

Fuel and physiochemical properties of mango (*Mangifera indica*) seed biodiesel and its blends with diesel

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Abstract: The aim of this work is to measure the fuel properties of mango seed biodiesel and its blends with diesel fuel. The oil was extracted by soxhlet extraction method. The high free fatty acid (FFA) value of the oil produced necessitated, acid pretreatment prior to base transesterification with methanol using sodium hydroxide as catalyst which resulted into a considerable reduction of the FFA value from 3.3% to 0.9% and a biodiesel yield of 25.84%. The produced biodiesel was washed, dried and blended with 10% (B10), 20% (B20) and 30% (B30) diesel. They were evaluated following the American Society for Testing and Materials (ASTM) and Europe Norm (EN) protocols. The result obtained shows that the kinetic viscosity at 40°C ranges between 2.86 and 4.32 cts, flash point ranges between 73°C and 145°C with the acid number ranging from between 0.445 and 0.558 mg KOH g⁻¹. Other results show that the biodiesel and its blend have fatty acid between 0.182 and 0.211% with the carbon residue between 0.05% and 0.09%. The properties of the biodiesel and its blend are close to those of diesel and can thus be used as alternative fuel for diesel engines.

Keywords: biodiesel, fatty acids ethyl esters, fuel properties, transesterification, extraction, pretreatment, mango seed

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1 Introduction

The present uncertainty in the world for fuel supply and the projected increase in demand, justify the search for alternative sources of fuel which will be readily accessible. Consequently, the search for alternative sources of renewable and sustainable energy has gained importance with the potential to solve many current social issues such as the rising price of petroleum crude and environmental concerns like air pollution and global warming caused by combustion of fossil fuels (Demirbas, 2005, Koh and Ghazi, 2011). This has led to the exploration for alternate fuel for diesel engines. Most researches have been focused on vegetable oil since it is

a renewable source from agriculture and it is evenly and abundantly distributed all over the world. All efforts to use vegetable oils to fuel diesel engines lead to problems associated to high viscosities lubrication oil contamination due to incomplete combustion and different chemistry of combustion. These factors give rise to several researches on alternative sources of energy that can safeguard ecosystems and public health. The best feasible method to surmount the disadvantages of vegetable oil is by converting it to esters. The advantage of using biodiesel compared to mineral - derived diesel fuel among others includes producing a range of organic raw materials including fresh or waste vegetable oils, animal fats, and oilseed plants. Others include reduced exhaust emissions, improved biodegradability, reduced toxicity and improved lubricity, higher flash point and lower vapour pressure (Margaroni, 1998; Knothe et al., 2005). Another important factor is its positive energy

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balance. Positive energy balance, signifies that the energy used in the production of the fuel is less than the energy value of the fuel produced collectively with many of the allied utilized by-products. The main predicament to the commercialization of biodiesel is its high cost due to the high price of virgin vegetable oils (Prokop, 2002). The cost of vegetable oils, which could be up to 75% of the total manufacturing cost, has led to the production costs of biodiesel becoming approximately 1.5 times higher than that for diesel (Ma and Hanna, 1999, Zhang et al., 2003). Here, the methods that permits minimization of costs of the raw material are of particular concern and can be reached by the use of mango seed oil which is an agro waste. The use of waste oils is a way that allows for the reduction in the production cost of biodiesel and thereby could aid in solving the problem of waste disposal. Every year during mango season, seeds wastes are collected in large volumes from homes, markets and processing industries to the landfills contributing to biochemical reactions which take place on landfill sites leading to the formation of methane and leachate which pollute the atmosphere and ground water (Sugumaran and Seshadri, 2010). Mangos belong to the genus *Mangifera indica* of the family Anacardiaceae. The genus *Mangifera indica* contains several species that bear edible fruit. Most of the fruit trees that are commonly known as mangos belong to the species *Mangifera indica*. The other edible *Mangifera indica* species generally have lower quality fruit and are commonly referred to as wild mangos. Mango is now cultivated throughout the tropical and subtropical world for commercial fruit production, as a garden tree, and as a shade tree for stock. Mangos are long-lived evergreen trees that can reach heights of 15-30 m. Most cultivated mango trees are between 3 and 10 m tall when fully matured, depending on the variety and the amount of pruning. Wild, non-cultivated seedling trees often reach 15 m when found in favorable climates, and they can reach 30 m in forest situations. The trees can live for over 100 years and develop trunk girths of over 4 m (Bally, 2006).

To utilize the mango seed oil in a common diesel cycle engine, without need of modifications in the engine,

it is obligatory to transesterify the vegetable oil, with the aim to lower its viscosity to a value close to that of mineral diesel oil as stated by Cono et al. (2005). Transesterification is the process of using an alcohol (e.g., methanol or ethanol) to chemically change the molecule of the raw vegetable oil into methyl or ethyl esters (biodiesel) of the vegetable oil with glycerol as a by-product in the presence of a catalyst, such as sodium hydroxide or potassium hydroxide. It has been described as the most feasible oil modification.

This work reports the production of vegetable oil from mango seeds and the use of the same oil to produce biodiesel via transesterification with methanol in the presence of sodium hydroxide as catalyst. The basic fuel and physicochemical properties of the biodiesel produced from mango seed oil was characterized through American Society for Testing and Materials (ASTM) standard tests.

2 Materials and methods

2.1 Sample collections and preparation

Mango fruit seeds were collected from waste bins in Masifa Quarters, Ogbomoso North local government area of Oyo State, Nigeria. The mango fruit seeds were sun dried for a week prior to the removal of the seeds manually from pulps so as to reduce the moisture content. It was then sun dried for two weeks until it was dry at relative humidity of 31.75% and mean daily temperature of 33.50°C. The mango seeds were cleaned and sorted by removing the immature, broken seeds and unwanted materials.

2.2 Oil extraction

The extraction of the oil was carried out from screen mango seed using Soxhlet method in accordance with the procedures of Onwuka (2005) using following materials: boiling flask, desiccators, petroleum ether and electric oven. The grounded pulp was packed into the extraction chamber of the soxhlet extractor; while the solvent (normal hexane) was poured into the round bottom flask of the extractor. The whole set-up was mounted on a heating mantle at 65°C and allowed to reflux for about eight hours. The extract was filtered (to remove impurities) and evaporated using a rotary evaporator to

isolate the free flow lipid from the solvent. The extracted oil was further evaporated in an oven at 150°C to eliminate any moisture and residue solvent that may be present. The weight of the oil produced and the residue were measured to ascertain the percentage of the oil content. The oil obtained was weighed and the percentage oil yield was calculated by Equation (1), as used by Haque et al. (2009).

$$\text{Oil yield (\%)} = \frac{W_2 - W_1}{W} \quad (1)$$

where, W = weight of mango seed used (g); W_1 = weight of beaker with glass ball (g); W_2 = weight of beaker with glass ball and oil (g); $W_2 - W_1$ = weight of oil. (g).

2.3 Transesterification procedure

A laboratory scale biodiesel processor was used to carry out the transesterification process. The reagent used was methanol. Methanol was used because of its low cost and short-chain alcohol that reacts fast. In order to convert some of the fatty acids (fatty acid value of the oil was 3.4 mg KOH g⁻¹ which was high) to ester, the oil was first pretreated using sulfuric acid as catalyst. Leung et al. (2010) wrote that pretreatment was necessary to reduce the content of free fatty acid (FFA) before base catalyzed transesterification. After, it was converted to alkaline transesterification with anhydrous methanol at a molar ratio of 6 to 1 and 3 g L⁻¹ of sodium hydroxide as catalyst. The seeds oil was heated to 110°C to remove any water vapour present and then cooled to 60°C. The processor was agitated at 600 rpm at 60°C for two hours. The mixture was then poured into a decanter and allowed to settle for 12 hours to enable the reaction to be driven to completion by allowing the mixture to separate into two layers of biodiesel and glycerol. The glycerol at the bottom was drained off by gravity and the surplus methanol in the biodiesel was removed in a flash evaporator. The biodiesel was washed in distilled and deionized water of volume ratio 3 to 1 for three times to enable the further removal of impurities. The washed biodiesel was dried by passing through anhydrous sodium sulphate (Na₂SO₄) (Schinas et al., 2009). The biodiesel produced and the glycerol was then weighed and percentage yield calculated using Equation (2) as used by Giwa et al. (2010).

biodiesel yield (%)=

$$\frac{\text{Weight of biodiesel}}{\text{Weight of biodiesel} + \text{weight of glycerol}} \quad (2)$$

2.4 Blending of biodiesel

Blending of biodiesel with conventional diesel was carried out using an in-tank method of petroleum blending. The biodiesel of mango seed oil obtained was blended with diesel obtained from fuel station at different proportions. They included 10% (B10), 20% (B20), 30% (B30) and unblended biodiesel (B100). The blends were centrifuged for homogeneity before proceeding with fuel analysis.

2.5 Determination of fuel and physiochemical properties

The fuel and physiochemical properties of the mango seed oil and biodiesel produced were determined following American Oil Chemists' Society (AOCS), ASTM, and European Norm (EN) methods. All measurements were carried out three times and the mean calculated.

2.5.1 Density and specific gravity

The sample biodiesel was cooled to 15°C using piece of ice blocks. An empty relative density bottle was first weighed, reweighed when filled with water and reweighed when filled with sample biodiesel. Density and specific gravity were estimated using the Equations (3) below:

$$\text{Specific gravity} = \frac{W_2 - W_0}{W_1 - W_0} \quad (3)$$

where, W_0 = weight of empty bottle (g); W_1 = weight of bottle (g); W_2 = weight of bottle and oil (g).

2.5.2 Kinematic viscosities

The kinematic viscosity was determined with a Herzog GmbH MP-480 that involves measuring the time for a fixed volume of the fuel to flow under gravity through a capillary at temperature of 40°C.

$$\text{Kinematic viscosity} = \text{Calibration constant (mm}^2 \text{ s}^{-1}) \times \text{mean time of flow (s)} \quad (4)$$

2.5.3 Flash point

Flash point measurements were done using Kehler Model K-16270 (Pensky-Martens Closed Flash Tester) according to method ASTM D6751.2.5.4 Higher and lower heating values.

The heating values were obtained using the oxygen bomb calorimeter (Parr Instrument Company, US.) following the ASTM D240 method.

2.5.5 Calculated cetane number

The cetane number of the biodiesel was calculated using Equation (5) (Willard, 1961).

$$CI = -420.34 + 0.016G^2 + 0.192G(\log T_{50}) + 65.01(\log T_{50})^2 - 0.0001809T_{50}^2 \quad (5)$$

where, G is the American Petroleum Institute (API) specific gravity and T_{50} is the distillation temperature at 50 vol. % fuel sample distilled and condensed in a unit of °F.

2.5.6 Acidic number

The acid number, which is the amount of KOH required to neutralize 1 g of fat and expressed as mg KOH g⁻¹, was determined by titrating with 0.01 N potassium hydroxide for the mixture of tested fuel and chemical reagents until the appearance of the color pink.

2.5.7 Carbon residue

Carbon residue was determined following standard procedure which involved heating a sample of the fuel to 500°C in nitrogen filled chamber at controlled rate to ensure that the sample cokes and does not combust. The volatile compounds formed were then flushed from the chamber with nitrogen after which the mass remaining was determined. The aim of the test was to simulate the formation of carbon deposits in the engine by the fuel.

2.5.8 Sulfated ash

Isotemp muffle furnace was used for the test. The residue was allowed to cool down and thereafter treated with sulfuric acid and heated to 750°C until oxidation of carbon was complete. The resulting ash was then cooled, retreated with sulfuric acid and heated to 750°C to constant weight. After which the percentage weight was calculated.

2.5.9 Cold flow properties

In measuring the pour point, a 45 mL sample initially at 45°C was cooled in Herzog HCP852 at specified rate and examined at interval of 3°C to check if the sample is still flowing. The cloud point was determined by a cloud point meter which comprises of a waveguide sensor of a total-reflection type, the wave guide sensor including a

wave guide having an incidence channel, an emergency channel and a detection surface all formed on a substrate, the incidence and emergency channels intersecting along the detection surface, an incidence optical fiber connected to the entrance of the incidence channel, and an emergency optical fiber connected to the exit of the emergence channel; and a cooling/heating means in contact with the waveguide sensor for cooling/heating the waveguide sensor within a desired temperature range. The cold soaked filter is a mandatory quality control test introduced by ASTM in 2008 to prevent particulate matters in biodiesel from precipitating at low temperature to clog filters and block fuel pipes thus cutting off fuel flow to the engine. A cold filter plug apparatus model MC840 with 0.8 micron filter was used to test 60 mL of each sample. It involved chilling the biodiesel to a predetermined point and then reheating to room temperature. The chilling and reheating processes formed mushy crystal-like material that could clog the fuel filter. The biodiesel is passed through the two filters and the time in seconds it takes for cold soaked biodiesel to pass through two 0.8 micron filters and the amount of matters collected are measured.

3 Results and discussion

The percentage oil yield of mango seed is 25.84%. This is 14.0% higher than, soybeans (19%-21%) (Nzikou et al., 2010) and cottonseed (15%-20%) (Shukla et al., 1992). However, the content is lower than 55% for jatropha (Pramanik, 2003), castor (55%) (Conceição et al., 2007), rubber seed (68.0%), coconut (60.0%), and castor seed (67.7%) as reported by Shukla et al. (1992). Oil content of agricultural materials provides an insight on whether it is reasonable to process oil industrially from a given seed or not. According to FAO (Akinoso and Raji, 2010), any seed containing greater than 17% of oil is considered to be an oil seed. For this reason, mango seed can be utilized for the industrial vegetable oil processing.

3.2 Characterization of the biodiesel and its blend with fossil diesel

The results obtained after characterization of the biodiesel and its blend with fossil diesel are shown in Table 1 and 2, and further discussion on the topic refers to it.

Table 1 Physicochemical properties of mango seed oil diesel and its blend with fossil diesel

Property	B100	B10	B20	B30
Density (g cm^{-3})	0.876	0.873	0.860	0.854
Specific gravity	0.874	0.871	0.870	0.868
Kinematic viscosity at 40°C (c.t.s.)	4.32	2.86	3.09	3.14
Lower heating value (MJ kg^{-1})	37.33	42.71	42.34	41.91
Higher heating value (MJ kg^{-1})	40.62	45.44	22.87	44.31
Refractive index	1.422	1.493	1.481	1.474
Carbon residue (%)	0.09	0.05	0.07	0.08
Sulfated ash	0.033	0.015	0.018	0.022

Table 2 Fuel properties of mango seed oil diesel and its blend with fossil diesel

Property	B100	B10	B20	B30
Flash point °C	145	73	78	93
Cloud point °C	7	5	5	5
Pour point °C	2	12	12	12
Calculated cetane number	59.67	45.98	48.12	47.25
Acid number (mg KOH g^{-1})	0.445	0.455	0.457	0.558

3.2.1 Density and specific gravity

It is an important parameter in relation to the weight of fuel delivered to the cylinder. B100 density of 0.876 g cm^{-3} reduced to 0.873, 0.860 and 0.854 g cm^{-3} for the B10, B20 and B30 respectively. The specific gravity of B100 and its blend with diesel all fell within the limits for biodiesel. The specific gravity was found to be 0.874, 0.871, 0.870 and 0.868 for B100, B10, B20 and B30 respectively and all the values are within the ASTM limits of biodiesel. These values must be within tolerable limits to allow optimum air to fuel ratio for complete combustion (Ramírez-Verduzeo et al., 2012) since high density biodiesel or its blends can lead to incomplete combustion and particulate matter emission. These results were indicators that biodiesel produced from mango seed oil might undergo complete combustion and produce less particulate matter. The density of the biodiesel was found to be consistent with the values of 0.876 g cm^{-3} reported by Adebayo et al. (2011) and also in agreement with 0.868 reported by Belewu et al. (2010). The specific gravity of the biodiesel was also compared favorably with those reported by Phan and Phan (2008) for waste cooked oil of 0.88 and Al-Widyan, and Al-Shyoukh (2003) for waste palm oil of 0.8737.

3.2.2 Kinematic viscosity

Kinematic viscosity is a measurement of fluid resistance to flow. It is the prime factor why biodiesel is

used as an alternative fuel instead of their vegetable oils and animal fats. Their direct use will eventually lead to operational problems such as engine deposits mainly at low temperatures when the increase in viscosity affects the fluidity of the fuel or leakage at high temperature when too thin. The kinematic viscosity measured at 40°C were found to be 4.32, 2.86, 3.09 and 3.14 cts for B100, B10, B20 and B30 respectively. The results were found to be in compliance with the biodiesel standards, ASTM D6751-02 and EN 14214. The kinematic viscosity of biodiesel with 4.8 cts was within the ASTM and EN limit of biodiesel. It was a little bit lower compared to 4.8 cts reported by Adebayo et al. (2011) and 4.7 cts reported by Samuel et al. (2013). The viscosity of biodiesel is usually higher compared to fossil diesel, inferring that biodiesel will have more lubricating effect in engines which will be an added advantage to the users, since it will reduce wear and tear in the engine.

3.2.3 Higher and lower heating values

Higher heating value (HHV) determines the appropriateness of biodiesel as an alternative to diesel fuels. Heating value is the enthalpy released after the complete combustion reaction of fuel at a constant pressure or volume. Fuels possessing higher heating values would require lower fuel flow rate than the fuel with lower heating value for the same engine power output. The higher and lower heating values for B100 was 37.33 and 40.62 MJ kg^{-1} respectively. The higher heating value for B10, B20 and B30 were 45.44, 44.87, and 44.32 MJ kg^{-1} respectively while their lower heating values were 42.71, 42.34 and 41.91 MJ kg^{-1} respectively. The heating values increased slightly with the increasing amount of diesel in the blend because diesel has higher heating values. The high heating value obtained was in agreement with 41.25 MJ kg^{-1} reported by Samuel et al. (2013) and 39.81 and 45.2 MJ kg^{-1} reported for tobacco seed oil biodiesel, and sunflower oil biodiesel by Usta (2005) and Rashid et al. (2009) respectively.

3.2.4 Carbon residue

The carbon residue test gives the indication of coking tendency of the fuel. It is the remaining part of the fuel when a sample of biodiesel has been subjected to thermal decomposition. The common cause of excessive carbon residue in biodiesel is inadequate purification process

which permits for excessive level of total glycerin soaps and other organic impurities. The carbon residue of the B100 was 0.09% while for the blends is 0.05%, 0.07%, and 0.08% for B10, B20 and B30 respectively. The value except for B10 was higher compared to 0.05% maximum standards (ASTM D4530). The result was however lower than 0.2% reported by Adebayo et al. (2011).

3.2.5 Sulfated ash

Sulfated ash is a vital test for biodiesel. It is the measure of the amount of metal contained in the fuel. The sulfated ash for B100 was 0.033 while for the blends were 0.015, 0.018 and 0.022 for B10, B20 and B30 respectively. The sulfated ashes for the four samples met the requirements for both ASTM D874 and EN ISO 3987 of 0.05 maximum. This implies that the four samples emissions from exhaust of vehicles will help reduce the pollution introduced to the atmosphere compared to that of fossil diesel.

3.2.6 Flash point

It is the lowest temperature at which fuel will ignite (flash) on application of ignition source. Flash point is not directly related to engine performance but is inversely related to fuel volatility (Ramirez-Verduzeo, 2012). From Table 2, B100 had a flash point of 145°C, the value for blends were 73°C, 78°C and 93°C for B10, B20, and B30 respectively. The obtained result for B100 was in agreement with ASTM biodiesel standard (of 130°C). Biodiesel has a high flash point which was more than 100°C, while conventional diesel fuel has a flash point of 77°C (Moser, 2000). However, the values for the blend were below the minimum for biodiesel. High flash point can prevent auto ignition and reduce fire hazards at high temperatures during transportation and storage periods. It is such a measure of flammability of fuels and a vital safety decisive factor. The result obtained of 144°C by Mukhtar et al. (2014) for ethanolysis of calabash seed oil and 139°C by Samuel et al. (2013) for restaurant waste cooking oil was in agreement with the obtained result. The result was however higher than 109°C reported by Al-widyan and Al-Shyoukh (2003), for waste palm oil.

3.2.7 Cloud and pour points

Cold temperature behavior of biodiesel is a critical quality condition, as fuel lines and filters might starve the engine of fuel due to blockage arising from frozen fuel.

The cloud and pour points of B100 were 7°C and 2°C respectively, while for the blend were 5°C and 12°C. This was in agreement with the report of Alamu et al. (2007) whom reported 6°C and 2°C for cloud and pour point of biodiesel production from Nigerian palm kernel oil. These points are all just a little bit above zero which limits their application in cold regions.

3.2.8 Calculated cetane index/number

The quality of the compression ignition of diesel fuel is frequently indicated by the cetane number, or cetane index (Lin 55% and Li, 2009). The calculated cetane index of the B100, B10, B20 and B30 were 59.67, 45.98, 48.12 and 47.25 respectively. The cetane number was above the minimum limit for biodiesel of 47 but reduced with blending since diesel has lower cetane number. The cetane index of these work fell between the cetane number biodiesel produced from soybean oil which ranges between 45.7 and 56.4 as reported by Graboski and McCormick (1998). Lin and Lin (2007) wrote that larger cetane number of a diesel fuel resulted in a longer ignition delay and duration of the combustion period, less occurrence of knocking, and lower formation of nitrogen oxides (NOx). Biodiesel has a higher cetane number of biodiesel fuel due to its oxygen content, and presents fatty acids which have very high cetane number.

3.2.9 Acid number

The acid value is simple technique for monitoring fuel quality. This is the quantity of base required to titrate a sample to a specified end point. It is a measure of FFA in biodiesel. Excessive free fatty acid in the fuel can react with any water present to form acidic solution making the biodiesel corrosive. The acid value of the biodiesel was 0.445 mg KOH g⁻¹ while the acid values of B10, B20 and B30 were 0.455, 0.457 and 0.558 mg KOH g⁻¹ respectively. The acid value increased with higher amount of diesel in the blends. This result satisfied the maximum acid value set in ASTM D6751 and EN 14214 standards. The results agreed with 0.459 mg KOH g⁻¹ reported by Samuel et al. (2013) and 0.43 mg KOH g⁻¹ reported by Phan and Phan (2008).

3.2.10 Refractive index

Reflective index value is an indicator of purity of the fuel. The refractive index of B100 was 1.422 but increased to 1.474, 1.481 and 1.493, for B30, B20 and

B10 after blending respectively. This was in agreement with the findings of Ogunsuyi (2012) who reported 1.42 for mango seed oil to biodiesel using acid and base catalysed transesterification.

4 Conclusions

Biodiesel was produced from oil extracted from *mangiferaindicavia* transesterification using sodium hydroxide (NaOH) as the catalyst. The derived biodiesel produced from *mangiferaindica* and its blend with diesel was compared to ASTM and EN standards and the result indicated that biodiesel and its blend with diesel show that most of the properties of the biodiesel are within the limits for biodiesel and can hence be used as alternative fuel for diesel engines. The results also showed that the higher the diesel in the blend the better the properties. Therefore, biodiesel production from the oil of *mangiferaindica* seed oil should be encouraged not only for creating treasure from trash but also serves as a means of relieving the environment of the discarded seeds of the fruit during the harvest season.

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