PRODUCTION AND CHARACTERIZATION OF COCONUT (*COCOS NUCIFERA*) OIL AND ITS METHYL ESTER

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ABSTRACT

This work considers the use of coconut oil for the production of alternative renewable and environmental friendly biodiesel fuel as an alternative to conventional diesel fuel. Test quantities of coconut oil biodiesel were produced through transesterification reaction. To overcome the high kinematic viscosity of the neat oil, a high molar ratio of 4:1 was used to produce the methyl ester (biodiesel). The biodiesel was characterized using the American Society for Testing and Materials (ASTM) D6751-02 limits for biodiesel. The results of the characterization obtained were within the ASTM D6751-02 limits for biodiesel and similar to those of diesel fuel, thus confirming that it can be used as alternative fuel for diesel engines.
Chromatography analysis of the coconut oil methyl ester shows that it has a total saturation and unsaturation of 94.8% and 5.2% respectively. The 94.8% level of saturation indicates oxidation stability of the coconut methyl ester while 5.2% of unsaturation indicates the level of reactivity to oxidation which makes it less prone to bacterial growth.

Keywords: Coconut oil, biodiesel, characterization, saturation, fatty acid profile.

INTRODUCTION
Background

Various global concerns such as the dwindling crude oil reserve, global warming and climatic change, air pollution and public health, and more importantly the steady rise in the cost of fossil fuel, have altogether received great interest in the use of renewable fuels. One of the most promising alternatives fuel that has properties close to those of diesel is vegetable oils and their derivatives (Leung et al., 2013; Koh and Ghazi, 2011; Van Gerpen et al., 2000). The pioneering investigation on the use of vegetable oil as fuel in compression ignition engine was undertaken by the inventor of the diesel engine -Rudolph Diesel, who used peanut oil to run his diesel engine. The long term use of vegetable oils has however led to operational problems such as injector coking and the thickening of crankcase oil which resulted in piston ring sticking. Therefore, neat vegetable oils are not used in diesel engines because of endurance issues (Ma and Hanna, 1999; Graboski et al., 1998). The use of vegetable oil as fuel for diesel engine did not prosper much then because of the discovery of diesel fuel. However, in 1912, Rudolf Diesel - who invented and designed the diesel engine, predicted that "*the use of vegetable oils for engine fuels may seem insignificant today, but such oils may become in the course of time as important as petroleum and the coal tar products of the present time*" To overcome the problems associated with the direct use of vegetable oil, various modifications of vegetable oils such as transesterification, micro-emulsion formation and the use of viscosity reducers have been tried. Among them, transesterification was considered the most suitable because the properties of the esters are similar to those of diesel (Ma and Hanna, 1999). Through transesterification, vegetable oils are converted to the alkyl esters of the fatty acids present in the vegetable oil (Ramadhas et al., 2005; Chhetri et al., 2008). These esters are commonly referred to as biodiesel. Biodiesel is an alternative fuel that is renewable in the sense that its primary feedstock has a sustainable source. Some other feed stocks that can be converted to biodiesel includes waste restaurant grease and animal fats, which are less expensive than vegetable oil.

In view of the recurrent instability in oil prices and the fact that crude oil is a wasting asset, biodiesel stands as an attractive and alternative source of energy. While it is worthy to note that biodiesel may not completely displace petroleum diesel at present, biodiesel has its place as an alternative fuel and can be a source of lubricity enhancing additive to diesel fuel. The emissions produced from biodiesel are cleaner compared to petroleum-based diesel fuel. Particulate emissions, soot, and carbon monoxide are lower since biodiesel is an oxygenated fuel and burns more completely. However, emissions of oxides of nitrogen (NO_X) are higher when biodiesel is used due to the high temperature and pressure in the engine that promotes dissociation during combustion (Antolín et al, 2002; Al-Widyan and Al-Shyoukh, 2002; Ma et al., 1999).

One particular problem of biodiesel is its cold flow properties. Neat biodiesel such as methyl soyate has a pour point (i.e. the lowest temperature at which the fuel is pourable) of -3°C (Peterson et al., 1990; Ramadhas et al., 2009). In colder climates, crystallization can occur, which can lead to the plugging of fuel filters and lines thus starving the engine of fuel. Considering that the production costs of biodiesel is higher than fossil diesel fuel, its use as a diesel replacement fuel still poises a major economic problem since fuel costs is often the largest part of the operating cost of transportation. The main benefits however, are in the area of energy security, saving in foreign exchange and the environment friendliness.

Chemical Properties of Vegetable Oils

Vegetable oil consists of triglycerides that are molecules of glycerol bounded to three fatty acid molecules. Different plant oils have different compositions and this explains why different triglycerides have different physico-chemical properties and they coagulates at different temperatures (Bello et al., 2014; Knothe, 2005).

The oxygen content is the most important difference in the chemical composition between fossil oils and vegetables oils. Vegetable oil contains 10% - 20% oxygen, whereas fossil fuel normally contain insignificant amounts of oxygen. The oxygen content affects both specific energy and combustion properties of the oils because the heating value of oxygen is lower than that of hydrogen and carbon molecules that are also present in the fuel. Vegetable oils are polar compounds that have high lubricity which helps to lubricate the fuel systems (Narasimharao et al., 2007; Koh and Ghazi, 2011).

Coconut oil

For many decades, coconut (*Cocos nucifera*) known as the “tree of life”, has been preferred raw material for the production of soap and cosmetics. As food item, coconut oil provides many health benefits being anti-viral, anti-bacterial, anti-fungal, anti-microbial and many more (Dr.Bruce, 2009). Coconut oil like any other vegetable oils and animal fats are triglycerides, inherently containing glycerin. Transesterification turns the oils into esters, separating out the glycerin from the main product (biodiesel). The heavier glycerin sinks to the bottom and the biodiesel floats on top and is separated using separating funnel. The process substitute's alcohol for the glycerin, using a catalyst. It has one of the highest oil contents among plant oils (Setiawan et al., 1997).

MATERIALS AND METHODS
Materials

Food grade coconut oil was procured from a local dealer and filtered to remove solid particles before use. Anhydrous Methanol of 99.95% purity, potassium hydroxide (KOH) and other chemicals used were all of analytical reagent grade and obtained from Finlab Nig. Ltd..

TRANSESTERIFICATION PROCESS
Transesterification and separation

Potassium methoxide was prepared by dissolving 2.5g of KOH in 1 litre of methanol. 20 ml of coconut oil was mixed with 100 ml of potassium methoxide and stirred at 600 rpm and a reaction temperature of 60^{o}C for 2 hours in a processor. The mixture was poured into a separating funnel and allowed to stay overnight for the reaction to be completed and for the mixture to separate into two layers of biodiesel and denser glycerol at the bottom. The glycerol was drained off and the biodiesel was washed with distilled water of volume ratio 3 to 1 three times by gently stirring to remove impurities such as diglycerine and monoglycerine, catalyst, soap and excess methanol, which can affect combustion and exhaust emission (Canakci and Van Gerpen, 1999; Anastopoulos et al., 2009). It was allowed to settle for 2 hours to separate into two layers of pure biodiesel and hydrated methanol, which was separated using separating funnel. The excess methanol in the ester was removed in a flash evaporator. The biodiesel yield was calculated from the weights biodiesel and oil using equation 2:

$$\text{Biodiesel yield \%} = \frac{Weight\ of\ oil\ used}{weight\ of\ biodiesel\ produced} \qquad 2$$

Characterization

The coconut oil and biodiesel were characterized according to ASTM D6751 and EN 14214 protocols.

Fatty Acid Profile

The components and composition of the coco nut oil and biodiesel were analyzed using Gas Chromatography (GC)-Mass Spectrometry (MS) and GC-FID (Flame Ionization Detector) respectively. The GC and MS were performed on an Agilent 7890A GC which was equipped with a FID and used Hydrogen as carrier gas for the GC and Agilent 5975 quadrupole Mass Selective Detector (MSD) with helium as carrier gas was used for the MS. Separation was achieved on a fused silica capillary type column HP-5 (25 m × 0.25 mm x 0.40 μm) with split ratio 40:1. The mass spectrometer was operated in the electron impact mode at 70 eV ionization energy and scanned from 30 Da to 600 Da. MS identification of the products was based on molecular mass, fragmentation patterns and by matching the spectra with a digital library. GC and MSD Chemstation software was used for data acquisition and analysis. The flow rate was 0.8 mL/min, injection volume was 1μL and pressure 5.6 psi. The oven initial temperature was at 60 ^{o}C and procedure for the analysis is as explained by Bello et al., (2013).

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RESULTS AND DISCUSSION

The Physico-chemical properties of the coconut oil and it's Methyl Ester (CME) (biodiesel) were measured. The methods and results obtained are shown in Table 4.1.

Table 4.1 Physico-chemical properties of Coconut Methyl Ester (CME) and coconut oil

Property	**ASTM Specification**	**Coconut oil**	**CME**	**ASTM Protocol**
Specific gravity 15 °C (Kg/m^3)	0.575 to 0.900	0.9251	0.8733	D 1298
Cloud point (°C)	–3 to +12	0.3	–3	D2500
Pour point (°C)	−15 to +10	−6	−12	D2500
Cold filter plugging point (°C)	−4 to −9	−12	−5	D 6371
Kinematic viscosity at 40 °C (mm^2/s)	1.9 to 6.0	27.23	2.83	D445
Flash point (°C)	100, min	266	110	D93
Fire Point (°C)	-	200	207	D93
Heating value (MJ/Kg)	-	37	36.1	D240
Cetane number	47, min	52	70	D 613
Moisture content (ppm)	360, max	332	300	D2709
Water content and residue (vol %)	0.05, max	0.04	0.0	D2709
Acid number (mgKOH/g)	$0.5, max$	2.1	0.18	D664
Iodine value (mgI_2/g)	-	10	8	EN14111
Perioxide value (meq/kg)	-	16	6.0	EN14111
Oxidation stability (IP, h)	6, min	12	9.2	EN14112
Saponification value (mgKOH/g)	120, max	190	93	EN1411
Unsaponifiable matter (%)	-	20	41	-
Copper strip corrosion (3 hrs at 100 °C)	< No. 1.0	1.0	1.0	D6751
Phosphorus (mg/kg)	0.001, max	0.0004	0.001	D4951
Sulphur (mass %)	0.05	1.3	0.002	D5453
Free glycerin (mass %)	0.020,max	0.062	0.008	D6584
Total glycerin (mass %)	0.24, max	0.32	0.24	D6584
Methanol (%)	-	-	0.085	-

Specific Gravity

The crude coconut oil has a specific gravity of 0.9251 kg/m^3 that reduced to 0.8733 kg/m^3after transesterification, which is still higher than 0.855 kg/m^3 for diesel fuel but can be further reduced by blending with diesel fuel (Bello and Makanju, 2011). Density is important in determining the mass of fuel flowing into the engine.

Cold Flow Properties

The cloud point is the temperature at which wax first become visible as the temperature is lowered, pour point is the temperature at which the oil has solidifies enough to resist flow while the cold filter plugging point is the temperature at which the fuel will solidify and block the flow of fuel through the filter. These properties can affect fuel flow and the performance of fuel pump and injector. The cloud point of -3^{o}C, pour point of -12 °C and cold filter plugging point of -12^{o}C makes it a suitable fuel for cold temperature and high altitude aviation applications.

Flash and Fire Points

Flash point, which is the temperature at which the fuel can ignite when exposed to a heat source is important from the point of view of safe handling, storage and transportation. The flash points of 266°C and 110°C for coconut oil and biodiesel respectively compared to 50°C for diesel fuel. Coconut oil biodiesel can be classified as a non hazardous fuel because of its high flash point. The fire point of 200^{o}C for the oil is also very high and makes it a suitable oil for deep frying of items like potatoes.

Heating Value

The heating value is the amount of energy produced when one kg of the fuel is burnt. Using a bomb calorimeter, the heating value obtained for biodiesel was 36.1 MJ/kg and is lower than the 42.5 MJ/kg for diesel fuel. The heating value is lower because of the effect of the about 10% oxygen content of biodiesel which has lower calorific value.

Cetane number

Cetane number is widely used as diesel fuel quality parameter related to ignition delay time and combustion quality. The Cetane number of biodiesel of 70 is one of the highest amongst vegetable oils. Even the 50 for the oil, which is above the minimum for biodiesel will allow is to be used directly in diesel engine with high combustion sufficiency, except of course for its adverse flow cold properties. High cetane number is good for improved burning ability, better fuel consumption, reduced engine noise, lower emission, wider energy release spectrum due to slower burning and hence better torque and power output characteristics (Dreamer et al., 2005; Bello and Agge, 2011).

Iodine value

Iodine value is the number of mg of KOH required to convert 1 gm of fat into glycerine/soap. It is hence a measure of the degree of unsaturated of vegetable oils, low iodine value means low content of unsaturated fat acids, hence reduced vacant bonds which translates to less reactivity of the fuel, tendency to polymerize, and better storage stability. The iodine values are 10 mgI_2/g and 8 mgI_2/g for the oil and biodiesel respectively these are some of the lowest for plant oils. These values are quite low compared to the acceptable maximum of 100 mgI_2/g for fuels to be used in internal combustion engines. The low iodine value will result in much reduced carbon deposits on engine internal parts and the tendency to block the holes on the injectors.

Peroxide value

This is an indicator of oil autoignition and high value is an indication of high degree of rancidity. The peroxide value of the oil was 16 meq/kg while that of the biodiesel was 6 meq/kg.

Saponification value

The saponification value is one of the highest for vegetable oils making it suitable feedstock for the manufacture of soaps, detergents and shampoo products. The oil has a value of 190 mgKOH/g which reduced to 93 mgKOH/g for the biodiesel.

Unsaponifiable matter

This is all the matter remaining after the oil has been saponified. This parameter is significant as it affects the properties of the fuel and glycerol. The oil contained 20% while the biodiesel contains 40%.

Acid Value

This is the quantity of base required to titrate a sample to a specified end point. It is a measure of free fatty acid in the biodiesel. High acid value of the fuel can be corrosive and may be a symptom of water in the fuel, poor production or oxidative degradation. Excessive free fatty acid in the fuel can lead to soap formation which tends to inhibits the transesterification process. The acid values for the oil and biodiesel are 2.1 mg/KOHg and 0.18 mg/KOHg respectively.

Water and residue and moisture contents

Water can be detrimental to the operation of fuel system because of its surface tension and its freezing point, which is above that of most biodiesel fuels. Water can react with fatty acids to form acidic solution which can degrade lubricating oil and as well as the biodiesel. The oil contained 0.04% water and residue but the biodiesel contained negligible amount. The moisture contents of the oil and biodiesel are 332 ppm and 300 ppm respectively. The quantity of water in the oil will depends on the method of manufacture with wet method being higher than the dry method.

Cold filter filtration

This test involved chilling down the biodiesel filtering it then warming it up again to remove any impurities such as sterols, which, if not removed, could the clogged filters. The cold filtration temperature of the coconut oil and its biodiesel are -12 and -5^{o}**C** respectively and are well below freezing point of water making a possible fuel in cold regions application.

Copper strip corrosion

This is a measure of the posterity of the fuel to corrode copper and copper alloyed components it comes into contact with. The values for the oil and biodiesel are both 1, which is lower than the value for diesel and within the limits for biodiesel.

Phosphorus

This element originates from the phospholipids in the fuel and has been reported to affect the effectiveness of the metals in exhaust systems catalytic converter. The value for the oil is 0.0004 mg/kg. and for the biodiesel 0.001 mg/kg. Both are low and below the maximum for biodiesel.

Sulphur

Sulphur originates from the soil on which the vegetable oil was grown and confers lubricity on diesel fuel and is mandatory in diesel in the US. It however, has adverse environmental effects as when it comes out of the exhaust system, it is air borned and can result in soiling when deposited on the soil and can cause acid rain. The value for the oil is 1.5 mass % and for the diesel 0.002 mass %.

Total Glycerol and Free glycerol

These are the sum of the concentrations of free glycerol and glycerol bounded in the form of mono-, di- and triglycerides. The concentration depends on the production process. Fuels out of specifications with respect to these parameters are likely to coke more and may thus cause the formation of deposits on injector nozzles, pistons and valves (Bello et al., 2013). Total glycerol affects storage stability which is a measure of how well the quality of fuel will be maintained in storage in contact with air and water. Free glycerin for the oil was 0.062 mass % while the biodiesel is 0.008 mass %. The total glycerol in the oil was 0.32 mass % and 0.24 mass % for the biodiesel.

Methanol

Vegetable oils contains negligible amount of methanol but the amount in biodiesel can be significant due to the use of methanol in the transesterification process that can become residualized and must hence be regulated. The amount in the biodiesel was 0.085 vol % even after washing.

Fatty acid profile

Table 4.2 shows the fatty acids profile of the oil and its biodiesel. Only very minor changes occurred in the % masses of the various fatty acids after transesterification. The dominant acid is lauric acid and the oil and biodiesel contained 54.5% and 48.83% respectively. Lauric acid is indispensable in soap making as good soap must contain at least 15% lauric acid for quick lathering while soap made for sea water use is virtually 100% lauric acid. It confers hardness, solubility and a feel of quality in soap.

Table 4.2: Fatty acid profile analysis of Coconut oil

Fatty Acid		Form	Coco nut Oil (Mass %)	Coco nut oil Methy ester (Mass%)
Caprylic acid	Octanoic	C8:0	4.8%	8.86%
Capric acid	Decanoic	C10:0	4.8%	6.17%
Lauric acid	Dodecanoic	C12:0	54.7%	48.83%
Myristic acid	Tetradecanoic	C14:0	18.8%	19.97%
Palmitic acid	Hexadecaonoic	C16:0	8.3%	7.84%
Stearic acid	Octadecanoic	C18:0	2.8%	3.06%
Oleic acid	9Z-octadecanoic	C18:1	5.0%	4.44%
Linoleic acid	9Z, 12Z-octadecadienoic	C18:2	0.8%	0.76%
Others	-	-	-	0.07%
Total saturation	-	-	94.2	94.8%
Total unsaturation	-	-	5.8	5.2%

The Peaks, Types and Structure of Fatty Acids

The chemical formulae and molecular weights of the components of the oil are as follows:

Caprylic Acid (8:0): Octanoic acid. The chemical formula is $C_8H_{16}O_2$ and molecular weight 146. It has a saturated linear chain structure with a hydroxyl group at the right hand end.
Capric Acid (10:0): Decanoic acid. The chemical formula is $C_{10}H_{20}O_2$ and molecular weight 132. It has a saturated linear chain structure with a hydroxyl group at the right hand end. It is also called Capric acid methyl ester; Metholene 2095; Methyl caprate; Methyl caprinate; Methyl decanoate; Methyl-n-caprate; Uniphat A30; Methyl n-decanoate; n-Capric acid methyl ester; Methyl tridecanoate
Lauric Acid (12:0): Dodecanoic acid. The chemical formula is $C_{12}H_{24}O_2$ and molecular weight 200. It has a saturated linear chain structure with a hydroxyl group at the right hand end. Other names are *n*-Dodecanoic acid, Dodecylic acid, Dodecoic acid, Laurostearic acid, Vulvic acid, 1-Undecanecarboxylic acid, Duodecylic acid.
Myristic (14:0): Tetradecanoic acid. The chemical formula is $C_{14}H_{28}O_2$ and molecular weight 228. It has a saturated linear chain structure with a hydroxyl group at the left hand end. Other names are Myristic acid; n-Tetradecanoic acid; n-Tetradecoic acid; Neo-Fat 14; Univol U 316S; 1-Tridecanecarboxylic acid; Crodacid; Emery 655; Hydrofol acid 1495; Hystrene 9014; n-Tetradecan-1-oic acid; Hystrene 9514; Philacid 1400; Prifac 2942; Prifrac 2942; NSC 5028; Tetradecanoic acid (myristic acid); Acide Myristique; Tetradecanoic (Myristic) acid; Myristic acid (tetradecanoic acid); Tetradecanoic acid (=Myristic acid)
Palmitic (16:0): n-Hexadecanoic acid. It has a saturated linear chain structure with a hydroxyl group at the right hand end. The chemical formula is $C_{16}H_{32}O_2$ and molecular weight 256.
Stearic (18:0). Octadecanoic acid, 2-hydroxyl-1, 3-propanediyl ester. It has a relatively long chain structure with no double bond, chemical structure $C_{39}H_{76}O_5$ and a relatively high molecular weight of 624. It has one hydroxyl and three propanediyl esters that are centrally located giving it a sort of a symmetry.

Oleic acid (C18:1): 9-Octadecenoic acid (Z)-, 2 hydroxyl-1-(hydroxymethyl) ethyl ester. The structure is not linear and it has a double bonds on site 9. Chemical structure $C_{21}H_{40}O_4$ and molecular weight of 354.
Linoleic (18:2): 9,12-Octadecadienoic acid (Z,Z)-. It has double bonds on sites 9 and 12, chemical structure $C_{18}H_{32}O_2$ and molecular weight of 280.

Distillation Characteristics

The distillation characteristic is shown in Fig.1. The distillation from 5 to 10% was very rapid, which shows that it contains very volatile components but thereafter the distillation was fairly uniform. The characteristic can be used to calculate the cetane number of the biodiesel (Willard, 2000).

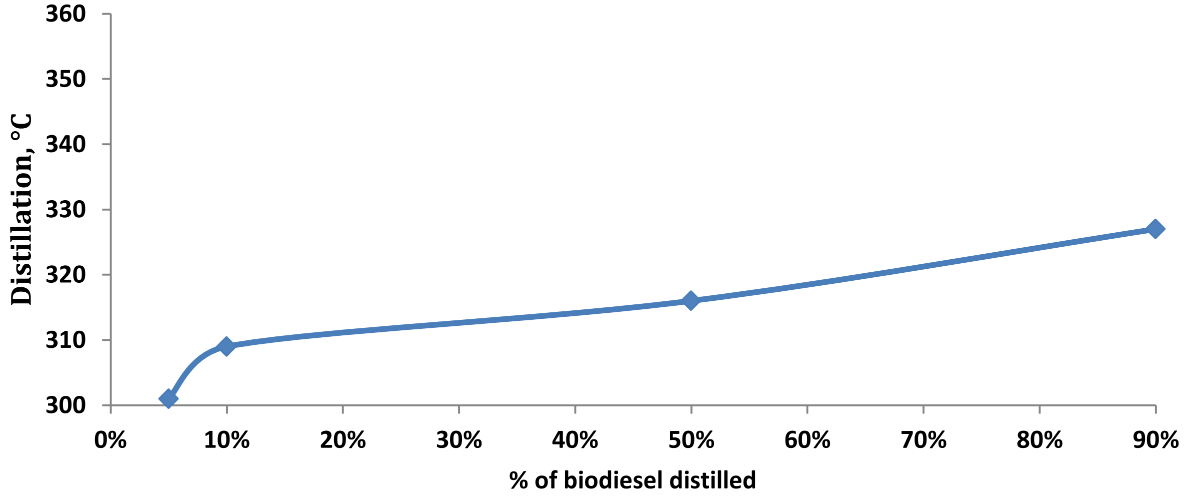

Fig. 1 Distillation Characteristics of coco nut oil biodiesel

CONCLUSIONS

Coco nut oil biodiesel has properties that are close to those of diesel fuel and mostly within the ASTM and EN limits of biodiesel. The biodiesel yield is 95% and the oil is 94.2% saturated with lauric acid constituting 54,7%. The high saponification value of 190 mg/KOHgm makes it a choice oil for the production of soap and allied products. The cetane number of 70 makes it a suitable alternative fuel for diesel engine. The iodine value for the oil and biodieseil are 10 and 8 respectively, which are one of the lowest among vegetable oils and would result in a clean combustion. The cold flow properties are below zero degree making is useable in cold regions.

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