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QUALITY ASSESSMENT OF BIODIESEL PRODUCED FROM *Caesalpinia pulcherrima* (Pride of Barbados)

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ABSTRACT
Alternative energy sources from vegetable products are developing as a response to the depleting fossil fuel reserves and the environmental damage caused by their burning. Among these different possible resources, biodiesel has received a lot of interest as a diesel engine fuel substitute because it is renewable, non-toxic, eco-friendly, etc. This study evaluated the quality assessment of production of biodiesel from Caesalpinia pulcherrima. The sample oil was extracted from the seeds of Caesalpinia pulcherrima using a soxhlet extractor with n-hexane; the results obtained showed low yield of 27 ± 0.1 % for the extracted oil. The physicochemical properties of the oils sample were found to be as follows: Saponification value: 142.87 mgKOH/g, peroxide value: 7.70 mEq O2/kg, acid value : 30.49 mg KOH/g, iodine value (IV): 23.15 g I2/100g, viscosity: 5.83 mm2 /sec, specific gravity: 0.90. The value of free fatty acid gotten was 15.43. The oil was transesterified using two-step transesterification due to their high free fatty acid (FFA) using sodium hydroxide as catalyst. The biodiesel produced was analyzed for its fatty acid profile using GC-MS and fuel properties using ASTM Methods. The Caesalpinia pulcherrima oil obtained for use in the production of biodiesel was clear, viscous, and yellowish in color. The result of the transesterification reaction was a transparent yellowish color liquid (biofuel) and the percentage yield was 71.73 %. The density of 0.86, pour point of -5.03 °C, cloud point of 5.83 °C, the specific gravity of 0.895, the kinematic viscosity of 5.37 mm^2 /s, and an acid value of 1.1 MgKOH/g were all within the ASTM D6751 specification for biodiesel fuel. respectively. Results obtained infer that oil from Caesalpinia pulcherrima possesses properties that are suitable for biofuel production using ASTM standard.
Keywords: Methylester, Caesalpinia pulcherrima, Biodiesel, fatty acid and fuel

INTRODUCTION

The need for energy has been steadily increasing around the world as industrialization and population have increased. Internal combustion engines, particularly diesel engines (compression ignition engines), have consumed a significant amount of energy (Hosseini *et al.,* 2019). Apart from the hydrogenation of energy, which has been inconsistent due to poor maintenance, the usage of renewable forms of energy is not well established in Nigeria. The government is attempting to launch programs to raise awareness and educate the public about the use of renewable energy resources, including building the policies, incentives, and regulatory environment required for biofuel to thrive in Nigeria (Onuoha, 2010)

Diesel engines have recently become the most used internal combustion engines in the transportation sector worldwide (Dogan, 2011). As a result, researchers have recently begun to concentrate on the use of biodiesel fuels rather than petroleum-based diesel fuel. The need for an alternative to diesel fuel has been fueled by the depletion of fossil fuel reserves and the possibility of becoming reliant on foreign energy sources (Yesilyurt and Cesur, 2020).

Biodiesels are bio-based diesels that are described as the ideal alternative fuel for diesel engines since they are environmentally friendly, biodegradable, have superior energy efficiency, and are practically acid-free (Igbum *et al.,* 2012). The efficiency of engine combustion, operation, and exhaust emission characteristics are all affected by the physicochemical qualities of seed oil (Rahman *et al.,* 2013).

The fatty acid composition and features of biodiesel are directly dependent on its chemical and physical properties, particularly cold flow behavior (Lanjekar and Deshmukh, 2016).

As a response to the dwindling fossil fuel supplies and the environmental harm resulting from their use, alternative energy sources derived from vegetable products are emerging. Because it is renewable, non-toxic, eco-friendly, etc., biodiesel has drawn the most interest among these various potential resources as a diesel engine fuel substitute. The present work is aimed at investigating the Biodiesel properties of *Caesalpinia pulcherrimma* seeds using Homogenous catalysts to determine their suitability for Industrial production. The chemical components of the resulting extract were analyzed using (GC-MS) for fatty acid profile.

MATERIALS AND METHOD

Sample Collection and Treatment

Pride of Barbados (*Caesalpinia pulcherrima*) seed was obtained from Samaru Market in Sabo Local government, Zaria, Nigeria. The sample was authenticated at the Department of Biological Sciences, Ahmadu Bello University, Zaria, Nigeria. The seed was subsequently powdered using a mortar and pestle then milled into a fine powder and stored in an airtight polythene bag prior to the extraction and analysis.

Extraction of Oils

Oils from the crushed seed were extracted using Soxhlet apparatus with n-hexane. The process was carried out by placing crushed powdered sample (200 g) in a thimble and inserting into a Soxhlet extraction chamber, 500 cm^3 of n-hexane was poured into the flat–bottom flask, and a condenser was mounted on it. The whole assembly was placed on a heating mantle and heated at 70 ^oC for six hours. The receiving flask was cooled and the thimble removed. The setup was reassembled for solvent recovery. The extraction was repeated until appreciable amount of oil from each of the samples was obtained. The solvent was recovered and oil content determined (Takadas and Doker, 2017).

Oil Yield

The extracted oil was transferred into a measuring cylinder which was placed over a water bath for 30 min at 70^oC to ensure complete evaporation of solvent and the volume of the oil was recorded and expressed as oil content (%) (Warra *et al.,* 2011). Weight times density were multiplied to obtain the oil's weight from its volume.

$$\text{Oil content} = \left(\frac{Weight\ of\ the\ oil}{Weight\ of\ sample}\right) \times 100$$

Physicochemical analysis of oil

Various physicochemical analyses were conducted to evaluate the quality of oil sample. The oil physicochemical properties were determined following standardized American Society of Testing and Materials (ASTM) test procedures. The physicochemical parameters analyzed were specific gravity, Iodine value, saponification value, acid value, peroxide value, color, and free fatty acid.

Specific gravity

A clean dry empty 50 cm^3 density bottle was weighed and the mass was recorded, it was filled up with distilled water and weighed, the mass of the bottle and water was taken (Onwuka, 2005). *Caesalpinia pulcherima* oil was also weighed in a separate density bottle which was weighed, the weight of bottle and oil, the specific gravity was evaluated using

$$\text{Specific gravity} = \left(\frac{Weight\ of\ oil}{Weight\ of\ Water}\right)$$

Saponification value

Exactly, 5 cm^3 of oil sample was weighed into a volumetric flask and 50 ml of 0.5N of alcoholic KOH was added from burette by allowing it to drain for 30 minutes and a blank was also prepared by taking only 50 cm^3 of 0.5N of alcoholic KOH allowing it to drain for 30 minutes. The flask was connected to the air condenser and boiled gently for about one hour. Then the flask and condenser were cooled, the condenser was rinsed with a little distilled water and then removed. Finally, 1 ml of phenolphthalein was added and titrated against 0.5 M of HCl until the pink color disappeared (AOAC, 2003).

$$\text{Saponification value} = \left(\frac{56 \times N(V_0 - V_1)}{W}\right)$$

Where; V_0 = the volume of the solution used for the blank test; V_1 = the volume of the solution used for determination; N = actual normality of the HCl used; W = Mass of the sample.

Acid value

Exactly, 2 cm^3 of oil sample was weighed in 250cm^3 conical flask separately, and each was dissolved in 25 cm^3 of alcohol. Then two drops of phenolphthalein indicator were added. The contents were titrated with 0.1N of alcoholic KOH. Blank titration was performed on 100 cm^3 of the titration solvent and 0.5 $cm^3$of the indicator solution, adding 0.1 cm^3 increments of the 0.1 M KOH solution. The KOH solution was standardized frequently to detect molarity change of 0.0005. The volume of 0.1cm^3 KOH (VA), for the sample titration, and volume for the blank (VB) was noted (AOAC, 2003)

Acid value = $(\frac{A \times M \times 56.1}{W})$

Where A = Amount (mL) of 0.1M KOH consumed by sample, M= Molarity of KOH, W= weight (g) of oil sample.

Peroxide value

Exactly 1.0 cm^3 of the seed oil was weighed into a clean dry boiling tube and 1g of powdered KI and 30 cm^3 of solvent (2 cm^3 of chloroform and 3cm^3 of glacial acetic acid) mixture was added. Hence, the tube was placed in boiling water so that the liquid boils within 30 seconds and allowed to boil vigorously for not more than 30 seconds. The contents were transferred quickly to a conical flask containing 20 cm^3 of 5% KI (5 g dissolved in 100cm^3 of H_2O) solution and the tube was washed twice with 25 cm^3 water each time and collected into the conical flask. Then, the solution was titrated against 0.001 M $Na_2S_2O_3$ solution until yellow color disappear and 0.5 cm^3 of starch was added with vigorous shaking and titrated carefully till the blue color disappear (AOAC, 2003).

Peroxide value =$(\frac{V \times M \times 1000}{W})$

V= Volume of sodium thiosulphate solution used, M= Molarity of thiosulphate, W=Weight of the oil sample.

Iodine value (IV)

Exactly 0.4 cm^3 of the *Caesalpinia pulcherima* oil sample was weighed into a conical flask and 20 cm^3 of CCl_4 was added to dissolve the oil. At the end of this period, 20 $cm^3$of 10% KI (10 g dissolved in 100 cm^3 of water) and 125 $cm^3$of distilled water were added using a measuring cylinder. The content was titrated with 0.1M sodium-thiosulphate solutions until the yellow color almost disappeared. A few drops of 1% starch indicator were added and the titration was continued by adding thiosulphate dropwise until blue coloration disappears after vigorous shaking. The same procedure was used for the blank test (AOAC, 2003).

Iodine value =$(\frac{12.69 \times C \times (V_0 - V_1)}{W})$

C = Concentration of sodium thiosulphate used, V_0 = Volume of sodium thiosulphate used for blank, V_1 = Volume of sodium thiosulphate used for determination, W =Mass of the sample.

Production of biodiesel using acid esterification

50 g of *Caesalpinia pulcherrima* oil was measured and weighed in a conical flask using a weighing balance. The conical flask containing the oil sample was placed on top of a magnetic stirrer, and then agitated and heated to a temperature of 60 ^{0}C. immediately after the agitation and heating started, 143.8 g of methanol and 3.2 g of sulphuric acid was added. The oil sample was esterified for five hours. The esterified biodiesel was tapped out, and the acid value was determined (Adepoju and Olawale, 2014).

Production of biodiesel using base transesterification

After the esterification reaction where the percentage of free fatty acid was reduced to a value less than 0.5%, further esterification was carried out. This was done by weighing 15% of methanol and 1% catalyst (KOH). Exactly, 40cm^3 of *Caesalpinia pulcherrima* oil was poured into a round-bottomed flask which was immersed in a water bath set at 55 °C and allowed to heat up until the temperature of the water bath was attained. 4 cm^3 anhydrous methanol was added into the flask and 0.4 g potassium hydroxide (KOH) was added to form methoxide; then condenser was fitted to the second neck of the flask; 0.4g of the KOH was dissolved in measured methanol and the mixture poured in to the oil. The solution was placed on a magnetic stirrer at 60^{0}C for agitation and heating for a period of 1 hour. The mixture was poured into a separating funnel after agitation, and allowed to stand for 1 hour for separation to take place. The lower layer (glycerol) was tapped off and the upper layer (biodiesel) was poured out. The volume and weight of obtained biodiesel were measured and recorded (Shikha and Chauhan, 2012).

Properties of Biodiesel

Density measurement (ASTM D445-12)

An empty beaker was weighed using an analytical weighing balance. 30cm3 of biodiesel was poured into a beaker, and the combined weight was measured. The difference between the weight of the beaker plus biodiesel and that of the empty beaker was obtained and recorded as the weight of the oil. The density was obtained by taking the ratio of the weight of the biodiesel and its volume (ASTM, 2010).

Density = $(\frac{Weight\ of\ measured\ biodiesel}{volume\ of\ measured\ biodiesel})$

Kinematic viscosity measurement (ASTM D445)

Exactly, 10 cm^3 of biodiesel was poured into a viscometer tube. The tube was immersed into a viscometer bath maintained at 40 °C. The oil in the tube was sucked up to the upper limit mark using a suction pump and allowed to drop under gravity. A stopwatch was started and the setup was monitored till the oil gets to the lower limit of the tube and the watch stopped. The time was recorded, and the procedure repeated twice. The kinematic viscosity was calculated using (ASTM, 2010)

Kinematic Viscosity = Time (s) × Tube constant
where, KV= kinematic viscosity, t = time in seconds, tube constant = 0.00768

Pour point (ASTM D6892-03)
Exactly 20 cm^3 of biodiesel was poured in a test jar with a thermometer clamped to it and was cooled at a constant temperature, forming wax crystals. The test jar was removed at every degree drop in temperature and tilted to check the surface movement. When the surface did not flow for 5 s, the temperature was recorded (ASTM, 2010).

RESULTS

The result of physicochemical properties of *Caesalpinia pulcherrima* seed oil studied was presented in Table 1. The percentage oil content of *Caesalpinia pulcherima* was 27.07 %. The oil content in this study was lower than the oil content of *C. pepo* (52.8%) reported by Bwade *et al* (2013) and higher than African locust bean seed (11.41%) as observed by Olowokere *et al.,* (2018). Oil yield can vary depending on species, environmental circumstances, particle size, and extraction process (Atta, 2003). This suggests that, in terms of production, *Caesalpinia pulcherrima* seed may not be a good oil feedstock for commercial use. Furthermore, *Caesalpinia pulcherrima* oil is dark brown in color and has a specific gravity of 0.90±0.00 which is lower than that of water, according to physical observations from this study. According to Birnin-Yauri and Garba, (2011), specific gravity is a critical quantity to assess since it determines the energy density (specific energy) of gasoline fuel using ASTM standard.

Acid values (AV) of 30.49±0.51 mgKOH/g obtained for *Caesalpinia pulcherrima* was higher than 7.09 mg KOH/g reported by Ottih *et al*., (2015) for *Hura crepitans* but lower than 36.2 mg reported for *Jatropha curcas* (Musa and Aberuagba, 2012). According to Ejilah *et al.,* (2012), the acid value indicates the extent of decomposition of the constituent glycerides by lipase activities. The acid value of the oil determines its, edibility, shelf life as well as industrial applications. High AV value renders the oil inedible, but useful for the production of paints, liquid soap, and shampoos (Aremu *et al.,* 2006).

The iodine value (IV) of 23.15±1.23 g/100g of oils extract of *Caesalpinia pulcherrima* plant gave lower values than *Cyperus esculentus* (71.82 g/100g) (Sidohounde *et al.,* 2018) and *Hura crepitans* (123.21 g/100g) (Sidohounde *et al.,* 2019), respectively. According to Jauro, and Adams, (2011) Oil classification depends on its Iodine values. Oil extracts with Iodine values less than 100 g/100g are non-drying, between 100-130 g/100g are semi-drying, and greater than 130 g/100g are considered drying respectively.

The saponification value (SV) of 142.87±1.45 mgKOH/g obtained from *Caesalpinia pulcherrima* was higher than 134.6 mgKOH/g reported by Jauro and Adams (2011) of *Balaenites aegyptiaca.* According to Ejilah *et al*., (2012), the Saponification value provides an index of the average molecular mass of fatty acids present in seed oil. The higher Saponification value implies higher molecular mass fatty acids. Oil extracts ranging from 130 to 193 mg KOH/g (Jauro, and Adams, 2011) and 220.19mgKOH/g (Ottih *et al.,* 2015) were considered suitable for use in biodiesel production. This is because higher saponification had been thought to improve the lubrication property of the oil, reduce engine wear, extend the operational life and efficiency of diesel fuel pumps and injectors (Ejilah *et al*., 2012)

In order to achieve the gasoline standard, viscosity is an important property that must be regulated in oil. The viscosity for *Caesalpinia pulcherrima* was 5.83±0.12 mm^2/sec, which was lower compared to the viscosity of *Cucurbita pepo* oil (93.65 mm^2/sec) according to Bwade *et al.* (2013). Low viscosity oil produced a sloppy effect (Yusup and Khan, 2010).

The peroxide value of *Caesalpinia pulcherrima* was 7.70 ± 0.06Meq/kg which is higher to Melon seed oil 5.63 Meq/kg as investigated by Oloefe et al (2012). This showed that the oil will have high level of deterioration when exposed to oxygen, since peroxide value indicated the level at which deterioration will take place as a result of oxidation owing to the availability of oxygen during storage (Atadashi, 2012).

TABLE 1: Physico-chemical properties of *Cesalpinia pulcherimma*

Properties	***Caesalpinia pulcherrima***
Specific gravity	0.90±0.00
Acid value (mg KOH/g)	30.49±0.51
Iodine value (g/100g)	23.15±1.23
Saponification Value (mg KOH/g)	142.87±1.45
Viscosity (mm^2/s)	5.83±0.12
Peroxide Value (Meq /Kg)	7.70 ± 0.21

Fuel properties

The fuel property of the biodiesel produced from *Caesalpinia pulcherrima* is shown in Table 2. The percentage biodiesel yield was recorded to be 71.37 ± 0.06 %., which was lower than the the percentage yield of biodiesel produced from the oilseeds of *Lagenaria Vulgaris* with 96.52% (Sokoto e*t al.,* 2013).

The specific gravity of the *Caesalpinia pulcherrima* methyl ester was 0.86±0.0*0* g/cm3 this is lower than the specific gravity of biodiesel produced from *Lagenaria vulgaris* with specific gravity of 0.8879g/cm^3 (Sokoto e*t al.,* 2013). This showed that the biodiesel produced could be securely handled and stored. In the management and storage of fuels, it was a key physical property (Nurhazirah, 2013).

The pour point of liquid fuel is the lowest temperature at which it loses its flow qualities. For the cold flow process, pour point is a crucial characteristic (Nick and Greg, 2008). The pour point of the *Caesalpinia pulcherrima methyl was* -5.03±0.12 $^{o}C.$ This is lower than ASTM standard which is 2.9 to 6.0, this shows that it does not meet pour point standard for production of biodiesel.

The cloud point *of the Caesalpinia pulcherrima was* 5.83±0.05 $^{o}C.$ Cloud point test characterized low-temperature operability of diesel fuel*.* In general, a fuel with a high cloud point has limited utility in cold climes. Nonetheless, the results were within range of -3 to 12 which is within ASTM standards*.*

The most important methylester characteristic is Kinematic viscosity, which affects fuel injection and sprays atomization, particularly at low temperatures (Atabani *et al.,* 2012). The kinematic viscosity of the *Caesalpinia pulcherrima* methyl esters is 5.37±0.07 which is between 1.9 to 6.0 biodiesel specification for ASTM, this indicates the presence of short-chain unsaturated methyl acid fatty esters and was likely to produce less deposit when burnt in combustion engines (Nick and Greg, 2008).

TABLE 2: Properties of Biodiesel

Properties	***Caesalpinia pulcherrima***	***ASTM Standard***
Biodiesel yield (%)	71.37	
Specific gravity (g/cm^3)	0.86±0.00	
Pour point (^{0}C)	-5.03±0.12	2.9 – 6.0
Cloud Point (^{0}C)	5.83±0.05	- 3 to 12
Kinematic viscosity (@40^{0}C)	5.37±0.07	1.9 – 6.0

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