



Production and Characterization of Biodiesel from Coconut (*Cocos nucifera*) Oil

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Abstract To have an environment free from pollutants, advocates of biodiesel claim that it is renewable, efficient, environmentally friendly and biodegradable fuel made from vegetable oil. The research focused on using green chemistry metrics in assessing biodiesel production process. Coconut oil was extracted by cold extraction from coconuts bought at Afo Obosima in Ohaji Egbema- a crude oil heavily polluted village in the oil rich Niger Delta Nigeria. Biodiesel was produced via base catalyzed transesterification. Both coconut oil and produced biodiesel were each characterized in parallel experiments using standard methods. Selected green chemistry metrics were used to assess the greenness of the process. Biodiesel showed that the coconut oil density; 0.89 g, viscosity; 21mm²/s, saponification value; 182 mg KOH/g, iodine value; 11.4 mgI₂/g, acid value; 14.3 mg KOH/g and flash point; 250 °C, smoke point; 160 °C, pH; 7.8, ester value; 167.7, free fatty acid; 7.2% and peroxide value; 5meq/kg while coconut oil had density; 0.75 g, viscosity; 2.7 mm²/s, saponification value; 85 mg KOH/g, iodine value; 9 mgI₂/g, acid value; 0.5 mg KOH/g and flash point; 125 °C, smoke point; 82°C, pH; 8.6, ester value; 82.75, free fatty acid; 0.25% and peroxide value; 0.15. The physicochemical properties of biodiesel produce from coconut oil compared with that of the standard biodiesel in the range of ASTM specifications. The FTIR shows that methyl ester functional groups are present in the biodiesel and absent in the coconut oil. The GC-Mass spectroscopy shows that fatty acid methylesters (67.8 %) are the most prominent component of the biodiesel. Results were within the ASTM specification for biodiesel and there biodiesels were successfully prepared from coconut oil. Atom economy, e-factor and green star for the process were assessed using appropriate factors and results showed that the process was generally adjudged to be a green process.

Keywords Biofuel, Cold extraction, Energy, Emission, Fuel properties, Greenometrics, Transesterification

1. Introduction

Biodiesel is a renewable, efficient, environmentally friendly and biodegradable fuel made from vegetable oil, including waste cooking oil. In the United States soya bean oil is the major oil being utilized for biodiesel. Fuel and

energy crisis and the concern of the society for the depleting world's non-renewable energy resources led to a renewed interest in the quest for alternative fuels. The first use of vegetable oil in a compression ignition engine was first demonstrated through Rudolph Diesel who used peanut oil in his diesel engine [1]. Biodiesel is defined by American Society for Testing and Material (ASTM) as a fuel composed of monoalkylesters of long-chain fatty acids derived from renewable vegetable oils or animal fats. Vegetable oil and animal fats are principally composed of triglycerols (TAG) consisting of long-chain fatty acids chemically bound to a glycerol backbone [2]. The chemical process by which biodiesel is prepared is known as the transesterification reaction, which involves a TAG reaction with a short-chain monohydric alcohol ($\text{CH}_3\text{-OH}$) normally in the presence of a catalyst at elevated temperature to form fatty acid alkyl esters (FAAE) and glycerol.

Coconut oil or copra oil is edible oil extracted from the kernel or meat of mature coconut harvest from the coconut palm. Because of its high saturated fat content, it is slow to oxidize and thus resistant to rancidification lasting up to six months at 24 °C (75F) without spoiling. The production of coconut oil can be obtained in two ways i.e dry process or wet process. The significance of this research work is to produce alternative source of energy which is less expensive, lower toxic emission, lower greenhouse gases, greater efficiency and a friendly habitat for human. Infact, because of the increase in the world population annually, the concentration of fossil fuel deposit is forecasted to finish in the few decades from now. These problems pose effects to our environment and at the same time create an unfriendly habitat for both man and plants [6].

Biodiesel is produced chemically by the process known as the transesterification which involves a triacylglycerol reacting with a short-chain monohydric alcohol normally in the presence of a catalyst at elevated temperature to form fatty acid alkyl esters and glycerols. Commonly used short-chain alcohols are methanol, ethanol, propanol and butanol but methanol is used commercially because of its low price [21]. Biodiesel can protect the environment by reducing CO_2 , SO_2 and hydrocarbons. The carbon cycle of biodiesel is dynamic through the photosynthesis process. Plants absorb CO_2 , which is more than those discharged by the biodiesel combustion process thus, using biodiesel can more effectively reduce the emission of CO_2 protect the natural environment and maintain the ecological balance, compared to the use of fossil fuel. The emission of SO_2 in the combustion process of biodiesel is much lower than normal diesel oil because of the low sulphur content in it. The use of biodiesel instead of normal diesel oil will effectively reduce acid rain, which is a serious threat to the environment and human in fracture in forms of acidification of soil, surface and ground water, forest and vegetation damage, increased corrosion of buildings and historical monuments made from calcium containing stones. CO , HC and particulate matters will be less discharged because ester compounds in biodiesel contain oxygen promoting clean burning. The use of biodiesel in a convectional diesel engine results in a substantial reduction of hydrocarbons, aromatic hydrocarbon, carbon monoxide, alkenes aldehydes, ketones and particulate matter [23].

The Specific objectives of the current work include the following: to extract using cold extraction method and to characterize oil from coconut; to carry out transesterification of the coconut oil using KOH . The biodiesel produced will be characterize using fuel characteristics and finally, both oil and biodiesel will be subjected to infrared spectroscopy (IR) and GC-Mass spectroscopy GC-MS analysis to enable.

2. Methodology

2.1 Equipment and Apparatus

Oven, Weighing balance, Rotary evaporator, Clinical flask, Shaker bath (temperature controlled), Reflux set up, Separating funnel, Burettes, Hot plate, Thermometer, Viscometer, Density bottle, Flash point tester, Beaker, Volumetric flask, Retort stand, Clamps and magnetic stirrer.

2.1.1 Sample collection

The sample, coconut was purchased at Afo Obosima Ohaji/egbema market, Imo State, Nigeria.

2.1.2 Drying

The drying of coconut for the production of biodiesel can be achieved by any of this means; oven drying, sun drying or shade drying. For this research work, sun drying was adopted.

2.1.3 Extraction

The extraction is done using cold extraction method but solvent extraction can also be used in the extraction of coconut oil.



2.1.4 Chemicals /Reagent

All the reagents used for this work were of analytical grade otherwise stated.

They includes potassium hydroxide (KOH), methanol (CH₃OH), coconut oil, hydrochloric acid (HCl), phenolphthalein, starch, potassium iodide, cyclohexane, wiggins solution, sodium thiosulfate (Na₂S₂O₃), sulphuric acid, diethyl ether, phenolphthalein. 0.5 M alcoholic potassium hydroxide, 0.2 M hydrochloric acid, 0.1 M potassium hydroxide, 1% Glucose, 10% potassium iodide, 1% phenolphthalein, 0.1 M sodium thiosulfate.

2.2 Transesterification of coconut oil

Potassium methoxide was prepared by dissolving 0.5 g of KOH in 100 cm³ of methanol. 20 ml of coconut oil was mixed with 100 cm³ of potassium methoxide and stirred at 600 rpm and a reaction temperature of 50 °C for 2 hours in a volumetric flask. The mixture was poured into a separating funnel and allowed to stay overnight for the reaction to be completed and for the mixture to separate into two layers of biodiesel and denser glycerol at the bottom. The glycerol was drained off and the biodiesel was washed with distilled water stir gently to remove impurities such as diglycerine and monoglycerine, catalyst, soap and excess methanol, which can affect combustion and exhaust emission (Canakci and Van Gerpen, 1999). It was allowed to settle for 2 hours to separate into two layers of pure biodiesel and hydrated methanol, which was separated using separating funnel.

2.2.1 Heating of the oil

In order to speed up the reaction, the oil was heated. The ideal temperature range is 120-140° F. The reaction can take days at room temperatures and will be inhibited above 140° F. Heating with electric elements was the easiest way to bring up the oil up to temperature required.

2.2.2 Mixing of methanol and catalyst.

The purpose of mixing methanol and the catalyst (KOH) is to react the two substances to form methoxide. Methanol and KOH are dangerous chemicals. Therefore, care was taken not to touch skin when handling. KOH does not readily dissolve into methanol and KOH was slowly added to it. The methoxide was ready to be used when the particles of KOH cannot be seen. The mixing process was achieved between 20- 30 minute.

2.2.3 Draining of glycerol

After the transesterification reaction, the glycerol was allowed to settle at the bottom of the separating funnel. This happen because glycerol is heavier than biodiesel. The settling begins immediately but the mixture was left a minimum of eight (8) hours preferably 12 hours to make sure all of the glycerol has settled out. The glycerol volume was approximately 20% of the original oil volume.

2.2.4 Washing of biodiesel product

The product of the transesterification reaction contains some impurities like un-reacted methanol; potassium methoxide and possibly potassium alkylate (soap). Therefore, it needs some form of purification before it can be used in diesel engine. Since all the impurities are polar group, water is a suitable solvent for dissolving. The following procedure was used in washing the biodiesel.

20 cm³ of water measured using a measuring cylinder and poured gently on the product sample. The mixture was gently stirred to avoid foam formation. Shaking rigorously is not advised. The mixture of water and biodiesel was left for 16 hours to settle into two phases via; water-impurities phase and biodiesel phase. The two-phase mixture was then separated using a separating funnel. The biodiesel layer was then heated to about 100 °C for 1 hour to evaporate the remaining water molecules in it. (Yusuf and Sirajo, 2009)

$$\text{Biodiesel yield(\%)} = \frac{\text{Weight of oil used}}{\text{Weight of biodiesel produced}}$$



2.3 Characterization of biodiesel

2.3.1 Determination of specific gravity/ density.

The analysis was carried out according to the method reported by Morris (1999).

50 ml viscometer bottle was washed thoroughly with detergent, water and petroleum ether, then dried and weighed.

The bottle was then filled with water and weighed before it was dried again. After the drying process, the bottle was filled with the oil sample and then weighed.

Calculation:

$$\text{Specific gravity} = \frac{\text{Weight of empty bottle} + \text{oil}}{\text{Weight of empty bottle} + \text{water}}$$

$$\text{Density (g/ml)} = \frac{\text{Weight of oil}}{\text{volume of oil}}$$

2.3.2 Determination of viscosity

This was done by preparing 1% glucose solution in 100 ml volumetric flask used as the standard. The flow rate of 1% glucose solution was determined using a 10 ml syringe. The flow rate of the test samples was determined using the same syringe. The viscosity of the test samples was calculated using the equation

$$\frac{V_s}{V_o} = \frac{F_s}{F_o}$$

where: V_s = Viscosity of sucrose, V_o = Viscosity of oil, F_s = flow rate of sucrose, F_o = flow rate of the oil.

2.3.3 Determination of the acid value (AOAC, 1990)

The acid value is the number of milligram of KOH necessary to neutralize the acid (free organic acid) in one gram of the sample. It is a measure of the free fatty acid present in an oil samples.

Procedures: 2 g of the test samples were weighed into a conical flask, 50 cm³ petroleum ether added and mixed gently, 50 cm³ ethanol was added into the mixture and titrated with 0.1 M KOH to pink colour.

Calculation:

$$\text{Acid value (mg KOH/g)} = \frac{\text{titre value} \times 56.1 \times \text{normality}}{\text{Weight of sample}}$$

$$\% \text{ free fatty acid} = \frac{\text{titre value} \times 28.2 \times \text{normality}}{\text{Weight of sample}}$$

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

1 cm³ of 1 M KOH = 56.1 mg of KOH

2.3.4 Determination of saponification value

This is the number of milligram of KOH required to react completely to saponify 1 g of oil. It is inversely proportional to the molecular weight of a sample; therefore, could be used to access the molecular weight of the sample oil under determination.

Procedure: The saponification value was determined by weighing 2 g of the test samples into a round bottom flask and 25 cm³ alcoholic KOH added.

The flask was then fitted onto a condenser and the solution refluxed for 10 min, 1 cm³ of phenolphthalein was added to the refluxed mixture and titrated with 0.2 M HCl and the titre value was recorded.

This was repeated with 25 cm³ KOH as blank and the test titre value taken. The difference between the blank and the tested titre gives the amount of KOH absorbed by the oil.

Calculation:

$$\text{Saponification value} = \frac{(S - B) \times M \times 56.1}{\text{Weight of sample used}}$$

where: B = Blank titre value, S = sample titre value, M = Molarity of the HCl,

56.1 = Molar mass of KOH.



2.3.5 Determination of flash point

The flash point was determined by the used of the automatic pensky marten flash point tester. The samples was poured into the cup of the tester and covered. A flame or electric spark of specified size was directed to the cup at interval until the vapour above the sample ignited. The thermometer reading was recorded and the flash point corrected.

2.3.6 Determination of iodine value (AOAC, 1990)

About 0.26 g of the test samples was weighed into glass stopper flask and dissolved in 10ml cyclohexane. 20 ml of Wijjis solution was added and the flask was stoppered and allowed to stand for 30 minute in the dark at 25 °C after which 20 ml of 10% KI solution was added. The mixture was titrated witty 0.1 M Na₂S₂O₃ using starch indicator. A blank was carried out and the iodine value was calculated using the following equation:

$$\text{Iodine value} = \frac{12.69 \times C(V_1 - V_2)}{\text{Weight of the sample}}$$

where: C = concentration of Na₂S₂O₃ solution, V₁ = volume of Na₂S₂O₃ used in blank, V₂ = volume of Na₂S₂O₃ used in the determination.

2.3.7 Determination of smoke point.

The smoke point determination was done using the method reported by Onwuka (2005).

10 ml of the oil sample was poured into an evaporating dish. A thermometer was then suspended at the centre of the dish ensuring that the bulb just dips inside the oil without touching the bottom of the dish. After which, the temperature of the oil was raised gradually using a stove.

The temperature at which the oil sample gave off a thin bluish smoke continuously was noted as the smoke point.

2.3.8 Determination of ester value

Ester value was obtained according to Akinola *et al.* (2010) by finding the difference between the saponification values and acid value.

2.4 Infrared spectroscopy (IR)

Infrared spectroscopy deals with the interaction between the molecule and radiation from the IR region of the electromagnetic spectrum (IR=4000-400cm⁻¹). This can be analyzed in three ways by measuring the absorption, emission and reflection. The main use of this technique is in organic and inorganic chemistry. It is used by chemists to determine functional groups in the molecules. IR spectroscopy measures the vibrations of atoms and based on this it is possible to determine the functional groups. IR causes the excitation of the vibrations of covalent bonds within that molecule. These vibrations include the stretching and bending bonds. The method use here to determine the functional group present is the fourier transform infrared (FTIR). The fourier transform infrared spectroscopy is one of the most widely employed techniques for functional group identification. FTIR spectra of coconut oil and coconut biodiesel were recorded on a Buck530FTIR spectrophotometer. Each sample of the coconut oil and biodiesel were mixed with potassium bromate (KBr) salt to form plate. The plate was loaded on the KBr plate and place in the chamber of FTIR spectrophotometer and scanned at room temperature with a scan range of 400-4000 cm⁻¹ at a resolution of 2cm⁻¹ for 4mins.

2.5 Gas chromatography–mass spectrometry (GC-MS)

Gas chromatography–mass spectrometry (GC-MS) is an analytical method that combines the features of gas-chromatography and mass spectrometry to identify different substances within a test sample. Applications of GC-MS include drug detection, fire investigation, environmental analysis, explosives investigation, and identification of unknown samples. The GC-MS is composed of two major building blocks: the gas chromatograph and the mass spectrometer.



3. Results

Table 1: Physico-chemical properties of Coconut Methyl Ester (CME) and coconut oil

Parameters	Coconut oil	Methyl-ester (Bio diesel)	ASTM standard	D6751
Viscosity 40°C (mm ² /s)	21	2.7	1.9 - 6.0	Viscosity 40°C (mm ² /s)
Flash point (°C)	250	125	93 min	Flash point (°C)
Acid value (mgKOH/g)	14.3	0.5	0.8 max	Acid value (mgKOH/g)
Iodine value (mgI ₂ /g)	11.4	9	14 max	Iodine value (mgI ₂ /g)
Saponification value (mg KOH/g)	182	85	120 max	Saponification value (mg KOH/g)
Smoke point (°C)	160	82	NA	Smoke point (°C)
pH	7.8	8.6	NA	pH
Ester value	167.7	82.75	NA	Ester value
Peroxide value (meq/kg)	5	0.15	NA	Peroxide value (meq/kg)
Free fatty acid %	7.2	0.25	NA	Free fatty acid %

NA=Not Available

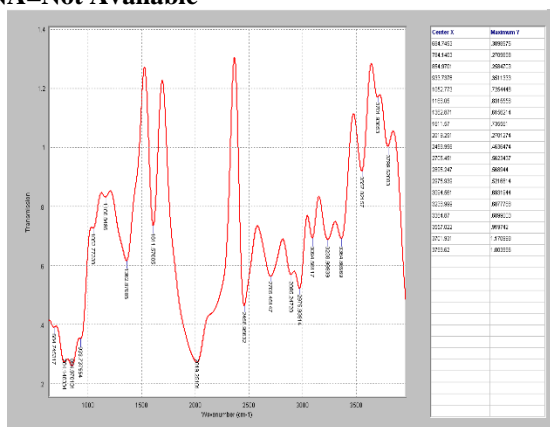


Figure 1: Infrared spectrum of coconut biodiesel

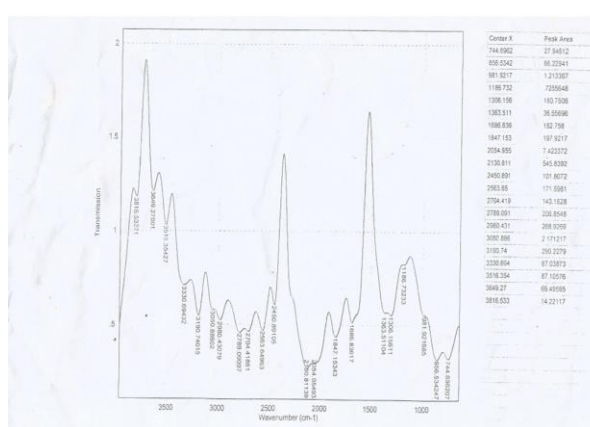


Figure 2: Infrared spectrum of coconut oil

Table 2: Data of biodiesel from GC-mass spectroscopy

Parameter	Biodiesel	
	Conc ppm	% composition
Glycerol	47.2307	20.416
Fatty acid methylester	157.0236	67.875
Alcohol	9.4846	4.100
Diglyceride	1.6078	0.695
Monoglyceride	5.6196	2.429
Triglyceride	10.3645	4.480
Total	231.3409	

4. Discussion and Conclusions

4.1.1 Density: The density of the coconut oil reduced from 0.89 to 0.75 after transesterification process to produce its biodiesel. Therefore, the density of the coconut oil biodiesel is within the range of ASTM standard.

4.1.2 Viscosity: Viscosity of the coconut oil is 23 mm²/s as seen in the table 1 above after transesterification it was reduced to 2.7 mm²/s. However, the viscosity of the biodiesel is low compared to the range of ASTM standard.



Viscosity is one of the important criteria in evaluating diesel quality. High viscosity leads to operational problems including engine deposits.

4.1.3 Acid Value: This is the quantity of base required to titrate a sample to a specified end point. It is a measure of free fatty acid in the biodiesel. High acid value of the fuel can be corrosive and may be a symptom of water in the fuel, poor production or oxidative degradation. Excessive free fatty acid in the fuel can lead to soap formation which tends to inhibit the transesterification process. The acid values for the oil and biodiesel are 14.3 mg/KOHg and 0.5 mg/KOHg respectively. Comparing with the ASTM standard, the acid value for the coconut oil biodiesel is very low. Therefore, coconut biodiesel is not corrosive.

4.1.4 Saponification value: The oil has a value of 182 mg KOH/g which reduces to 85 mg KOH/g for the biodiesel. Coconut oil have high saponification value compared to the ASTM standard.

4.1.5 Iodine value: It is a measure of the degree of un-saturation of vegetable oils, low iodine value means low content of unsaturated fat acids, hence reduced vacant bonds which translates to less reactivity of the fuel, tendency to polymerize, and better storage stability. The iodine values are 11mgI₂/g and 9 mgI₂/g for the coconut oil and biodiesel respectively. The iodine value of the coconut oil and biodiesel is less compared to the ASTM standard. The low iodine value will result in much reduced carbon deposits on engine internal parts and the tendency to block the holes on the injectors.

4.1.6 Flash Points: flash point, which is the temperature at which the fuel can ignite when exposed to a heat source, is important from the point of view of safe handling, storage and transportation. The flash points for coconut oil and biodiesel is 250 °C and 120 °C respectively. Coconut oil and biodiesel compared to the ASTM standard can be classified as a non-hazardous fuel because of its high flash point.

4.1.7 Specific Gravity: The coconut oil has a specific gravity of 0.94 kg/m³ that reduced to 0.90 kg/m³ after transesterification. Density is important in determining the mass of fuel flowing into the engine.

4.1.8 Peroxide value: This is an indicator of oil autoignition and high value is an indication of high degree of rancidity. The peroxide value of the oil was 5 meq/kg while that of the biodiesel was 0.15 meq/kg.

4.1.9 Smoke point: The smoke points for the coconut oil and the methyl ester are 160 °C and 85°C.

4.1.10 Ester value: Ester value is the number of milligrams of potassium hydroxide required to combine with fatty acids present in glyceride form in 1g sample of oil or fat. The coconut oil has an ester value of 167.7 and 82.75 Of the methyl ester.

Table 3: IR absorption data for coconut oil and coconut oil biodiesel

Biodiesel absorption data		Coconut oil absorption data	
Absorption frequencies (cm ⁻¹)	Possible functional groups	Absorption frequencies (cm ⁻¹)	Possible functional groups
684.7	S-OR Ester	744.6	C-Cl Stretch
784.1	C-Cl Stretch	981.9	P-H Phosphine
933.7	P-H Phosphine	1186.7	C-N Aromatic secondary amine
1052.7	C-N Stretch	1306.1	C-C Stretch
1166.0	P=O Phosphine oxide	1363.5	C-C Stretch
1611.5	C=N	1847.1	Open-chain acid anhydrides
2019.2	Cyanide ion	2130.8	C=C Stretch
2458.9	R ₃ C-H	2450.8	R(C=O)O-H Carboxylic acid
2706.4	O=C-H Stretch	2562.6	S-H Thiols
2895.2	C-H Stretch	2704.4	H-C=O Aldehyde
2975.9	C-H Stretch	2789.0	H-C=O Aldehyde
3034.6	C-H Stretch	2980.4	C-H Stretch
3238.9	C-H Stretch	3050.8	O-H Stretch
3336.4	O-H Stretch	3190.7	O-H Stretch
3537.0	O-H Stretch	3330.6	O-H Stretch
3701.9	O-H Stretch	3516.3	O-H Stretch
3330.6	O-H Stretch	3649.2	O-H Stretch
3516.3	O-H Stretch	-	-
3649.2	O-H Stretch	-	-



IR Analysis

IR analysis is a most reliable proof that transesterification took place by displaying relevant absorption peaks. In fig.1 present the IR spectrum of coconut biodiesel and fig. 2 present the IR spectrum of coconut oil. The peaks and their absorption frequencies are indicative of the presence of functional groups present in the coconut biodiesel and coconut oil. The salient groups present in the coconut biodiesel include the ester groups and associated bonds, the hydrocarbon component and its associated bonds, each bond type identified by its absorption frequency. In the IR spectrum of the coconut biodiesel, shows the functional group of fatty acid ethyl esters (1300 cm^{-1}) while the coconut oil does not show the functional group of fatty acid ethyl esters. Both the coconut oil and coconut biodiesel have the O-H group in the IR spectrum. The $3500\text{-}3450\text{ cm}^{-1}$ band is attributed to the presence of small quantity of remnant water used during washing of the coconut biodiesel. The coconut oil contain aromatics secondary amines (C-N) of 1186 cm^{-1} absorption frequency while the coconut biodiesel does not. 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 total absence of aromatics.

GC-Mass Spectroscopy

The analysis of transesterification product using GC-mass spectroscopy are presented in table 2 above. From the data the it was identified that fatty acid methylester is the most prominent component of the sample which is in abundance having 67.8%. glycerol is 20.4% which indicate the complete transesterification of the coconut oil.

Conclusions

At the end of the research work, the methyl ester (coconut oil biodiesel) was discover to have certain properties attributable to the diesel fuel. The Coconut oil biodiesel can be classified as a non-hazardous fuel because of its high flash point. The saponification value is one of the highest for vegetable oils making it suitable feedstock for the manufacture of soaps, detergents and shampoo products. The density and viscosity of the coconut oil and it methyl ester was found to be low. The iodine value is high meaning, high content of unsaturated fat acids. Hence, the low acid value of the fuel tells us that the fuel can't be corrosive because high acid value of the fuel can be corrosive and may be a symptom of water in the fuel. Finally, coconut oil can be classify as one of the best vegetable oil for the production of biodiesel. Coconut oil was extracted and biodiesel was produced from it and also characterized in this study. From the results obtained and discussed, it is very evident that coconut oil is a good feedstock for biodiesel production and the biodiesel can be used in convectional diesel engine without modification because of close fuel properties.

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