Physicochemical Properties of Shea Butter Synthesized Biodiesel

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Physicochemical Properties of Shea Butter Synthesized Biodiesel

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ABSTRACT

Base – catalyzed transesterification of Shea (*Vitellaria paradoxa*) seed fat was carried out at a methanol/oil ratio of 5:1 (V/V) at 70^{o}C to synthesize the corresponding methyl esters (biodiesel). The percentage yield of approximately 87%, was recorded after ninety minutes, indicating that Shea fat is a good biodiesel feedstock. The physicochemical properties of the Shea biodiesel were determined. The colour was pale yellow while the relative density (870 Kg/m^3), kinematic viscosity (2.66 mm^{2}s^{-1} 40^{0}C), acid value (0.19 mg KOH/g), peroxide value (0.52 meq/kg) and cetane number (68.10) were observed. The cloud point was found to be 9.3^{0}C, while the flash point of 156.67^{0}C, iodine value of 35.29 mg/100g and energy value of 39.3 MJ/Kg were recorded. All these value compare well with previous works and are within acceptable limits as specified by the American Society for Testing and Materials (ASTM). The current research indicated that Shea butter has biodiesel potential aside its uses in culinary and cosmetics applications.

Keywords: Biodiesel, Physicochemical, Shea, Synthesize, Transesterification

Introduction

Shea tree has been identifies as native to over sixteen west African countries (Benin, Burkina Faso, Cameroon, Central African Republic, Chad, Cote d'lvoire, Ethiopia, Ghana, Guinea, Mali, Niger, Nigeria, Senegal Sudan, Togo and Uganda) and found growing wildly in many Nigerian states (Niger, Nasarawa, Kebbi, Kwara, Kogi, Adamawa, Benue, Edo, Katsina, Plateau, Sokoto, Zamfara, Taraba, Borno and Oyo) (Ekeh, 2010). Oil and butter being components of shea nuts from the shear tree *Vitellaria paradoxa* formally *Butryospermium paradoxum* (Warra, 2011), has found many usefulness inclusive but not exhaustive in the production of creams, pharmaceutical, paint, vanish, animal feed, soap, confectionery products, wood polish and of recent biodiesel. Production of biodiesel using various catalytic processes has been researched viz: homogenous catalyst method (Ejeh and Aderemi, 2014), catalyzed and supercritical transesterification synthesis processes (Odisu et al., 2019), and transesterification reaction under different operational conditions of temperature, reaction time and catalyst (NaOH) (Yaro *et al*., 2019) among others. The current research involve synthesis and establishment of the physicochemical properties of biodiesel from Nasarawa Eggon shea butter popularly sold in the Mada Station Market of Nassarawa State, Nigeria. Biodiesel being fuel generated from plants and plant based materials is seen as a better option for the future as it is not only environmental friendly compared to the fossil stock but also renewable.

Materials and Methods

Shea butter Source

The shea butter (which is the main feedstock for biodiesel production) was purchased from Mada Station Market, Nasarawa Eggon Local Government Area of Nasarawa State, Nigeria

Shea Biodiesel Production

A five hundred millilitre three-necked round-bottomed flask was used as the reactor for the transesterification reaction. Shea butter fat was first molten and a volume (500ml) of 470g weight was measured into the reactor and preheated at $50^{o}C$ for 30mins. Methoxide (100 ml) solution prepared by dissolving 4.25g of KOH in hundred millilitres of methanol, was added and fitted with a condenser. The temperature was raised to 70^{o}C maintaining continuous stirring with a magnetic spin stirrer. After 90 mins, the reaction mixture was slightly acidified with 1M H_2SO_4 solution to neutralize the base, and transferred to a separating funnel. Biodiesel separation was carried out by addition of warm distilled water on the mixture, which was then left to stand overnight. A dark brown coloured, cloudy denser lower layer (glycerol and soap) was tapped off leaving a light yellow upper layer (biodiesel). The biodiesel was concentrated and its fuel properties were determined.

Characterization of Shea Biodiesel

Determination of density/relative density

The biodiesel was determined according to a method described by Onwuka (2018). A pycometer (density) bottle of 50ml capacity was thoroughly washed with detergent, water and petroleum ether, dried and weighed. The clean dry pycometer bottle was filled with 50ml of synthesized biodiesel and weighed again. Weighed biodiesel was gotten by subtracting the weight of bottle from the weight of biodiesel and density bottle. Density was determined by taking the ratio of the weight of the biodiesel to the known volume (50ml) according to the formula:

$$Density = \frac{Biodiesel\ Weight}{Biodiesel\ \ Volume}$$

Specific gravity was also determined using the 50ml capacity pycometer bottle .The weight of the empty bottle and that of 50ml of biodiesel had been taken earlier. Now, after washing the bottle and dry,the bottle, it was filled with water (50ml) and weighed . Specific gravity was calculated using the formula: ‘

$$Specific\ Gravity = {Weight\ of\ Biodiesel\ (50\ ml)}\Big/{Weight\ of\ Water\ (50\ ml)}$$

Determination of Refractive index

Refractive index was determined using a digital table top refractometer (HI96800) manufactured by Hanna Instruments, Romania. The device was initially calibrated to zero using distilled water. The biodiesel was then placed at the glass prism and the refractive index read off.

Determination of Moisture

Dry oven method moisture determination by Wick (1985) was used. Synthesized biodiesel (5g) was weighed into an already weighed petri dish (a). The sample in petri dish was transferred into an oven and left for an hour at 105^0C. The sample was allowed to cool in a desiccator. The second weight of the biodiesel was taken after oven heating (b). Percentage moisture was calculated using the formula:

$$\%\,Moisture = \frac{a-b}{Initial\ Sample\ weight} \times 100\,\%$$

Determination of Saponification Value

Saponification value was determined according to the method of Boerlage and Broeze, (1990). This is the milligram of KOH required to neutralize the fatty acids resulting from the complete hydrolysis of 1g of the sample. Saponification value is the measure of the molecular weight of the fatty acid. Briefly, a quantity (0.5g) of the biodiesel was weighed into a conical flask and 50ml of 0.5N ethanolic KOH was added to it. The mixture was refluxed to saponify the sample. The unreacted KOH was titrated back with 0.5N hydrochloric acid using three drops of phenolphthalein as indicator. The saponification value of the biodiesel was calculated thus:

$$Saponification\ Value = \frac{(Titre\ Value)(Nomality\ of\ NaOH)(56.1)}{(Weight\ of\ Biodiesel)}$$

Determination of Kinematic Viscosity

U-tube viscometer manufactured by Poulten Selfe and Lee Ltd (PSL ASTM-IP 350) was used to determine the kinematic viscosity of the shea biodiesel. Micropipette was used to introduce the sample into the viscometer. Sample flow time was determined in seconds at 40^0C.

$Kinematic\ Viscosity = ct\ (mm^2\ s^{-1})$

Where c is viscosity constant 0.4891

Determination of Acid Value

The acid value which is the number of milligram of KOH required to neutralize the free fatty acid in 1g of the biodiesel sample was determined according to ASTM (2002). Briefly, shea biodiesel (0.5g) was weighed into a conical flask and 20ml of ethanol and three drops of phenolphthalein indicator added. Titration of the solution with constant agitation was carried out until a faint, pink end point which persists for thirty seconds appeared. The volume of the titrant at end point was recorded. From the readings obtained, the acid value was evaluated using the equation below:

$$Acid\ Value\ = \frac{(Acid\ Titre\ Value)(Nomality\ of\ NaOH)(56.1)}{(Biodiesel\ Weight)}$$

Determination of Peroxide Value

Peroxide value was determined according to the method of Boerlage and Broeze (1990). The peroxide value is the measure of the peroxides contained in the oil. Glacial acetic acid (25ml) and chloroform were mixed together in the ratio 2:1 into a conical flask added to 0.5g of synthesized biodiesel. A volume (1ml) of 10% potassium iodide solution was added shaken vigorously, covered and kept in the dark for one minute after which starch indicator (35ml) was added. The whole solution (V_1) was titrated with 0.02N sodium thiosulphate solution as it turned from pale black to white. Titration was also carried out on the blank. Peroxide value was calculated from the equation below:

$$Peroxide\ Value\ = \frac{(100)(V1 - V2)(Normality\ of\ Titrant)}{(Sample\ Weight)}$$

Determination of Iodine Value

Boerlage and Broeze (1990) described the protocol employed in this research. Briefly, 15ml of chloroform was added to 0.5g of synthesized biodiesel. To the resulting mixture was added 25ml of Wiji's solution, and vigorously agitated. The mixture was covered tightly and placed in the dark cupboard for thirty minutes. Measured volume (20ml) of 10% KI was added followed by 150ml of distilled water. Resultant red colouration was observed after which 5ml of 5% starch solution indicator was added and observed for blue-black colour change. The solution was titrated with 0.1N sodium thiosulphate solution until black precipitates appear in colorless solution. Titration was also made for the blank. Iodine value was calculated as:

$$Iodine\ Value\ = \frac{(12.69)(V2 - V1)(Normality\ of\ Titrant)}{(Weight\ of\ Biodiesel)}$$

Determination of Cloud Point

The synthesized biodiesel sample was placed in a medium sized test tube and the test tube with its content placed in a test tube rack and placed in a refrigerator and monitored. The temperature at which the heavier components formed mass of colloids is the cloud point. This temperature was determined using mercury-in-glass thermometer.

Determination of Flash/Fire point

Flash (fire) point were determined by pouring the biodiesel sample in a glass petri dish so that the surface of the dish was covered. A mercury-in-glass thermometer was immersed into the sample in the petri dish so that the tip of the thermometer just touched the biodiesel sample. The thermometer was held in position using a retort stand and clamp. The sample was placed on a laboratory heating mantle and was gradually heated and light source was applied at intervals. The lowest temperature at which the sample just ignited' and went off was recorded as the flash point.

Determination of Aniline point

Aniline point was determined according to ASTM (2004). Aniline oil was poured into a test tube. The shea biodiesel sample was also introduced into the test tube. Both samples were initially not mixable and so formed double phase. The test tube was placed in a test tube rack and placed in a refrigerator. The lowest temperature at which the aniline oil and biodiesel mixed together was recorded as the aniline point.

Determination of API gravity

The American Petroleum Institute (API) gravity was determined from the value of the specific gravity of the biodiesel using the formula:

$$API = {}^{141.5}\!/_{Specific\ Gravity\ at\ 60^{o}F)} - 131.5$$

Determination of Cetane index/Cetane number

The cetane index is a measure of the ignition quality of diesel fuel. The higher the cetane index, the easier it is to start a standard diesel engine. Cetane index determined according to the method of Boerlage and Broeze (1994). Using the formular:

$$Cetane\ Index\ = \frac{Aniline\ Point\ (°C)\ \times API\ Gravity}{10}$$

Determination of Energy Value

The energy value was determined using a bomb colorimeter. After standardization of the biodiesel with benzoic acid, a quantity (0.5g) of the biodiesel was burned in the calorimeter and values taken. Measurements were repeated three times.

Characterization of Shea butter

Very essential characterization properties determinations carried out in the synthesized biodiesel were equally repeated with shea butter samples.

Statistical Analysis

Data obtained were expressed as mean $\pm$ standard deviation (SD).

Results

The Physicochemical properties of the Shea Biodiesel.

The physicochemical properties of Shea biodiesel and those expected of standard biodiesels are presented in Table 1. All the fuel properties of Shea biodiesel such as density (0.87±0.002 kg/m^3), specific gravity (0.88), acid value (0.19 mgKOH/g), cloud point (9.33^{0}C), kinematic viscosity (2.66mm^2/s^{-1}), flash point (156.67^{0}C), cetane number (68.10) and energy value (39.3MJ/Kg) all fell within the acceptable range for standard biodiesels.

Table 1: The Physicochemical Properties of synthesized Shea Biodiesel values compared with the Standard Biodiesel (Values/Range).

Properties	Shea Biodiesel (Mean ± SD)	Standard Biodiesel (Values/Range)
Density (Kg/m^3)	870 ± 2.15	575 to 900
Specific gravity	0.88	0.88
Refractive index @ 29^{0}C	1.46	-
Moisture (%)	Nil	< 0.05
Saponification value (mg KOH/kg)	184.18 ± 0.25	
Viscosity@ 40^{0}C (mm^{2}s^{-1})	2.66	1.9-6.0
Acid value (mg KOH/g)	0.19 ± 0.01	< 0.5
Peroxide value (meq/kg)	0.52 ± 0.01	-
Iodine value (mg/100g)	35.29 ± 0.94	-
Cloud point (^{0}C)	9.33 ± 0.29	-3 to 12
Flash point (^{0}C)	156.67 ± 1.53	100-170
Aniline point	23	-
API gravity	29.61 ± 0.93	-
Cetane index	68.10 ±2.15	47 minimum
Specific gravity	0.88	-
Energy value (MJ/Kg)	39.3	40-50

American Society for Testing and Material (ASTM) D6751 for biodiesels.

Table 2: The Physicochemical Properties Value of Shea Butter

Properties	Shea Butter Mean ± SD	
Viscosity at 40°C (mm^2s^{-1})	114.40 ±1.58	
Density (kg/m^3)	920 ± 0.003	
Energy Value (MJ/Kg)	33.9	
-	-	
Cloud Point (°C)	24	
Acid Value (mgKOH/g)	0.47 ± 0.02	
Flash Point (°C)	339.33 ± 2.08	
Perioxide Value (meq/kg)	0.85 ± 0.02	
Saponification Value (mgKOH/Kg)	181.98 ± 0.48	
Iodine value (mg/100g)	31.19±0.56	
Refractive index at 29°C	1.47±0.48	
Moisture	0.10±0.01	

The values are presented as mean ± standard deviation

Discussion

The yield of Shea biodiesel synthesized with methanol was 87%. The results of the characterization of the shea biodiesel are presented in table 1. The properties determined include: Density, acid value, specific gravity, iodine value, moisture content, cloud point, flash point, peroxide value, energy value, cetane index, saponification value, kinematic viscosity, refractive index, aniline point, API gravity.

Density is an indicator of the physical thickness of solid, liquid or gas measured by its mass per unit of volume. The density of the shea biodiesel produced was 870 kg/m^3. The finding from this research also conforms to the value range (575 – 900) specified by American Society for Testing and Materials (ASTM) D6751 and European Standard (EN) 14214 (Jenvanitpanjakul, 2009) as well as 887.61 kg/m^3 (Datti *et al.*, 2020) from similar research. The density (920Kg/m^3) of the shea butter itself is above the recommended limits and different from the value (895.4 kg/m^3) obtained by Ejeh and Aderemi, (2014) in their research.

The specific gravity of the shea biodiesel was 0.88 which is agreement with the finding of Odisu *et al.* (2019). They produced biodiesel from shea butter using acid-catalysed transesterification and recorded a value of 0.87. The specific gravity obtained from the current research also agrees with values of previous work: 0.90 (Ejeh and Aderemi, 2014), and is in conformity with the specification by the American Society for Testing and Materials (ASTM) D6751 and (EN) 14214 (Jenvanitpanjakul, 2009) for biodiesels.

Acid value is the mass of potassium hydroxide in milligram that is 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. An acid value of 0.19 mg KOH/g was obtained for the shea biodiesel produced. This value also conforms to that (0.5 maximum) specified by the American Society for Testing and

Materials (ASTM) D6751 and (EN) 14214 (Jenvanitpanjakul, 2009) as well as 0.37 KOH/g (Datti *et al*., 2020). The acid value (0.47 mg KOH/g) was also in normal range in the stock (shea butter) table 2.

Iodine value is the mass of iodine in grams that is consumed by 100 grams of a chemical substance. It is used to determine the degree of unsaturation of fatty acids present. Iodine value of the shea biodiesel was found to be 35.29 mg/100g similar to 34.24 mg/100g by Datti *et al*. (2020) as well as 76.14 mEq/g by Ejeh and Aderemi (2014) conforming to American Society for Testing and Materials (ASTM) D6751 and (EN) 14214 International Standard. The value from the shea butter stock was 31.19 mg/100g thus illustrating that the synthesized biodiesel is more unsaturated than its source.

Moisture is the presence of a liquid, especially water, often in trace amounts or amount of water vapour present in the air. Moisture of the shea biodiesel was 0.06 (%) which is slightly higher than (0.05%) specified by the American Society for Testing and Materials (ASTM) D6751 and (EN) 14214 International Standard. The cause of this disagreement is not traceable to an error in the main process of the product synthesis but rather in downstream processing where the moisture would have been retained. It also depends on the level of synthesized shea butter biodiesel yield during concentration of product which could differ between researcher and varied methodologies adopted. Further concentration of the shea biodiesel can address this problem.

Cloud point refers to the temperature below which wax in diesel or biodiesels forms a cloudy appearance. Cloud point of the shea biodiesel was 9.33^0C and fell within the specifications by ASTM D6751 and (EN) 14214 International Standard (Jenvanitpanjakul, 2009).

Flash point is the lowest temperature at which vapours of the material will ignite, when given an ignition source or when exposed to a spark of flame. This property is one of the main properties of fuels and determines the likelihood or tendency of a fuel sample to ignite accidentally. It therefore expresses how safe the storage and handling of the sample is. The flash point of the shea biodiesel from this research was 156.67^0C whereas 110^0C and 96 ^{0}C were reported by Ejeh and Aderemi, (2014) and (Datti *et al*., 2020) respectively. These value recorded in the current research is quite in conformity to that (93^0C minimum) specified by ASTM) D6751 and (EN) 14214 International Standard (Jenvanitpanjakul, 2009). The result from this research showed that there is more stability and safety of the synthesized biodiesel from shea butter against possible ignition. It is thus indicative of a high value biodiesel.

Peroxide value is widely used to measure the extent which an oil or biodiesel sample has undergone primary oxidation. Peroxide value of the shea biodiesel was 0.52 meq/kg. This is a good property since this value is minimal indicating non rancidity formation of the synthesized product. The aniline point was 23. Refractive index from the synthesized work was 1.46 very comparable to 1.441 by Datti *et al.* (2020).

Energy value it is expressed in K calories (Kcal) and in Kilo Joules (KJ). It is important in knowing the heat of combustion not only of foodstuff but also of any combustible material. Energy value of the shea biodiesel was 39.3 MJ/Kg which is very close to that from a previous work by Ejeh and Aderemi, (2014) who reported an energy value of 37.2 MJ/kg for their own shea biodiesel. The value from this work is though lower but do not deviate significantly from the range (40 – 50) specified by ASTM and EN (Jenvanitpanjakul, 2009).

Cetane number is an indicator of the combustion speed of diesel fuel and compression needed for ignition. It is an inverse of the similar octane rating for gasoline and is important in delay period of fuels. Cetane number of the shea biodiesel was 68.10 and conforms to ASTM) D6751 and (EN) 14214 International Standards (Jenvanitpanjakul, 2009) since it is above the standard minimum of 47. The works by Ejeh and Aderemi, (2014) however, have cetane number (46.8374) which was lower than that obtained in this research. Thus its combustion is expected to be in moderate proportion to the combustion time necessary to give the required output should it be used to power any energy generating equipment or machine.

Saponification value (SV) is the number of milligrams of potassium hydroxide required to saponify 1g of fat under the condition specified. It measure average molecular weight. Saponification value of the shea biodiesel was 184.18 mgKOH/kg. This property is more important in oils that are being tested for suitability in soap making. SV of shea butter (181.98 mgKOH/Kg) and other oil properties are presented in Table 2, and are pointers to various uses of the shea fat including cosmetics and culinary dimensions.

Viscosity of a fluid is a measure of it resistance to deformation at a given rate. For liquid, it corresponds to the informal concept of thickness. Viscosity of the shea biodiesel was 2.66 mm^2s^{-1} at 40^0C. This value agrees well with that (2.88 mm^2s^{-1}) reported for another produced shea biodiesel by Odisu *et al.* (2019) and however, though within range, the value (3.62 mm^2s^{-1})by Datti *et al*. (2020) was quite higher than that of this research. These values are within the range

(1.9 – 6.0) specified by American Society for Testing and Materials (ASTM) D6751 and (EN) 14214 International Standard (Jenvanitpanjakul, 2009). Differences though quite insignificant in the two shea biodiesel generated by Odisu *et al.* (2019) and of this work could be due to locality and climatic condition differences.

API gravity is the acronym for American Petroleum Institute gravity. It is an inverse measure that is used to determine the weight of petroleum liquid in comparison to water. API gravity of the shea biodiesel was 29.61. This parameter was necessary for calculating the cetane number.

Conclusion

Shea butter biodiesel of acceptable quality was successfully produced through transesterification in a 90 mins batch run using KOH as catalyst, and methanol at a reaction temperature of 70°C. Shea biodiesel yield of 87% was achieved agreeing with previous works that shea fat is a good feedstock for biodiesel production. Majority of the characterized physicochemical properties are within the acceptable range of standard biodiesel. Shea butter biodiesel could thus be utilized as an addition in bio-based biodiesel for energy generation in machineries and various equipment.

Declarations:

Ethics approval and consent to participate

Not Applicable

Consent for publication

All the authors have given their consent through the corresponding author for this research work to be published under the Bioresources and Bioprocessing Journal.

Availability of data and materials

The data and materials collected were from the values and environment where the research was carried out. There is no dispute or ethical violation in both the data and materials used as recorded in the manuscript.

Competing interests

The authors declare that they have no competing interests.

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Authors Contribution

IGO designed the study. EKD carried out the field/laboratory operations. Data analysis were by CSE and IGO. Supporting literature was by CSE. Preparation of manuscript was carried out by CSE, All authors read and approved the final manuscript.

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