



PRODUCTION AND CHARACTERIZATION OF BIODIESEL FROM MARULA (*Sclerocarya birrea*) SEED OIL

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Abstract

The main objective of this work is to evaluate the marula oil yield and physicochemical properties of the marula oil biodiesel in Nigeria. The marula oil with methanol in ratio 6:1 and potassium hydroxide catalyst of concentration of 1.4 wt% were heated in a beaker placed on a hot plate with magnetic stirrer for 30 minutes at a temperature of 60 °C and operated at a speed of 305.5 rpm. The most common physicochemical properties of the marula biodiesel were determined using standardized laboratory methods of the Association of Official and Analytical Chemist. The oil yield of the marula was found to be 58.7%. The saponification value, iodine value, free fatty acid, acid value, peroxide value, cetane number, density, viscosity, calorific value, flash point, and pour point for both marula oil and marula biodiesel were found to be (187.72 mgKOH/g), (61.77 g/100 g), (1.72% and 0.21%), (3.42 mgKOH/g and 0.44 mgKOH/g), (10.67 meq/kg and 2.4 meq/kg), (51 and 55.1), (943 kg/m and 850 kg/m), (41 mm²/sec and 5.0 mm²/sec, (38.40 MJ/kg and 42.89 MJ/kg), (240°C and 175°C) and (5°C and 3°C) respectively. The results of the analysis fall within the specification range of international standards for biodiesel specifications.

Keywords: Marula oil, characterization, biodiesel, physio-chemical, transesterification

Introduction

The continued rise of fuel prices, environmental pollution, depletion of fossil oil and increase in population and energy demand has stirred up active research interest in non-petroleum, renewable, and less polluting fuels. (Singh and GU, 2010). Conventional energy source, such as coal, gas and oil, remain the predominant global energy consumed in the world today have limited reserves that are expected not to last for an extended period (Yunus and Zuru, 2017). Also, greenhouse emission from the use of fossil fuel contributes significantly to climate change, and this has led to the search for alternative source of energy to the non-renewable energy sources. Among these alternative sources are biofuel, bio-ethanol and bio-oil mostly obtainable from biomass which has contributed greatly to the biofuel industry (Akinola and Fapetu, 2015; Fapetu *et al.*, 2018). Vegetable oils occupy a prominent position in the development of alternative fuels. Although, there have been many problems associated with using it directly in compression ignition (CI) engine (especially in direct injection engine). These include; Coking of injectors leading to poor fuel atomization as a result of plugged orifices, Carbon deposits, Oil ring sticking, Thickening or gelling of the lubricating oil as a result of contamination by vegetable oils, and Lubricating problems (Singh and GU, 2010). Other disadvantages to the use of vegetable

oils and especially animal fats are the high viscosity (about 11–17 times higher than diesel fuel), lower volatilities that causes the formation of deposits in engines due to incomplete combustion and incorrect vaporization characteristics. These problems are associated with large triglyceride molecule and its higher molecular mass. These problems can be overcome by chemical modification mainly transesterification, pyrolysis and emulsification which all converts the oil into biodiesel (Atasie *et al.*, 2004). Many vegetable oils (soybean oil, palm oil, castor oil, neem oil and coconut oil) and animal oils have been investigated for the production of biodiesel and are widely reported (Raja *et al.*, 2011; Aransiola *et al.*, 2012; Fapetu *et al.*, 2018).

Transesterification is a catalytic reaction that converts lipids into fatty ester methyl esters and glycerol as co-product through four steps. The first step is preparing the catalyst and mixing the catalyst with alcohol. The second step is the transesterification reaction that takes place through the reaction between the mixture of catalyst / alcohol and with the fatty acid (triglycerides). The optimum temperature range is about 40°C – 60°C for transesterification reaction and the rate of reaction increased with increase temperature. The third step is to separate glycerol from biodiesel, and the fourth step

concerns the addition of water to both the biodiesel and glycerol for purification, especially the glycerol that may remain in the biodiesel (Manirafasha *et al.*, 2020).

Sclerocarya birrea (Marula) plants are grown in the west, north-east and east tropical Africa across a range of vegetation types, principally wooded tropical Africa and sub-Saharan region (Shackleton *et al.*, 2001) and this includes northern Nigeria. The fruit is round or oval drupe, usually wide with a diameter of 30-40 mm, the shape and number of nuts per stone determine the final shape of the fruit (Elijah *et al.*, 2012). It normally grow up to 18-20 meters in height in a sandy loam soil and leafless during winter. The fruit consist of a hard woody seed covered by pulp and juice which makes the fleshy part of the fruit. The fleshy part of the fruit contains a high concentration of vitamin C. The fruits are edible and yellow in colour with a plain tough skin and glutinous flesh which encloses 2-3 white kernels which are rich in oil and protein. Juice, wine and snacks are produce from the pulp (Nyoka *et al.*, 2015). The barks, leaves and roots are used in treating some human ailments such as malaria, fever, stomach problems, headache, toothache, rheumatism and body pains (Attigbo and Tahir, 2016).

Attigbo and Tahir (2016) reported on the evaluation of the physicochemical properties of Northern Ghana *Sclerocarya birrea* seed oil and proximate analysis of the process waste. It was concluded that the physicochemical properties of the extracted oil compared favourably with those reported by other researchers. Ahmed *et al.*, (2015) worked on the

physicochemical evaluation of seeds and oil of non-traditional oil seeds. It was inferred that all parts of marula fruit are edible with the seeds being very rich in fat (56 – 61%) and protein (28 – 31%). Silitonga *et al.*, (2013) reported that biodiesel is completely miscible with diesel and the blending in different concentrations by volume is possible in order to optimize the fuel qualities. However, different chemical nature of biodiesel and diesel may cause differences in the physicochemical properties that will affect the engine performance and pollutant emissions produced. However, marula plants widely available in the northern part of Nigeria are not being used meaningfully, and are mostly consumed by animals. This paper therefore is aimed at the production and characterization of biodiesel and its blends from marula (*Sclerocarya birrea*) seed oil for energy application, but limited to when B100 (marula oil applied as 100% biodiesel) is compared to 100% petrodiesel and standards.

Materials and Methods

Sample Collection

Fresh fruits were collected from the premises of Waziri Umaru Federal Polytechnic Birnin Kebbi, and its environment in Nigeria. The fruits were crushed using mortar to remove its outer cover, the wet exposed fruit were then sun dried and a hammer mill was used to break the shell to expose the kernels. Figures (1-4) show the ripe marula fruits, wet exposed marula fruit, dry exposed marula fruit and marula kernel respectively.



Figure 1: Ripe Marula Fruits



Figure 2: Wet Exposed Marula Fruit



Figure 4: Marula Kernels



Figure 3: Dry Exposed Marula Fruit

Oil Extraction

Solvent extraction method was used to determine actual oil yield levels of the kernels using soxhlet apparatus under standard conditions as recommended by (Luque-Rodriguez *et al.*, 2005). The soxhlet apparatus is shown in Plate 1



Plate 1: Soxhlet Apparatus

The soxhlet solvent extraction set up comprises of extraction chamber, thimble flask, heating mantle and condenser connected with water recirculation system. The extraction process employed consist of six steps; kernel grinding, solvent preparation, oil extraction, filtration, distillation and purging. A 500 g of Marula kernel were grounded into powder using a mini-grinding machine for 5 minutes. The solvent was prepared by mixing approximately 500 ml of hexane, 100 ml of isopropyl alcohol in a 1000 ml flask and 3 g of anti-bumping stones were added to the mixture to regulate boiling of the solvent during oil extraction. 100 g of powdered sample was charged into a thimble and placed inside a soxhlet setup. The 500 ml of solvent was heated to 60°C for 6 hours of the extraction process. Filtration process was then performed to eliminate any possibility of solid particles in the oil rich solvent using filter paper. The separation of solvent from the oil was achieved through distillation process carried out using a rotary evaporator. However, to ensure that the oil sample is solvent free, the oil was purged with nitrogen gas for approximately 30 minutes.

Transesterification

The experiment was conducted in a laboratory scale apparatus in batch. The transesterification was carried out in a 1000 ml reaction flask equipped with reflux condenser and a digital electric magnetic stirrer. 95 g of marula oil was weighed and preheated to 100°C to eliminate water. The oil was allowed to cool to 60°C. A solution of methanol of 99.5% purity and 8 g of potassium hydroxide of 1.4 wt% was used as catalyst and was added to the preheated marula oil in order of 6 : 1 potassium methoxide to oil volume ratio and the total mixture was stirred for 30 minutes at a speed of 305.5 rpm and controlled temperature of 60°C.

After transesterification, the mixture was poured into a separating funnel and allowed to settle for about 12 hours for the complete separation of glycerol from marula oil methyl ester (MOME). On settling, the biodiesel forms as the top layer and glycerin and other impurities remain as the bottom layer. The bottom layer was removed and MOME as biodiesel was collected. About 50 ml of 50°C distilled water was added to the product of MOME and swirled slowly and left for 2 hours in order to wash and remove the excess methanol and soap formed. This washing process was repeated until the final product of MOME was pure at pH value between 6 and 7. A small quantity of anhydrous magnesium sulphate was added and stirred for 5 minutes. The biodiesel was later filtered using cotton wool to separate the biodiesel from the un-hydrated magnesium sulphate and was subjected to heating at 100°C in an oven to remove excess alcohol and water. The yield of the biodiesel was calculated using Equation 1

$$\% \text{ yield of biodiesel} : \quad (1)$$

$$= \frac{\text{Volume of biodiesel}}{\text{Volume of oil}} \times 100$$

Characterization of the Marula Oil and Biodiesel Produced

The characterization of the Marula Oil and Biodiesel produced were done according to the AOAC (1998; 2000) (Association of Analytical Chemists) Method. Physical and Chemical properties analyzed were saponification value, iodine value, peroxide value, acid value, free fatty acid value, centane number, moisture content, density, kinematic viscosity, and calorific value, flash point, pour point and cloud point.

Saponification Value (S.V.)

According to AOAC (2000), 2 g of oil sample was placed in a 250 ml conical flask, and added with 25 ml of 0.5 M methanolic potassium hydroxide solution, then boiled for 45 minutes for complete saponification. The potassium hydroxide was titrated with 0.5 N hydrochloric acid using 1.0 ml phenolphthalein indicator. A blank determination was carried out under the same condition simultaneously. The procedure was repeated for triplicate samples and the saponification value was calculated using Equation 2 (AOCS, 1998; Li *et al.*, 2013).

$$\text{Saponification Value (S.V)} = \frac{(B-S) \times N \times 56.1}{\text{weight of sample (g)}} \quad (2)$$

where ; B is Blank titre and S is Sample titre value

Iodine Value

The method used for the determination of the iodine value was in conformity with AOAC standard (AOAC, 2000). One gram of oil was placed in a 500 ml conical flask, and 25 ml of iodine monochloride solution was added to the measured oil sample, and the contents were

mixed and placed in the drawer for 30 minutes. Potassium iodide solution (15 ml of 15% w/v) was added to the flask washing down any iodide that may be found on the stopper. This was titrated against 0.13 M $\text{Na}_2\text{S}_2\text{O}_3$ until the solution became light yellow. Starch indicator (1%, 2 ml) was added and the titration continued until the blue colors just disappeared. A blank determination was carried out under the same conditions. The titre value was recorded and used to calculate the iodine value. This laboratory procedure was repeated for replicate test samples, and the iodine value was calculated using Equation (3)

$$\text{Iodine Value (I.V)} = \frac{(B-S) \times M \times 12.69}{\text{weight of sample (g)}} \quad (3)$$

where; M is Molarity of $\text{Na}_2\text{S}_2\text{O}_3$ used, B is Blank titre and, S is Sample titre value.

Peroxide Value

According to AOAC (2000), 5.00 g of oil sample was placed in a 250 ml conical flask, and 30 ml of the acetic acid - chloroform solution was added to the flask. The flask was swirled until the oil was completely dissolved. 0.5 ml of saturated potassium iodide solution was also added and swirled for exactly one minute. Shortly afterwards, 30 ml of distilled water was added into the conical flask to liberate the iodine from the chloroform layer. The solution was initially a light amber color; then 1 ml of starch solution was added as an indicator and titrated with 0.1 M sodium thiosulfate until the blue gray color disappears in the aqueous (upper layer). A blank determination was carried out under the same condition before repeating the procedure in triplicate samples, and subsequently calculating the peroxide value with Equation 4

$$\text{Peroxide Value (P.V)} = \frac{(\text{ml blank (B)} - \text{ml sample (S)}) \times N \times 1000}{\text{weight of sample (g)}} \quad (4)$$

Acid Value (A.V)

According to AOAC (1998), 2 g of oil sample was placed in a 250 ml conical flask and warmed. 50 ml of methanol was added with thorough stirring, and subsequently followed by five drops of phenolphthalein indicator before titrating with 0.13 N potassium hydroxide solutions, until a permanent light pink colour appeared. The procedure was repeated in triplicate samples and the acid value was calculated using relationship in Equation 5 (Banani et al., 2015).

$$\text{Acid Value} = \frac{(V_{eq} - b_{eq}) \times N \times 56.1}{\text{Weight of sample (g)}} \quad (5)$$

where; v_{eq} is volume of titrant (ml) consumed by the crude oil sample and

b_{eq} is Volume of titrant (ml) consumed by spiking solution.

Free Fatty Acid (FFA)

The AOAC method was used for the determination of the Free Fatty Acid (AOAC, 1998). Here, 4 g of oil sample was placed in a 250 ml conical flask and

warmed. 50 ml of methanol was added with thorough stirring and followed by adding 5 drops of phenolphthalein indicator and titrated with 0.1 N potassium hydroxide solution. A permanent light pink colour appeared. The end point of the titration was recorded and used to calculate the FFA value. This procedure was triplicated and the free fatty acid value was calculated using Equation 6

$$\text{Free Fatty Acid \%} = \frac{V \times N \times 56.1}{\text{Weight of sample}} \quad (6)$$

where; N is molarities of base and V is Volume of potassium hydroxide solution

Cetane Number

The Cetane number indicates ignition delay; i.e. the time elapsed between the injection of fuel into the combustion chamber and self-ignition of the fuel-air mixture using Equations (7 - 8).

$$CN = CI \times 0.72 + 10 \quad (7)$$

$$CI = 46.3 + \frac{5458}{SV} - 0.225IV \quad (8)$$

where; CI is Cetane index, SV is Saponification Value and IV is Iodine Value

Density

The density of fuel samples was determined using gravimetric method. The weight of a small empty bottle was determined using an electronic weighing balance. Subsequently, the bottle filled with oil samples in a defined volume was also weighed on the weighing balance. The density was calculated using Equation 9.

$$\text{Density } (\rho) = \frac{W_2 - W_1}{V} \quad (9)$$

where; W_2 is weight of bottle and oil, W_1 is weight of empty bottle and V is volume of the oil

Kinematic Viscosity

Kinematic viscosity was determined by employing ASTM D2171 (ASTM, 2008) standard. U-tube viscometer was used by measuring the time taken for a fixed volume of a sample to flow through the capillary viscometer under an accurately reproducible head, and at a closely controlled temperature. Similar procedure was applied for water. The viscosity value of the test oil samples was calculated from the results obtained using Equation (10).

$$\nu = \frac{T_s - T_w}{T_w} \quad (10)$$

where; ν is Kinematic Viscosity (mm^2/s), T_s is Time to run samples and T_w is Time to run water

Calorific Value

To determine the calorific value, the sample was poured in the capsule of the bomb calorimeter. A fuse wire was inserted in the sample which was then placed in the bomb. The bomb was charged with oxygen from the oxygen cylinder after which it was covered. The calorimeter bucket was then filled with 2 liters of water. The bomb was lowered partially in the bucket. The two ignition lead wires were then pushed into the terminals of the bomb head. The wires were positioned away from the stirrer shaft, to keep them un-entangled. The calorimeter cover was then closed. The calorimeter took over the running of the test, and the calorific values of oils samples were subsequently read.

Flash point

Pensky-Martens closed-cup method was employed to determine the flash point of the fuel. 100 ml conical flask with a branch opening and a cork with an opening, a thermometer was inserted in to the flask through the cork opening in such a way that its tip does not touch the bottom of the flask. The set up was placed on a hot plate. About 5 ml of MOME was put inside the flask and heated. The test flame was applied every 2°C rise in temperature. The flash point was taken at the lowest temperature, as the application of the test flame caused

the vapour leaving the flask opening to ignite (ASTM, 1990).

Cloud Point

The cloud point was determined by putting 5 ml each of the extracted oil and MOME into two test tubes and a thermometer with calibration below 1°C was inserted into each test tube. The set ups were in turn inserted in two beakers containing ice. The temperature at which the oil observed to form a cloud of gel was recorded as the cloud point (ASTM, 1990).

Pour Point

This was determined by putting 5 ml each of the extracted oil and MOME into two test tubes respectively and a thermometer with calibration below 1°C was inserted into each test tube. The two test tubes were inserted in a beaker containing ice to solidify; the test tubes were removed and tilted. The lowest temperature at which the oil was observed to flow was recorded as the pour point (ASTM, 1990).

Results and Discussion

The results obtained are presented in Tables 1 and 2 for both physical and chemical properties of the extracted marula oil and the transesterified marula oil in comparison with diesel and standard limits.

Table 1: Physical Properties of Extracted Marula Oil and Transesterified Marula Oil

S/N	PARAMETER	UNITS	EXTRACTED MARULA OIL	TRANSESTERIFIED MARULA OIL (B100)	DIESEL	ASTM LIMITS	DIN EN 14214
1	Specific Gravity	-	0.850	0.943	0.880		
2	Kinematic Viscosity @ 40°C	mm ² /s	41.0	5.0	4.0	1.9-6.0	3.5-5.0
3	Pour Point	(°C)	5	3	-20	-	-
4	Cloud Point	(°C)	10	8	-12		
5	Smoke Point	(°C)	140.00	66.00	-	-	
6	Flash Point	(°C)	240	175	68	>130	>120
7	Fire Point	(°C)	160.00	72.00	-	-	
8	Moisture Content	(%)	4.2	0.04	0.02	<0.03	<0.05
9	Cetane Number		54.3	55.1	46.0	47	51
10	Density	kg/m ³	943	880	850	-	
11	Calorific Value	(MJ/kg)	38.4	42.89	46.0	42	-

Table 2: Chemical Properties of Extracted Marula Oil and Transesterified Marula Oil

S/N	PARAMETER	UNITS	EXTRACTED MARULA OIL	TRANSESTERIFIED MARULA OIL (B100)	DIESEL	ASTM LIMITS	DIN EN 14214
1	Acid Value	(mgKOH/g)	3.42	0.44	0.35	<0.8	<0.5
2	Free Fatty Acid	(mg/g)	1.72	0.21	-		
3	Peroxide Value	(mgKOH/g)	10.67	2.4	-	-	-
4	Saponification Value	(mgKOH/g)	187.72	-	159.89		
5	Iodine Value	(mgI ₂ /g)	61.77	-	-	115 max	120 max
6	Ester Value	(mgKOH/g)	171.194	161.568	-		-

Discussions

Saponification Value

The saponification value of extracted marula oil was 187.72 mgKOH/g, which agrees with 187-196 mgKOH/g range according to (Penfield *et al.*, 1990), 187 - 191 mgKOH/g (Sesame indicum L. seeds), 179 – 220 mgKOH/g (Detarium microcarpum and Moringa oleifera), and 185 – 200 (Shea butter oil) (Akpabio, *et al.*, 2011; Fapetu *et al.*, 2018). High saponification improves lubrication property of the oil, reduces engine wear, extend the operational life and efficiency of the diesel fuel pumps and injector (Ejilah *et al.*, 2012). It also gives tendency of soap formation and lowers the yield by increasing the formation of emulsions in the product making it difficult to separate biodiesel from glycerin (Shah *et al.*, 2004). The result obtained is higher than 154.6 mgKOH/g balanites aegyptiaca by Jaruo and Adams (2011), 137 mgKOH/g Cashew nut oil (Akinhanmi and Atasie, 2008); and lower than 330.99 mgKOH/g Raffia regalis oil by Fapetu *et al.*, (2018), 216.43 mgKOH/g balanites aegyptiaca by (Chomini *et al.*, 2020) and 190.00 mgKOH/g by Musa and Aberuagba (2012).

Iodine Value

The iodine value refers to an index of amount of double bonds in biodiesel. Thereby, the parameter quantifies the degree of unsaturation of the biodiesel. Higher iodine value implied higher number of unsaturated bonds and makes the oils samples susceptible to oil oxidation. The iodine value of marula oil is 61.77 gI₂/100g this indicates low degree of unsaturation and that the oil is non-drying since the iodine value is less than 100 gI₂/100g (Pehrsson *et al.*, 2000; Fapetu *et al.*, 2018). This value is also lower than 100.33 gI₂/100g (Ejilah *et al.*, 2012), 84 gI₂/100g (Ghobadian *et al.*, 2009) and 67.5 gI₂/100g (Haile *et al.*, 2019). The results show that iodine value of biodiesel purely depended on the source from which biodiesel is produced.

Free Fatty Acid

The percentage FFA for Marula oil was 1.72%. This enable the direct transesterification of the oil to form biodiesel since from literature, FFA above 5% requires acid esterification before a base transesterification. Higher FFA content lead to soap formation as a result the separation of biodiesel from glycerol would be more difficult and the oil yield would be less. Marula Oil Methyl Ester was 0.21%. This value fell within the limits recommended by ASTM and EN standards.

Acid Value

Acid value is an important factor to evaluate the quality of biodiesel as it measures the amount of acids present in the fuel, which are hydro peroxide in biodiesel that further oxidized into acid. Acid value of the extracted marula oil was 3.42 mgKOH/g. This was out of specifications of EN14214 biodiesel standard (0.5 mgKOH/g minimum) and ASTM D665 (0.8

maximum). This shows why transesterification was needed. An acid value of 0.44 mgKOH/g was obtained for the Marula Oil Methyl Ester (MOME). This value fell within the specification of EN and ASTM standards. Acid value indicates the extent of decomposition of the constituent glycerides by lipase activities (Ejilah *et al.* 2012). It determines its edibility, shelf-life, and industrial application. Low acid value oils (<4.0 mgKOH/g) are edible to human and livestock (Nwe *et al.*, 2019). The acid value is quite impressive when compared to about 2.18 mgKOH/g for balanites aegyptiaca oil (Chomini *et al.*, 2020), and 0.995 mgKOH/g for balanites aegyptiaca oil (Jauro and Adams 2011).

Peroxide Value

Peroxide value of oil indicates the degree of primary oxidation and its likeliness of becoming rancid (Musa *et al.*, 2016; Fapetu *et al.*, 2018). A lower number of peroxide values indicate a good quality of oil and a good preservation status (Lanjekar and Deshmukh, 2016). The peroxide values of both marula oil and Marula Oil Methyl Ester were 10.67 mEq/kg and 2.4 mEq /kg respectively. The obtained values compared favourably with about 5 mEq/kg for jatropa (Lanjekar and Deshmukh, 2016), and that of 4.84 mEq/kg for balanites aegyptiaca oil (Chomini *et al.*, 2020). However, it is higher when compared to 1.5 mEq /kg of groundnut oil (Akinhanmi and Atasie, 2008).

Density Value

Fuel density is the mass per unit volume, is an important design parameter for diesel fuel injection system. The density of Marula Oil Methyl Ester (880 kg/m³) was higher compared to that of diesel (850 kg/m³) but lie within ASTM standard (860 – 900 kg/m³). Biodiesel density is usually higher than that of petro-diesel (Elsolh, 2011). Haile *et al.*, (2019) in their research reported 906 kg/m³ for moringa stenopetala biodiesel, he concluded that the higher density may also be attributed to the higher molecular weight of triglyceride molecules present in biodiesel which strongly depends on temperature, fatty acid composition, and purity which in turn affects the quality of atomization and combustion fuel in compression ignition engine (Sandu and Chiru, 2007).

Viscosity Value

Kinematic viscosity is a very important property since it directly affects combustion of the fuel in the engine. A fuel with high viscosity flows with difficulty; while fuels with lower viscosity flow more easily. The kinematic viscosity of Marula Oil and Marula Oil Methyl Ester is 41 and 5.0 mm²/sec at 40°C respectively. The (MOME) value is within the biodiesel specification standard prescribed by the EN 14214 of 3.5 - 5.0 mm²/sec and ASTM D6751 of 1.9 - 6.0 mm²/sec (ASTM D445 method).

The viscosity of (MOME) value of 5.0 mm²/sec is lower than 5.91 mm²/sec for Hara crepitans (Ottih *et al.*, 2015), 5.7 mm²/sec for jatropha (Sivaramakrishnan and Ravikumar, 2012), and 6.2 mm²/sec for waste cooking oil (Nguyen *et al.*, 2020), but higher than 4.56 mm²/sec for palm oil (Vargas, *et al.*, 2019) and 4.05 mm²/sec for coin oil (Santos *et al.*, 2014). With this it shows that the viscosity of oil extracts depends on the nature of the constituent triglycerides, as well as their chemical properties (Azuaga *et al.*, 2018).

Calorific Value

The gross calorific values of crude marula oil and marula biodiesel were found to be 38.40 and 42.89 MJ/kg respectively. The calorific value of (MOME) fell within 42 MJ/kg specified by ASTM. The result is also lower than 44.70 MJ/kg for petro-diesel which agree with conclusion drawn by researchers that calorific value of biodiesel appeared to be less than natural diesel which could be due to the difference in their chemical composition from that of diesel or the difference in the percentage of carbon and hydrogen content, and the presence of oxygen molecule in the molecular structure of marula oil and biodiesel (Musa *et al.* 2016).

Cetane Number

Cetane number predicts the ignition delay quality for fuels. This is one of the important parameters, which is considered during the selection of fatty acid methyl esters (FAMES) for uses as biodiesel. Generally, FAMES with higher Cetane number (CN) are favoured for use as biodiesel. Cetane number of Marula oil and Marula Oil Methyl Ester are 51 and 55.1 respectively, which is higher than 46 for petro-diesel. The result also shows quantitative proximity with 47.7 for waste cooking oil (Nguyen, *et al.*, 2020) and 54.18 for Balanites aegyptiaca oil (Chomini *et al.*, 2020). Biodiesel sample exhibited higher Cetane number than conventional diesel fuel and these results in higher combustion efficiency and smoother combustion (Ariza-Montobbio and Lele, 2010).

The higher Cetane number observed in biodiesel as compared to diesel fuel was because of its oxygen content and the presence of fatty acid (Savaramakrishnan and Ravikumar, 2012). The longer the carbon chains and the more saturated the molecules, the higher the Cetane number.

Flash Point, Cloud point and Pour Point

Flash point is a measure of volatility and residual alcohol in the fuel, and a determinant of flammability classification of materials. Flash points of crude marula oil and marula biodiesel were determined to be 240°C and 175°C, and were quite high compared to 68°C for the diesel fuel. A fuel becomes difficult to handle if the flash point falls below 130°C limit set by ASTM standards (Bello *et al.* 2011). This high value

makes the oil less flammable which would make handling and transportation safer.

The pour points of all samples were determined to be 5°C for crude marula oil, 3°C for B100 and -16°C for diesel fuel. This might be due to the presence of wax, which begins to crystallize with decrease in temperature. The high pour point of marula oil sample constrains oil performance at low temperature conditions. The problems of higher pour point of marula biodiesel could be overcome by blending with diesel (Gawrilow, 2003).

Conclusions

Biodiesel from marula was produced and characterized, and the following conclusions were drawn. High saponification and viscosity values would enhance the lubricity of engine parts which reduces friction and engine wear, and the high Cetane number compare with conventional diesel showed that there will be no ignition delay. The calorific value of the marula biodiesel fell within the standard recommended by ASTM standard and the acid value of marula biodiesel fell within the specification of EN and ASTM standards, suggesting that marula biodiesel can be used as feedstock for biodiesel production. The lower peroxide value of marula indicates a good quality of oil and a good preservation status. The With the low value of free fatty acid, direct transesterification of the marula oil to biodiesel could be done while the flash point of marula will be safer in handling and less flammable than conventional diesel.

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