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Synthesis and Evaluation of Biodiesel Derived from Shea Butter (Vitellaria paradoxa)

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Abstract
This study explored the potentials of converting shea butter into biodiesel to address both energy demands and environmental concerns. 200 ml of shea butter oil was mixed with 100 cm^3 of potassium methoxide. The results showed that biodiesel obtained from shea butter has a density of 900 kg/m^3, viscosity of 4.3 mm^2/s, cloud point of 11 ºC, acid value of 0.28 mgKOH/g, iodine value of 34.26 mg/100g, saponification value of 266.48 mgKOH/g, cetane index and cetane number of 58.22 and 55.62 respectively, and energy value of 37.72 mj/kg. These results were found to be within the ranges of American society for Testing and Materials (ASTM) specification.

Keywords: Biodiesel, Shea Butter Oil, Transesterification, Potentials, Biodiesel

Introduction

The use of biofuels has become increasingly important in the global effort to lessen reliance on fossil fuels and slow down climate change. Interest in biofuels made from biological sources has increased due to the growing need for sustainable energy on a worldwide scale (Enweremadu & Alamu, 2010). Shea butter is made from the nuts of the Shea tree (Vitellaria paradoxa), which is mostly found in Africa. Shea butter is often used in food and cosmetics because of its high fatty acid content.

Extracting biofuel from shea butter is not just a novel approach to energy sustainability, but it also aligns with global efforts to reduce carbon emissions, reliance on nonrenewable energy sources, job creation and economic development. Shea butter is an excellent choice for transesterification based biodiesel synthesis. This technique transforms the triglycerides in shea butter into fatty acid methyl esters (FAME), which are used to produce biodiesel. The development of biofuels has the potential to significantly impact their economies by generating new sources of income and stimulating local economies (Ogunsola et al., 2022).

Several studies have investigated the extraction methods of shea-butter for biofuel production. Tulashie et al. (2018) applied double stage acid alkaline catalysis to synthesize biodiesel from shea butter. Fourier Transform Infrared (FTIR) spectroscopy was used to analysed the synthesized biodiesel. Also, the density, flash point, cetane number, kinematic viscosity, iodine value, high heating value and methyl ester content of the synthesized biodiesel were investigated at different high temperature. This method was considered environmentally friendly and can produce high-quality oil, while Okoye et al. (2020) extracted biodiesel from shea butter through base-catalyzed transesterification method carried out at methano/oil ratio of 5:1 (V/V) at 70 ºC. The study reported 87% biodiesel yield after 90 mins, indicating that shea butter is a good biodiesel feedstock. Additionally, the outcome of the study showed that the cloud point and flash point was 9.3 ºC and 156.67 ºC, respectively. Also, the iodine and energy value were observed to be 35.29 mg/100g and 39.3 mj/kg, respectively.

Ogunsola et al. (2023) studied the utilization of heterogeneous catalysts in the transesterification process and discovered that the use of heterogeneous catalysts can increase the efficiency and sustainability of the transesterification process. This is because heterogeneous catalysts can easily be removed from the reaction mixture and reused, eliminating the need for costly and environmentally hazardous homogeneous catalysts. In addition, Ibrahim et al. (2022) investigated the optimization of reaction conditions for the transesterification process of waste cooking oil to biodiesel, and they found out that the optimization of reaction conditions, such as methanol/oil ratio, catalyst, temperature and reaction time is critical for achieving high yields and quality of biodiesel. The authors used response surface methodology to optimize the reaction conditions and found out that a temperature of 50-70 °C and a reaction time of 1-15 hours resulted in the highest yield and quality of biodiesel. The quality and properties of biodiesel are crucial factors that determine its suitability as a diesel fuel substitute. ASTM fuel tests, as mentioned in a study by Datti et al. (2020), plays a vital role in evaluating biodiesel properties such as density, viscosity, acid value, flash point, iodine value, saponification value e.t.c. These properties are essential for ensuring that biodiesel meets the required standards for use in diesel engines. Hence, the objective of this research was to synthesize an alternative fuel for diesel engine that is environmentally friendly to substitute diesel obtained from petroleum processes. The present study examined the extraction and conversion processes of shea butter as alternative renewable bioenergy.

Materials and Methods

Sample Collection

The shea-butter (which was the main feedstock for the biodiesel production) was purchased from Nupawa Street at Central Market in Kaduna State, Nigeria.

Sample Preparation

The shea-butter was stored away directly from heat source and sunlight and was brought to the laboratory. 400g of the shea-butter was cut, weighed and melted with the help of a magnetic stirrer hot plate at a temperature of 40 ⁰C. After melting, the shea butter oil was collected in a measuring cylinder. The melted shea-butter oil was pretreated by sieving it with the help of a Sieve cloth to remove all unwanted, undissolved solid impurities from the oil.

Methods and Materials

Transesterification method described by Datti et al. (2020) was implored in this study. 200 ml of shea-butter oil was mixed with 100 cm³ of potassium methoxide (obtained by dissolving 2.5g of KOH in 100 cm³ of methanol). The mixture was stirred with a magnetic stirrer at a reaction temperature of 60 ⁰C for one hour in a volumetric flask. On completion of the reaction, the mixture was transferred into a separating funnel and then allowed to stay for about 24 hours to ensure the complete separation of the mixture. The mixture was separated into two layers which is the biodiesel at the upper layer and the dense glycerol settling at the bottom layer. The glycerol was drained off, while the biodiesel was washed with distilled water to remove catalysts present, glycerol and water soluble impurities after which the biodiesel was heated to remove water present due to washing and allowed to settle.

Characterization of the Biodiesel

Determination of Density

The density of the biodiesel was measured with the help of a density bottle. The volume and weight of the biodiesel was recorded, and the density was calculated using equation 1:

$$Density = \frac{Weight\ of\ Biodiesel}{\text{Volume of Biodiesel}} \quad (1)$$

Determination of viscosity

The viscosity of the biodiesel was measured using a digital viscometer. 25 ml of the biodiesel was measured out into a beaker to test the viscosity noting the temperature.

Determination of cloud point

10 ml of the biodiesel sample was placed in a medium sized test tube and was placed in a refrigerator while monitoring the temperature at which solidification began using a mercury -in- glass thermometer.

Determination of Acid value

0.5g of the biodiesel was weighed and transferred into a conical flask. 20ml of ethanol and 3 drops of phenolphthalein indicator was added. The solution was titrated against 0.1M KOH to pink color. The volume of the titrant at end point was recorded and calculated using equation 2:

$$\text{Acid value (A.V)} = \frac{\text{Weight of biodiesel}}{\text{Acid titre value} \times \text{Normality of KOH} \times 56.1} \quad (2)$$

Determination of Iodine value

10 ml of chloroform was added to 0.5g of the biodiesel. 20 ml of iodine monochloride dissolved in acetic acid was added to the resultant mixture of chloroform and biodiesel. The mixture was covered tightly and kept in a dark cupboard for 30 minutes. Then 20 ml of 10% potassium iodide (KI) solution was added to the solution and

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titrated against 0.1M sodium thiosulphate ($Na_2S_2O_3$) using starch solution as the indicator. The whole procedure was carried out for a blank and the iodine value was calculated using equation 3:

$$\text{Iodine value (I.V)} = \frac{12.69 \times (V1 - V2) \times \text{Normality of } Na_2S_2O_3}{\text{Weight of biodiesel}} \quad (3)$$

Where,
V1= Volume of $Na_2S_2O_3$ used for the Blank
V2 = Volume of $Na_2S_2O_3$ used for the Sample

Determination of saponification value
0.5g of biodiesel was weighed into a conical flask and 50ml of 0.5M ethanolic KOH was added and heated under reflux for 30 minutes to saponify the sample. The solution was titrated with 0.5M hydrochloric acid (HCL) using three drops of phenolphthalein as indicator. The saponification value was calculated using equation 4:

$$\text{Saponification value (S.V)} = \frac{\text{Titre value} \times \text{Normality of HCL} \times 56.1}{\text{Weight of biodiesel}} \quad (4)$$

Determination of cetane index/cetane number
The cetane index is a measure of the ignition quality of the biodiesel. The Cetane index (CI) was determined from the correlation in equation 5:

$$CI = 46.3 + (5458/S.V) - 0.25\ I.V \quad (5)$$

where, S.V – Saponification value I.V – Iodine value

The Cetane number (CN) was then obtained from a correlation reported by (Ejeh & Aderemi 2014).

$$CN = CI - 2.6 \quad (6)$$

Determination of Energy value
The energy value of the biodiesel was also determined according to the model presented in equation 7:

$$HHV = 49.43 - (0.015\ I.V) - (0.042\ S.V) \quad (7)$$

where
S.V – Saponification value
I.V – Iodine value

Results
Table 1 below presents the characterization results of shea butter biodiesel alongside with the ASTM standard range for biodiesel. It provides a summary of the physiochemical fuel properties of the shea butter biodiesel.

Table 1 Results for the characterization of the shea-butter biodiesel

S/N	Properties	Shea-butter biodiesel	ASTM biodiesel standard range (D6751) (Sakthivel et al., 2018)
1	Density (kg/m³)	900	575 – 900
2	Viscosity (mm²/s)	4.30	1.9 – 6.0
3	Cloud point (⁰C)	11	-3 – 12
4	Acid value (mgKOH/g)	0.28	0.8 maximum
5	Iodine value (mg/100g)	34.26	–
6	Saponification value (mgKOH/g)	266.48	120 maximum
7	Cetane index	58.22	47 minimum
8	Cetane number	55.62	
9	Energy value (mj/kg)	37.72	

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From Table 1 above, the results for the characterization of shea butter biodiesel revealed several key physiochemical properties that align well with ASTM standards for biodiesel, though some variations were observed when compared to other biodiesel produced from other feedstocks.

Discussion

Density indicates the weight of the oil or fat and the solid content at a given temperature. The density of the biodiesel was found to be 900 kg/m³, which fell within the upper limit of the ASTM range (575–900 kg/m³). Density is a critical property that affects combustion efficiency and engine performance. This value is comparable to findings by Adebayo et al. (2021) for neem seed biodiesel, with densities ranging from 880 to 895 kg/m³, but it was slightly higher than the density of castor oil biodiesel (870–880 kg/m³) reported by Manu et al. (2023). The higher density in this study is likely due to the specific triglyceride structure and higher saturated fatty acid content in the shea butter. A low-density value indicates a more complete ester conversion.

Furthermore, viscosity is the measure of a fluid's resistance to deformation at a given rate. 4.30 mm²/s was obtained as the viscosity value for the synthesized shea butter biodiesel. This value falls within the ASTM range of 1.9-6.0 mm²/s. Viscosity influences the fuel flow and injection characteristics, making it a crucial property for engine performance. This result is consistent with the viscosities of palm oil biodiesel (4.2-4.5 mm²/s) as reported by Olaniyi et al. (2019). The slight differences in viscosity values across feedstocks are influenced by the length and saturation level of the fatty acid chains. The balanced composition of saturated and unsaturated fatty acids in shea butter contributes to the moderate viscosity, making it suitable for engine use. Additionally, the temperature at which biodiesel begins to cloud or become hazy due to the formation of wax crystals is known to be cloud point. Cloud point is an important indicator of biodiesel suitability in cold climates. 11 °C was obtained as the cloud point value for the synthesized biodiesel. While this value is within the ASTM range of -3 to 12 °C, it is relatively higher compared to other biodiesels, such as soybean biodiesel (8 °C) which was reported by Adegoke et al. (2022) and rapeseed biodiesel (5 °C) reported by Egbo et al. (2020). The elevated cloud point of shea butter biodiesel is attributed to its higher saturated fatty acid content, particularly stearic acid which increased the crystallization temperature. This characteristic may limit its application in colder regions unless blended with lower cloud point fuels.

Also, the acid value is the mass of potassium hydroxide in milligram required to neutralize one gram of a chemical substance. It is an indicator for the level of free fatty acid in a biodiesel or oil sample. The acid value of 0.28 mg KOH/g was obtained for shea butter biodiesel which was similar to 0.37 KOH/g and 0.19 mg KOH/g reported by Datti et al. (2020) and Okoye et al. (2020). These values conform to the ASTM value 0.5 mg KOH/g, indicating excellent fuel stability and minimal risk of corrosion. This value aligns closely with coconut oil biodiesel (0.25 mg KOH/g) reported by Adejumo et al. (2019). Variations in acid value are largely influenced by the feedstock's quality and the efficiency of the transesterification process. Shea butter lower free fatty acid content contributes to its lower acid value, enhancing its long-term storage stability.

In term of iodine value, 34.26 mg /100 g obtained coincide with 35.29 mg/100g reported by Okoye et al., 2020 conforming to ASTM D6751 and (EN) 14214 International Standard. The relatively high iodine value in this study can be attributed to the presence of monounsaturated fatty acids, such as oleic acid in shea butter, which enhances its combustion properties but may increase susceptibility to oxidative polymerization.

Furthermore, the amount of alkali required to saponify a biodiesel is known as saponification value. The saponification value of 266.48 mg KOH/g obtained was significantly above the ASTM standard of 120 mg KOH/g, suggesting a higher molecular weight of fatty acids in the biodiesel. This value is higher than the saponification value of shea butter biodiesel extracted through traditional method (167.4 mg KOH/g) and solvent extraction method (202.9 mg KOH/g) reported by Ajala et al. (2016). The elevated saponification value can be attributed to the unique lipid profile of the shea butter, which includes shorter fatty acid chains, thereby increasing the saponification potential.

In addition, 58.22 and 55.62 was obtained as the cetane index and cetane number of the biodiesel, respectively. These values exceeded the ASTM minimum requirement of 47. High cetane numbers indicate excellent ignition quality and smooth combustion, comparable to castor oil biodiesel. However, these values are slightly higher than castor biodiesel, which typically ranges from >47 to 50 (Margaret & Mbamalu, 2025). The higher cetane numbers for shea butter biodiesel can be attributed to its higher saturated fatty acid content, which enhances combustion efficiency and reduces ignition delay. Finally, the energy value of 37.72 MJ/kg was within the acceptable ASTM

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range for biodiesel. The high energy content of shea butter biodiesel is influenced by its triglyceride structure and fatty acid composition, making it a viable alternative to conventional diesel fuels.

The variations observed between the results of in this study and those from other related works can be attributed to several factors which are, the fatty acid composition of shea butter which differs significantly from other feedstock. Shea butter contains a higher proportion of saturated fatty acids such as stearic acid, which influences properties like density, viscosity, and cloud point. In contrast, feedstocks like soybean and rapeseed, with higher unsaturated fatty acid content, exhibit lower densities and cloud points. Secondly, the biodiesel production process, including catalyst type, reaction temperature, and transesterification efficiency played a crucial role. Variations in these parameters can result in differences in acid value, iodine value, and saponification value. For example, a more efficient transesterification process reduces free fatty acid content, resulting in lower acid values. Lastly, the quality of the feedstock, including its purity and pretreatment affects the final biodiesel properties.

Conclusion

Biodiesel based shea-butter was synthesized using transesterification method with potassium hydroxide (KOH) as a catalyst and methanol at a reaction temperature of 60°C. Majority of the characterized physiochemical properties are within the acceptable range of standard biodiesel. However, shea butter being a semi-solid fat may require different processing techniques and catalyst compared to liquid oils like soybean or palm oil, which could influence the saponification and cetane values. Shea butter biodiesel could thus be utilized as an addition in bio-based biodiesel for energy generation in machineries and various equipment

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