

Lipid Extraction and Fatty Acid Methyl Ester (FAMES) Production from Coconut

¹Adekanmi Abideen Adeyinka, ²Orimogunje Samuel Akin, ²Agbesanya Oluwadare, ²Esho, Ezekiel Olubayode and ²Oyero Gbenga Micheal

¹Raw Materials Research and Development Council (RMRDC) Abuja, Nigeria

⁴Department of Science Laboratory Technology, Osun State College of Technology, Esa-Oke, Osun Nigeria

Corresponding Author: Name; Adekanmi Abideen Adeyinka, Phone; +2348060214264, E-mail; yinklabl234@gmail.com

Abstract: Biodiesel fits in as an ideal replacement as they are produced from non-toxic, biodegradable and renewable resources providing clean and environmentally friendly energy sources. This study is aimed at extracting oil from coconut, trans-esterifying the oil to Fatty Acid Methyl Ester and characterization of the diesel produced. The coconuts were de-husked and the shell split open in order to remove the kernel (flesh). The kernel was grinded into fine particles with a blender and sun-dried for 7 days. The oil extraction was determined by solvent extraction methods. Trans esterification of coconut 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 49.00% saturated, 29.90% monounsaturated and 21.10% polyunsaturated fatty acids. The fatty acid content of biodiesel had 45.10% saturated, 31.90% monounsaturated and 23.00% polyunsaturated fatty acids. The physicochemical properties of the methyl ester produced were : Colour (Colorless), specific gravity (0.8600), kinematic viscosity (1.83 mm²/s at 40°C), flash point (98°C), cloud point (-5°C), pour point (-9°C), refractive index (1.44), heat of combustion (36.98MJ/kg), density (0.8600 g/cm³), ash content (0.02%) , acid value (0.20mgKOH/g), saponification value (220 mgKOH/g), peroxide value (1.50 meq/kg), iodine value (41.81mgI₂/g), free fatty acid (1.14 %), cetane number (70), oxidative stability (6.56 hrs), long chain saturated factor (24.2 °C), cold-filter plugging point (66.2°C), degree of unsaturation (65.80), high heating value (41.24°C) and water content (0.09). Coconut biodiesel was found to have better fuel quality that has potentials to fuel a diesel engine.

Keywords: Coconut, Lipid extraction, Transesterification, Methyl esters, diesel engine

1. INTRODUCTION

The current problem ravaging the world has been attributed to the environmental pollution as a result of increased in the use of energy from fossil sources. Energy from petroleum source is rising and increasing at geometrical rate, and this can be connected to evolving of various industrial sectors, development of urban areas, new technology, and advancement of new equipment at all sectors of life. The development and evolution experienced is for betterment and improvement of human race but these new invention required the use of energy, so, as there is advancement also is increase or more use of energy. The introduction of new technology attracted use of more energy unlike the old one.

The energy available worldwide for human use is petroleum fossil fuel, most of the new industries depend heavily on conventional energy to power their various machineries and for their day to day activities. The problem linked to fossil energy includes depletion of ozone layers, release of poisonous gasses and various health issues. The hazards traced to conventional energy is not limited to aforementioned problems but also the exploration and processing processes. This has affected means of sustenance of people living in areas where those oils are explored and their operation has also worsened or complement the problems. The land in the affected areas are not fertile for farming as a result of oil spillage and exploration activities while most of water sources have been polluted and not more fit for drinking and others human use.

The aquatic environments are not left out with the problems associated with continuous use of fossil fuel because oil spillage and washing away of oil to aquatic and riverine areas has affected the lives of aquatic organisms and this has bring set back to aquatic farmers while human being that fed on fish and other aquatic organisms affected with oil spillage suffered various degree of health challenges. Scarcity and hike in price are two majors problems hindered availability of the fossil fuel despite barriers and side effects associated with their use. The search for environmental-friendly resources that have potential and capability to substitute mineral oil in various industrial applications is currently being considered in the fuel and energy sector [1].

An alternative fuel to petrodiesel must be technically feasible, economically competitive, environmentally friendly, and easily available. Biodiesel is the best alternative for diesel fuels in diesel engine [2]. Biodiesel is defined as the mono alkyl esters of long

fatty acids derived from renewable lipid feedstock such as vegetable oils or animal fats, for use in compression ignition (diesel) engines [3].

The advantage of biodiesel includes lower exhaust emissions, renewability, biodegradability, higher lubricity and higher combustion efficiency [2]. Biodiesel can be used in its pure state or blended with petroleum-based diesel fuel (B20 is assigned for 20 vol. % biodiesel and 80 vol. % petroleum-based fuel blend) [4]. Biodiesel can offer other benefits, including reduction of greenhouse gas emissions, regional development and social structure, especially to developing countries [5, 6]. Nevertheless, the advantages of biodiesel supersede the disadvantages generally on the environmental aspects, making it a very popular alternative to petroleum derived-diesel oil [2].

Currently, supply of these non-renewable energy sources is threatening to run out in a foreseeable future [7]. Also, it has been widely reported that not less than ten major oil fields from the 20 largest world oil producers are already experiencing decline in oil reserves. Recently published data also revealed a total of 29 major world oil producing countries already experiencing declining oil reserves from year 2005 to 2007 [8]. Despite the above stated set back on use of petroleum fossil fuel, the effort on renewable and bioenergy is not yielding expected result, hence the search for renewable sources of energy should be a continuous while more studies and technologies should be targeted towards improving on existing work done on biofuels.

The kernel of coconut is the main source of the products which are mainly coconut oil and desiccated coconut or dried kernel (copra). The copra which is mainly used for oil extraction contains about 65 to 75% oil [9]. The different fatty acids present in coconut oil range from C6 to C18 [10] and approximately 50% of the fatty acid is lauric acid [9]. The aforementioned property of kernel of coconut is an indication that they are useful and good starting materials in biodiesel production. Most of the available works on Coconut kernel are mainly on lipids/oils while there is little or no reported work done on biodiesel from coconut kernel. Hence, the current finding is aimed at extracting oil from coconut and trans-esterifying the oil to Fatty Acid Methyl Ester before characterization. This is achieved through the following objectives: Extraction of oil from coconut copra (dry flesh), Trans-esterification of coconut oil to Fatty Acid Methyl Ester and Physicochemical characterization of the diesel produced from coconut oil.

2. MATERIALS AND METHODS

2.1 Sample collection

The coconuts were obtained from Ilesha market, Osun state, Nigeria and taken to laboratory (biology) at Department of Science Laboratory Technology, Osun State College of Technology, Esa Oke. The area lies within the latitude and longitude of 7°37'40.40" N and 4°44'29.80" E respectively. The nuts were de-husked and the shell split open in order to remove the kernel (flesh). The kernel was grinded into fine particles with a blender and sun-dried for 7 days. The dried fine particles were further grounded into powdery form through blender.

2.2 Extraction of oil from Coconut

The modified method of Bligh and Dyer [11] by Nascimento *et al.*, [12] was used for the extraction of oil from powdery form of coconut kernel. To 1 gram of grounded coconut kernel, a mixture of 2 ml methanol and 1 ml chloroform (2:1) were added. The mixture was kept for 24 h at room temperature to dissolve the lipids properly. This was followed by centrifugation for 10 minutes at 3000 rpm and supernatant was collected. The process was repeated again by adding 2 mL of chloroform to the pellet and shaken properly. It was again centrifuged for 5 minutes at 3000 rpm and supernatant was separated. The former and latter supernatants were added together. Two separate layers were formed after addition of 2 mL of 1% potassium chloride (KCL) to the supernatants. The upper layer (methanol/kernel) was decanted and the lower chloroform layer containing the extracted lipids was collected in another test tube. The upper layer (methanol/kernel) was extracted again using the same procedure but with 1 mL of 1% potassium chloride (KCL). The bottom phase (chloroform/lipids) were combined and evaporated for 24 hours in a drying oven at 80°C. Then, the Lipid/oil was obtained after evaporation of the chloroform

2.3 Trans-esterification of Coconut oil (Lipid) to Fatty Methyl Ester (FAME)

This was carried out by the Lepage and Roy [13] method, with slight modifications by Anitha and Sriman [14]. The crude oil (lipid) (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 % (Scharlau Chemie, reagent grade) were added as catalyst, for the trans-esterification of about 10 mg of lipids. 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 laboratorial hood, at room temperature (of about 25 °C).

2.4 Gas Chromatography and Mass spectroscopic of Coconut Fatty Acid Methyl Ester

This was done by method of Medina *et al.* [15] 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 was 230°C, and this was further increased to 300°C at a temperature gradient of 15°C/min. Lipid samples (100 µL) was placed into capped test-tubes, saponified with 1 ml of saturated KOH-CH₃OH solution at 75°C for 10min, and then subjected to methanolysis [16] 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.

2.5 Physicochemical Characterization of the Coconut Methyl Esters Produced

2.5.1 Specific Gravity

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

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

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

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

2.5.2 Relative density

This was carried out using the method described by ASTM D6751 [17]. The mass was obtained by weighing the empty and filled (Coconut methyl ester) Erlenmeyer flask. The difference was the mass of the algal methyl ester. With these two values, the density was obtained, which is mass divided by volume.

2.5.3 Moisture content

This was determined by method described by AOCS. Cd 3-25 [18] 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.

$$\text{Moisture content (\%)} = \frac{(W_1 - W_2) (g)}{W_2(g)} \times 100$$

Where,

W_1 = Original weight of the sample before drying

W_2 = Weight of the sample after drying

2.5.4 Viscosity

This was done using the method of AOAC [19]. The 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:

$$\text{Viscosity (\%)} = \frac{v p_1 t_1}{p_2 t_2} \times 100$$

Where,

n = Viscosity of the methyl ester mm²/s

v = Viscosity of water mm²/s

p₁ = Density of the methyl ester, Kg/m³

p₂ = Density of water, Kg/m³

t₁ = Time taken for the coconut methyl ester to flow back

t₂ = Time taken for the water to flow back

2.5.5 Flash Point

The flash point was determined according to open cup method described by ASTM D6751, [17]. 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.

2.5.6 Cloud Point

This was carried out based on the ASTM D6751, [17] 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.

2.5.7 Pour Point

This was carried out using method described by ASTM D 97-96a [20]. 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.

2.5.8 Percentage ash content

The ASTM D874, [21] method was used for the determination of percentage ash content. To a washed porcelain crucible, One gram of coconut methyl ester was added. It was weighed after drying and cooling in an oven at 100°C and a dessicator 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:

$$\text{Ash content (\%)} = \frac{A-B}{C} \times 100$$

Where,

A = Weight of crucible + ash,

B = Weight of crucible

C = Weight of original sample

2.5.9 Refractive Index

This was determined according to the method of Alamu *et al.* [22] 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

2.5.10 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 ($\mu\text{S}/\text{cm}$).

2.5.11 Heat of Combustion

This was carried out using a bomb calorimeter by method of AOAC [19]. 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:

$$\text{Heat of Combustion} = \frac{E\Delta T - 2.3V}{g(\text{KJ}/\text{Kg})} \times 100$$

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

2.5.12 Acid Value

This was determined by method of ASTM D6751 [17]. 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 (colourless to pink) was recognized. The total acidity (acid number) in KOH/g was calculated using below equation.

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

Where,

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

M= Weight of the sample in gram

N= Concentration of ethanolic KOH

2.5.13 Peroxide Value

The method of AOAC [19] 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 ml

M = Molarity of Sodium thiosulphate

2.5.14 Iodine Value

This was determined using the method of AOAC [19]. 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 stoppered 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

2.5.15 Percentage Free Fatty Acid

This was carried out using the method of AOAC [19]. 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.

2.5.16 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) were determined by equations proposed by Islam *et al.* [23]:

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

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

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

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

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

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

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 microalgae oil.

The kinematic viscosity (ν), density (ρ) and higher heating value (HHV) of each Fatty Acid Methyl Ester (FAME) were determined by equation proposed by Ramirez-Verduzco *et al.* [24]:

$$\ln(\nu_i) = -12.503 + 2.496 + \ln(M_i) - 0.178 \times N$$

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

$$HHV_i = 46.19 - 1794/M_i - 0.21 \times N$$

Where,

(ν_i is the kinematic viscosity of at 40 °C in mm²/s; ρ_i is the density at 20 °C in g/cm³; HHV_i 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.* [25]:

$$OS = 117.9295 / (\text{wt\% } C_{18,2} + \text{wt\% } C_{18,3} + 2.5905) \quad (0 < 100)$$

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. RESULTS AND DISCUSSION

The composition, quality and quantity of fatty acids in biodiesel of coconut oil were examined through gas Chromatography and Mass spectroscopy analysis. The biodiesel extracted from the coconut oil had ten fatty acids, namely Palmitic acid: 33.80%,

Palmitoleic acid: 2.40%, Stearic acid: 10.60%, Oleic acid: 26.00%, Linoleic acid: 17.10%, Linolenic acid: 4.00%, Arachidic acid: 3.4%, Gondoic acid: 1.5%, Behenic acid: 0.5% and Linoceric acid: 0.7%. The highest carbon Carbon chain $C_{16} - C_{18}$ of 95.4% was recorded in coconut diesel while percentage composition of saturated, monounsaturated and polyunsaturated fatty acids in the biodiesel extracted from lipids were 49%, 29.9% and 21.10% respectively (Table 1).

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

Structure	Fatty Acids	Molecular Formular	<i>Coconut biodiesel</i>
			Percentage Composition of each Fatty Acids
$C_{16:0}$	Palmitic Acid	$C_{16}H_{32}O_2$	33.80
$C_{16:1}$	Palmitoleic Acid	$C_{16}H_{30}O_2$	2.40
$C_{18:0}$	Stearic Acid	$C_{18}H_{36}O_2$	10.60
$C_{18:1}$	Oleic Acid	$C_{18}H_{34}O_2$	26.00
$C_{18:2}$	Linoleic Acid	$C_{18}H_{32}O_2$	17.10
$C_{18:3}$	Linolenic Acid	$C_{18}H_{30}O_2$	4.00
$C_{20:0}$	Arachidic Acid	$C_{20}H_{40}O_2$	3.4
$C_{20:1}$	Gondoic Acid	$C_{20}H_{38}O_2$	1.50
$C_{22:0}$	Behenic Acid	$C_{22}H_{44}O_2$	0.5
$C_{24:0}$	Linoceric Acid	$C_{24}H_{48}O_2$	0.7

$C_{16} - C_{18}$ (%)	95.4
Saturated Fatty Acid (%)	49
Monounsaturated Fatty Acid (%)	29.9
Polyunsaturated Fatty Acid (%)	21.10

The specific gravity of the coconut biodiesel is 0.8600. The specific gravity obtained conforms satisfactorily to stipulated standard as required by US (ASTM D6751-08) and EUROPE (EN14214) (Table 2). The specific gravity was found to be lower than those of palm kernel biodiesel (0.883), rapeseed biodiesel (0.876) [26], and sunflower biodiesel (0.876) [27], but was higher than that of petrol diesel (0.853) [26] and almost the same with that of canola biodiesel (0.869) [27].

The kinematic viscosity is 1.83 (Table 2). The viscosity of the coconut methyl esters at 40 °C was 1.83 mm²/s; this value is far lower than that of the oil, but was found to be within the limits of ASTM D6751 (1.9 – 6.0 mm²/s) biodiesel fuel standard. The viscosity of the coconut biodiesel was found to be lower than those of Jatropha oil biodiesel (4.80 mm²/s) as reported by Rashid *et al.* [28], canola oil biodiesel (4.892 mm²/s) [26], olive oil biodiesel (4.00 mm²/s), [29] and then that of rapeseed biodiesel (6.170 mm²/s) [26].

The flash point (134) of biodiesel obtained is within the US (ASTM D6751- 08) 130min and EN 14214 120min biodiesel standard (Table 2). The value was also found to be above the minimum value (120 °C) of the EN 14214 and (130°C) of the ASTM D6751 biodiesel fuel standard. The flash point of the coconut biodiesel was reported by Alamu *et al.* [26] to be lower than that of palm kernel biodiesel (167°C), Canola biodiesel (177°C), sunflower biodiesel (178°C), and olive biodiesel (182°C), and rapeseed methyl ester (181°C) as observed by Anastopoulos *et al.* [29]. These relatively higher flash point values of coconut methyl ester is indicative of the presence of little or no residual alcohol and is of prime importance for prevention of fire outbreak in the compressor engine when used as fuel, also important for storage and transportation of the fuel [30].

The cloud point (-5°C) of the biodiesel in this study is within the limits of ASTM D6751 (-3 to 12°C) biodiesel fuel standard. This was similar to reported work of Alamu *et al.* [26] that the cloud point of coconut biodiesel was higher compared to that of canola ethyl esters (-6 °C), rapeseed ethyl esters (-10 °C) and extremely higher than that of petrol diesel (-12°C) but lower to those of palm kernel biodiesel (6°C).

The pour point of (-9) biodiesel satisfied required standard range (Table 2). The lowered pour point and cloud point implies that the coconut oil ethyl ester can be used as fuel in regions where the temperature is within the range of -5 to -10°C. The pour point of the coconut oil ethyl ester was found to be within the limits of ASTM D 6751 (-15 to 10°C) biodiesel fuel standard. The value was lower compared to those of sunflower ethyl esters (-6°C), rapeseed ethyl esters (-8°C) and olive ethyl esters (-5°C) as reported by Anastopoulos *et al.* [29], but higher than that of petrol diesel which is -12°C as shown by Alamu *et al.* [26] and coconut methyl esters (12°C) being reported by Kumar *et al.* [31].

The volatile matter for the coconut biodiesel is 99.65.

The value of refractive index (1.44) obtained for Coconut biodiesel conform satisfactorily to required biodiesel standard (US (ASTM D6751- 08) (Table 2). The refractive index of coconut methyl ester is 1.44. This value was almost the same to that of palm oil methyl ester (1.430) and ghee methyl ester (1.431) [32], but lower than that of groundnut oil methyl ester (1.463) [33].

The standard value for heat of combustion as stipulated by US (ASTM D6751- 08) and EN 14214 is min 35, the value recorded for Coconut diesel (36.98) meet the stipulated standard (Table 2). The heat of combustion of the coconut biodiesel is 36.98 MJ/kg. Although, this value was lower than that of petrol diesel (45MJ/kg) as reported by Lang *et al.* [27], the heat of combustion of the coconut biodiesel in this study is comparable to those of sunflower (38.6 MJ/kg), olive methyl ester (39.2 MJ/kg) [29] rapeseed methyl ester (40.97 MJ/kg) and linseed methyl ester (39.65 MJ/kg) as reported by Lang *et al.* [27].

The density of Coconut biodiesel is 0.8600 and it fall within the required standard range (0.86 – 0.90g/cm³) (Table 2).

The ash content is a measure of the amount of residue left when the fuel is heated to 600°C [34]; this was found to be 0.02% for the coconut oil ethyl ester (Table 2). The ash content of the coconut methyl ester was found to be above the limit of ASTM D 6751 (0.01%), but was within the limit of EN 14214 (0.02%) biodiesel fuel standard. The ash content of the coconut methyl ester was also comparable to that of jatropha (0.016%) as reported by Rashid *et al.* [35]. The slight increase of the ash content of the coconut biodiesel above the biodiesel fuel standard could be as a result of the presences of little amount of metal contaminants.

The peroxide value of the coconut oil ethyl ester was 1.50meq/Kg. This value was lower when compared to that of the coconut oil and groundnut oil methyl ester (3.23meq/kg) as reported by Ibeto *et al.* [33]. The low peroxide value of the coconut 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.

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 [34]. The conductivity of the ethyl ester IS 0.00µS/cm, this could be due to proper drying and short storage time before the test was carried out.

The acid value (0.20mgKOH/g) is within the ASTM D6751- 08 and EN 14214 standard value of 0.5 for biodiesel (Table 2). The acid values of the coconut oil ethyl ester was found to be within the limits of the ASTM D6751 (0.5mgKOH/g maximum) and EN 14214 (0.5mgKOH/g maximum) biodiesel fuel standards and almost the same to that of canola oil ethyl ester (0.265mgKOH/g) [27]. The acid value of the coconut oil ethyl ester was lower to those of sunflower oil ethyl ester (0.610mgKOH/g), linseed oil ethyl ester (0.324mgKOH/g) as observed by Lang *et al.* (2001) and rapeseed oil ethyl ester (1.02mgKOH/g) as reported by Anastopoulos *et al.* [29]. The acid value of the coconut oil ethyl ester was also lower to that of its oil but higher than that reported for coconut oil methyl ester (0.18 mgKOH/g) as observed by Kumar *et al.* [31]. The low acid value of the coconut oil ethyl ester

indicates that the fuel contains relatively little or no water which could hydrolyse the biodiesel to free fatty acids. It could also indicate that the acid pretreatment done reduced the free fatty to the minimal. This high value, though, lower than that of the coconut oil, indicates a low average molecular weight.

The saponification value (mgKOH/g) of 220 was obtained for biodiesel extracted from oil coconut while value recorded for Peroxide (meq/kg) is 1.50 (Table 2). The saponification value of the coconut 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) [29].

The biodiesel had iodine values (41.81mgI₂/g) within the ASTM D6751- 08 and EN 14214 standard value of 120 (maximum). The result recorded for free fatty acids (%) in biodiesel of coconut diesel is 1.14 (Table 2). The iodine value of the coconut oil ethyl ester was found to be 1.81mgI₂/g; this implies lower degree of unsaturation and better oxidative stability of the coconut oil ethyl ester. The iodine value of the coconut oil ethyl ester was found to be within the limits of EN 14214(120 mgI₂/g maximum) biodiesel fuel standard, but was lower than that of Jatropha methyl ester (104 mgI₂/g) [36].

The value of cetane number of biodiesel obtained in this study fall within the ASTM D6751- 08 and EN 14214 biodiesel standards. Bello *et al.* [37] 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 coconut oil biodiesel had empirically calculated cetane number of 70 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 coconut biodiesel (methyl ester) was found to be higher than some conventional biodiesels such as jatropha oil biodiesel (55) [38] and waste cooking oil biodiesel (10.96) [39]. Thus, the higher cetane number of coconut biodiesel indicates a shorter ignition delay time.

Oxidative stability (hr) of 6.56 observed in biodiesel obtained from Coconut diesel conforms satisfactorily within ASTM D6751-08 and EN 14214 biodiesel standard (Table 2).

The biodiesel obtained from Coconut had 24.2 long chain saturated factor (LCSF) while 66.21 was recorded for Cold filter plugging point (Table 2). The degree of unsaturation and higher heating value of Coconut biodiesel are 65.8 and 41.24 while water content is 0.09 (Table 2).

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

S/N	Physicochemical parameters	Coconut diesel	USA (ASTM) D6751-08	EUROPE (EN 14214)
1	Specific Gravity	0.8600	0.88	
2	Kinematic Viscosity (mm ² /s) at 40 ⁰ C	1.83	1.9-6.0	3.5-5.0
3	Flash Point (°C)	134	130min	120min
4	Cloud Point (°C)	-5	-3-+12	-
5	Pour Point (°C)	-9	- 15 - +10	-
6	Volatile Matter (%)	99.65		
7	Refractive Index	1.44	1.479	
8	Heat of Combustion (MS/kg)	36.918	35min	35min
9	Conductivity (MS/cm)	0.00		

10	Density (g/cm ³)	0.8600	0.86-0.90	
11	Ash Content (%)	0.02	0.01	0.02
12	Acid Value (mgKOH/g)	0.26	0.5	0.5
13	Saponification Value (mgKOH/g)	220		
14	Peroxide Value (meq/kg)	1.50		
15	Iodine Value (mgI ₂ /g)	41.81		120max
16	Free Fatty Acid (%)	1.14		
17	Cetane Number	70	47min	51min
18	Oxidative Stability (hr)	6.56		3.0min
19	Long Chain Saturated Factor (LCSF) ⁰ C	24.2		
20	Cold-Filter Plugging Point (CFPP)	66.21		
21	Moisture Content (%)			
22	Degree of Unsaturation	65.8		
23	High Heating Value (HHV)	41.24		
24	Water Content	0.09	500ppm	500max
25	Calorific Value (Kcal/kg)			

4. CONCLUSION

The process of biodiesel production from coconut kernel is result oriented. Various characterization methods adopted for determine the quality of methyl ester revealed biodiesel of quality standard compare to fossil fuel biofuel. This is an indication that the lipid /oil extracted from kernel of coconut that formed diesel through transesterification process, is one of the measures to put an end to environment hazards and havoc experienced as result of continuous reliance on fossil fuel. From the findings of the above work, it can be suggested that the coconut kernel is one of the viable and starting raw materials 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).

5. REFERENCES

1. Jha, M. K., Gupta, A. K. and Kumar, V. (2007). Kinetics of transesterification on *Jatropha curcas* oil to biodiesel fuel. *World Congress on Engineering and Computer Science (WCECS)*, **99**: 978-988.
2. Demirbas, A. (2009). Progress and recent trends in biodiesel fuels., *Energy Conversion and Management*, **50**: 14-34.
3. National Biodiesel Board (1996). *Biodiesel Report*. Summary of 1998 USDA/DOE Biodiesel Life Cycle Analysis. National Biodiesel Board.
4. Issariyakul, T., Kulkarni, M. G. Dalai, A. K. and Bakhshi N. N. (2007). Production of biodiesel from waste fryer grease using mixed methanol/ethanol system. *Fuel Processing Technology*, **88**: 429-436.
5. Demirbas, A. and Demirbas I. (2007). Importance of rural bioenergy for developing countries. *Energy Conversion and Management*, **48**: 2386-2398.

6. Ma, F. and Hanna, M. A. (1999). Biodiesel production: A review. *Bioresource Technology*, **70**:1–15.
7. Munack A., Schroeder O., Krahl J., Buenger J., *Comrison of relevant exhaust gas emissions from biodiesel and fossil fuel*, Agricultural Engineering International, 2001, Available at: <http://cigr-ejournal.tamu.edu/Volume3.html>.
8. Alamu, O. J., Waheed, M. A., Jekayinfa, S. O. and Akintola, T. A. (2007). Optimal transesterification duration for biodiesel production from Nigerian palm kernel oil. *International Commission of Agricultural Engineering (CIGR, Commission Internationale du Genie Rural) E-Journal* **9**: 225-239.
9. Mandal, M. D. and Mandal, S. (2011). Coconut (*Cocos nucifera* L.: Arecaceae): In: Health promotion and disease prevention. *Asian Pacific Journal of Tropical Medicine*, **4**: 241-247.
10. Russell, P. J. and Williams, A. (1995). *The Nutrition and Health Dictionary*. Chapman and Hall, New York. Pp. 443-446.
11. Bligh, E. G. and Dyer, W. J. A. 1959. A rapid method of total lipid extraction and purification, *Canadian Journal of Biochemistry and Physiology* **37**:911-917
12. Nascimento IA, Marques SSI, Cabanelas ITD, Pereira SA, Druzian JI, de Souza CO, Vich DV, de Carvalho GC, Nascimento MA. 2013. Screening microalgae strains for biodiesel production: lipid produc- tivity and estimation of fuel quality based on fatty-acids profiles as selective criteria. *Bioenerg Res* **6**:1–13
13. Lepage, G., Roy, C.C., 1984. Improved recovery of fatty acid hrough direct transesterification without prior extractionor purification. *J. Lipid Res.* **25**, 1391-1396
14. Anitha, S* and Sriman Narayanan, J. 2012. Biodiesel Production From Chlorella Vulgaris With Special Emphasis On Immobilized Lipase Catalyzed Transesterification, *International Journal of Recent Scientific Research Research*, Vol. 3, Issue, 9, pp. 733 - 737, September, 2012
15. Medina AR, Grima EM, Gimenez AG, Ibanez MJ. Downstream processing of algal polyunsaturated fatty acids. *Biotechnology Advances* 1998; **16**(3):517–80.
16. Schreiner, M. (2006) Optimization of solvent extraction and direct transmethylation methods for the analysis of egg yolk lipids. *Int J Food Prop* **9**, 573– 581.
17. ASTM Standard D6751 2009. Standard specification for biodiesel fuel blend stock (B100) for middle distillate fuels. ASTM International, West Conshohocken, PA. <http://www.astm.org/standard/D6751.htm>. downloaded on November 15th, 2012.
18. AOCS, Cd 3-25 1998. Official and tentative methods of American oil Chemists Society. 3rd edition. Champaign, IL.
19. Association of Official Analytical Chemists 1998. Official methods of analysis of the Association of Official Analytic Chemists (AOAC). 16th Edn, Gaithersburg, MD.
20. American Society for Testing Material, (1998). D 97-96a, D 613, D 93, D 5773 and D 664. In: Annual book of ASTM Standards, vol. 05.01. ASTM, Philadelphia, PA, USA.
21. ASTM Standard D874. 2007. Standard test methods for sulfated ash from lubricating oils and additives. ASTM International, West Conshohocken, PA. <http://www.astm.org/standard/D874.htm>. Downloaded on November 15th, 2012.
22. Alamu OJ, Waheed MA, Jekayinfa SO (2008). Effect of ethanol-palm kernel oil ratio on alkali-catalyzed biodiesel yield. *Fuel*, **87**(8-9): 1529- 1523
23. Islam, M.A., Magnusson, M., Brown, R.J., Ayoko, G.A., Nabi, M.N., Heimann, K., 2015. Microalgal species selection for biodiesel production based on fuel properties derived from fatty acid profiles. *Energies*, **6**(11), 5676-5702.
24. Ramírez-Verduzco, L. F., Rodríguez-Rodríguez, J. E. and Jaramillo-Jacob, A. R. 2012. “Predicting Cetane Number, Kinematic Viscosity, Density and Higher Heating Value of Biodiesel from Its Fatty Acid Methyl Ester Composition,” *Fuel*, **91** (1) :102-111.
25. Park, J. Y., Kim, D. K., Lee, J. P., Park, S. C., Kim, Y. J. and Lee, J. S. 2008. Blending effects of biodiesels on oxidation stability and low temperature flow properties. *Bioresource Technology*, **99**: 1196–1203
22. Alamu OJ, Waheed MA, Jekayinfa SO (2008). Effect of ethanol-palm kernel oil ratio on alkali-catalyzed biodiesel yield. *Fuel*, **87**(8-9): 1529- 1523

27. Lang, X., Dalai, A. K., Bakhshi, N. N., Reaney, M. J. and Hertz, P. B. (2001). Preparation and characterization of bio-diesels from various bio-oils. *Bioresource Technology*, **80**:53-62.
28. Rashid, U., Anwar, F., Jamil, A. and Bhatti, H. N. (2012). Jatropha curcas seed oil as a viable source for biodiesel. *Pakistan Journal of Botany*, **42**(1): 575-582.
29. Anastopoulos, G., Zannikou, Y., Stournas, S. and Kalligeros, S. (2009). Transesterification of vegetable oils with ethanol and characterization of the key fuel properties of ethyl esters. *Energies*, **2** :362-376.
30. Moser, B. R. (2009). Biodiesel production, properties, and feedstocks. *In Vitro Cellular and Developmental Biology—Plant*, **45**:229–266.
31. Kumar, G., Kumar, D., Singh, S., Kothari, S., Bhatt, S. and Singh, C. P. (2010). Continuous low cost transesterification process for the production of coconut biodiesel. *Energies*, **3**:43-56.
32. Deshpande, P. and Kulkarni, K. (2012). Production and evaluation of biodiesel from palm oil and ghee (clarified butter). *Chemical and Process Engineering Research*, **2**:33-42.
33. Ibeta, C. N., Ofoefule, A. U. and Ezeugwu, H. C. (2011). Fuel quality assessment of biodiesel produced from groundnut oil (*Arachis hypogea*) and its blend with petroleum diesel. *American Journal of Food Technology*, **6**(9): 798-803.
34. Sanford, S. D., White, J. M., Shah, S. P., Wee, C., Valverde, M. A. and Meier, G. R. (2009). Feedstock and biodiesel characteristics report. Renewable Energy Group, Inc, 11-136 www.regfuel.com.
35. Rashid, U., Anwar, F., Jamil, A. and Bhatti, H. N. (2010). Jatropha curcas seed oil as a viable source for biodiesel. *Pakistan Journal of Botany*, **42**(1): 575-582.
36. Singh, B. K. and Padhi, S. K. (2009). Characterization of jatropha oil for the preparation of biodiesel. *Natural Product Radianance*, **8**(2):127-132.
37. Bello, I. E., Mogaji, S. T. and Agge, M. (2011). The effects of transesterification on selected fuel properties of three vegetable oils. *Journal of Mechanical Engineering Research*, **3**(7):218-225.
38. Reddy, J.N. and Ramesh, A. (2005). Parametric studies for improving the performance of a jatropha oil fuelled compression ignition engine. *Renewable Energy*, **31**:1994-2016.
39. Owolabi, R. U., Osiyemi, N. A., Amosa, M. K. and Ojewumi, M. E. (2011). Biodiesel from household/restaurant waste cooking oil (WCO). *Journal of Chemical Engineering Technology*, **2**:4-10