Extraction and characterization of biodiesel from *Borassus aethiopum* and *Cassia sieberiana*

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Biodiesel was successfully produced from *Borassus aethiopum* and *Cassia sieberiana* seeds using the trans-esterification method. The biodiesels produced were characterized by physico-chemical properties of acid value, iodine value, specific gravity, refractive index, viscosity, density, flash point, cloud point and pour point. Results showed that the biodiesels produced have values which compared well with the European Biodiesel Standard. The free fatty acid from *B. aethiopum* oil and *C. sieberiana* oil are 2.68 and 13.97%, respectively, while the iodine values are 47.93 gl²/100 g and 48 gl²/100 g, respectively. Also, their saponification values are 201.69 mg KOH/g and 208.56 mg KOH/g, respectively, while their acid values are 5.38 and 28.04, respectively. The calorific value for *B. aethiopum* biodiesel and *C. sieberiana* biodiesel are 42568 and 41427 KJ/kg, respectively, while their fire point are 90 and 50°C, respectively.

**Key words:** Biodiesel, *Borassus aethiopum*, *Cassia sieberiana*, trans-esterification.

**INTRODUCTION**

Currently, due to gradual depletion of world petroleum reserves and the impact of environmental pollution due to increasing exhaust emissions, there is an urgent need to develop alternative sources of energy such as biodiesel fuel. Biodiesel (vegetable oil) is a promising alternative because it has several advantages such as renewability, environmentally friendly and ease production in rural areas where there is an acute need for modern forms of energy (Stavros and John, 2002).

Biodiesel is a renewable clean-burning diesel made from a diverse mixture of biomass materials. Solvent extraction is a relatively easier method that has been widely used for lipid extraction from plant seeds and considered economical for commercial scale systems (Renil and Ben, 2015). Biodiesel can be used alone or blended with petrol diesel in any proportion. Biodiesel blends can also be used as heating oil. It is meant to be used in standard diesel engines. Biodiesel has different solvent properties than petrol diesel.

Trans-esterification is the term used to describe the chemical process of biodiesel production. The oil contains concentrated oleic acid which has the potential to be used as biodiesel (Pranab, 2011; Patrick, 1998; Azad et al., 2015). Several processes for biodiesel fuel production have been developed, among which transesterification using alkali-catalyst gives high levels of conversion by triglycerides to their corresponding methyl esters in short reaction time. This process has therefore...
been widely utilized for biodiesel production in many countries. Using base catalyzed transesterification to produce biodiesel is the most economical process, as it requires only low temperatures and pressures. This process produces up to 98% production yield. A successful trans-esterification reaction is signified by the separation of the ester and glycerol layers after the reaction time. The heavier co-product, glycerol settles out and may be sold as is or it may be purified for use in other industries, like cosmetics (Emil et al., 2009; McCormick, 2006).

Chemical equation (Equation 1) of transesterification (Emil et al., 2009) shows the chemical process for methyl ester biodiesel. The alcohol must be added in excess to derive the reaction towards the right and ensure complete conversion because the reaction between the fat/oil and the alcohol is a reversible one (Antony et al., 2011; Meena and Amarendra, 2010).

\[
\text{CH}_2\text{O} \xrightarrow{\text{Catalyst}} \text{CH} = \text{O} \text{C} \text{R} + \text{CH}_2\text{OH} \xrightarrow{\text{OH}} 3\text{CH}_2\text{O} \text{C} \text{R} \xrightarrow{\text{OH}} \text{CH}_2\text{OH} \text{Glycerol} \]

(1)

\[
\text{CH}_2\text{O} \xrightarrow{\text{Catalyst}} \text{CH} = \text{O} \text{C} \text{R} + \text{CH}_2\text{OH} \xrightarrow{\text{OH}} 3\text{CH}_2\text{O} \text{C} \text{R} \xrightarrow{\text{OH}} \text{CH}_2\text{OH} \text{Glycerol} \]

Borassus aethiopum (Figure 1), commonly known as African fan palm, is a species of Borassus palm from Africa. It grows swelling, solitary trunks to 25 m in height and 1 m in diameter at the base. The leaves are armed with spines with a spherical crown shaft of about 7 m. The fruit is yellow in color and dark brown when dried. In Nigeria, B. aethiopum can be mostly found in the east, north and some parts of the west. The fruit is edible and the wood is used for construction (it is termite proof) (Sanjay, 2013).

Cassia sieberiana seeds and plant (Figure 2) commonly known as drumstick, is a tree in the fabaceae family also native to Africa. It ranges from 10 to 20 m in height. The color of the bark (the outermost part of the tree or the covering) ranges from grey to black. The lenticels are horizontal and reddish. The plant has both flowers and fruit. It has bright yellow flowers during the dry season which are arranged mostly upright. The fruit ranges from a dark brown to black color. The fruit is indehiscent (that is, it stays attached to the tree for an extended period of time) (Sanjay, 2013; Nisha and Gupta, 2012).

B. aethiopum and C. sieberiana tree/seed are biodegradable and non-toxic, have low emission profiles and so are environmentally beneficial (Garba et al., 2015). They are readily available and fruits almost all year round. This makes it possible to produce the fuel at any time of the year. Both plants are drought and fire resistant. Palm biodiesel is an oxygenated fuel suitable for use in diesel engines to enhance combustion efficiency and decrease polycyclic aromatic hydrocarbons (PAH). Fuels from both plants reduce the amount of particle matter in automotive diesel engine (Gerald and Makeme, 2013; Knothe, 2001). The by-products of both can be sold to other industries such as cosmetics and pharmacy (Gerald and Makeme, 2013; Raul, 2001).

In this work, we aim to extract biodiesel from B. aethiopum and C. sieberiana using traditional solvents; compare the amount and quality of biodiesel obtained.
from *B. aethiopum* and *C. sieberiana*; characterize the produce for physical properties and compare these properties with that of standard biodiesel.

**MATERIALS AND METHODS**

**Preparation of seed**

The seeds of the samples were collected from the plants, sundried, crushed and then ground into powder form.

**Extraction of oil**

The manual method of extraction was employed in the extraction of oil from the two samples. 2.6 and 2.4 kg weight of each sample (powdered form) of *B. aethiopum* and *C. sieberiana*, respectively were poured in a transparent bucket. An industrial hexane was added to each sample in turn (Figure 3) and mixed for about 1 h to aid extraction of oil from the samples. The mixture was allowed to settle and the liquid part of it which contains the extracted oil and the hexane was then filtered carefully into a bottle to prevent evaporation.
Hexane extraction through soxhlet

The filtrate was passed through the soxhlet extractor so that the oil and the hexane will be separated. After the separation, the oil samples were taken to the oven for drying (Figure 4).

Reagents used

Materials used are methanol, petroleum ether, potassium hydroxide, sodium hydroxide pellets, industrial hexane and anhydrous sodium sulphate, diethyl ether, phenolphthalein, sodium-thiosulphate, toluene, sodium chloride solution, distilled water, carbon tetrachloride, potassium iodide, glacial acid, HCL solution all of analytical grade.

Equipment/apparatus used

Equipment/apparatus used are measuring cylinder (for measuring samples), constant temperature magnetic stirrer (for stirring samples at constant temperature), retort stand, separating flask (for separating mixtures of different densities), beaker, pH paper (for testing pH value), pipette, glass wood, adiabatic bomb calorimeter (for checking calorific value), rotary evaporator, viscometer, oven, thermometer, soxhlet extractor, vacuum pump, density bottle, infrared spectrophotometer, reflux denser, glass column, refractor.

Purification of oil extracted from *B. aethiopum* and *C. Sieberiana*

Degumming of oil

Phospholipids, fatty acids, sterols and other impurities contained in the extracted oil were removed in order to improve the physical stability of the oil and also facilitate further refining, through the process of degumming. This was carried out as follows: 20.1 and 10.5 ml of water were heated to 70°C, 100 and 50 ml of *B. aethiopum* and *C. sieberiana* oil were also measured and heated at the same temperature. The 20.1 and 10.5 ml of water were then added to *B. aethiopum* and *C. sieberiana* respectively in drop wise and continuously stirred for 30 min using the constant temperature magnetic stirrer, maintaining the temperature of 70°C. The mixtures were then put in a separating flask and allowed to stay under gravity for 24 h. Two layers were formed, the lower layer which contains gum (phospholipids, sterol and other impurities) was run off and the upper layer which contains the oil was collected for further refining (Figure 5).

Alkali refining of oil

41 and 11 ml of oil from *B. aethiopum* and *C. sieberiana* were measured and heated to 80°C. 310 and 77 ml of 0.8 M KOH were added into the heating oil of *B. aethiopum* and *C. sieberiana* respectively in drop wise and stirred at a constant temperature of 80°C for 5 min. The mixtures were poured into the separating flask and left for 72 h (unlike other products which usually separate after 24 h, the two samples separated slowly) (Pranab, 2011; McCormick, 2006). The mixtures separated into three layers. The bottom layer was the aqueous layer, the middle layer was the soapy layer and the upper layer was the oil. The middle and the bottom layer were run off and the upper layer was collected (Figure 6).

Purification of alkali refined oil

Petroleum ether was added to the oil and it was filtered and poured in separating flask. Distilled water heated to 70°C was used to wash the oil until the aqueous layer became clear and neutral to litmus paper. Sodium sulphate was then added to the washed oil to remove traces of water. Petroleum ether, an organic solvent, was removed from the samples through distillation.
Trans-esterification of oil from *B. aethiopum* and *C. sieberiana*

0.7 g of NaOH was dissolved in 200 ml of methanol in a conical flask with a stirrer. 60 ml of *B. aethiopum* oil and 20 ml of *C. sieberiana* oil was then added to 180 and 60 ml of the methanol solution, respectively, and stirred at a constant temperature of 60°C for 1 h. Some of the water that was not removed during the washing of the oil by the sodium sulphate was evaporated during the heating and stirring. The mixture was then put in a separating flask and allowed to stay for 24 h. Two layers were formed, the lower layer which consist of glycerin, excess alcohol, catalyst, impurities and traces of unreacted oil, the upper layer consist of biodiesel, alcohol and traces of soap. Oil methyl esters of the two samples were formed during the trans-esterification (Farooq and Umer, 2007). The biodiesel was then washed with hot water to remove unreacted alcohol. It was then allowed to stay under gravity for 24 h.
The biodiesel was collected and taken for characterization.

**Biodiesel conversion and analysis: Proximate analysis**

It is very important to check the physical properties of vegetable oils after trans-esterification. This is to check whether or not it has met the standard before usage. The biodiesel produced from the two samples were analyzed in the laboratory and compared with the European Biodiesel Standard.

The analyzed properties were specific gravity: specific gravity can be seen as a ratio of the density of a substance to the density of a reference substance. It is important to specify the temperature and pressure of the sample and the reference when obtaining specific gravity (Antony et al., 2011; Singhal and Rahman, 1994). Mathematically, specific gravity is given as:

\[ \frac{\text{density of sample}}{\text{density of reference}} \]

**Viscosity**

It is resistance of a liquid to flow. It is also known as friction in liquids. Kinematic viscosity is the ratio of the dynamic viscosity to the fluid density, that is,

\[ V = \frac{u}{\rho} \]

where \( V \) is velocity of liquid, \( u \) is viscosity constant and \( \rho \) is density.

**Refractive index**

This is the ratio of speed of light in vacuum to the speed of light in a substance, that is,

\[ N = \frac{C}{V} \]

Where \( C \) is the speed of light in vacuum and \( V \) is the velocity of light in the medium (Pranab, 2011; Mathew and Saleh, 2006).

**Saponification value**

It is the number of milligrams of potassium hydroxide required to saponify 1 g of fat under specific conditions. It is the measure of the average molecular weight of all fatty acids present in a substance (Ajeet et al., 2014).

**Flashpoint**

This is the lowest temperature at which a given liquid will ignite.

**Cloud point**

This is the temperature at which dissolved solids are no longer soluble, thereby precipitating as a second phase giving the fluid a cloudy appearance.

**Pour point**

It is the temperature at which liquid becomes semi-solid and loses its flow characteristics.

**Calorific value**

This is also known as the heating value or energy value of a substance. It is the amount of heat released during the combustion of a specific amount of substance. It is measured in (kJ/g) (Ajeet et al., 2014; Monyem and Gerpen, 2001).

**Percentage of free fatty acid**

This is given by:

\[ \frac{T \times M \times MM}{MASS} \]

Where \( T \) is the titre value, \( M \) is molarity and \( MM \) is the molar mass of the oil.

**Fuel properties of B. aethiopum oil, biodiesel and petrol diesel**

Table 2 shows the fuel properties (physico-chemical properties) of B. aethiopum oil, B. aethiopum biodiesel, petrol diesel and biodiesel standard EN 14214.

**Fuel properties of C. sieberiana oil, biodiesel and fossil fuel**

Table 2 shows the fuel properties of C. sieberiana oil, biodiesel, petrol diesel and biodiesel standard EN 14214 (Pranab, 2011).

**RESULTS AND DISCUSSION**

**Comparison of biodiesel extracted from B. aethiopum and C. sieberiana with commercial biodiesel**

Biodiesel obtained from B. aethiopum was more than the one obtained from C. sieberiana. Though they have similar fuel characteristics, C. sieberiana tends to have fuel properties more closely to that of the Biodiesel Standard EN 14214 in some ways, and B. aethiopum has closer values to that of the biodiesel standard EN 14214 in some ways (Rajagopal and Zilberman, 2007). For example, from Tables 1 and 2, it can be seen that the biodiesel standard for viscosity was 3.5-5.0, C. sieberiana biodiesel falls within that range (4.9) while biodiesel from B. aethiopum slightly passes the range (5.1). It can also be seen that B. aethiopum has closer value of specific gravity (0.906) to the biodiesel standard (0.86-9.0) than C. sieberiana (1.1) (Aliyu et al., 2013). Taking a look at the cloud point, it was observed that both B. aethiopum and C. sieberiana met the biodiesel standard. The calorific value of B. aethiopum was 42568 KJ/kg which was closer to the biodiesel standard than that of C. sieberiana which was 41427 KJ/kg. Figure 7 shows the comparison of the specific gravity of the various samples. It was observed that C. sieberiana has the higher viscosity, while B. aethiopum and the biodiesel standard have almost the same value (Gerald and Makeme, 2013). Figure 8 was a chart of viscosity in which B. aethiopum oil shows higher viscosity than the rest. The viscosity of B. aethiopum
### Table 1. Physico-chemical properties of *B. aethiopum* oil.

| Property                        | *B. aethiopum* oil | *B. aethiopum* biodiesel | Petrol diesel | Biodiesel standard EN14214 |
|---------------------------------|--------------------|--------------------------|---------------|-----------------------------|
| Specific gravity (kgm⁻³)        | 0.899              | 0.906                    | 0.850         | 0.86-0.90                   |
| Viscosity (mm²s⁻¹)              | 5.200              | 5.100                    | 2.600         | 3.5-5.0                     |
| Refractive index                | 1.475              | 1.343                    | 1.370         | 1.330                       |
| Acid value (MgKOHg⁻¹)           | 5.300              | N/A                      | >3.500        | >2.000                      |
| Saponification value            | 201.690            | N/A                      | -             | -                           |
| Pour point (°C)                 | -                  | -5                       | -20           | -                           |
| Cloud point (°C)                | -                  | 0                        | -12           | -                           |
| Flash point (°C)                | -                  | 150                      | >40           | >120                        |
| Calorific value (KJkg⁻¹)        | -                  | 42,568                   | 44,800        | -                           |
| Free fatty acid                 | 2.680              | -                        | -             | -                           |
| Fire point (°C)                 | -                  | 50                       | -             | -                           |
| Iodine value (g1/100 g)         | 47.930             | -                        | -             | -                           |

### Table 2. Physico-chemical properties of *C. sieberiana* oil.

| Property                        | *Cassia sieberiana* oil | *Cassia sieberiana* biodiesel | Petrol diesel | Biodiesel standard EN 14214 |
|---------------------------------|--------------------------|-------------------------------|---------------|-----------------------------|
| Specific gravity (kgm⁻³)        | 0.882                    | 1.150                         | 0.850         | 0.86-0.90                   |
| Viscosity (mm²s⁻¹)              | 4.951                    | 4.910                         | 2.600         | 3.5-5.0                     |
| Refractive index                | 1.460                    | 1.360                         | 1.370         | 3.330                       |
| Acid value (MgKOHg⁻¹)           | 2.800                    | -                             | >3.500        | >2.000                      |
| Saponification value            | 208.560                  | -                             | -             | -                           |
| Pour point (°C)                 | -                        | -6.000                        | -20.000       | -                           |
| Cloud point (°C)                | -                        | -4.000                        | -12.000       | -                           |
| Flash point (°C)                | -                        | 190.000                       | >40.000       | >120.000                    |
| Calorific value (KJkg⁻¹)        | -                        | 41,427                        | 44,800        | -                           |
| Free fatty acid                 | 13.970                   | -                             | -             | -                           |
| Fire point (°C)                 | -                        | 90                            | -             | -                           |
| Iodine value (g1₂/100g)         | 48.380                   | -                             | -             | -                           |

**Figure 7.** Specific gravity of *Borassus aethiopum* and *Cassia sieberiana* oil, biodiesel with biodiesel standard.
biodiesel and *C. sieberiana* biodiesel has almost the same value with that of biodiesel standard.

Figure 9 shows the refractive index of the different oil. It could be observed that refractive index of the oil (*B. aethiopum* and *C. sieberiana*) was higher than that of the biodiesels standard. *C. sieberiana* biodiesel has the highest refractive index among the biodiesels.

Figure 10 was a chart of flash point in which *C. sieberiana* biodiesel has the highest value, followed by *B. aethiopum* biodiesel, biodiesel standard and petrol diesel, respectively.

**Conclusions**

Biodiesel was successfully produced from *B. aethiopum* and *C. sieberiana* seeds using the trans-esterification method. The biodiesels produced were characterized for physical-chemical properties of acid value, iodine value, specific gravity, refractive index, viscosity, density, flash point, cloud point and pour point. Results showed that biodiesel obtained from *B. aethiopum* and *C. sieberiana* seeds compared well with the biodiesel standard EN 14214. Results also show that alkaline catalyzed
transesterification is a promising area of research and has potential for the production of biodiesel in large scale. The study also shows that *B. aethiopum* produced more oil than *C. sieberiana*. 200 g of oil was extracted from 2.6 kg sample of *B. aethiopum*, whereas 105 g of oil was extracted from 2.6 kg sample of *C. sieberiana*. The produced biodiesel can best be used when blended with petrol diesel in any proportion due to the slight difference in the biodiesel standard range.

**Conflict of Interests**

The author have not declared any conflict of interests.

**REFERENCES**

Ajeet P, Sandeep B, Jagvijay S, Priyanka T (2014). Biodegradation potential of petroleum hydrocarbons by bacteria and mixed bacterial consortium isolated from contaminated sites. Turkish J. Eng. Environ. sci. 38:41-50.

Aliyu AO, Nwaedozie JM, Ahmed A (2013). Quality parameters of biodiesel produced from locally sourced Moringaoleifera and CITrulluscolocynthis L. Seeds found in Kaduna, Nigeria. Int. res. J. pure appl. Chem. 3:377-390.

Antony SR, Robinson DSS, Lindon CRL (2011). Biodiesel production from jatropha oil and its characterization. Res. J. chem. Sci. 1:81-87.

Azad Ak, Rasul MG, Islam R (2015). Propect of Moringa Seed Oil as a Sustainable Biodiesel Fuel in Australia: A Review. Procedia Eng. 105:601-606.

Emil A, Zahira Y, Siti KK, Manal I, Jumat S (2009). Characteristics and composition of JatrophaCurcas oil seed from Malaysia and its potential as biodiesel feedstock. Euro. J. Sci. Res. 29(3):396-403.

Farooq A, Umer R (2007). Physico-chemical characteristics of Moringaoleifera seeds and seed oil from a wild provenance of Pakistan. Pak. J. Bot. 39:1443-1453.

Garba AA, Medugu DW, Gwaski PA, Amusat RO (2015). Extration and characterization of Moringa oleifera seed oil. Appl. Res. J. 9:473-477.

Gerald K, Makeme M (2013). Influence of fatty acid profiles during supercritical transesterification of conventional and non-conventional feedstocks. Am. J. Anal. Chem. 4:469-475.

Knothe G (2001). Historical perspectives on vegetable oil-based diesel fuels. INFORM 12:103-1107.

Mathew KB, Saleh B (2006). Selected physio-chemical properties of petrol diesel. Int. J. Food Properties 9(4):701-713. DOI: 10.1080/10942910600575641

McCormick RL (2006). Biodiesel Handling and Use Guide, third edition. pp. 12-18.

Meena M, Amarendra NM (2010). Jatropha: The Biodiesel plant biology, tissue culture and genetic transformation. Int. J. Pure Appl. Sci. Technol. 1:11-24.

Monyem A, Gerpen J (2001). The effect of biodiesel oxidation on engine performance and emissions. Biomass Bioenergy 20:317-325.

Nisha SP, Gupta CV (2012). Nutrient content, mineral content and antioxidant activity cassia siberiana. Res. J. med. Plants. pp.253-259.

Patrick D (1998). On the constitution of stearin. Quart. J. Chem. Soc. Lond. 5:303.

Pranab KB (2011). Biodiesel from seeds of Jatropha found in Assam, India. Int. J. Energy Inf. Commun. 2:3-66.

Rajagopal D, Zilberman D (2007). Review of environmental, Economic and policy aspect of biofuels. Policy research working paper 4341.

Raul FC (2001). Trans-esterification of Bio-oil to Biodiesel. Biomass Energy. pp. 344-345.

Renil A, Ben S (2015). Solvent extraction and characterization of neutral lipids in Oocystis sp. Bioenergy and Biofuels. doi.org/10.3389/fenrg.2014.00064.

Sanjay B (2013). Transesestertification with heterogeneous catalyst in production of biodiesel. J. Chem. Pharmaceut. Res. 5:1-7.

Singhal KC, Rahman A (1994). “Plant oil as Diesel-Engine fuel”. National Conference on I.C Engines and Combustions. pp. 55-60.

Stavros L, John T (2002). Characterization of *Moringa oleifera* seed oil Variety “Perlyakulam 1”. J. Food Composition Anal. 15:65-77.