c	1.99	1.9-6.0	3.5-5.0
3	Flash Point (^{0}C)	133	130min	120min
4	Cloud Point (^{0}C)	-5	-3-+12	-
5	Pour Point (^{0}C)	-5	- 15 - +10	-
6	Volatile Matter (%)	99.60		
7	Refractive Index	1.41	1.479	
8	Heat of Combustion (MS/kg)	36.50	35min	35min
9	Conductivity (MS/cm)	0.00		
10	Density (g/cm^3)	0.861	0.86-0.90	

11	Ash Content (%)	0.01	0.01	0.02
12	Acid Value (mgKOH/g)	0.38	0.5	0.5
13	Saponification Value (mgKOH/g)	221		
14	Peroxide Value (meq/kg)	1.9		
15	Iodine Value (mgI$_2$/g)	46.81		120max
16	Free Fatty Acid (%)	1.24		
17	Cetane Number	73	47min	51min
18	Oxidative Stability (hr)	6.80		3.0min
19	Long Chain Saturated Factor (LCSF)^{0}C	27.41		
20	Cold-Filter Plugging Point (CFPP)	69.30		
21	Moisture Content (%)			
22	Degree of Unsaturation	64.52		
23	High Heating Value (HHV)	41.25		
24	Water Content	0.04	500ppm	500max
25	Calorific Value (Kcal/kg)			

4. CONCULUSION

The results obtained suggest that the palm oil could be considered as a viable raw material for biodiesel production; this is due to the fact that its ethyl ester (biodiesel) meets the standard specification of the American Society for Testing and Material (ASTM). The acid catalyzed transesterification reaction of palm oil with ethanol gave higher yield of biodiesel at higher oil to ethanol volume ratios. However, it is advised to increase the concentration/volume of the methanol than that of the oil because increase in the oil concentration/volume favors saponification reaction which is a side reaction to the overall process. This side reaction depletes the oil in the reaction; thereby, reducing the yield of biodiesel.

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