



Synthesis and Characterisation of Biodiesel from Cameroon Palm Kernel Seed Oil

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Authors' contributions

This work was carried out in collaboration between all authors. Author AMB did the sample collection, carried out all the laboratory analyses, wrote the first draft and managed the literature search. Author NMK designed the study and reviewed the manuscript. Author PTN wrote the protocol and reviewed the paper. Author YMS managed all the analyses and proof-read the manuscript. All authors read and approved the final manuscript.

Article Information

DOI: 10.9734/AJB2T/2018/40200

Editor(s):

(1) Fernando Jose Cebola Lidon, Professor, Faculdade de Ciencias e Tecnologia Universidade Nova de Lisboa, Campus da Caparica, Portugal.

Reviewers:

(1) Yash Gupte, India.

(2) Benard A. Udeh, Cyprus International University, Turkey.

Complete Peer review History: <http://www.sciedomains.org/review-history/23824>

Original Research Article

Received 26th December 2017

Accepted 8th March 2018

Published 26th March 2018

ABSTRACT

Aims: This study was aimed at synthesizing and characterizing a biodiesel from Cameroon palm kernel oilseed.

Study Design: Synthesis was carried out on purified kernel oil by trans-esterification using methanol in basic medium.

Place and Duration of Study: Laboratory work was effectively carried out in the Department of Chemical Engineering (Ahmadu Bello University, Nigeria) in the early part of the year 2017.

Methodology: Oil was extracted from Cameroon palm kernel seed, which constitutes an agricultural industrial waste, by mechanical expression, was purified and analysed. Palm kernel oil (PKO) biodiesel was synthesised by trans-esterification in methanol with potassium hydroxide (KOH) as a base at the mass ratio PKO to methanol to KOH of 100:20:1.

Results: The yield of optimised synthesis of biodiesel was 97%, with a useful by-product (glycerol) which has potential pharmaceutical, cosmetic and engineering applications. PKO biodiesel was

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analysed using UV and IR spectroscopy and gas chromatography-mass spectrometry (GC-MS). Biodiesel had the following physical characteristics: specific gravity 0.884 g/mL, ASTM colour 1.5, viscosity at 40°C 4.9080 cSt, viscosity at 100°C 1.7914 cSt, cloud point 17°C, pour point 0°C, flash point 50°C, aniline point 90°F, cetane number 28, and diesel index 25. The following chemical parameters were obtained for the synthesized PKO Biodiesel: sulphur content 0.0328% w/v, acid value 0.4488 mg KOH/g, free fatty acid (FFA) content 0.2244 mg KOH/g, iodine value 7.36 mg I₂/g, saponification value 168.30 mg KOH/g, ester value 154.84 mg KOH/g, hydroxyl value 51.89 mg OH/g and peroxide value 3.20 meq/kg.

Conclusion: These results of physical and chemical characterisation of PKO biodiesel fell in the range reported in the literature for biodiesel and were close to those of diesel fuel.

Keywords: Biodiesel; palm kernel oil; trans-esterification; viscosity; saponification value; glycerol.

1. INTRODUCTION

Worldwide increasing trends in industrialisation, modern technology, environmental concerns and demand for clean energy have continued to fuel research into energy sources to address such human pre-occupations as the gradual depletion of crude oil reserves compounded by the steady surge in the cost of these fossil fuels [1-6]. Vegetable oils and their derivatives are plausible alternatives to diesel fuel [6-9]. Peanut oil was one of the first vegetable oils to be used in a Rudolph Diesel compression ignition engine [10-11]. Since then, a number of attempts have been made to use such oils as coconut, soybean, sunflower, safflower, peanut, linseed, rapeseed and palm oil inter alia in compression ignition (CI) engines. The extended use of vegetable oils in compression ignition engines is undesirable as it has such adverse effects as injector coking and the thickening of crank-case which gives rise to piston ring sticking [11] due to high viscosity. Such predicaments can be overcome by derivatising vegetable oil. A more convenient scientific technique is trans-esterification which converts the vegetable oils to alkyl esters of fatty acids called biodiesel [11-14]. This technique reduces viscosity by 80 to 90%. The properties of these alkyl esters closely resemble those of petro-diesel. Biodiesel is an alternative fuel that is renewable by virtue of the fact that its primary feedstock is sustainable and renewable. Other potential feed stocks that are convertible to biodiesel are waste restaurant grease, animal fat [11,15] and agro industrial waste from palm oil processing plants such as palm kernel seeds. Dara [16] reported that energy production from industrial waste is highly desired due to the attendant environmental sanitisation and a plethora of other socio-economic benefits.

Biodiesel may not completely replace petroleum diesel as an alternative fuel but can act as a

lubricant when used as an additive to petroleum diesel in various ratios. The emissions produced from biodiesel are much cleaner compared to those emanating from petroleum-based diesel fuel. Particulate emissions such as soot and carbon monoxide are lower since biodiesel is much more oxygenated than petroleum-based fuel; the oxygen content of the vegetable oil ranges from 10 to 20% [6,8,17]. This causes the emission of noxious nitrogen oxides when biodiesel reacts with atmospheric nitrogen in the compression engine at high temperatures. Hence, some slight engine modification may be required to include a chamber in the exhaust system that adsorbs nitrogen oxides and chemically transforms them into harmless products. Some feed stocks for biodiesel synthesis such as canola oil, sunflower, amongst others, are regular food sources and their use in energy production can promote a potential food crisis [2,18]. Alternative feed stocks like waste cooking oil, and tallow from animal fat cannot lead to large scale biodiesel production due to their limited supply. A salient feed stock for biodiesel production is palm kernel seed oil because it is sustainable, is readily available, is an industrial waste product, and does not conflict with food security concerns. Furthermore, it has environmental sanitization potential. Palm oil processing plants in Cameroon produce a good quantity of palm kernel seed waste, part of which is used to extract palm kernel oil for the soap and medicinal industry; some of it is burnt to supply heat energy in thermal batch processes and the rest is dumped into the environment, constituting land pollution (industrial waste). There has been a recent increase in the production of palm oil in Cameroon due to a drop in the prices, in the early 1990s, of cocoa and coffee which are major commercial crops [19-21]. The above implies a proportional increase in industrial waste from the palm oil processing. In a recent study, Nchanji et al. [21] showed that the extraction of palm kernel

oil is only 4% efficient at the artisanal level. A field outreach in the Widikum sub-division (Cameroon) by the researcher revealed that artisanal palm oil processing used about only 20% of palm kernel to produce kernel oil and related products.

Dara [16] recommended that biomass (such as palm kernel seed) be preferred whenever energy can be produced as a by-product of waste from industrial and related sectors. If this waste could be converted into a vital energy source, the benefits will be manifold, encompassing environment sanitisation through pollution control, an additional return on agricultural investment, an advancement in waste transformation, and a positive contribution to global climate regulation through low gas emission and cleaner fuel. The synthesis and physico-chemical characterization of biodiesel from Cameroon palm kernel seed raw material is reported in this paper.

2. MATERIALS AND METHODS

2.1 Materials

Palm kernel nuts were collected from the dumping site at an artisanal oil mill in Widikum, Momo division, North West region of Cameroon. The nuts were mechanically cracked to separate the seeds from the hard shells. The seeds were sun-dried for two days to reduce the moisture content. A high moisture content hydrolyses the triglycerides and raises the free fatty acid content which reduces the quality of the oil. The dried palm kernel seeds were then kept under anhydrous conditions to be used to extract palm kernel oil by mechanical pressure and solvent extraction. Reagent-grade anhydrous methanol (99.95% pure), potassium hydroxide (KOH), ethanol, ether, and chloroform were used as

purchased without further purification. The extracted palm kernel oil was purified and characterised by physico-chemical methods.

2.2 Methods

2.2.1 Trans-esterification of palm kernel oil

Palm kernel oil methyl ester (biodiesel) was synthesised by a modified method [1,6]. 150 mL of purified PKO was transferred into a heating pan and warmed to 50°C (below the boiling point of methanol). Then, 35 mL of methanol (excess) was transferred into a beaker. 1.3851 g of potassium hydroxide (KOH) was added to the beaker containing methanol. The mixture was magnetically stirred until methanol completely dissolved potassium hydroxide. Warm PKO was transferred into the reaction vessel (500-mL round bottom distillation flask) and the alcoholic KOH solution was added. The contents of the reaction vessel were swirled vigorously for five minutes. The reaction mixture was then refluxed in a water bath at 70°C for 45 minutes during which trans-esterification took place. The electric heater was turned off, the reaction mixture was allowed to cool down slowly. The contents of the reaction vessel were transferred into a separating funnel and visible separation of biodiesel as the supernatant liquid was observed after 10 minutes. The separating funnel was allowed to stand overnight. The reaction mixture separated into two main layers with a tiny debris layer between the upper biodiesel layer (amber coloured) and the lower brown coloured glycerol layer. The reaction products were separated from each other by carefully opening the tap of the separator funnel and allowing each fraction to flow into separate labelled beakers.

The stoichiometric equation for the trans-esterification reaction is depicted as in Fig. 1.

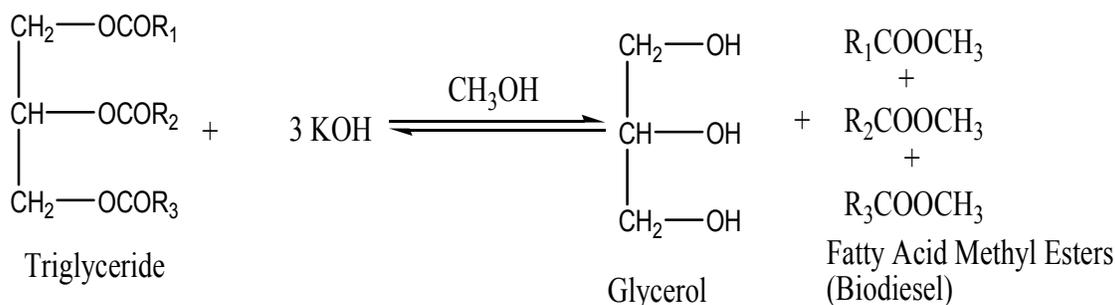


Fig. 1. Trans-esterification stoichiometric reaction

2.2.2 Biodiesel purification

The crude biodiesel was purified by washing with warm distilled water [22] in order to remove inorganic contaminants, soaps, and excess methanol. Excess methanol was then removed by heating the product at 100°C, beyond the boiling point of methanol. 50 mL of hot distilled water was poured into 100 mL of crude biodiesel in a separating funnel; water, containing dissolved impurities, settled to the bottom while the washed biodiesel floated. The separated biodiesel was heated to 110°C to expel all water and the volume of the purified biodiesel was measured after cooling. The yield y of biodiesel was then calculated using equation (1) [1,23].

$$y = \frac{V_p}{V_s} \times 100 \quad (1)$$

where V_p is the volume of biodiesel produced and V_s the volume of sample oil used for the synthesis.

2.2.3 Physical and chemical characterisation of the synthesized PKO biodiesel

The physical and chemical characterizations were carried out using standard test procedures. The specific gravity of synthesised biodiesel was determined using a 50-mL capacity specific gravity bottle. The specific gravity ρ of the biodiesel was calculated using equation (2) [2,23,24].

$$\rho = \frac{m \text{ (g)}}{v \text{ (mL)}} \quad (2)$$

The ASTM colour of PKO biodiesel was determined using the ASTM D1500 test method [25,26]. This test method covers the visual determination of the colour of a wide variety of petroleum and other natural products such as lubricating oils, heating oils, diesel fuel oils, and petroleum waxes, not excluding biodiesel and biolubricants.

The combined water and sediment content of biodiesel was determined using the ASTM D1796 analytical method as outlined in Idris and Mataka [26]. This is a laboratory test for the determination of water and sediment in fuel oils using the centrifuge method in the range from 0 to 30% volume.

The kinematic viscosity of biodiesel from palm kernel oil was determined at 40°C and at 100°C

using ASTM D445 [25] in a Cannon-Fenske viscometer. The kinematic viscosity η was calculated by equation (3) [26].

$$\eta = C \times t \quad (3)$$

where t is the average flow time in seconds and C the viscometer constant.

The flash point of Cameroon PKO biodiesel was determined using the ASTM D93 analytical method [25,26]. This test was conducted in a semi-automated Pensky-Martens closed-cup apparatus [26].

The cloud point of Cameroon PKO biodiesel was determined according to the ASTM D2500 analytical procedure of [25,26]. The pour point of biodiesel was determined using the ASTM D97 analytical procedure [25,26]. This test method is used for any natural or synthetic petroleum product such as crude, fuel oil, biofuel, lubricants, and biolubricants. The pour point gives the temperature below which the fuel cannot be used.

The aniline point of biodiesel was determined using the ASTM D611 analytical procedure. The aniline point is used to estimate the aromatic content of an oily sample [27,28], an indication of the environmental friendliness of the product. The U-tube method, which is suitable for transparent samples not darker than ASTM colour 6.5, was used.

The diesel index of the biodiesel sample was obtained by calculation from previously data of biodiesel in conformity with IP21 standard analytical method [25]. The diesel index was calculated from the American Petroleum Institute (API) value (equation 4) and then the diesel index was used to calculate the cetane number (equation 5). The cetane number is an indication of the ignition characteristics of a fuel oil in a combustion engine.

$$API = \left(\frac{141.5}{\rho @ 60^\circ F} \right) - 131.5 \quad (4)$$

$$DI = \frac{\text{aniline point } (^\circ F) \times API}{100} \quad (5)$$

Finally the cetane number was calculated from diesel index value using equation (6).

$$\text{Cetane number} = (DI \times 0.72) + 10 \quad (6)$$

The sulphur content of biodiesel was determined by the technique of energy-dispersive X-ray Fluorescence Spectrometry as described in ASTM D4294 analytical method and realised with the help of the Sulphur-in-Oil Analyser-SLFA-2800 machine. The applicable concentration range is 0.015-5.0% weight/volume of sulphur. The biodiesel was analysed immediately after pouring into the sample cell and waiting for stability. Care was taken not to touch the inside of the sample cell or portion of the window film in the cell or in the instrument window that is exposed to X-rays as oil or grease from fingerprints can lead to faulty readings. The film was stretched firmly to avoid wrinkles because they could affect the intensity of X-rays transmitted. The prepared sample cell was placed so that the transparent window film faced downwards into the X-ray detector compartment, and then it was closed. When the READY menu was displayed, the MEASURE key was pressed. The measurement indicator showed up and the first measurement was made. The machine took three measurements and computed the average and standard deviation. When the measurement was completed the result was printed out.

The free fatty acid composition of biodiesel was determined by titration with potassium hydroxide solution with phenolphthalein as indicator [29,30]. The acid value refers to the mass of KOH in milligrams that neutralises all the free fatty acids that are present in 1 g of biodiesel or oil sample. The total acid number was determined by titration and the data collected from this experiment was used to compute the acid value of biodiesel using equation (7) [31]

$$\text{TAN} = \frac{V \times C \times 56.10}{m} \quad (7)$$

where V is the average titre value, C the concentration of KOH solution, m the mass of biodiesel used and 56.10 the equivalent weight of KOH.

The iodine value refers to the number of grams of iodine absorbed per 100 g of fat or wax. It is a measure of the unsaturation in the fatty sample under investigation. Higher unsaturation would mean that the product is predisposed to any rancidity-type reaction with attendant shorter shelf life of the biofuel. The iodine number for biodiesel synthesised was determined using the Wij's titration method [32] and the iodine value was calculated using equation (8):

$$\text{IV} = \frac{(V_B - V_S) \times N \times 12.69}{m} \quad (8)$$

where V_B is the average titre value from the titration of the blank, V_S the titre value from the titration of the biodiesel sample, N the normality of sodium c and m the mass of the biodiesel sample.

The saponification value of biodiesel refers to the number of milligrams of potassium hydroxide required to neutralise all the fatty acids resulting from the complete hydrolysis of 1 g of biodiesel sample. The saponification value gives an indication of the average molecular weight of constituent fatty acid moieties in the biodiesel sample [6]. It therefore depends on the molecular weights of the fatty acid constituents of biodiesel. The greater the molecular weight, the smaller the saponification value and vice versa. The Saponification Value for PKO biodiesel was determined following the method described by Umeh and Ogbuagu [33]. The ester value of biodiesel refers to the number of milligrams of potassium hydroxide required to saponify all the esters in 1 g of the biodiesel sample. The ester value (EV) is usually calculated from the Saponification Value (SV) and the Acid Value (AV) using equation (9) [31].

$$\text{EV} = \text{SV} - \text{AV} \quad (9)$$

The UV-Visible spectrum of synthesised biodiesel was realised with the aid of the Agilent Technologies Cary Series UV-Visible spectrophotometer. IR spectroscopy analysis of biodiesel was carried out on the Fourier Transform Infrared Spectrometer manufactured by Agilent Technology; model number Cary 630 FTIR in the wavelength range from 600 to 4000 cm^{-1} . The equipment had a interface with a macro lab PC.

The chemical composition of the biodiesel produced from Cameroon palm kernel oil was determined using the combined gas chromatography-mass spectrometry analytical technique on the GC-MS-QP2010 Gas Chromatography-Mass Spectrometer with helium gas as the mobile phase.

3. RESULTS AND DISCUSSION

Cameroon PKO Biodiesel was synthesised in various batches. This was done by varying the volume of purified PKO from 50 mL to 500 mL and calculating the corresponding volumes of

methanol used as well as the corresponding masses of the KOH required by the stoichiometry of trans-esterification. The volumes of PKO and methanol as well as corresponding KOH masses are displayed in Table 1.

The results show that the synthesis of biodiesel using the small batch process gave higher yields probably due to the simplicity of the operation. Generally, for any volume of palm kernel oil taken, the required stoichiometric amount of methanol, potassium hydroxide and the right reaction conditions gave the ultimate yield. The synthesis of biodiesel from palm kernel oil was generally efficient with some slight variations within experimental errors. The methanol was in excess to drive the reaction kinetics and the reaction equilibrium in the forward direction favouring the formation of product. This agrees

with theoretical principles for maximising the conversion of reactants into the desired products.

Table 2 shows results of the physic-chemical properties of biodiesel alongside those of palm kernel oil from which biodiesel was synthesised.

A cursory look at these results reveals that major physical and chemical properties are drastically changed on conversion of palm kernel oil to biodiesel, thus demonstrating the importance of chemical derivatisation in the formation of new products with desirable properties. An example is the viscosity of palm kernel oil, 26.02 cSt which is too high to be used in compression ignition engines but when trans-esterified in the presence of methanol, the viscosity at 40°C drops to 4.90 cSt, representing a percentage decrease of 81.2%, more than a five-fold drop in viscosity

Table 1. Quantities of reagents used and the yield of biodiesel

Trial n°	Vol. PKO (mL)	Vol. CH ₃ OH (mL)	Mass of KOH (g)	Vol. of crude biodiesel (mL)	Vol. of pure biodiesel (mL)	Crude yield (%)	Yield of pure biodiesel (%)
1	50	12	0.4617	54	48	108	96
2	100	24	0.9234	100	95	100	95
3	150	35	1.3851	152	145	101	97
4	200	48	1.8468	205	180	103	90
5	250	60	2.3085	250	235	100	94
6	300	74	2.7702	300	250	100	83
7	350	82	3.2319	350	263	100	75
8	400	98	3.6936	400	330	100	83
9	450	105	4.1553	453	426	101	95
10	500	118	4.6170	500	460	100	92

Table 2. Physico-chemical properties of PKO and PKO methyl esters (Biodiesel)

Property	Palm kernel oil	PKO biodiesel
Specific gravity 15°C	0.9234 g/cm ³	0.8740 g/cm ³
Colour	< 2.5	1.5
Kinematic viscosity at 40°C	26.02 cSt	4.90 cSt
Kinematic viscosity at 100°C	5.93 cSt	1.79 cSt
Cloud point	29°C	17°C
Pour point	20°C	0°C
Flash point	200°C	50°C
Water and sediment	0.90%v/v	0.70%v/v
Aniline point	105 F	89 F
Diesel index	23	25
Cetane number	27	28
Sulphur content	0.016% w/v	0.033% w/v
Acid number	17.95 mg KOH/g	0.4488 mg KOH/g
Free fatty acid	8.98%	0.2244%
Iodine number	2.10 mg I ₂ /g	7.36 mg I ₂ /g
Peroxide value	2.10 meq/kg	3.20 meq/kg
Saponification value	140.95 mg KOH/g	168.30 mg KOH/g
Hydroxyl value	93.4 mg OH/g	51.89 mg OH/g
Ester value	123.0 mg KOH/g	154.84 mg KOH/g

[6,30]. Some properties of the synthesised biodiesel agreed with or were very close to values reported in the literature while other values of the synthesised biodiesel significantly differed from values reported by previous research work but were within reasonable range as highlighted in the discussions that follow.

3.1 Physical Properties

3.1.1 Specific gravity

The specific gravity of crude palm kernel oil, 0.9234 kg/m^3 reduced to 0.8720 kg/m^3 after trans-esterification. Hence the specific gravity of vegetable oils can be brought into acceptable ranges suitable for fuel purposes by trans-esterification. The specific gravity of PKO biodiesel was however slightly lower than 0.8740 kg/m^3 for petro-diesel. Blending of biodiesel with petro-diesel could equilibrate some of these properties [6]. Such blends would be homogeneous given that their densities are quite close.

3.1.2 Cold flow properties of PKO biodiesel

The cloud point refers to the temperature at which wax first becomes visible as the temperature of PKO biodiesel is lowered while the pour point is the temperature at which the biodiesel solidifies enough to resist flow. The cold filter plugging point is the temperature at which the fuel will solidify and block flow through the filter [6]. The cloud, pour and cold filter plugging points of PKO biodiesel can affect fuel flow and the performance of fuel pump and injector. Trans-esterification brought down the cloud point from 29°C for palm kernel oil to 17°C for biodiesel and reduced the pour point from 20°C for PKO to 0°C for biodiesel. The cold filter plugging point for PKO biodiesel was not directly determined but could be inferred from the cloud and pour points. These properties make PKO biodiesel fuel suitable mostly for the tropical climates in which ambient temperatures seldom go below 20°C . One problem of PKO biodiesel is its cold flow properties [11]. This is due to the fact that outside the tropics, temperatures can drop to 0°C or lower and should neat PKO biodiesel be used in such climatic conditions, the fuel would freeze and not be able to flow. Hence, in colder climates, crystallisation can occur to plug fuel filters and lines, causing automobile mobility problems. In order to improve flow properties, biodiesel can be blended with petro-diesel such as B20 blends which are 20% biodiesel in diesel fuel [11,34,35].

3.1.3 Flash point of PKO biodiesel

The flash point of biodiesel refers to the temperature at which the fuel can ignite when exposed to a heat source. The flash point is of relevance for safe handling, storage and transportation of fuel. The flash points of PKO and PKO biodiesel were respectively 200°C and 50°C . Trans-esterification greatly enhanced the volatility of the final product. Ambient temperatures average 30°C in the tropics; therefore PKO biodiesel can be classified as non-hazardous because of its high flash point which confers thermal stability with attendant safety in transportation in hot climates. The flash point of PKO biodiesel is however lower than that of petroleum diesel (84°C) which was also analysed. Hence blending is necessary to obtain a hybrid product that has more desirable characteristics. The flash point of PKO biodiesel is also much lower than that of biodiesel from other sources such as coconut oil which has been reported as 110°C [6].

3.1.4 Kinematic viscosity of PKO biodiesel

The values of viscosity obtained for PKO biodiesel were 4.90 cSt at 40°C and 1.79 cSt at 100°C respectively. The value at 40°C was very close to that obtained by Alamu et al. [1] and Viele et al. [24] for PKO biodiesel which were respectively 4.84 and 4.26 cSt but differed from those of Bello et al. [6] and Musa et al. [30] which were respectively 2.82 and 2.7 cSt for biodiesel from coconut oil. The viscosity of the precursor oil (PKO), 26.02 cSt reduced to 4.90 cSt after trans-esterification representing a 81.2% reduction. The trans-esterification reaction drastically modifies the viscosity of vegetable oils and consequently improves their fuel properties as they become less liable to clog injectors and related tubing in the automobile engine [36]. Other authors have reported a similar drastic drop by more than 88% in viscosity during the trans-esterification of coconut oil [6,30].

3.1.5 Aniline point, diesel index and cetane number

These parameters are mathematically related and so affect one another. The aniline point gives an indication of aromatic content of hydrocarbon mixtures and consequently its toxicity level [27]. Aromatic hydrocarbons exhibit the lowest aniline values; paraffins exhibit the highest values whilst cycloparaffins and olefins exhibit intermediate values. A high cetane number is good for improved burning ability, better fuel consumption,

reduced engine noise, reduced emission and a broad energy release spectrum due to slower burning with attendant better torque and power output [6]. The aniline point of PKO biodiesel was high (89°F) showing the absence of aromatic hydrocarbons in palm kernel oil biodiesel and hence its suitability in diesel compression ignition engines. A high diesel index is an indication of the high ignition quality of the fuel. The diesel index of PKO biodiesel was 25, much lower than 47 for petroleum diesel.

The cetane number of a diesel fuel is used as a quality parameter related to ignition delay time and combustion quality [6]. The cetane number of PKO biodiesel was 28, lower than 44 for petroleum diesel. The higher the cetane number, the better the fuel. Some appropriate measures may be needed to be taken to adjust the cetane number of PKO biodiesel upwards.

3.1.6 Water and sediment content

Water in biodiesel can be detrimental to the operation of the fuel system because it reacts with the methyl esters of fatty acids to form acidic mixtures which can degrade lubricating oil causing corrosion of engine parts with an attendant upsurge in maintenance costs and reduced life span of the engine. The water and sediment content of PKO biodiesel was 0.7% v/v which is almost negligible but stringent manufacturing procedures need to reduce this amount to trace levels.

3.1.7 Sulphur content

The sulphur content of vegetable oil originates from the soil on which the crops grow and confers lubricity to fuel [6]. Significant amounts of sulphur in fuels have adverse environmental effects. From the exhaust system, sulphur compounds are pollutants which cause acid rain, destroying vegetation and engineering structures. The value obtained for palm kernel oil was 0.016% w/v (160 ppm) and that of PKO biodiesel was 0.033% w/v (330 ppm). The slight increase in the sulphur content of biodiesel may be due to the removal of the glycerol backbone which was acting as a diluting factor and thus its removal tends to increase the mass of sulphur per unit volume. This sulphur content for biodiesel is low enough not to cause environmental problems but higher than the ASTM D 5453 requirement (15 ppm) for ultra-low sulfur diesel determined by the Ultraviolet Fluorescence Method [25].

3.1.8 Iodine value

The iodine number is a measure of the degree of unsaturation in vegetable oils. Low iodine value implies low content of unsaturated fatty acids and hence reduced reactivity of the fuel to polymerisation, thermal oxidation and other forms of rancidity. Low iodine value also implies better storage stability. The iodine values were 2.67 and 9.77 mg I₂/g for palm kernel oil and PKO biodiesel respectively. These values are very low compared to the acceptable 100 mg I₂/g, the permissible maximum for fuels to be used in internal combustion engines [6]. This low iodine number for Cameroon PKO biodiesel is advantageous because it results in much reduced carbon deposits on engine internal parts and a reduced tendency to block the holes on the injectors. The value for biodiesel value is higher than the PKO value suggesting that some multiple bonds were created in the course of trans-esterification due to thermal events.

3.1.9 Acid value and free fatty acid

This is the number of milligrams of KOH required to react with all the free fatty acids in one gram of oil. The higher the acid number, the higher the free fatty acid concentration in the biodiesel sample, leading to corrosion of engine components. This is usually a symptom of water in the fuel due to poor production or oxidative degradation. A significant level of free fatty acid in the fuel often results to soap formation with the attendant propensity to inhibit the trans-esterification reaction. The acid values determined for palm kernel oil and biodiesel were respectively 17.95 and 13.46 mg KOH/g. The acid value of PKO biodiesel corresponds to a free fatty acid level of 0.673% FFA. The value is slightly larger than 0.18 mg KOH/g reported by Kumar et al. [37] and Musa et al. [30]. However, the acid value for Cameroon PKO biodiesel falls in the range of internationally accepted standards [37].

3.1.10 Saponification value

The saponification value obtained for biodiesel was 168.30 mg KOH/g while that of PKO was 140.95 mg KOH/g. This high value shows that the PKO biodiesel consists of mostly short chain fatty acid methyl esters. The magnitude of the saponification number varies inversely with the average chain length of the constituent fatty acids in the oily sample [38]. Hence the higher the saponification number, the shorter the

average chain length [39]. Consequently, coconut oil, with a value of 190 mgKOH/g, contains shorter fatty acid molecules compared to PKO. The high saponification value for the precursor oil implies that PKO can be better suited for the manufacture of soaps, detergents, and shampoo.

3.1.11 Ester value of PKO biodiesel

The closer the ester value of a biodiesel sample is to the saponification value, the better the quality because this implies that the free fatty acid content of the biodiesel is low. This is a desirable condition of biodiesel because it is not detrimental to the operation of the ignition compression engine. The ester value for Cameroon PKO biodiesel obtained in this study was 154.84 mg KOH/g which is quite close to its saponification value of 168.30 mg KOH/g. This is another indication that Cameroon PKO biodiesel is clean due to negligible free fatty acid content [31] and possesses the requisite fuel properties for diesel compression ignition engines.

3.1.12 Peroxide value of PKO biodiesel

The peroxide value is a measure of the amount of peroxides that are found in biodiesel as a result of some depreatory reactions that might have taken place between the fatty acid moieties and atmospheric oxygen. The formation of peroxides is a result of rancidity of the precursor oil molecules; a biochemical reaction between fats and oxygen promoted by microorganisms in the sample. In the process, long chain fatty acids are degraded to short chain compounds. The peroxide value of biodiesel in this study was 3.20 meq/kg which is small compared to 9.2 meq/kg for *Jatropha curcas* oil biodiesel reported by Baroi et al. [2]. The value is also smaller than 6.0 meq/kg for coconut biodiesel [6]. The PKO biodiesel obtained is therefore not rancid and possesses greater thermal stability.

3.1.13 Hydroxyl value of the synthesized PKO biodiesel

The hydroxyl value refers to the number of milligrams of potassium hydroxide required to neutralise the acetic acid taken up on acetylation of one gram of oil that contains free hydroxyl groups. Low hydroxyl values are desirable as this signifies fewer free hydroxyl groups and consequently lower free fatty acids in an oil. The hydroxyl value obtained for PKO in this work was 51.89 mg OH/g. This value falls in the range of safe values for good fuel quality [33].

3.2 Chemical Composition of PKO Biodiesel

The chemical composition of the synthesized biodiesel was investigated by IR analysis and the combined gas chromatography and mass spectrometry.

3.2.1 IR analysis of biodiesel

IR analysis is a most reliable proof that trans-esterification took place by displaying relevant absorption peaks. Fig. 2 presents the IR spectrum of Cameroon PKO biodiesel. The peaks and their absorption frequencies are indicative of the presence of functional groups present in biodiesel. The salient groups present in biodiesel include the ester groups and associated bonds, the hydrocarbon component and its associated bonds, each bond type identified by its absorption frequency. On the IR spectrum of biodiesel, the absorption bands 1300 to 1000 cm^{-1} represent the ether bond C-O-C found also in esters [40]. Esters show carbonyl bands at or in the proximity of 1740 cm^{-1} . They also show strong absorptions in the C-O single bond in the region from 1300 to 1000 cm^{-1} that may be used to differentiate them from ketones. This band is usually of comparable breath and intensity to the carbonyl band. It can be seen that biodiesel contains methyl esters of fatty acids, some of which are saturated and others not; some are aliphatic while a few are ringed structures. None are aromatic since the precursor molecules come from palm kernel seed oil.

The IR spectrum above confirms the stoichiometric relationship of triglycerides reacting with hydroxide to give methyl esters of fatty acids. The peaks and related information confirm the formation and presence of esters in biodiesel, given that there is a total absence of aromatics. The information from this spectrum is summarised Table 3.

3.2.2 GC-MS analysis

GC-MS also gives additional proof that the trans-esterification reaction is largely effective and gives products that are predicted by the stoichiometric reaction of trans-esterification of Cameroon palm kernel oil. Methanol was used for the trans-esterification reaction and that's why there is a preponderance of methyl esters in the biodiesel product. However, ethyl esters were observed in the biodiesel chemical composition

even though ethanol was not used. This may be due to some minor polymerisation reactions that might have led to an increase in the hydrocarbon chain length. Other distractive reactions that occurred at a minor scale giving rise to some impurity compounds in the biodiesel include redox reactions (oxidation, reduction, and disproportionation reactions) involving oxygen, hydrogen and halogens. The following Tables 4 and 5 show that the composition of Cameroon PKO biodiesel includes saturated methyl esters, saturated non-methyl esters, unsaturated methyl and non-methyl esters, and some impurities due to simultaneous aberration reactions.

3.2.3 Non-ester components in palm kernel oil biodiesel (impurities)

Some impurity compounds in the PKO synthesised biodiesel identified by the GC-MS which are normally not part of the chemical composition of biodiesel include alcohols, ketones, fatty acids, ethers, halo-alkanes and alkenes. These impurities are a strong indication

that distractive reactions do take place alongside the main trans-esterification reaction responsible for the synthesis of biodiesel. Alcohols and ketones may result from reduction reactions of fatty acids and esters while alkenes may result from oxidation of the hydrocarbon components of the esters by hydrogen abstraction processes. For high yields of biodiesel, the process should be optimised by carefully studying and applying reaction conditions so as to minimise these side reactions and favour the trans-esterification reaction.

3.2.4 Mass spectra of molecular species in biodiesel from Cameroon palm kernel oil

The following mass spectra (Figs. 3-16) corroborate the fact that PKO biodiesel is principally made up of alkyl esters with some related molecules due to distractive reactions. The mass spectra further illustrate the authenticity the trans-esterification stoichiometry.

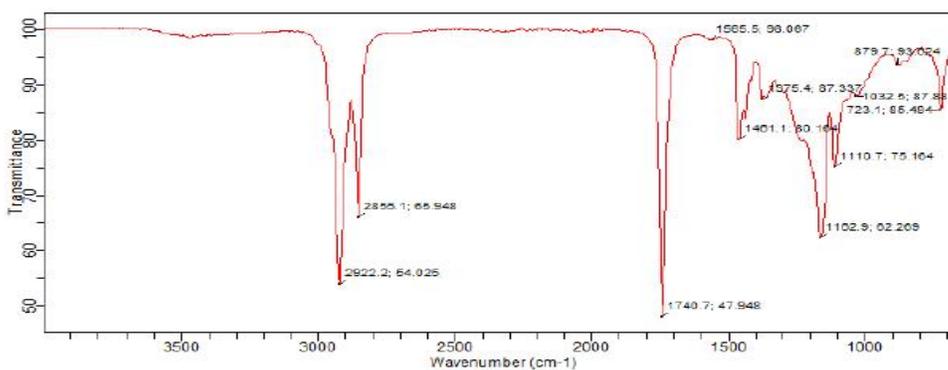
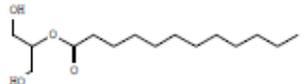
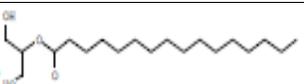
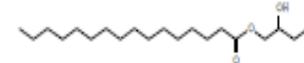
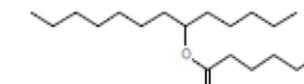
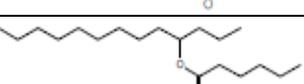


Table 4. Saturated methyl esters detected in Cameroon PKO biodiesel

S/N ^o	Ester	Molecular formula
1	Butanoic acid methyl ester	CH ₃ (CH ₂) ₂ COOCH ₃
2	Hexanoic acid methyl ester	CH ₃ (CH ₂) ₄ COOCH ₃
S/N ^o	Ester	Molecular formula
3	Heptanoic acid, 2-propylmethyl ester	CH ₃ (CH ₂) ₄ CH[(CH ₂) ₂ CH ₃]COOCH ₃
4	Octanoic acid methyl ester	CH ₃ (CH ₂) ₆ COOCH ₃
5	Nonanoic acid methyl ester	CH ₃ (CH ₂) ₇ COOCH ₃
6	Decanoic acid methyl ester	CH ₃ (CH ₂) ₈ COOCH ₃
7	Undecanoic acid methyl ester	CH ₃ (CH ₂) ₉ COOCH ₃
8	Dodecanoic acid methyl ester	CH ₃ (CH ₂) ₁₀ COOCH ₃
9	Tridecanoic acid methyl ester	CH ₃ (CH ₂) ₁₁ COOCH ₃
10	Pentadecanoic acid, 14-methyl-, methyl ester	CH ₃ CH(CH ₃)(CH ₂) ₁₂ COOCH ₃
11	Tetradecanoic acid methyl ester	CH ₃ (CH ₂) ₁₂ COOCH ₃
12	Hexadecanoic acid methyl ester	CH ₃ (CH ₂) ₁₄ COOCH ₃
13	Hexadecanoic acid, 15-methyl-, methyl ester	CH ₃ CH(CH ₃)(CH ₂) ₁₃ COOCH ₃
14	Heptadecanoic acid, 16-methyl-,methyl ester	CH ₃ CH(CH ₃)(CH ₂) ₁₄ COOCH ₃
15	Octadecanoic acid methyl ester	CH ₃ (CH ₂) ₁₆ COOCH ₃
16	Octadecanoic acid, 2-oxo, methyl ester	CH ₃ CO(CH ₂) ₁₅ COOCH ₃
17	Eicosanoic acid methyl ester	CH ₃ (CH ₂) ₁₈ COOCH ₃
18	Heneicosanoic acid methyl ester	CH ₃ (CH ₂) ₁₉ COOCH ₃
19	Docosanoic acid methyl ester	CH ₃ (CH ₂) ₂₀ COOCH ₃
20	Heptacosanoic acid methyl ester	CH ₃ (CH ₂) ₂₅ COOCH ₃
21	Tetracosanoic acid methyl ester	CH ₃ (CH ₂) ₂₂ COOCH ₃

Table 5. Saturated non-methyl esters detected in Cameroon PKO biodiesel

S/N ^o	Higher saturated esters of fatty acids	Molecular / Structural formula
1	Octanoic acid ethyl ester	CH ₃ (CH ₂) ₆ COOCH ₂ CH ₃
2	Decanoic acid ethyl ester	CH ₃ (CH ₂) ₈ COOCH ₂ CH ₃
3	Tetradecanoic acid ethyl ester	CH ₃ (CH ₂) ₁₂ COOCH ₂ CH ₃
4	Valeric acid, 2-pentadecyl ester	$\text{CH}_3(\text{CH}_2)_{12}\overset{\text{CH}_3}{\text{CH}}-\text{O}-\overset{\text{O}}{\parallel}{\text{C}}-(\text{CH}_2)_3\text{CH}_3$
5	Dodecanoic acid, 2-hydroxy-1-(hydroxymethyl) ethyl ester	
6	Pentadecanoic acid, 2-hydroxy-1-(hydroxymethyl) ethyl ester	
7	Hexadecanoic acid, 2-hydroxy-1-(hydroxymethyl) ethyl ester	
8	Hexadecanoic acid, 2,3-dihydroxyl propyl ester	
9	Tetradecanoic acid, 2,3-dihydroxyl propyl ester	
10	Hexanoic acid, 6-tridecyl ester	
11	Hexanoic acid, 4-tridecyl ester	

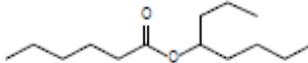
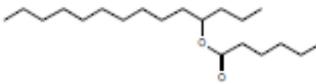
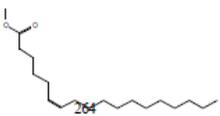
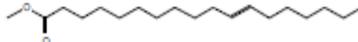
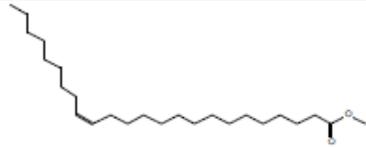
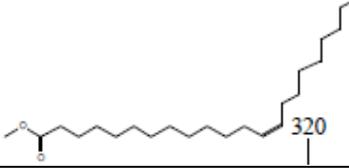
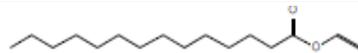
S/N ^o	Higher saturated esters of fatty acids	Molecular / Structural formula
12	Hexanoic acid, 4-octyl ester	
13	Hexanoic acid, 4-tetradecyl ester	
14	Oxalic acid, isobutyl nonyl ester	

Table 6. Unsaturated alkyl esters in Cameroon PKO biodiesel

S/N ^o	Unsaturated esters in biodiesel	Structural formulae
1	6-Octadecenoic acid methyl ester	
2	9-Octadecenoic acid methyl ester	
3	10-Octadecenoic acid methyl ester	
4	11-Octadecenoic acid methyl ester	
5	13-Octadecenoic acid methyl ester	
6	15-Tetracosenoic acid methyl ester	
7	13-Docosenoic acid methyl ester, (Z)	
8	Myristic acid, methyl vinyl ester	

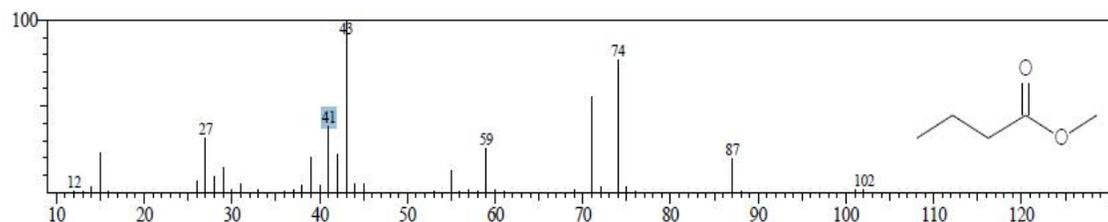


Fig. 3. Mass spectrum of Butanoic acid methyl ester

Formula of compound: $C_5H_{10}O_2$, molecular weight: 102 and names of compound: Butyric acid methyl ester, methyl butanoate, methyl n-butyrate; $n-C_3H_7COOCH_3$

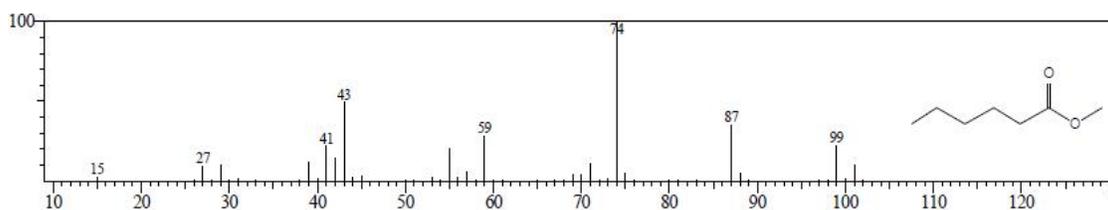


Fig. 4. Mass spectrum of Hexanoic acid methyl ester

Formula of compound: $C_7H_{14}O_2$, molecular weight: 130 and other acceptable names of the compound are Methyl caproate or Methyl hexoate or Methyl n-hexanoate ($C_5H_{11}COOCH_3$)

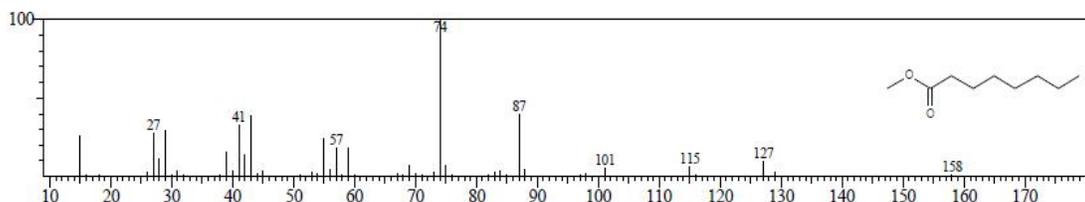


Fig. 5. Mass spectrum of Octanoic acid methyl ester

Formula of compound: $C_9H_{18}O_2$, molecular weight: 158; other acceptable names of the compound include: Caprylic acid methyl ester, Methyl caprylate, Methyl n-octanoate or Methyl octanoate ($C_7H_{15}COOCH_3$)

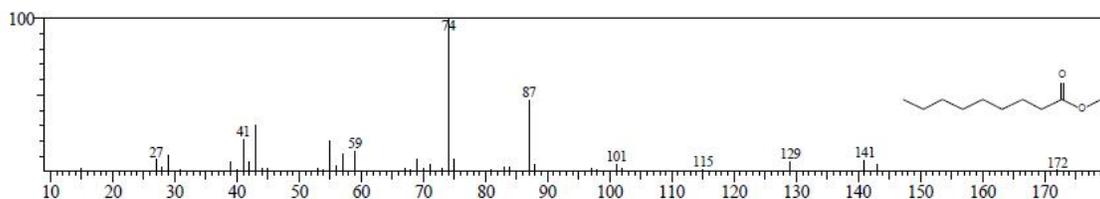


Fig. 6. Mass spectrum of Nonanoic acid methyl ester

Formula of compound: $C_{10}H_{20}O_2$, molecular weight: 172. Other acceptable names of the compound include; Methyl n-nonanoate, Methyl nonanoate; Methyl nonylate, Methyl pelargonate, Pelargonic acid methyl ester ($C_8H_{17}COOCH_3$)

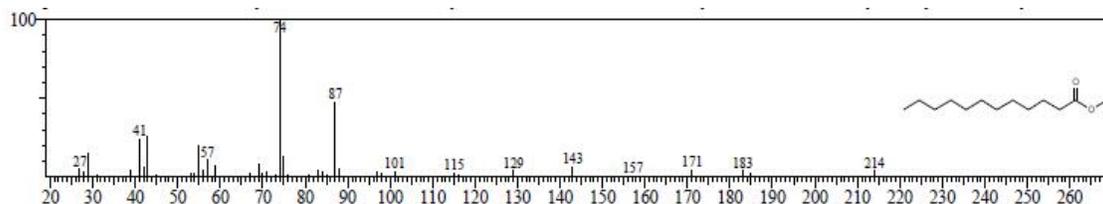


Fig. 7. Mass Spectrum of Dodecanoic acid methyl ester ($CH_3(CH_2)_9COOCH_3$)

Formula of compound: $C_{13}H_{26}O_2$; molecular weight: 214. Alternative names of compound are Lauric acid methyl ester, Methyl dodecanoate, Methyl dodecylate, and Methyl laurate

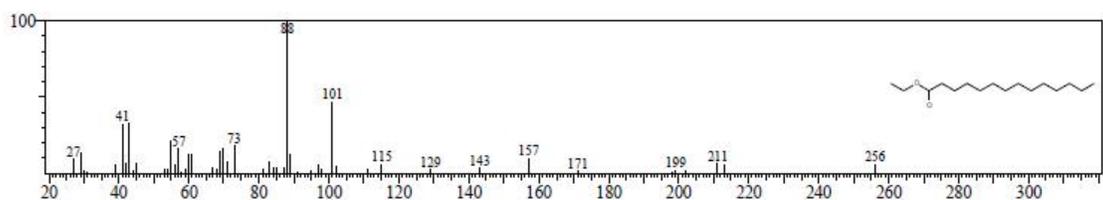


Fig. 8. Mass spectrum of Tetradecanoic acid ethyl ester

Formula of compound: $C_{16}H_{32}O_2$, molecular weight: 227. Alternative names: Myristic acid, ethyl ester, Ethyl myristate, Ethyl tetradecanoate, Ethyl ester of tetradecanoic acid

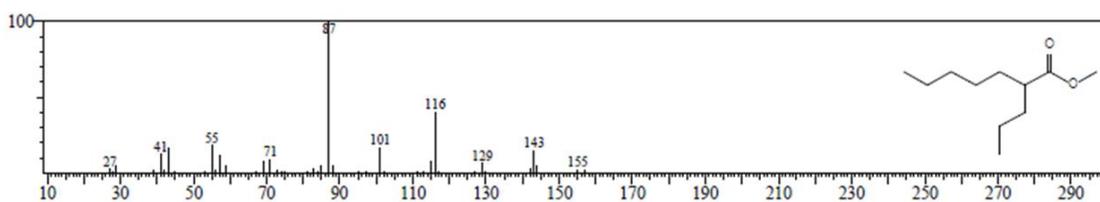


Fig. 9. Mass spectrum of Heptanoic Acid 2-Propyl methyl ester

Formula of compound: $C_{11}H_{22}O_2$, molecular weight: 186. Alternative name: Methyl 2-propylheptanoate

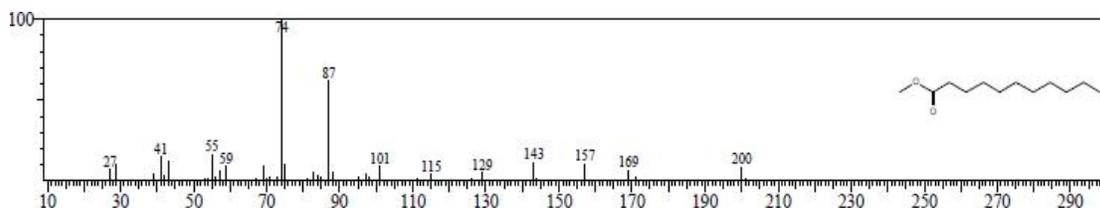


Fig. 10. Mass spectrum of Undecanoic acid methyl ester ($CH_3(CH_2)_9COOCH_3$)

Formula of compound: $C_{12}H_{24}O_2$, Molecular Weight: 200. Alternative names of compound are: Methyl undecanoate, n-Undecanoic acid methyl ester, Methyl ester of undecanoic acid

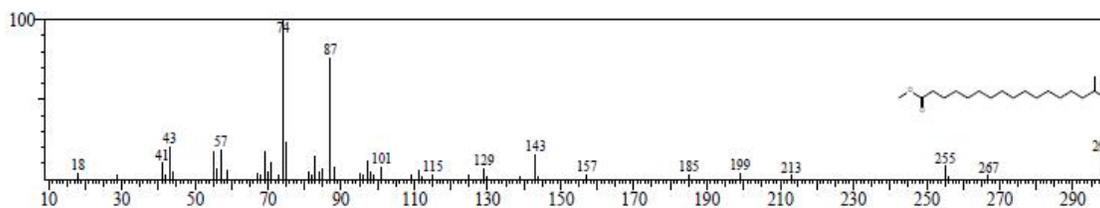


Fig. 11. Mass spectrum of Heptadecanoic acid, 16-methyl- methyl ester

Formula of compound: $C_{19}H_{38}O_2$, molecular weight: 298, alternative names of compound include Methyl isostearate, Methyl 16-methylheptadecanoate

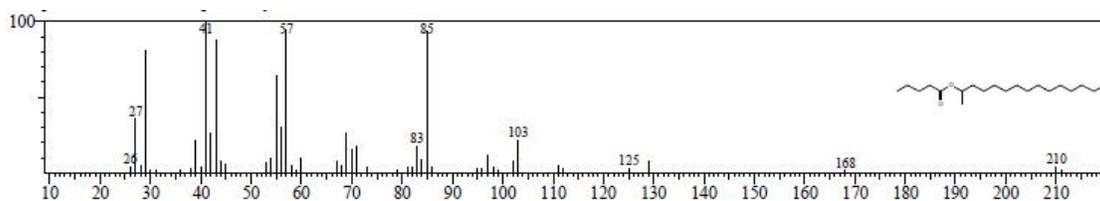


Fig. 12. Mass spectrum of Valeric acid, 2-pentadecyl ester ($CH_3(CH_2)_3COOCH(CH_3)(CH_2)_{12}CH_3$)

Formula of compound: $C_{20}H_{40}O_2$, molecular weight: 312

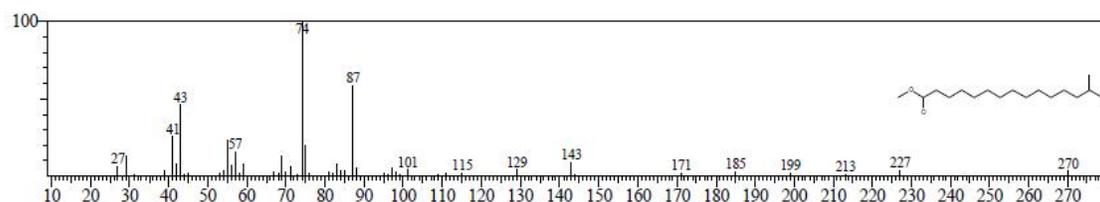


Fig. 13. Mass spectrum of Pentadecanoic acid 14-methyl- methyl ester

Formula of compound: $C_{17}H_{34}O_2$, molecular weight: 270, alternative name of the compound is Methyl 14-methylpentadecanoate

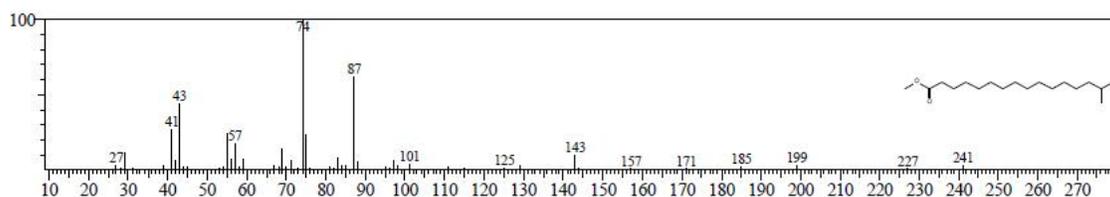


Fig. 14. Mass spectrum of Hexadecanoic acid 15-methyl-methyl ester

Formula of compounds: $C_{18}H_{36}O_2$, molecular weight: 284, alternative names of the compound are Methyl isoheptadecanoate, Methyl 15-methylhexadecanoate

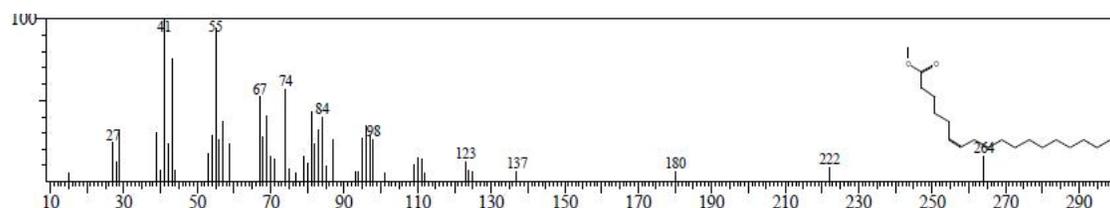


Fig. 15. Mass spectrum of 6-Octadecenoic acid methyl ester

Formula of compound: $C_{19}H_{36}O_2$, Molecular Weight: 296, other coherent names of the compound include Methyl cis-6-octadecenoate, Methyl (6Z)-6-octadecenoate

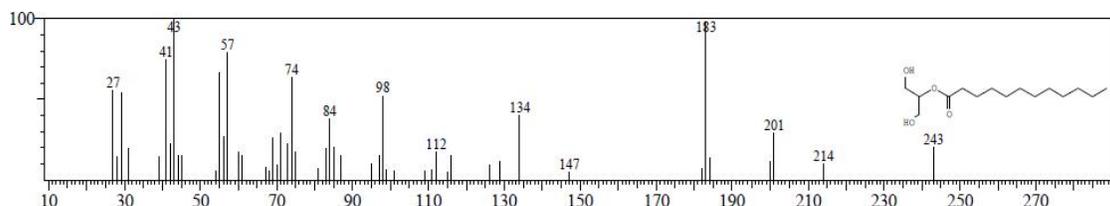


Fig. 16. Mass spectrum of Glycerol 2-laurate

Formula of compound: $C_{15}H_{30}O_4$, molecular weight: 274, alternative names to the compound include Dodecanoic acid, 2-hydroxy-1-(hydroxymethyl) ethyl ester, beta.-Monolaurin, Glycerol 2-laurate

4. CONCLUSION

The results obtained in this study agree with literature values for biodiesel production from vegetable oils, thereby attesting to the motivation and driving force of this research; that PKO biodiesel can be used as alternative fuel in diesel engines. Results from instrumental analyses clearly confirmed the stoichiometry of biodiesel synthesis. The generation of some useful by-products from the biodiesel synthetic reaction implies that the production of biodiesel from Cameroon palm kernel oil can generate and promote allied industries.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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