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ORIGINAL ARTICLE

Assessing the Potential of Oils from Palm Kernel, Tiger Nut and Castor Seed for Biodiesel Production: A Comparative Analysis

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Abstract

The depletion of fossil fuel supplies, together with expanding demand, the unpredictability of supply, high pricing, and environmental concerns, has prompted a quest for acceptable alternative fuels. Petroleum-based fuels now account for more than 90% of transportation fuel and create large amounts of greenhouse emissions. Biodiesel, a non-petroleum-derived alternative fuel, is being researched as a potential option to reduce greenhouse gas emissions. In this study, the biodiesel quality of three feedstocks well grown in Nigeria—palm kernel oil (PKO), castor seed oil (CSO), and tiger nut oil (TGO)—was compared to international standards and conventional petro-diesel. The biodiesel was produced by a two-step transesterification reaction, followed by an examination of its physical properties and characterization by employing Fourier transform infrared spectroscopy (FT-IR) and gas chromatography-mass spectrometry (GC–MS). The characteristics of biodiesel fuel were established using ASTM/EN standards. The study's findings revealed that biodiesel created from PKO, TGO, and CSO feedstocks had acceptable saponification, iodine, and flash point values. GC–MS analysis of the biodiesel's fatty acid composition revealed that methyl petroselinate (15%), oleic acid (13.73%), and ricinoleic acid (57.9%) were the most prevalent in PKO, TGO, and CSO biodiesels, respectively. FT-IR spectroscopy was also employed to identify the functional groups of the triglyceride. Overall, this research sheds light on the appropriateness of biodiesel produced from PKO, TGO, and CSO feedstocks as fuel for compression ignition engines, as well as the possibility of these feedstocks as alternate sources of biodiesel production.

Keywords Biodiesel · Feedstock · Transesterification · FT-IR · GC–MS

1 Introduction

The importance of diesel fuel in powering the economies of many countries around the world cannot be overlooked. Diesel fuel powers more than two-thirds of the energy for off-road machinery and equipment used in agriculture, construction, and mining [1]. However, diesel fuels (and fossil fuels in general) not only make up the majority of the planet's energy supply, but their continual production and use have resulted in numerous problems, including health issues, deterioration of the environment, the earth's climate change, and greenhouse gas emissions [2]. Concerns over global fuel resource depletion, market volatility, and negative environmental effects have prompted both developing and developed nations to seek alternative fuels [3]. The main repercussions of utilizing fossil fuels include the potential of these fuels to emit toxic gases such as CO2, SOx, CO, and NOx, as well as certain chemical groups like aldehydes, monocyclic aromatic hydrocarbons (MAHs), and polycyclic aromatic hydrocarbons (PAHs). These compounds have a toxic effect on the environment and an impact on the greenhouse system, leading to acid rain, environmental and water pollution, global warming, and other problems. Their toxic effects lead to human health issues like cancer, lung problems, vascular diseases, and others. [4]. It is also predicted that the planet is likely to face an energy supply collapse in the coming future due to over-reliance on fossil fuels.

Consequently, biofuels, particularly biodiesel, have been given more attention as alternative fuels [5]. Alternative fuels include producer gas, ethanol, biogas, methanol, and

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vegetable oils. Yet, because it is a renewable and non-polluting fuel, biodiesel is preferred above the others as a replacement for petroleum-based diesel [6].

Biodiesel is a mono-alkyl ester of fatty acids with long chains produced during the transesterification process, according to the American Society for Testing and Materials (ASTM) [7]. It is an alternative, sustainable diesel fuel made from vegetable or animal fats. Transesterification, a procedure, is typically used to turn vegetable oils into biodiesel. The calorific value and cetane number of vegetable oils derived from most seeds are comparable to diesel, and they can be used with minimal modification in distribution and car fuel systems [8]. The main disadvantages of this fuel include its high viscosity, which makes it difficult for the fuel injection system to operate, as well as its inability to start cold [9]. A proven procedure such as transesterification can overcome issues with the physical properties of plain vegetable oil.

Nigeria is a significant agricultural country that relies primarily on agriculture based on rain [10]. This makes her easily susceptible to the negative effects of climate change, such as flooding, extreme heat, and drought, all of which have contributed to soil degradation and low agricultural production. Finding renewable energy sources with low environmental impact is becoming increasingly important, which necessitates investigating alternate renewable fuel sources from the nation's large agricultural resources.

Initially, biodiesel was mostly produced from food oils such as safflower oil, soybean oil, palm oil, sunflower oil, coconut oil, etc. It became tricky to produce biodiesel using food oils as a feedstock. since it competed with the food supply, resulting in a high price for edible vegetable oil as well as a proportional rise in biodiesel production costs [11]. As a result, coordinated research efforts are being made to discover and evaluate non-edible seed oils as feasible feedstocks.

Non-edible oil plants are numerous all over the world. Plants such as neem (*A. indica*), palm kernel oil, mahua (*M. indica*), jatropha tree (*J. curcas*), castor, linseed, rubber plant (*H. brasiliensis*), microalgae, etc. have all been considered for biodiesel production. [12].

Palm kernel oil (PKO), castor seed oil (CSO), and tigernut oil (TGO) have been widely studied as feedstocks for producing biodiesel due to their abundance and high oil content. PKO has been extensively researched as a feedstock for biodiesel production due to its high oil yield and availability. Several studies have investigated the optimization of PKO biodiesel production processes using different methods such as transesterification with alkali and acid catalysts, enzymatic transesterification, and microwave-assisted transesterification [13–15]. The physicochemical properties of PKO biodiesel have been stated to be similar to those of petroleum diesel, with low sulfur and nitrogen contents, a high cetane number, and good lubricity [16].

CSO is another potential feedstock used in producing biodiesel, with a high oil content and low viscosity. Studies have shown that biodiesel produced from CSO has good fuel properties, such as a high cetane number, low sulfur and nitrogen content, and good oxidative stability. However, the high acid value and viscosity of CSO can pose challenges during the transesterification process, necessitating the use of acid catalysts or co-solvents to improve the yield and quality of biodiesel. Sharma et al., [17] employed a two-stage conversion technique to improve the yields and properties of CO biodiesel. The catalyst $CaCu(OCH_3)_2$ was prepared from waste eggshell and synthesized with copper oxide in the presence of methanol. The castor oil was subjected to pyrolysis at 450–500 °C and then transesterified in the presence of a modified catalyst. Vasquez et al. tried to identify the varieties of castor oil plants that could be cultivated and harnessed as bioenergy crops in Tlaltenango (Zacatecas, Mexico) [18]. Depending on the methanol-to-oil ratio, catalyst loading, reaction time, temperature, and other factors, up to 100% of the biodiesel was successfully produced as a consequence of the transesterification investigation.

TGO, also known as chufa oil, is a relatively new feedstock for biodiesel production. TGO has a high oil content and a low iodine value, making it a suitable feedstock biodiesel with good oxidative stability and low exhaust emissions [19]. However, the high free fatty acid content of TGO can lead to challenges during transesterification, requiring the use of acid catalysts or pretreatment methods such as esterification or acid-catalyzed pre-treatment to improve the biodiesel yield and quality. A study by Shi et al. [20], revealed that MgAl-LDHx is a promising catalyst for both decompositions of tigernut and hydrodeoxygenation of bio-oil, providing a potential strategy for bio-oil production. So as to maximize the yield of methyl ester (biodiesel) from the oil, Adedara et al. [21] evaluated the transesterification process of tiger nut waste for the production of biodiesel.

The primary intention of this research is to provide a comprehensive verification of the biodiesel properties reported in previous studies while expanding upon the existing knowledge. We aim to contribute valuable insights and identify any potential gaps in understanding. By doing so, we aim to enhance the understanding of PKO, CSO, and TGO biodiesels as alternative fuels and their viability for use in compression ignition engines. Moreover, our research highlights the potential of these feedstocks as alternate sources for biodiesel production and identifies any novel findings not previously reported in the literature.

2 Materials and Methods

2.1 Materials Used

Methanol (99.8%), n-hexane (97%), and Sulfuric acid (98%) from BDH Limited Poole (United Kingdom) were used. Potassium hydroxide (≥ 85%), chloroform (≥ 99.5%), potassium iodide (99.99%), sodium thiosulfate (99%), glacial acetic acid ((≥ 99%), and iodine (≥ 99.5%) were supplied by Sigma Aldrich. Phenolphthalein solution, starch solution, Wiji's solution, and distilled water prepared in the laboratory were used. All the reagents were of analytical grade.

2.2 Sample Collection and Treatment

A total of 5 kg of castor and tigernut seeds were purchased from the Agric-Osun Training School in Osogbo, Osun State. The 3 kg of cracked palm kernel seeds were sourced from local oil merchants in Iwo, Osun State, Nigeria. The seeds were thoroughly sorted to eliminate any damaged seeds. They were then cleaned, sundried, and crushed. Thereafter, they were dried in a 105 °C oven until they attained a uniform weight.

2.3 Extraction of the Oil

A cold extraction method using n-hexane as a solvent was used to extract oil from the seeds. In a 4-L extracting jar, one kg of fresh powdered seeds was extracted with 2 L of n-hexane. The jar was covered and allowed to sit at room temperature for 72 h. The resultant solution was decanted from the jar and filtered with a muslin cloth followed by Whatman filter paper. The crude oil was recovered by concentrating the filtrate (oil and n-hexane) by distillation using a rotary evaporator. For each extraction, a fresh n-hexane solvent was employed. The extracted oil was placed in the fume cupboard for 72 h to remove any traces of solvent from the oil; this was then decanted to obtain clear oil, which was stored in a cool, dry place.

2.4 Transesterification Process

The transesterification reaction was carried out using a 500 mL round bottom flask fitted with a magnetic stirrer and a thermometer inserted through the cork stopper. Each oil sample underwent a two-step transesterification process to minimize free fatty acid (FFA) content and hence significantly reduce soap formation (saponification) during biodiesel manufacture.

The average molecular weight of the oil was calculated, and this determines how much oil would be consumed in the reaction. After preheating 150 g of oil for 30 min, 1:6 oil to methanol was added, and 1% w/w H2SO4 acid catalyst was stirred for two hours at 65 °C with a magnetic stirrer. After settling for two hours, the upper layer containing the oil sample with reduced FFA separated from the lower layer. The now esterified oil sample was heated to 100 °C to evaporate remnant methanol and impurities and then allowed to cool to 45 °C. Methanol was added using a 1:6 methanol to oil ratio and a 2% w/w KOH catalyst, and the mixture was stirred vigorously at 300 rpm for 2 h by a magnetic stirrer at 65 °C. It was then moved to a separatory funnel, where it was given 24 h to settle. The yellow biodiesel was then obtained by removing the glycerol-containing bottom layer.

2.5 Biodiesel Washing

The separating funnel was used again for this process. The produced biodiesel and an equal volume of warm distilled water were added to help wash away any remnants of the unused catalyst. After swirling the separating funnel several times and leaving it for 5 min, the mixture split into a lighter upper phase and heavier lower phase of biodiesel and milky water respectively. This was repeated twice more.

2.6 Biodiesel Physicochemical Properties

2.6.1 Determination of Acid Value and Percentage Free Fatty Acid

A conical flask was loaded with 5 g of biodiesel sample. 2–3 drops of phenolphthalein indicator were added to 50 mL methanol followed by 1–2 drops of 0.1 M KOH to create a subtle pink hue. The mixture was placed in a water bath until the biodiesel globulized. Titration with 0.25 M KOH was carried out until we achieved pink colour which lasted for about a minute. The formula below was then used to determine the acid value and percentage fatty acid (ASTM standard methods, ASTM D664);

$$\text{Acid value } = \frac{titrevalue\ (mL) \times M \times 56.1}{weightofoil\ (g)} \tag{1}$$

$$\%\text{ free fatty acid } = \frac{Acid\ value}{1.99} \tag{2}$$

where M denotes the molarity of KOH

2.6.2 Calculating the Iodine Value

A conical flask was filled with 1.0 g of biodiesel and 25 cm^3 of carbon tetrachloride in order to dissolve the sample. A measuring cylinder was used in a fume chamber to add 25 cm^3 of Wijj's Reagent to the flask. Before installation, the cork was moistened with potassium iodide and the flask's contents were vigorously swirled. The flask was then moved into a cabinet and kept in the dark for 30 min before 20 cm^3 of 10% aqueous potassium iodide and 100 cm^3 of water were added. After adding 3 drops of freshly prepared 1% starch solution, the content was titrated using a solution of 0.1 M sodium thiosulphate. Drop by drop, with steady shaking, sodium thiosulphate was added until the blue-black coloration vanished. To titrate a blank sample, the procedure was repeated. Iodine content was determined using:

$$\text{Iodine value} = \frac{12.69 \times C \times V2 - V1}{weight\ of\ biodiesel(g)} \tag{3}$$

where C is the concentration of sodium thiosulphate, V1 is the volume of sodium thiosulphate for the sample, V2 is the volume of sodium thiosulphate for the blank, and 12.69 is the constant.

2.6.3 Determination of Saponification Value

Mixing 20 g of KOH and 22.5 g of CuO yielded an alcoholic KOH solution. The mixture was carefully crushed before being dissolved in a small quantity of methanol. This was then transferred to a volumetric flask with a capacity of 1000 mL and filled with methanol. Before leaving the flask overnight, it was violently shaken for a few minutes. The solution was filtered the next morning. 2.0 g of biodiesel with 25 mL of alcoholic KOH was measured into a conical flask and refluxed for 1 h with the use of a magnetic stirrer. Two drops of phenolphthalein indicator were added after cooling, and 0.5 M HCl was used to titrate the solution. The titer value was established by a color change from pink to colorless.

The titration was repeated three times, with the average titer value recorded. The result was computed using Eq. 4:

$$\text{S.V.} = \frac{(B - S) \times\ 56.1 \times M}{weight\ of\ biodiesel(g)} \tag{4}$$

where B is the blank titer value, S is the sample titer value, 56.10 is the molar mass of KOH, and M is the molarity of HCL

2.6.4 Specific Gravity (relative density) of Biodiesel (@ 40 °C)

A density bottle with a volume of 25 cm^3 was weighed while empty and marked as W_o in order to calculate the specific gravities of water and biodiesel samples. The bottle was filled with distilled water, sealed, and heated to 40 °C in a water bath. After being dried off, the bottle was weighed as W_1. After repeating the procedure with biodiesel in place of water, the weight was noted as W_2. Using the results, an appropriate expression was employed to determine the specific gravity of each of the samples

$$\text{S.G.} = \frac{W2 - W0}{W1 - W0} \tag{5}$$

S.G. is the specific gravity of the oil; (W_2–W_o) is the mass of the sample, and (W_1–W_o) is the mass of an equivalent amount of water.

2.6.5 Flashpoint and Smoke Point (°C) (ASTM D93)

On a regulated hot plate, a clean, dry crucible with 20 mL of biodiesel within was heated. A thermometer was clamped into the biodiesel without coming into touch with the bottom of the crucible. The sample was agitated, and a flame was passed over it at intervals of 3 °C. A "smoke point" was defined as the temperature at which the sample started to emit smoke, while a "flash point" was defined as the temperature at which the flame flashed as it was passed over the crucible.

2.6.6 Cloud and Pour Point (°C)

A Biodiesel was half-filled into a 20 mL clean, dry test tube; a cork attached to a thermometer was used to cover the test tube, with the thermometer bulb allowed to drop all the way to the bottom. A cold bath was used to immerse the whole test tube. The test tube was removed every $3\,^{\circ}C$ and examined for the cloud before being promptly reinstalled. Once a whitish/milky cloud appeared on the bottom wall of the test tube, the temperature was recorded as the "cloud point." This was continued until wax crystals developed. The test tube was withdrawn and inverted every $3\,^{\circ}C$ to check for flow. The "pour point" was the temperature at which the biodiesel stopped flowing.

2.6.7 The Cetane Number

Cetane number of biodiesel was calculated using the equation below;

$$\text{Cetane number} = 46.3 + \frac{5458}{saponificationvalue} - 0.225 \times \text{ iodine value} \tag{6}$$

where 46.3, 5458 and 0.225 are constants

2.6.8 Higher Heating Value (MJ/Kg)

Higher heating value (HHV) was calculated using Demirbas[13] equation;

$$\text{HHV (MJ/Kg)} = 49.43 - 0.041(\text{Saponification value}) - 0.015(\text{Iodine value}) \tag{7}$$

where 49.43, 0.041, and 0.0015 are constants.where M is the mass of water in grams, C is the specific heat capacity of water (which is 4.186 j/g °C), ΔT = T2–T1

It is important to keep in mind that the actual HHV of biodiesel might change based on the fuel's unique composition and the circumstances in which it is burnt. However, the calculated value provides a useful estimate of the potential energy content of the fuel.

2.6.9 Ash Content

In a muffle furnace (preheated at 450 °C), the sample (5 g) was placed inside a preweighed quartz crucible. After half an hour, the crucible was removed after the biodiesel had completely burned to ash. The crucible was covered and allowed to cool in a fume cupboard after which it was reweighed. The ash content was determined using Eq. 8 below.

$$\text{Ash content of biodiesel \%} = \frac{Finalweightofthecrucible - Initialweightofthecrucible}{Weightofthebiodiesel} \times 100 \tag{8}$$

2.7 Analytical Method

The physicochemical properties of the biodiesel from PKO, TG, and CSO were determined by ASTM standard methods. Comparisons were also made with the European biodiesel standard EN14214 in mind.

Fourier transform infrared spectroscopy FT-IR was used to compare the constituents of biodiesel with those of standard diesel. The purified fatty acid methyl ester (FAME) was analyzed by an Agilent 630 Cary FT-IR. The crystal was cleaned using N-hexane. All spectra were recorded within a range of 4000–600 cm^{-1}. The identification of biodiesel compounds was carried out using GCMS. For the GCMS, an Agilent Technologies inert XL EI/CI mass selective detector (MSD) (5975B, Agilent Technologies Inc., Palo Alto, CA) was connected to a gas chromatograph (6890N, Agilent Technologies Network). The components of biodiesel were identified by matching the retention times and mass spectra to those of the NIST 11 mass spectral database. A single microliter of the sample was injected into column A using the proper injection techniques. When the analysis was finished, the results were automatically printed. The integrator chart speed was set to 2 cm/min, the injector temperature to 220 °C, and the detector temperature to 250 °C. The GC was given time to warm up while the oven was prepared to 180 °C. The following activities are scheduled during warming up Signal 181 °C as the end temperature, 180 °C as the beginning temperature, 1 min as the final time, 15 min as the initial time, and a rate of 0 °C/min.

3 Result and Discussion

3.1 Physiochemical Properties

Using Table 1 as a reference, the acid value of the biodiesel obtained was 1.1 mg KOH/g (PKO), 1.2 mg KOH/g (TG), and 1.5 mg KOH/g (CSO). The acid value is an essential metric that determines the quality of biodiesel [22]. A very high acid value has the potential to cause engine rubber seals and hoses to break prematurely due to their strong solvency [23]. The acid values for PKO, TGO, and CSO samples are all relatively low, which is desirable for biodiesel production. According to a study published in Renewable and Sustainable Energy Reviews [24], the acid value for various biodiesel

Table 1 Physicochemical properties of produced biodiesel and standard

	PKO	TGO	CSO	Biodiesel standard limits ASTM D6751
Acid Value (mgKOH/g)	1.1	1.2	1.5	0.8
Percentage FFA (%)	1.1	1.4	1.6	–
Saponification Value (mgKOH/g)	179	136	114	370 max
Specific gravity	0.92	0.92	0.94	0.86–0.90
Iodine value (mgKOH/g)	46	101	114	120 max EN 14214
Smoke point °C	75	100	123	–
Flashpoint °C	146	180	200	130 min
Cloud point °C	8	9	6	− 3 to − 12
Pour point °C	0	6	− 3	− 15 to − 16
Cetane number	67	64	40	47 min
Higher heating value (MJ/kg)	41	42	36	35 min
Ash content (%)	0.02	0.09	0.1	0.02%

samples typically ranges from 0.1 to 2.5 mgKOH/g, which is consistent with the values observed in Table 1.

A larger concentration of free fatty acids results in a higher acid value, which causes severe corrosion in an engine's fuel delivery lines. The FFA content of PKO biodiesel in the current study was 1.1%, which is slightly lower than the FFA content reported in a study by Li et al. [24], where the FFA content of PKO biodiesel was found to be 1.3%. The FFA content of TGO biodiesel in the current study was 1.4%, which is comparable to the FFA content reported in a study by Mofijur et al. [25], where the FFA content of TGO biodiesel was found to be 1.4%. The FFA content of CSO biodiesel in the current study was 1.6%, which is slightly higher than the FFA content reported in a study by Atabani et al. [26], where the FFA content of CSO biodiesel was found to be 1.4%.

A high SV suggests that the oil has a larger concentration of low molecular weight fatty acids or vice versa [27]. PKO (179 mg KOH/g), TG (136 mg KOH/g), and CSO (287 mg KOH/g) were the SVs obtained from the biodiesel samples. These results are higher than the ASTM standard of 100 mg KOH/g, indicating that due to their relatively high saponification values, all of the biodiesel samples are largely composed of fatty acids with fairly long chain lengths (C16-C20). An implication of this includes; higher viscosity, a higher cetane number, lower oxidative stability, and higher low-temperature properties. A study by Bong et al. [28], determined the saponification value of Cameroon PKO biodiesel to be 168.30 mg KOH/g.

The mix of fatty acid compounds determines the specific gravity of biodiesel, and biodiesel densities vary with fatty acid quantity and purity. Specific gravity is commonly employed as a predecessor to a variety of other fuel qualities, including heating value, viscosity, and cetane number [29]. The specific gravities derived for the biodiesel sample are PKO (0.92), TGO (0.92), and CSO (0.94), which are in close proximity to the ASTM D6751 standard (0.86–0.90). This suggests that biodiesel will burn correctly and flow well in a typical diesel engine. Since exact fuel amounts must be supplied via injection systems, pumps, and injectors in order for the engine to burn fuel efficiently, SG plays an important role in this efficiency [30]. Soot and ignition delay can also result from high specific gravity. The SG of biodiesel from PKO was reported to be 0.879 in one by Alamu [14], which is similar to the value obtained in Table 1. For TGO biodiesel, the specific gravity was reported to be 0.916 by Adelekan [19], which is also similar to the value obtained in the table provided. However, there are few studies on the specific gravity CSO produced biodiesel, making it difficult to make a comparison.

The iodine value of oil evaluates its unsaturated fatty acid content and indicates how easily oil oxidizes when exposed to air. It is used to figure out how much of a fatty acid is unsaturated. The iodine value increases with the amount of unsaturated fatty acids present. The iodine content in petroleum diesel fuel is quite low, whereas the maximum iodine content of biodiesel for EN 14214 is 120 mg KOH/g. PKO (46 mg KOH/g), TGO (101 mg KOH/g), and CSO (114 mg KOH/g) were determined for the biodiesel samples. The three samples had lower iodine values than the EN suggested level (120 mg KOH/g), suggesting that they will be more stable to oxidation. As a result, there is mild unsaturation and appropriateness for fuel use. The iodine value of biodiesel from PKO was reported to be 38.9 by Alamu, which is lower than the value obtained in the table provided. However, Abdullah reported a similar value of 48.2 for PKO biodiesel. For TGO biodiesel, the iodine value was reported to be 97.2 in one study Adelekan et al. [19], a value higher than that presented in Table 1 above. For CSO biodiesel, the iodine value was reported to be 76.0 in a study by Gbadamosi et al. [16], which is also lower than the value obtained in Table 1 above.

The "smoke point" is the temperature at which smoke appears for the first time when oil is heated. The biodiesel obtained had a smoke point value of PKO (75 °C), TGO (100 °C), and CSO (123 °C).

Fuel flashpoint is a measure of flammability and an essential metric for identifying dangers regarding fuel storage and transportation. It is the temperature that fuel must attain before the gas and oxygen mixture above the fuel may be ignited, and it is important as fuels are categorized according to their hazards for distribution, storage, and transportation. The flash point has no direct effect on combustion; rather, fuels are safer to store, handle, and transport when they have higher values. The flash points for the biodiesel produced were PKO (146 °C), TGO (180 °C), and CSO (200 °C), all of which were greater than the ASTM D6751 minimum requirement (130 °C). The lower the flash point in terms of safety, the greater the expense of the storage system.

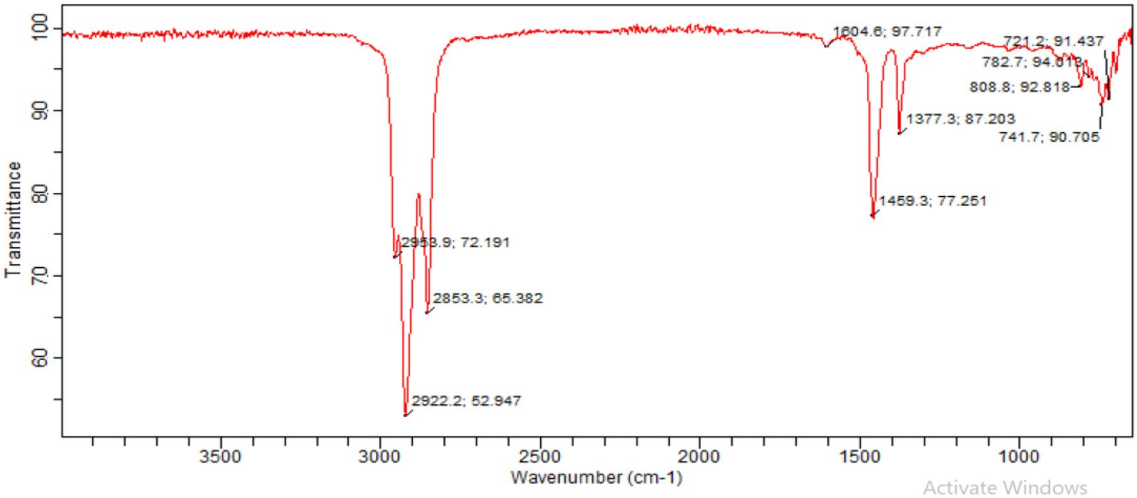
The temperature at which fuel crystals first begin to form is known as the cloud point (CP). This happens when the biodiesel's temperature drops to the point where wax crystals begin to form, which causes a hazy suspension at that temperature. When below the CP, these crystals may accumulate at the bottom of a storage tank or clog the engine's filters. The CP is the most commonly used metric to determine a fuel's ability to operate at very low temperatures. PKO (8 °C), TGO (9 °C), and CSO (6 °C) are the cloud points determined from biodiesel samples. Depending on the feedstock used, the cloud point of biodiesel may differ from that of petroleum diesel but is normally higher.

The "pour point" is the temperature at which fuel has become so clogged with crystals that it stops flowing. This happens when the temperature drops below the cloud threshold and the microcrystals combine to form huge clusters, which can impede the movement of biodiesel through the engine's fuel system pipes. Pour point values are influenced by the feedstock from which the biodiesel was produced, much like cloud point values are. Pour points for biodiesel samples are PKO (0 °C), TGO (6 °C), and CSO (− 3 °C). Despite the temperature dropping below the cloud point, fuel could still work in an engine. However, below the pour threshold, the fuel cannot operate [31]. Although European EN 14214 does not have a minimum standard for cloud and pour points, the American ASTM D6751 standard is − 3 to − 12 °C and − 15 to − 16 °C for cloud and pour points, respectively. The values for the biodiesel samples exceed the ASTM standard, with CSO exhibiting the highest cold weather performance of the three samples, which makes it more recommended at locations with very low ambient temperatures.

Fuel's ignition quality is indicated by its cetane number [32], as it indicates the quality of fuel ignition for compression ignition engines. Biodiesel samples had cetane numbers of PKO (67), TGO (64), and CSO (40). Starting the engine may be difficult if the biodiesel has a low cetane number. The engine may run noisily and inefficiently, especially at low temperatures. An overly high cetane number causes the ignition to happen too quickly before a good mix with air, which leads to incomplete combustion and more exhaust smoke. Unburned gasoline fragments may clog injector nozzles, potentially damaging the engine. The cetane number of the PKO and TG samples was higher than the minimum set by ASTM D6751 and EN 14214, which are 47 and 51 minimum, and was close to the 70 derived by Bello et al. [33]. The CSO biodiesel sample falls below the minimum values set at 40, which may lead to some delay between fuel injection and ignition and also cause increased pollution.

"Higher Heating Value" is a metric that quantifies a fuel's energy content. It is the amount of thermal energy that is released per unit of fuel when the fuel has completely burned and the combustion products have cooled to the combustible mixtures' initial temperature [34]. As per ASTM biodiesel standards, there is no specification of the heat of combustion. European rules, on the other hand, mandate that biodiesel have a minimum heating value of 35 MJ/kg. The biodiesel sample heating values were as follows: PKO (41.41 MJ/kg), TGO (42.34 MJ/kg), and CSO (35.91 MJ/kg). While it is less than diesel (49.65 MJ/kg), the HHV of the biodiesel samples above is still reasonably high and exceeds the minimum level established. Alamu indicated that the greater heating value of biodiesel from PKO was 37.1 MJ/kg, which is a little bit less than the number seen in the accompanying Table 1. Abdullah [15] did, however, publish a comparable figure of 39.8 MJ/kg for PKO biodiesel. The higher heating value for TGO biodiesel, which is somewhat less than the figure seen in Table 1, was reported by Adelekan to be 38.4 MJ/kg. Gbadamosi claimed that the greater heating value for CSO biodiesel was 34.5 MJ/kg, which is likewise less than the figure seen in the presented Table 1.

The ash contents determined for the biodiesel samples were; PKO (0.02%), TGO (0.09%), and CSO (0.1%). These were substantially identical to the fuel standards, suggesting that there were no concentrated abrasive particles, catalyst residues, or dissolved metal in the samples that may produce ash when burnt during the combustion process, and hence no engine deposits. The ash content of biodiesel from PKO was reported to be 0.010% in one study [13], which is lower than the value obtained in Table 1 provided. However, Alamu reported a similar value of 0.015% for PKO biodiesel. For TGO biodiesel, the ash content was reported to be 0.012% by Adelekan, which is also similar to the value obtained in the table provided. For CSO biodiesel, the ash content



Activate Windows

Fig. 1 FTIR Spectrum of Petroleum diesel

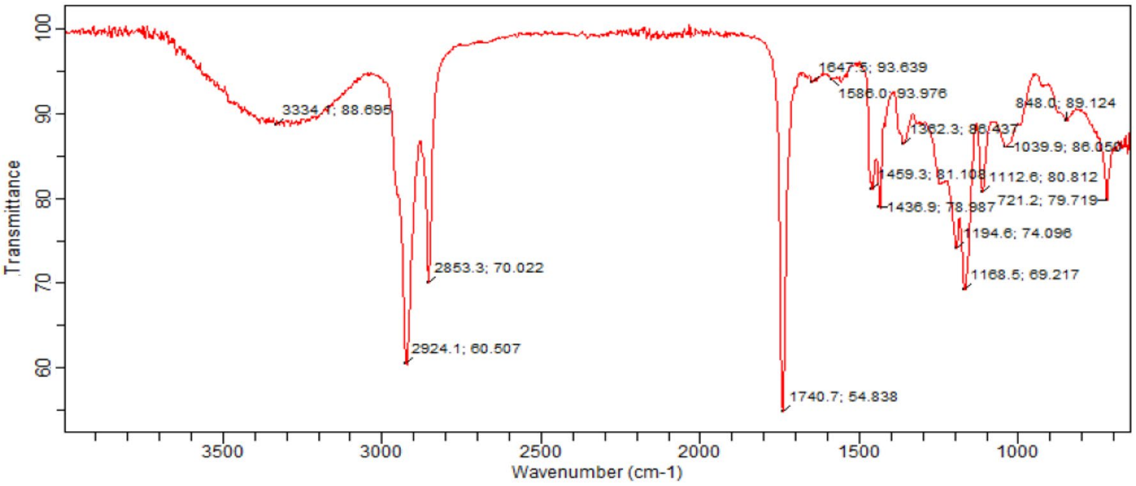


Fig. 2 FTIR Spectrum of PKO biodiesel

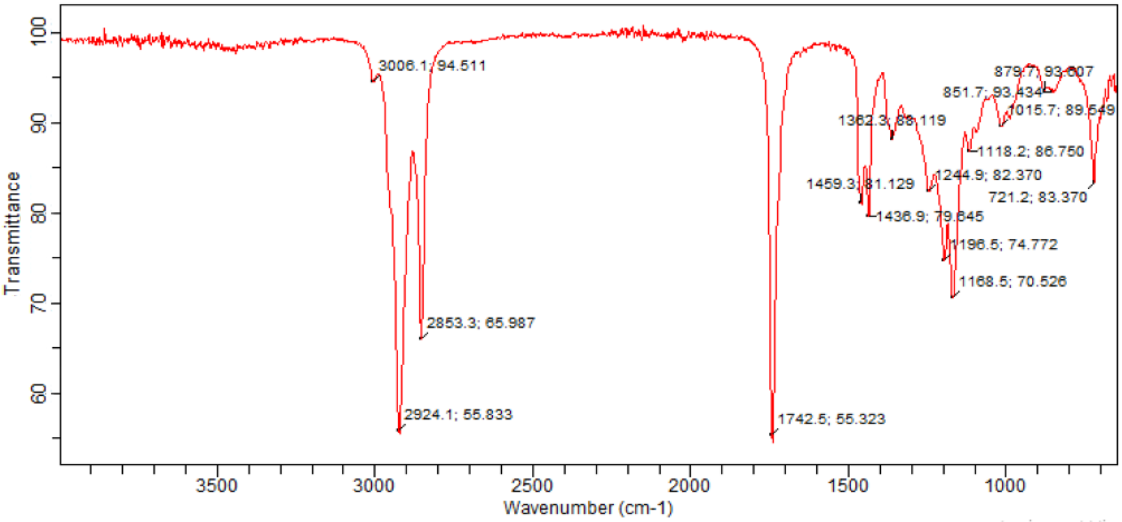


Fig. 3 FTIR Spectrum of TGO biodiesel

was reported to be 0.023% by Gbadamosi, which is slightly higher than the value obtained in the table provided.

3.2 FT-IR

FT-IR is used to analyze and simulate the existence of different carbon isomers in biodiesel. Figures 1, 2, 3 and 4 shows typical FT-IR spectra ofconventional petroleum diesel, PKO biodiesel, TGO biodiesel, and CSO biodiesel, indicating the existence of volatile chemicals at various mixing vibrations.

Table 2 shows the complete conclusions of the FTIR spectrum.

The O–H stretching vibration of the free fatty acids contained in the biodiesel sample is responsible for the medium and broad peaks found at 3334.4 cm^{-1} and 3383.9 cm^{-1} in the FT-IR spectra of biodiesel derived from palm kernel oil and castor seed oil, respectively. These free fatty acids may have developed as a result of insufficient transesterification of the oil's triglycerides during the biodiesel synthesis process. It is also possible that water is present in the sample, which might contribute to the observed peak, as water also exhibits an O–H stretching vibration at around 3400 cm^{-1}. However, this is less likely to be the case since biodiesel samples are typically dried prior to FT-IR analysis to prevent interference from water vapor.

However, an interesting observation is the absence of a similar peak in the TGO biodiesel spectrum, despite having a comparable free fatty acid (FFA) content and the presence of FAMEs (fatty acid methyl esters) containing hydroxy-groups, as indicated by the GC–MS evaluation.

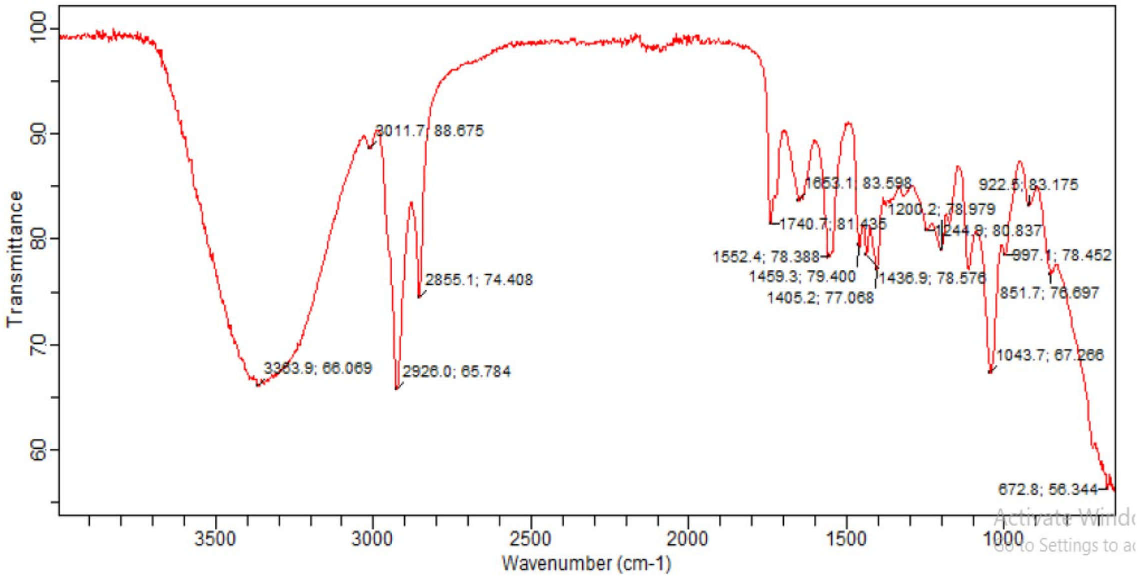


Fig. 4 FTIR Spectrum of CSO biodiesel

Table 2 FTIR spectrum of produced biodiesel

No	Wave length (cm^{-1})	Inferences of FTIR Spectrum	Signal Characteristic
1	3334.4 (PKO) 3383.9 (CSO)	Glyceride ester carbonyl absorption	Medium, Broad
2	2922.2(Petrol diesel), 2924.1(PKO), 2924.1(TG) 2926.0(CSO) 2853. (diesel, PKO, TG), 2855.1(CSO)	Stretching vibrations of C-H that are asymmetric and symmetric in CH2 and CH3 groups, respectively	Strong
3	1740.7 (PKO & CSO) 1742.5 (TGO)	C=O group of triglycerides and esters	Strong
4	1600–1400	CH2 and CH3 aliphatic groups,	Small
5	1362.3(TG, PKO) 1377.3(petrodiesel)	HCH	Small
6	1500–900	chemical differences between biodiesel and vegetable oil	Small

This discrepancy suggests that factors other than FFA content alone may influence the presence or intensity of the O–H stretching peak in the FT-IR spectra. One possible explanation for this difference is the varying composition of the FAMEs in the different feedstocks. The presence of hydroxy-groups in FAMEs from TGO might result in altered molecular interactions or structural arrangements that affect the O–H stretching vibrations and lead to changes in the FT-IR spectrum. Additionally, it is important to consider that the FT-IR technique may have limitations in capturing subtle differences in specific functional group vibrations, which could contribute to the disparity observed.

Further investigations are required to fully elucidate the reasons behind the absence of an O–H stretching peak in the TGO biodiesel spectrum despite the presence of hydroxy-containing FAMEs. Future studies could explore more comprehensive characterization techniques or consider additional factors that may impact the observed FT-IR spectra differences, such as molecular conformation, hydrogen bonding, or intermolecular interactions.

The bands at 2922.2 cm^{-1}(Petrol diesel), 2924.1 cm^{-1} (PKO), 2924.1 cm^{-1} (TGO), and 2926.0 cm^{-1} (CSO) in the spectra are attributed to stretching vibrations of C-H that are asymmetric and symmetric in CH2 and CH3 groups, respectively [35]. The strong bands at 1740.7 cm^{-1} (PKO & CSO) and 1742.5 cm^{-1} (TGO) demonstrate a stretching vibration band of triglycerides and esters. The bending vibrations of the aliphatic groups CH2 and CH3 were confirmed by the peaks at 1600–1400 cm^{-1}, whereas the bending of HCH occurred at 1362.3 cm^{-1} (TGO & PKO) and 1377.3 cm^{-1} (petrodiesel). The area of the spectrum between 1500 and 900 cm^{-1} elucidates the chemical differences between biodiesel and vegetable oil [36]

Table 3 Biodiesel major resolved peak areas from PKO gas chromatogram and suggested compounds from NIST11 LIBRARY

Fatty acid methyl esters (FAME)	Retention time (mins)	FAME yield (%)	Molecular formula	Molecular weight
Octanoic acid methyl ester	7.963	4.447	$C_9H_{18}O_2$	158.2
Dodecanoic acid methyl ester (Lauric acid)	11.640	9.265	$C_{13}H_{26}O_2$	214
Methyl tetradecanoate	12.896	6.870	$C_{15}H_{30}O_2$	242
Pentadecanoic acid 14-methyl, methyl ester	13.977	7.673	$C_{17}H_{34}O_2$	270.5
6-Octadecanoic acid, methyl ester, (z)- (Stearic Acid)	14.994	15.282	$C_{19}H_{36}O_2$	296.5
9-Octadecenoic acid, methyl ester, (E)- (Methyl elaidate)	15.292	3.343	$C_{19}H_{36}O_2$	296.5
Methyl stearate	15.379	7.303	$C_{19}H_{38}O_2$	298
cis-13-Eicosenoic acid, methyl ester	16.535	2.924	$C_{21}H_{40}O_2$	324.5
Eicosanoic acid, methyl ester (Arachidic acid)	16.717	7.090	$C_{21}H_{40}O_3$	340.5
Hexadecanoic acid, 2-hydroxy-, methyl ester	17.286	4.463	$C_{17}H_{34}O_3$	286.5
Octadecanoic acid, 9–10-dihydroxy-, methyl ester	17.842	1.803	$C_{19}H_{38}O_4$	330.5

3.3 GC MS Characterization of Biodiesel

The fatty acid methyl ester profile is one of the crucial factors used to determine if a feedstock is suitable for use in the manufacturing of biodiesel fuel [33]. According to the data in Table 3, the most prevalent ester in PKO biodiesel are 6-octadecanoic acid methyl ester (z)- (methyl petroselinate), and dodecanoic acid methyl ester (Lauric acid) with 15% and 9% composition respectively. Given the high percentage of saturated fatty acids in PKO biodiesel, it is likely that combustion will occur with a high heat of combustion and a quick release of heat.[37], which is supported by the sample's comparatively high HHV and cetane numbers. This is excellent news for biodiesel stability because due to the substantial polymerization tendency generated by peroxidation, a higher degree of unsaturation in fatty acid methyl esters restricts their usefulness as a fuel.

The methyl ester compound present in PKO biodiesel bears great similarities to those found in white and black PKO [38], which contained octanoic acid methyl ester, decanoic acid methyl ester, dodecanoic acid methyl ester, methyl tetradecanoate, hexadecanoic acid, 9-octadecanoic acid, and eicosanoic acid methyl ester. The reduced presence of unsaturated fatty acids is important since heating higher unsaturated fatty acids is known to result in glyceride polymerization. This frequently results in the production of deposits in the engine or lubrication degradation [39]. Similar results were also recorded by Aledetuyi [40] with PKO biodiesel containing methyl octanoate, methyl dodecanoate, methyl tetradecanoate, 14-methyl pentadecanoate, 6-octadecanoate, and 2-hydroxyl-1-methyl hexadecanoate among major fatty acid esters.

The major fatty acid methyl ester compounds detected in Tiger nut biodiesel are listed in Table 4 above, with the most abundant in the sample being 9-octadecanoic acid (z) methyl

Table 4 Biodiesel major resolved peak areas from TGO Gas chromatogram and suggested compounds from NIST11 library

Fatty acid methyl esters (FAME)	Retention time (mins)	FAME yield (%)	Molecular formula	Molecular weight
Octanoic acid methyl ester	7.943	1.226	$C_9H_{18}O_2$	158.2
Dodecanoic acid, methyl ester	11.686	4.942	$C_{13}H_{26}O_2$	214
Octanoic acid, 6,6-dimethoxy-, methyl ester	12.244	1.697	$C_{11}H_{22}O_4$	218
Methyl tetradecanoate	12.941	3.381	$C_{15}H_{30}O_2$	242
Hexadecanoic acid, methyl ester	13.961	5.383	$C_{17}H_{34}O_2$	270.5
Pentadecanoic acid, 14-methyl-, methyl ester	14.276	3.583	$C_{16}H_{32}O_2$	256
Hexadecanoic acid, 15-methyl-, methyl ester	14.589	1.435	$C_{17}H_{34}O_2$	270.5
6-Octadecenoic acid, methyl ester, (z)-	15.730	6.731	$C_{19}H_{36}O_2$	296.5
10-Undecenoic acid, 2-methoxy-, methyl ester	16.125	4.290	$C_{13}H_{24}O_3$	228
Eicosanoic acid methyl ester	16.831	7.617	$C_{21}H_{42}O_2$	326.5
9-Octadecanoic acid (z), methyl ester (Oleic acid)	16.925	13.736	$C_{19}H_{36}O_2$	296.5
Octadecanoic acid, 9,10-dihydroxy-methyl ester	17.945	8.959	$C_{19}H_{38}O_4$	330.5

ester (13.736%), octadecanoic acid, 9,10-dihydroxy-methyl ester (8.959%), eicosanoic acid methyl ester (7.617%), and 6-octadecenoic acid methyl ester, (z)- (6.731%).

According to the data presented in Table 4, it is observed that tiger nut biodiesel contains a significant percentage of monounsaturated oleic acid, along with a substantial proportion of long-chain saturated fatty acids. The presence of these fatty acids may contribute to a lower affinity for oxygen, potentially leading to a higher susceptibility to rapid peroxidation in the biodiesel sample.

Furthermore, it is noted that all three biodiesel samples exhibit higher cloud point temperatures, which can be attributed to their elevated saturated fatty acid content. This property indicates that these biodiesel samples may have limited suitability for regions with extremely low temperatures, such as Canada or parts of Europe, where temperatures can reach as low as − 40 °C. In regions of Middle and Eastern Europe, for instance, temperatures can frequently drop below − 3 °C, with maximum temperatures during some winter days reaching as low as − 20 °C or even lower during the night. Similarly, in certain areas of Canada, minimum temperatures as low as − 40 °C can be experienced, depending on the specific location and prevailing weather conditions.

Considering these extreme temperature conditions, the cloud point temperature of the biodiesel samples, which indicates the temperature at which solid particles start to form and can potentially clog fuel lines and filters, might pose challenges for their direct use in colder climates. Additional measures, such as cold flow improvers or blending with additives, would be necessary to enhance the low-temperature operability of these biodiesel fuels in regions characterized by severe winter conditions (see Table 5).

Ricinoleic acid has been discovered as a significant component of castor oil in various studies and is shown to be the most prevalent in the castor oil biodiesel sample, accounting for 57.9% of the total composition. Ricinoleic acid is an unsaturated fatty acid that may account for the variations in CSO biodiesel over PKO and TGO, and as castor oil's methyl esters have very low cloud and pour points, this biofuel is a great alternative for winter conditions. The cetane number of CSO is lower, which may be related to the existence of the castor oil molecule's free hydroxyl group and the ricinoleic acid double bonds. Because the value is lower than the norm, blending with petrodiesel [41] might be an option to meet the standard.

4 Conclusions

In this study, palm kernel oil, tiger nut oil, and castor seed oil were evaluated as potential feedstocks for biodiesel production in Nigeria. The transesterification of these feedstocks resulted in the production of fatty acid methyl esters (FAME) that exhibited some properties within the range of ASTM biodiesel standards. However, upon careful evaluation of the measured characteristics and the referenced standards, it is evident that some properties need to meet the specified limits, indicating potential issues for direct use without modifications.

It is important to note that specific measured properties raised concerns. The acid values were found to be relatively high, and the density exceeded the referenced ASTM standard. Additionally, the ash content exceeded the recommended limits for two out of the three biodiesels produced. Furthermore, properties such as oxidative stability, storage stability, and combustion characteristics were not directly measured in this study, highlighting the need for further investigations. The estimation of specific characteristics, such as cetane number and higher heating value, based on parameters like iodine value and saponification value, can provide approximate values but cannot guarantee acceptable properties. Therefore, a comprehensive assessment of these properties, including oxidative stability and combustion performance, through engine tests or other relevant methods, is crucial to better understanding the behavior of biodiesel fuels.

Also, exploring the use of additives, such as antioxidants, and considering fuel blending strategies could address the identified shortcomings and enhance the overall performance and stability of biodiesel fuels.

Table 5 Biodiesel major resolved peak areas from CSO gas chromatogram and suggested compounds from NIST11 library

Fatty acid methyl esters (FAME)	Retention time (mins)	FAME yield (%)	Molecular formular	Molecular weight
Dodecanoic acid, methyl ester	11.647	2.548	$C_{13}H_{26}O_2$	214
Methyl tetradecanoate	12.899	1.432	$C_{15}H_{30}O_2$	242
Tetracosanoic acid, methyl ester	13.557	2.955	$C_{25}H_{50}O_2$	382.6
Pentadecanoic acid,14-methyl-, methyl ester	14.017	7.129	$C_{17}H_{34}O_2$	270.5
9-Octadecanoic acid (z), methyl ester (Oleic acid)	14.975	21.160	$C_{19}H_{36}O_2$	296.5
cis-10-Nonadecenoic acid, methyl ester	15.752	1.704	$C_{20}H_{38}O_2$	310.5
Methyl 12-hydroxy-9-octadecenoate (ricinoleic acid)	16.676	57.888	$C_{19}H_{36}O_3$	312.5

The results of this study indicate that Nigeria possesses the necessary resources to produce its own alternative energy source that can rival those of most countries in the world. This would not only play a role in combating global warming and climate change but would also be a significant boost to the country's GDP. The use of biodiesel as an alternate fuel source, sourced from sustainable feedstocks, can be an important step towards reducing the reliance on diesel and fossil fuels and promoting sustainable energy development in Nigeria.

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Declarations

Conflict of interest The authors have no competing interests to declare that are relevant to the content of this article.

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