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Production and Characterization of Friendly Environmental Biodiesel from *Azanza Garkeana* (Snot Apple) Seed Using Methanol as Estrification Agent

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Abstract: The research was aimed at producing biodiesel from Azanza garkeana Seed using methanol as esterification agent. The Azanza garkeana (snot apple) seed oil has been extracted using solvent extraction. The physicochemical properties of the oil were determined to ascertain its suitability for use in biodiesel production. The results of the analyses of the oil that were obtained for saponification value, peroxide value, acid value, free fatty acid, Iodine value, specific gravity and density, are 163.81 mg/g KOH, 90.24 mg/L, 4.24 mg/g, 2.14 mg/g, 44.69 mg/g, 0.912 and 0.936 g/cm^3 respectively. This shows that it is a good raw material for biodiesel production. The production of biodiesel was carried out using the extracted oil through transesterification process by catalyst, and the analysis of the produced biodiesel showed that its pH, cloud point, pour point, flash point, Density, Specific gravity, Saponification Value, Peroxide value, Acid value, free faty acid and Iodine value are, 5.51, 2 ^{o}C, 5 ^{o}C, 168 ^{o}C, 0.811 g/cm^3, 0.861, 159.90 mg/g KOH, 44.00 mg/L, 3.92 mg/g KOH, 1.96 mg/g and 47.71 respectively. The results of the analyses of the produced biodiesel, which were found to compare very well with the standard values, indicated that the biodiesel produced using the extracted Azanza garkeana oil was of standard quality.

Key words: Biodiesel, Snot apple, Production, Transesterification

1. Introduction

As the world population increases, the energy consumption also increases. In any nation, energy is the most fundamental requirement for human existence and activities (Ribeiro *et al.*, 2011). Unfortunately, the non-renewable energy sources that contribute over 86 % of the global energy supply are depleting (Atadashi *et al.*, 2012). In addition, apart from price hike in the depleting petroleum based products, greenhouse gas emission being emitted as a result of the products

contributes significantly to climate change and ozone depletion. This problem has resulted to intense search for alternative feedstock and sustainable technology that can counter the shortcomings of non-renewable energy sources. One of the alternative energy types considered to replace the dwindling conventional transportation fuel is biodiesel (Moser, 2009).

Biodiesel, as an alternative fuel, has been currently receiving much attention owing to the limited availability of conventional petroleum diesel and environmental concerns. It can be directly used to replace petroleum diesel without modifying diesel engines since their properties, e.g., specific gravity, cetane number, viscosity, cloud point, and flash point, are similar (Simasatitkul *et al.*, 2011). It is a promising alternative or extender to conventional petroleum based diesel fuel.

Biodiesel is produced from vegetable oils or animal fats and an alcohol, through Trans-esterification reaction. Biodiesel is a renewable fuel made from any biologically based oil, and can be used to power any diesel engine. It is a mono-alkyl ester which is produced mainly by transesterification of vegetable oils or animal fats. This reaction is carried out by heating feedstock (oil or fat) and alcohol with suitable catalyst. Over time, edible oils were used but this has resulted to food crisis causing the research on biodiesel production to shift to non-edible seed oils and even the waste oils (Ibrahim *et al.*, 2016).

Therefore, it is imperative to examine the biodegradability of biodiesel fuel and their biodegradation rate in natural environment so as to have an idea of how persistent they would be when discharged into the environment. Biodiesel is derived from vegetable oil or fat via transesterification reaction. However, increasing interest and the used of petro diesel necessitate the search for other viable feedstock from the abundant and versatile renewable resource specially plant seeds. In tropical Africa, the *Azanza garckeana* (Snot Apple) fruit tree is a popular multipurpose fruit tree that is characterized by edible fruits, plant parts with medicinal uses, and products sold to local markets (Glew *et al.*, 2005). The ripe fruit is used as food additives in Sudan (Suliman *et al.*, 2012). *Azanza garckeana* fruits are sold in local markets in Botswana, Kenya, Zambia and Zimbabwe. The species is semi domesticated in Botswana, Nigeria, Zambia and Zimbabwe where local people grow the species in home gardens and crop fields.

2. Materials and Method

2.1 Materials/ Equipment

Mate Conical flask, Density bottle, Separation funnel, Stirrer, Burette, Retort stand measuring cylinder, Heating mantle, Mortar and pestle, Cotton wool, Erlenmeyer flask, Soxhlet extractor, Oven, Weighing balance, Beaker 250 ml Volumetric flask.

2.2 Chemicals and Reagents

Sodium hydroxide, Methanol, Calcium hydroxide, Hydrochloric acid, Magnesium sulfate, Ethanol, Starch indicator, Phenolphthalein indicator, Saturated potassium iodide solution, Glacial acetic acid, Chloroform, 0.2 N Sodium thiosulphate Ethanol potassium hydroxide solution (0.5M in 95% ethanol), Anhydrous chloroform, 5 % potassium iodide and 1 % starch indicator**.**

3. Preparation of reagents

3.1 Preparation of Wij's Reagent

The reagent is prepared by dissolving 2 g of iodine and 6 g of potassium iodide in 100 ml of distilled water.

3.1.1 Preparation of 0.1 M Sodium Thiosulfate Solution

A 25 g of sodium thiosulfate was weighed and dissolved in sufficient amount of water in a 1 dm^3. It was made up to the mark with distilled water.

3.1.2 Preparation of 0.5 M Aqueous KOH Solution

Potassium hydroxide (2.8g) was dissolved in about 40 ml of distilled water in a 100 ml volumetric flask and made up to mark with distilled water.

3.1.3 Preparation of 0.5 Ethanolic KOH Solution

A 2.8 g of Potassium hydroxide was weighed and dissolved in a 40 ml of ethanol in 100 cm^3 volumetric made up to the mark with ethanol.

3.1.4 Preparation of 0.5 M Hydrochloric Acid

A 4.180 cm^3 of concentrated hydrochloric acid was measured into a 100 ml volumetric flask and diluted with water to the mark.

5 % Potassium Iodide Solution

A 5 g of Potassium iodide was weighed and dissolved in 50 ml of distilled water. It was then transferred quantitatively into a 100 ml volumetric flask and made up to mark with distilled water.

30 % Potassium Iodate Solution

A 30 g of Potassium iodate was dissolved in 40 ml of distilled water in a 100 cm^3 volumetric flask and made to up to mark with distilled water.

1 % Starch Indicator

A 1 g of Starch indicator was dissolved in 50 ml of distilled water in a 100 cm^3 volumetric flask and made up to mark with distilled water.

4. Methods

4.1 Sample Collection

Snot apple seed was collected from Shan gon Local Government Area Gombe State, Nigeria. The sample was washed, air-dried, pulverized using a mortar and pestle, the powder was stored in an air tight container for subsequent experiments.

4.1.1 Oil Extraction

The oil was extracted from the Snot apple (150 g) in a Soxhlet extractor using n-hexane as the solvent until it was certified that at least 90 % of the oil is extracted. The oil was filtered to remove impurities and the solvent was removed by the use of rotary evaporator. The oil to be recovered was evaporated using an oven at a temperature of 105 ^{0}C.

4.1.2 Production of Biodiesel

Methanol (100ml) was added to 1.00 g of KOH in a conical flask with a slight heating and slow stirring until completely dissolved. The mixture was added to the oil (90 ml) with slow stirring and this was continued for about 2 hours 15 minutes after which the mixture was slowly

transferred into a separation funnel and allowed to stand overnight. The mixture was separated into two distinct layers, the glycerol layer (the lower layer) was drained off. Water was slowly added to the glycerol and swirled slowly so as not to form an emulsion and the water drained off with remaining glycerol. This process was repeated severally until the product was clear. The fatyy acid methyl ester (FAMES) was dried to obtained a pure biodiesel.

5. Fuel Qualify Parameter Test/ physical Properties Test

5.1 Pour Point (P.P) Test

The cylindrical test tube was filled with the biodiesel to a specific level (10 ml) and clamped with wooden clamp bearing a thermometer. The sample was then allowed to cool below 0 °C in an ice/salt bath. At this point it was removed and tilted on the clamp and the set up observed at intervals. The lowest temperature at which the oil was observed to flow was recorded as the pour point (Aalam and Saravanan 2016).

5.2 Cloud Point

The cylindrical test tube was filled with the biodiesel to a specific level (10 ml) and clamped with wooden clamp bearing the thermometer. The test tube was placed in the ice/salt bath and the set up inspected at intervals for cloud formation. The temperature at which a distinct cloud appeared at the bottom of the test tube was observed and recorded as the cloud point of the oil (Aalam and Saravanan 2016).

5.3 Flash Point (F.P)

A 50 ml beaker was filled to a specific level (10 ml) with the biodiesel and was heated at a slow constant rate on the hot plate. The flash point was taken at the lowest temperature when an application of the test flame caused the vapor above the sample to ignite (Aalam and Saravanan 2016).

pH

The seed oil (10 ml) was measured in to a 50 ml beaker, the electrode of the pH meter was cleaned and dried, it was then introduced into the sample and the reading was recorded (Moser, 2009).

6. **Specific gravity**

The specific gravity bottle was oven dried to remove existing moisture after which its mass (empty) was measured and recorded as W_1. It was then filled with water to its volume and the mass was recorded as W_2. The specific gravity bottle was then filled with equal volume of oil extract while its mass was measured and recorded as W_3. The specific gravity was calculated using the formula below:

$$\text{Specific gravity} = \frac{W3 - W1}{W2 - W1}$$

Where, W_1 = Mass of empty specific gravity bottle

W_2 = Mass of specific gravity bottle + water

W_3 = Mass of specific gravity bottle + Oil

7. **Density**

Specific gravity bottle was washed, rinsed with acetone and dried in an oven. The bottle was cooled to room temperature in a desicator and the weight of the empty bottle was determined on weighing balance. The weight of the bottle filled with the biodiesel was recorded. The volume of the oil in the bottle was recorded and the density was computed as follows ((Aalam and Saravanan 2016).

Density= (weigh of bottle + oil) – weigh of empty bottle / volume of oil.

8. **Characterization of the Biodiesel**

8.1 Determination of percentage yield

The initial mass of sample was weighed and recorded before extraction (after oven drying). The mass of oil extract obtained was also weighed and the percentage yield calculated using the equation below:

$$\text{Percentage Yield} = \frac{mass\ of\ oil\ extracted}{initial\ mass\ of\ sample\ used} X\ 100$$

8.2 Acid Value

The sample (5 g) was accurately weighed and placed in a 250 ml flask. 50 ml of a mixture of equal volumes of ethanol and ether, which had been neutralized by 0.5 N of potassium hydroxide, was added. The resulting mixture was heated for 10 minutes to allow for complete dissolution of the sample and then cooled. 1 ml of phenolphthalein was added as indicator while shaking the contents vigorously. The mixture was titrated with 0.5 M potassium hydroxide until a pink colour, which persisted for 15 seconds, was obtained. The entire procedure was repeated without the sample (blank). The acid value was calculated using the formula:

$$\text{Acid value} = \frac{TDX\ N\ X\ 56.1}{M}$$

Where, TD = Titre Difference = B - S

B = Titre value blank; S = Titre value with sample

N = Normality of titrating solution (KOH used herein)

M = Mass of sample (g)

8.3 Peroxide value

The sample (5 g) was accurately weighed and placed in a 250 ml flask. 30 ml of glacial acetic acid-chloroform solution was added while swirling the flask and carefully warming the mixture with an electric heating mantle until the sample was completely dissolved. 0.5 ml of saturated potassium iodide solution was added and the contents swirled for exactly I minute. 30 ml of distilled water was then added and the contents shaken vigorously to liberate iodine from the chloroform layer.1 ml of starch solution was added as indicator. The resulting mixture was titrated with 0. I M sodium thiosulphate until the blue-gray colour disappeared in the aqueous layer. The entire procedure was repeated without the sample (blank). The peroxide value was calculated using the formula.

$$\text{Peroxide Value} = \frac{TDX\ N\ X\ 1000}{M}$$

Where, TD = Titre Difference = B - S

B = Titre value blank; S = Titre value with sample

N = Normality of titrating solution (KOH used herein)

M = Mass of sample (g)

8.3.1 Saponification value

The sample (2g) was accurately weighed and placed in a 250 ml flask. 25 ml of a mixture of equal volumes of ethanol and potassium hydroxide was added. The mixture was heated in a water bath (coupled to a reflux condenser from the soxhlet extractor) for 30 minutes while being stirred continuously. 1 ml of phenolphthalein was added as indicator. The resulting mixture was titrated with 0.5 M hydrochloric acid. The entire procedure was repeated without the sample (blank). The saponification value will be calculated using the formula given.

$$\frac{S - BXMX56.1}{Wiegh\ of\ Sample}$$

Where, S = sample titre value (mL), B = blank titre value (mL), M = molarity of the HCl

8.3.2 Iodine value

The sample (2 g) was accurately weighed and placed in a 250 ml flask. 20 ml of chloroform was added to the sample. 25 ml of Wijis reagent was added with the aid of a pipette. The resulting mixture was stirred and stored in a dark place at 25 ^{0}C for 30 minutes. I ml of 30 % potassium iodide was then be added to the mixture as well as 100ml of distilled water. The mixture was titrated with 0. I M sodium thiosulphate until the yellow colour almost disappeared. 1 ml of starch solution was then added and the mixture was titrated further until the blue starch-iodine colour disappeared. The entire procedure was repeated without the sample (blank). The Iodine value was calculated using the formula below:

Iodine Value = $\frac{TD\ X\ 1.269}{M}$

Where, TD = Titre Difference = B - S

B = Titre value blank; S = Titre value with sample

N = Normality of titrating solution (KOH used herein)

M = Mass of sample (g)

8.3.3 Free fatty acid (FFA)

The free fatty acid value is usually regarded as half the acid value of the oil extract

9. Result and Discussion

9.1 Result

Azanza garckeana oil was extracted from Azanza garckeana (snot apple) seed using soxhlex apparatus and biodiesel was produced from the oil using methanol as esterification agent. The physiochemical properties of the oil and biodiesel are presented in tables 1 and 2 respectively while the fuel quality parameters are presented in table 3.

Table 1: Physicochemical properties of Snot Apple Seed Oil

Properties	**SASO**
Colour	Pale Yellow
Odour	odour less
Density (g/cm^3)	0.936
Specific Gravity	0.912
Saponification Value (mg/g KOH)	163.81
Peroxide Value (mg/L)	90.24
Acid Value (mg/g KOH)	4.24
Free fatty acid(FFA)	2.14
Iodine Value (mg/g)	44.69

Key: SASO= Snot Apple Seed oil

Table 2: Physicochemical Properties of Biodiesel and ASTM Standard

Properties	Biodiesel	ASTM Standard
Colour	Pale Brown	Pale Brown
Odour	Odour less	Odour less
Density (g/cm^3)	0.881	0.900-1.00
Specific gravity	0.861	0.880
Saponification Value (mg/g KOH)	159.90	194.72
Peroxide value (mg/L)	44.00	
Acid Value (mg/g KOH)	3.92	0.1-0.2
FFA (mg/g)	1.96	0.5-1.5
Iodine Value (mg/g)	47.71	- -

Key ASTM: American Standard method of testing materials

Table 3: Fuel Quality Parameters of the Biodiesel Produced and ASTM Standard

Fuel Property	Biodiesel	ASTM Standard
% Yield	66.25	-

pH	5.51	5.7-6.2
Flash Point (^{0}C)	168	100-170
Cloud Point (^{0}C)	2	-3-15
Pour Point (^{0}C)	5	-5-10

Key ASTM= American Standard for Testing Material

10. Discussion

The iodine value in table 1 was found to be (44.69 mg/g) indicates that the oil is a non drying type with a very low degree of unsaturation. Oils are classified based on their iodine value as nondrying oils (I.V less than 100), drying (I.V. 130 and above) and middle drying oils (I.V. between 100 and 130). Based on research, the more unsaturated, the higher the iodine value and the low iodine value of the oil is highly advantageous because the oil would be stable. (Jauro and Haruna 2011). The result was similar to reports (Berbeker, 2013; Mohamed and Mohammed, 2018). Oils with iodine values lower than 100 are non-drying, while those having values range between 100 -130 are semi-drying and those with values above 130 are termed drying oils (Jock, 2011).

The peroxide value of an oil or fat is used as a measure of the extent to which rancidity reactions have occurred in the oil and fat during storage (Giwa *et al.,* 2014). The peroxide value of the seed oil in table 1 was found to be 90.24 mg/L, which is lower than 100 mg/L which may be due to the freshness of the seed. This shows that the oils would not easily go rancid when stored properly and show a good potential for production of biodiesel. The results of this study agree with similar reports by (Yau *et al.,* 2020) who reported 87.3 mg/L and (Mohammed *et al.,* 2017), but far lower than reported by (Barbeker, 2013) and (Manji *et al.,* 2013) who respectively reported 63.5 and 56.6 mg/L).

The acid value is used as acid present in a chemical substance. The acid value of the seed oil in table 1 was found to be 4.2 mg/g KOH and it has free fatty acid composition of 2.14 mg/g which is not within the acceptable range (0.1 by the ASTM and that of European standard 0.1-0.5). The result reveals that the acid value is not good enough for seed oil to serve as a good feedstock for the production of biodiesel. Similarly, (Yau *et al.,* 2020) reported that the acid value of

Balanite aegytica was 3.06 mgKOH/g and observed in his study is just below the FAO/WHO standard of 4 mgKOH/g, but shows that the oil is stable (Haftu, 2015). However, the results for the acid value and the free fatty acids obtained in this study are in agreement with similar findings by (Manji *et al.*, 2913) and (Jock, 2011) but lower than that research by (Ogala *et al.*, 2018).

From table 1 the saponification value of the seed oil was found to be 163.81 mg/g KOH which is lower than that of ASTM (212. 60 mg/g KOH). Majority of oils used in biodiesel production, their saponification number is within the range of 130 to 193 meq/kg (Jauro and Momoh 2011). This suggest that the oil is suitable for use in biodiesel production. Moreover, [14] reported that saponification value was found to be 198 mgKOH/g for *Balanites aegyptica* kernel oil analyzed, and this value is within the FAO/WHO range of 195–205 mgKOH/g for edible oils.

The Iodine value of the biodiesel in table 2 was found to be 47.71 mg/g indicate that the biodiesel is a non-drying type with a very low degree of unsaturation. It is shown that the more unsaturated, the higher the iodine value and the more prone the oil to rancidity by oxidation. The low iodine number of the oil is highly advantageous because the oil would be stable to polymerization and/or oxidation. Moreover, (Mohammed *et al.*, 2017) reported that saponification value of Biodiesel produce from oil *Balanie aegytica* was found to be 17 mg/g, and this value is far below with result shows in table 2.

The peroxide value of the biodiesel is used as a measurement of the extent to which rancidity reaction have occurred during storage in a vehicle engine. The peroxide value of the biodiesel in table 2 was found to be 44.00 mg/L which is lower than that of ASTM Standard for biodiesel (40-100 mg/L), the peroxide value is within the range of the standard and this result was closed to that of (Jauro and Momoh 2011) reported that the peroxide value of biodiesel produce from Calabash seed oil was 38.00 mg/L.

The saponification value of Biodiesel in table 2 was found to be 159.90 mg/g KOH which is lower than that of ASTM Standard (194.72 mg/g KOH). For a biodiesel, their saponification values are within the range of 130 to 194 mg/g KOH. This shows that the biodiesel is suitable for use and (Jauro and Momoh 2011) produce biodiesel from calabash seed oil with saponification number of 126 mg/g KOH which lower that the biodiesel reported in table 2 above.

The acid number is used to quantify the amount of acid present in a chemical substance. Acid value should not be more than 1.50 mg/g since the FFA produced may corrode automotive parts. The acid value of the biodiesel in table 2 was found to be 3.92 mg/g which is higher than that of acceptable limits (0.1-0.2 mg/g) which may lead to the corrosion of engine vehicle. Similarly, (Jauro and Momoh 2011) reported that the acid value of biodiesel produce from calabash seed oil was 0.28 mgKOH/g and observed in his study is just above ASTM standard of 0.1-0.2 mgKOH/g, but still shows that the acid value of the biodiesel is very close.

Moreover, the specific gravity of the biodiesel in table 2 was found to be 0.861 which is compared with that of ASTM Standard which is closed to the standard (0.880) indicating that the biodiesel is good. Apart from that, the density of the biodiesel as, given in Table 2, was found to be 0.881 g/cm^3 close to that of ASTM Standard, which is (0.9-1.00 g/cm^3), indicating that the produced biodiesel would be a very good alternative to petrodiesel (Jauro and Momoh 2011).

The flash point is used in assessing the overall flammability of a material. Higher flash point indicate material that is less likely to ignites accidentally auro and Momoh 2011). ASTM standard requires a minimum 100-170 °C. the flash point of the biodiesel was found to be (168°C) and is within the ASTM standard (Table 3). Biodiesel tends to freeze at higher temperatures than petrodiesel. This is one of the major factors affecting the use of biodiesel. The cloud point (CP) is the temperature of the fuel at which small solid crystals can be observed as the fuel cools. The observed cloud point of biodiesel in table 3 was found to be 2 °C which has been found to be within ASTM standard (-3-15).

Table 3 shows the properties of the produced biodiesel, and it was deduced from the results that the low pour point (5 ^{0}C) of the biodiesel could make it usable for engines at cold regions. Besides, the other parameters determined for the biodiesel were found to compare well with the standards ASTM (-5-10).

11. Conclusion

The physicochemical properties of the oil determined were within the accepted limits for biodiesel production except the acid values that were high.

The results obtained from the preliminary investigation carried out in this work revealed that Snot apple seed oil was an economically viable oil source because its oil content was found to be high

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