Physiochemical properties of biodiesel produced from ogbono (*Irvingia gabonesis*) seed oil

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Abstract

Biodiesel is a promising alternative fuel and has gained significant attention due to the predicted depletion of conventional fossil fuels and environmental concerns. This study aims to produce biodiesel from ogbono seed oil (using 98 ml methanol and 2g potassium hydroxide (KOH) as a catalyst) via transesterification process and to determine the physiochemical properties of the biodiesel produced. The physiochemical properties of the feedstock (extracted ogbono seed oil) were also determined before the transesterification process. The physiochemical properties of the produced biodiesel showed that it has a density of 0.5±0.00 g/cm³, pour point of 2.0±0°C, saponification value of 58.90±0.06 mg KOH/g, ester value of 98.0±0.5% (m/m), iodine value of 26.64±0.15gI₂/100g, acid value of 0.28±0.05 mgKOH/g, moisture value of 0.0006 ±0.0% and trace amounts of ash content. The results of the physiochemical properties of the produced biodiesel agree with ASTM-D6751 and EN 14214 standard. Thus, it was concluded that ogbono seed oil is an excellent feedstock for biodiesel production via base catalyzed transesterification process.

Keywords: Biodiesel, Ogbono, Extraction, Transesterification, Physiochemical, *Irvingia gabonesis*
INTRODUCTION

The biggest challenge faced by modern industrial society today is the decline and exhaustion of the fossil power resources. The primary sources of energy that power our civilization today are from fossil fuels (Singh et al., 2020). The International Energy Agency has forecasted that the world's overall energy necessities will rise by half in the next 25 years. On the other hand, BP's Energy Outlook to 2035, has forecasted that world energy consumption will rise by 34% between 2014 and 2035. It is anticipated that global energy demand will keep increasing because of increasing population growth, ever-increasing urbanization and better living standards (Khan et al., 2014, BP Energy Outlook, 2016, Keneni and Marchetti, 2017). Therefore, persistent use of petroleum-sourced fuels is now extensively identified as unsustainable due to depleting supplies and increasing demand (Manzanera et al., 2008, Keneni and Marchetti, 2017). Sustainability is a key precept in natural resource control, and it has turned out to be more and more apparent that persisted dependence on fossil fuel energy resources is unsustainable, because of depleting resources and the contribution of those fuels to environmental and health problems. Thus, alternative and renewable energy sources such as the transesterification process (final product similar to petroleum-based diesel fuel) is increasingly researched as a means of producing an environmentally ideal source of energy (Narwal et al., 2015) and to meet the anticipated large energy demand and also handle the fast diminution of fossil fuels (Khan et al., 2014).

Biodiesel is an aggregate of fatty acid alkyl esters acquired from transesterification (ester exchange) of vegetable oils and animal fats (Mahlia et al., 2020). These lipid feedstocks are composed through 90–98% (weight) of triglycerides and small quantity of mono and diglycerides, free fatty acids (1–5%), and residual quantities of phospholipids, phosphatides, carotenoids, tocopherols, sulphur compounds, and traces of water. In the transesterification reaction, a homogeneous or heterogeneous acid or basic catalyst is commonly used to enhance the reaction rate, even though some processes no longer require the use of a catalyst (Mata and Martins, 2010). Bio-derived catalyst are also explored for biodiesel production (Alagumalai et al., 2021). Triglycerides used for the production of biodiesel come from various sources such as edible oils, non-edible oils, waste/used oils, animal fats, microorganisms (Adetuyi et al., 2014), microalgae (Muhammad et al., 2021), enzymes (Lv et al., 2021) and insects (Kalu-Uka et al., 2021). The technical characteristics of biodiesel are similar and/or almost the same to that of fossil diesel and some properties of biodiesel are slightly higher than that of fossil diesel. However, biodiesel is biodegradable, nontoxic and produced from renewable source (Pedro et al., 2008, Siraj et al., 2013, Singh et al., 2020). In fact, biodiesel is the most advantageous renewable energy source (Ananthi et al., 2021). Furthermore, biodiesel can be mix with petrol diesel fuel, as it has almost the same characteristics (Selvakumar and Alexis, 2016). Thus, making a promising energy source. Ogbono seed (Irvingia gabonensis) is obtained from African bush mango tree of 15-40 cm with a bole slightly buttressed (Joseph and Aworth, 1991). The tree is largely distributed in Africa. The fruit is spherical with a smooth yellow fibrous mesocarp and hard endocarp when ripe, its often harvested during dry season and sold in Nigerian market. The fatty acid content of the seed (the seed is regarded as waste to best of our knowledge with no specific and/or commercial used) makes it applicable in biodiesel production. It’s worth nothing that Ogbono seed (which is a waste, with no commercial application) was used for the study not ogbono itself used in making soup. Thus, there is no feed and food competition. Oil from the seed was extracted for the study. Moreover, any oil bearing seeds that can produce up to 30% oil yield is considered suitable for industrial biodiesel production, this form one of the bases of the feedstock selection (Matchet, 1963).

The aim of this study is to produce biodiesel from ogbono seed oil using 98 ml methanol and 2 g potassium hydroxide (KOH) as catalyst and to also determine the physicochemical properties of the biodiesel. The overall goal is to determine the potential of ogbono seed oil as feedstock for biodiesel production.

MATERIALS AND METHODS

Sample preparation
Ogbono seed was procured from Dankure market, Sokoto State Nigeria and identified in the herbarium of Usmanu Danfodio University, Sokoto. The seed was dried and grounded into fine powder for extraction.

Chemicals and reagents used for research

All chemicals and reagents used i.e methanol of 99.5% purity, sodium hydroxide with 95% purity, sulphuric acid with 98% purity, hydrochloric acid of 36% purity, potassium hydroxide, n-hexane and phenolphthalein indicator were of analytical grade manufactured by British Drug House (BDH, London, United Kingdom. They were used as procured without further purification.

Oil Extraction

Soxhlet extraction technique was used for the oil extraction. Fourty grams of the grinded powdered sample and 250 ml of n-hexane (extracting solvent) were used for each extraction (five different extractions were carried out). The solvent was recovered using rotary evaporation. The oil was dried at 60 °C for 30 minutes and then weighed after cooling. The percentage oil yield of the seed was calculated according to equation 1.

\[
\text{% oil yield} = \frac{\text{wt of oil (g)}}{\text{wt of sample (g)}} \times 100 \quad (1)
\]

Acid pre-treatment

The oil was poured into a beaker, then 2% sulphuric acid was added into the oil, then poured into a reflux apparatus for two hours. Free fatty acid (FFA) value was determined again after the acid pretreatment.

Transesterification process

Prepared solution of methanol (98 ml) and 2g of KOH catalyst were added to the warmed oil in a round bottom flask. The mixture was then placed on water bath and the reaction conditions were maintained with constant stirring at 60 °C. After the reaction is complete (1 hour later), the content was transferred into a separating funnel and allowed to settle for 24 hrs. This allowed the glycerol to settle down since it is denser than the biodiesel. The glycerol was then removed from the separating funnel. Percentage yield of biodiesel was calculated according to equation 2.

\[
\% \text{ yield of biodiesel} = \left(\frac{\text{weight of biodiesel (g)}}{\text{weight of oil (g)}}\right) \times 100 \quad (2)
\]

Purification of the Biodiesel

The biodiesel was washed three times with warm distilled water to remove any residual glycerol, alcohol, catalyst and soap. It was then dried with anhydrous sodium sulphate and filtered prior to the characterization.

Ogbono oil and biodiesel physiochemical properties determination

Physicochemical properties of the oil and biodiesel were determined according to American society for testing and materials (ASTM) standard method. Properties determined are free fatty acid (FFA) value (ASTM D5555-95), density (ASTM D1298), pour point (ASTM D97), ash content (ASTM D428), saponification value (AOAC Method 920:160), ester value using European standard (EN) (EN 14103), iodine value (ASTM D5768), acid value (ASTM D664) and moisture content (ASTM D2709).

RESULT AND DISCUSSION

Physicochemical properties of ogbono seed oil

Table 1 shows the physicochemical properties of ogbono seed oil. Percentage (%) yield of the oil is 62.2±1 with a free fatty acid (FFA) value of 12.00±0.26mgKOH/g. Although the percentage oil yield is high and suitable for industrial application (Matchet, 1963), the high FFA value shows the needs for esterification and/or acid pre-treated. The value (FFA) of the oil was successfully reduced to the acceptable level of 1.00±0.26mgKOH/g after acid pre-treatment. With this value the oil was trans-esterified easily to biodiesel using methanol and KOH as catalyst. The oil density 0.83±0.07g/cm³ means it’s quite dense. Pour point of the oil is quite low as depicted in table 1. This was expected, as bio-oils tend to have generally low pour point (Fagernas, 1995). The high percentage of ash in the oil clearly means the presence of alkali metals. Generally, the higher the ash content the lower the bio-oil yield will be and vice versa (Chouhan, 2013). Saponification value of the oil 65±0.04mgKOH/g is lower than that of most of the vegetable oils reported (Karmaker et al., 2010). Ester value, iodine value and acid value of

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the oil are 97.5±1.0% (m/m), 35±0.12 gla/100g and 10.08±0.23 mgKOH/g respectively. Moisture in oil comes directly from the original moisture from feedstock used in oil production. The water content is significant because it affects other physical properties. It rises the pH, reduces the heating value and the viscosity, water content also affects both chemical and physical stability, and can affect the subsequent upgrading processes (Fagernas, 1995).

**Physicochemical properties of biodiesel generated from ogbono seed oil**

Table 2 shows the physiochemical properties of biodiesel produced from ogbono seed oil. The parent oil has a density of 0.83±0.07 g/cm³ but when trans-esterified the density reduced to 0.5±0.00 g/cm³. The biodiesel density is within the range of ASTM D6751 and EN 14214 standard. The pour point value of both the oil and biodiesel are also within the standard biodiesel range of ASTM D6751 and EN 14214. However, it’s worth noting that the pour point of the oil 7.2±0°C is higher than that of the biodiesel generated 2.0±0°C. The decrease in the pour value is clearly the effect of the transesterification process. The pour point of an oil is an essential property of oil and diesels, because it determines how it will flow at a given temperature. This is particularly used in a system where temperature can fluctuate due to the internal and external environmental conditions. For example, car and aerospace engine (Zahrani, 1998).

Trace to almost zero amount of ash was recorded in the produced biodiesel. This falls within the ASTM D6751 standard of 0.01%. The later also indicates that there is little metallic constituents, contaminants or impurities in the biodiesel generated.

| S/N | Parameters                          | Value    |
|-----|-------------------------------------|----------|
| 1   | Oil yield (%)                       | 62.2±1   |
| 2   | Free Fatty Acid Value (mg KOH/g)    | 12.0±0.26|
| 3   | Density (g/cm³)                     | 0.83±0.07|
| 4   | Pour Point (°C)                     | 7.2±0.0  |
| 5   | Ash Content (%)                     | 2.0±0.0  |
| 6   | Saponification value (mg KOH/g)     | 65±0.04  |
| 7   | Ester value (% (m/m))               | 97.5±1.0 |
| 8   | Iodine value (gI²/100 g)            | 35±0.12  |
| 9   | Acid value (mg KOH/g)               | 10.08±0.23|
| 10  | Moisture (%)                        | 3.0±0.0  |

Values are means ± SD triplicates analysis of samples

| S/N | Parameters                          | Biodiesel Extracted from Ogbono Seed | Biodiesel Standard (ASTM D6751) | Biodiesel Standard (EN 14214) |
|-----|-------------------------------------|--------------------------------------|---------------------------------|-------------------------------|
| 1   | Density (g/cm³)                     | 0.5±0.00                             | 0.88                            | 0.86-0.90                     |
| 2   | Pour Point (°C)                     | 2.0±0                               | -15-10                          | -                             |
| 3   | Ash Content (%)                     | Trace                               | 0.01                            | -                             |
| 4   | Saponification value (mgKOH/g)      | 58.90±0.06                          | -                               | -                             |
| 5   | Ester value (% (m/m))               | 98.0±0.5                             | -                               | 96.5minimum                  |
| 6   | Iodine value (gI²/100g)             | 26.64±0.15                           | 130maximum                     | 120maximum                    |
| 7   | Acid value (mgKOH/g)                | 0.28±0.05                            | 0.5maximum                      | 0.5maximum                    |
| 8   | Moisture (%)                        | 0.0006±0.0                           | 0.05maximum                     | 0.05maximum                   |

Values are means ± SD triplicates analysis of samples

Saponification value of 58.90±0.06 mgKOH was recorded and ester value of 98.0.5±0.5 %/(m/m). The later (ester value) fall within the EN 14214 minimum standard range. Iodine value
(26.649±0.15 gI/100g) of the biodiesel is within the ASTM and EU standard range. Iodine value was introduced in biodiesel quality standards to determined biodiesel stability to oxidation. Biodiesel can be easily oxidized in contact with air when it has high iodine value. However, iodine value greatly relies on the nature and ester composition of the feedstocks used in biodiesel production. Consequently, the iodine value is limited in different regions of the globe contingent to certain conditions. For example, the standard value for Europe and Japan is a maximum of 120. However, the value rises to 130 in Europe for biodiesel used as heating oil. The standard value is 140 in South Africa while Brazil has no limit. Biodiesel with high iodine value tends to polymerize and form deposits on injector nozzles, piston rings and piston ring grooves. The potential of polymerization rises with the degree of unsaturation of the fatty acids. The low iodine value of biodiesel reported in this study agrees with the low level of unsaturation in the fatty acids in the biodiesel (Muhammad et al., 2019). It was not surprising to find the acid value 0.28±0.05 mgKOH/g of the generated biodiesel from ogbono seed oil to be within the ASTM and EN standard range. This is because the freshness of the oil is related to acid value of the oil. Oils may generate free fatty acids during longer storage due to hydrolysis reaction. Hence, the acid value becomes one of the most important quality targets to determine the purity of oil.

High moisture content in biodiesel can cause problems such as water accumulation and microbial growth in fuel handling, storage, and transportation equipment. However, the results of the moisture of the oil reduced remarkably after transesterification showing that the biodiesel produced has good combustion properties and less or no contaminants. Due to the nature of the chemical structure of biodiesel, it has the characteristic of absorbing more moisture than petroleum diesel. The maximum amount of allowable water content in biodiesel as specified in ASTM standard D6751 is 0.05 % vol. The issue of high moisture content in biodiesel is often caused by improper treatment after processing (Gerpen, 2005) or by the absorption of atmospheric moisture during storage (Gerpen, 2005). Condensation and precipitation may occur if the moisture content in biodiesel is beyond its saturation point as its storage temperature decreases. Microbial growth, another effect of high moisture content as mentioned previously, causes fuel filter plugging, corrosion of storage containers and engine fuel systems (Gerpen, 2005). The moisture content of the biodiesel produced from ogbono seed oil is within the range of ASTM D6751 and EN 14214 standards.

Direct comparison of the present study with previous studies is not possible due to difference in feedstock, catalyst and other conditions. However, previous studies such as those conducted by Muhammad et al. (2019), with Abelmoscus esculentus seed oil as feedstock and Aworanti et al. (2019), using vegetable and palm waste frying oils as feedstock for bio-diesel production are similar to the present study with regards to physiochemical properties of biodiesel produced.

CONCLUSION

Ogbono seed oil is an excellent feedstock for biodiesel production via base catalyzed transesterification process. This is because the physiochemical properties of the produced biodiesel such as density, pour point, ash content, iodine value and moisture content are within the standard range of ASTM D6751 and EN 14214 for biodiesel.

REFERENCES

Aladetuyi A., Olutunji, G. A., Ogunniyi, D. S., Odetoye, T. E., and Oguntoye, S. O. (2014). Production and characterization of biodiesel using palm kernel oil; fresh and recovered from spent bleaching earth. Biofuel Research Journal, 4:134-138.

Alagumalai, A., Mahian, O., Hollmann, F. and Zhang, W. (2021). Environmentally benign solid catalysts for sustainable biodiesel production: A critical review. Science of The Total Environment, 768:144856.

Ananthi, V., Brindhadevi, K., Pugazhendhi A. and Arun, A. (2021). Impact of abiotic factors on biodiesel production by microalgae. Fuel, 284:118962.

Aworanti, O. A., Ajani, A. O., Agarry, S. E., Babatunde, K. A., Akinwumi, O. D. (2019). Evaluation of process parameters for biodiesel production from vegetable and palm waste frying oils using a homogeneous catalyst. International Journal of Energy Engineering, 9(2):25-35.
BP Energy Outlook (2016). BP Energy Outlook 2035, BP PLC. Available from: https://www.bp.com/content/dam/bp/pdf/energy-economics/energy-outlook-2016/bp-energy-outlook-2016.Accessed January 3, 2021.

Chouhan, A. P. S. and Sarma, A. K. (2013). Critical analysis of process parameters for bio-oil production via pyrolysis of biomass: a review. Recent Patents on Engineering, 7(2):1-17.

Eka, O. U. (1980). Proximate composition of seeds of bush mango tree and some properties of dika fats. Nigerian Journal of Nutritional Science, 1:33-6.

Fagernas, L. (1995). Chemical and physical characterization of biomass-based pyrolysis oil: literature review. VTT Technical Research Center of Finland, Espoo, Finland.

Gerpen, J. V. (2005). Biodiesel processing and production. Fuel Processing Technology, 86:1097–107.

Joseph, K. and Aworth, O. C. (1991). Composition, sensory, quality and respiration during ripening and storage of edible wild mango (Irvingia gabonensis). International Journal of Food Science and Technology, 26 (3):337-342.

Kalu-Uka, G. M., Kumar, S., Kalu-Uka, A. C., Vikram, S., Okorafor, O. O., Kigozi, M., Ihekweme, G. I. and Onwuala, A. P. (2021). Prospects for biodiesel production from Macrotermes nigeriensis: Process optimization and characterization of biodiesel properties. Biomass and Bioenergy, 146:105980.

Karmakar, A., Karmakar, S. and Mukherjee, S. (2010). Properties of various plants and animals’ feedstocks for biodiesel production. Bioresource Technology, 101(19):7201–7210.

Keneni, Y. G. and Marchetti, J. M. (2017). Oil extraction from plant seeds for biodiesel production. AIMS Energy, 5(2):316-340.

Khan, T. M. Y., Atabani, A. E., Badruddin, I. A. (2014). Recent scenario and technologies to utilize non-edible oils for biodiesel production. Renewable Sustainable Energy Reviews, 37:840–851.

Lv, L., Dai, L., Du, W., and Liu, D. (2021). Progress in Enzymatic Biodiesel Production and Commercialization. Processes, 9(2):355.

Mahlia, T. M. I., Syazmi, Z. A. H. S., Mofijur, M., Abas, A. E. P., Bilad, M. R., Ong, H. C. and Silitonga, A. S. (2020). Patent landscape review on biodiesel production: Technology updates. Renewable and Sustainable Energy Reviews. 118():109526.

Manzanera, M., Molina-Muñoz, M. L., and González-López J., (2008). Biodiesel: An alternative fuel. Recent Patents on Biotechnology Bentham Science Publishers Ltd, pp 25-34.

Mata, T. M. and Martins, A. A. (2010). Biodiesel production processes. Recent Progress in Chemical Engineering. Studium Press, India. Pp 314-343

Matchet, J. R. (1963). Industrial utilization of seed oil. Economic Botany, 17(1):23-30.

Muhammad G., Asraful M. A., Mofijur, M., Jahirul, M. I., Lv, Y., Xiong, W., Ong, H. C. and Xu, J. (2021). Modern developmental aspects in the field of economical harvesting and biodiesel production from microalgae biomass. Renewable and Sustainable Energy Reviews, 135:110209.

Muhammad, A. M., Jodi, L. M., Hassan, L. G., Abubakar, L. and Sokoto, A. M. (2019). Optimization of reaction variables of ultrasound-assisted transesterification of Abelmoscus esculentus seed oil into biodiesel. Biofuels.

Narwal, S. K., Saun, N. K., Dogra, P., Chauhan, G., and Gupta, R. (2015). Production and characterization of biodiesel using nonedible castor oil by immobilized lipase from bacillus aerius. BioMed Research International, 2015, Article ID 281934.

Pedro, B., Aguado, J. and Aguado, A. (2008). Basic properties of palm oil biodiesel–diesel blends. Fuel, 87:2069–2075.

Selvakumar, M. J. and Alexis, S. J. (2016). Renewable fuel production technologies. Middle East Journal of Scientific Research, 24(8):2502–2509.
Singh, D., Sharma, D., Soni, S.L., Sharma, S., Sharma, P.K. and Jhalani, A. (2020). A review on feedstocks, production processes, and yield for different generations of biodiesel. *Fuel, 262*:116553.

Siraj, R. S., Gitte, B. M., Joshi, S. D., Dharmadhikari, H. M., (2013). Characterization of biodiesel: a review. *International Journal of Engineering Research & Technology (IJERT), 2*(10):2077-2082

Zahrani, S.M. and Al-Fariss, T. F. A. (1998). General model for the viscosity of waxy oils. *Chemical engineering and Processing, 37*:433-438