

Synthetic extraction and Characterization of Biodiesel from Luffa (*Luffa cylindrica*) Seed Oil

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Abstract: In this study, oil was extracted from luffa seed by solvent extraction method using N-hexane as solvent. Physiochemical properties of the oil were determined; these include moisture content, viscosity, density, specific gravity, smoke point, flash point, acid value, free fatty acid, saponification value, peroxide value and iodine value. The oil was then converted to biodiesel through a two-step transesterification process. Physiochemical properties of the biodiesel were determined and compared with biodiesel ASTM6751 standard. The obtained values for the physiochemical properties of the extracted oil are; 4.82% (moisture content), 5.6mm²/s at 40°C (viscosity), 0.91g/cm³ (density), 0.89 (specific gravity), 165°C (smoke point), 173°C (flash point), 11.05mgKOH/g (acid value), 5.525 (free fatty acid), 162mgKOH (saponification value), 292meq/kg (peroxide value) and 122% (iodine value) respectively. The properties of biodiesel determined were viscosity, specific gravity, density, cloud point, pour point, flash point, acid value, free fatty acid and cetane index. Their corresponding values are 7.0mm²/s at 40°C, 0.90, 0.96g/cm³, 10°C, -6°C, 136°C, 4.68mgKOH/g, 2.525 and 51.63 respectively. The biodiesel produced has the potential to be used to power diesel engines and be stable during storage as the high flash point of 136°C of the biodiesel would ensure safety during storage and prevent explosions. The biodiesel has a valid cetane number which ensures a good ignition quality of the fuel and is of importance in the satisfactory operation of a diesel engine.

1. INTRODUCTION

Luffa (*Luffa cylindrical L.*) is an annual climbing crop from the cucumber and marrow family grown for its multipurpose fruit and fibre in many tropical countries (Mazali and Alves, 2005). It is commonly referred to as a sponge gourd, *loofa*, vegetable sponge, bath sponge or dish cloth gourd. They can grow to a length of 15m and produce fruits of cylindrical, smooth, and dehiscent capsule; which are 50cm long and 10cm broad, with a characteristic fibrous mesocarp containing at least thirty seeds when matured (Achigan-Dako *et al.*, 2011; eFlores, 2014). Luffa requires a very long growing season and it is cold sensitive. The crop has a long history of cultivation in the tropical countries of Asia and Africa (Oboh and Aluyor, 2009). *Luffa Acutangula* (L.) and *Luffa Cylindrica* (L.) are the only domesticated species in the genus and most common and popular.

Luffa is primarily grown for its fibre production. The young fruits and leaves can be cooked as a vegetable or eaten fresh but it has to be picked before the fibrous vascular bundles harden and before purging compound develop. In India and China the fruits are used to prepare curry spice by peeling, slicing and frying. When the fruits mature it becomes fibrous; the fibre is used for scrubbing utensils and as a sponge for bathing

(especially in rural parts of western Nigeria). In central Africa, luffa fibre is used to brush clothes. It is also used

to make hats, insoles of shoes, car-wipers, mats, sandals and gloves. The fibre has shock and sound absorbing properties that can be used in helmets and armoured vehicles. Fungal biosorbents can be immobilized on cylindrical sponges made of luffa in order to absorb heavy metals from wastewaters, including those from olive oil meals. Luffa oil meal is suitable as a fertilizer according to Achidan-Dako *et al* (2011). It also has numerous uses in the field of medicine health but commonly used in treating and preventing colds, arthritis, muscle and chest pains. It can also help nursing mothers increase milk flow.

According to Elemo *et al* (2011), luffa seeds can be extracted for their oil which is rich in linoleic acid and has a high unsaturated: saturated fatty acid ratio. However luffa seeds and oil meal contain bitter substances that may be toxic to livestock.

A biofuel is a fuel that is produced through contemporary biological processes, such as agriculture and anaerobic digestion, rather than a fuel produced by geological processes such as those involved in the formation of fossil fuels, such as coal and petroleum, from prehistoric biological matter. Example of biofuel is bioethanol, biodiesel, green diesel and biogas. Biofuels are increasingly making headway into the energy sector, and the demand for them as alternatives to petroleum fuels is increasing, prompting the placements of policies in various countries and regions to promote adoption and utilization of biofuels (Sorda *et al.* 2010; Rajagopal and Plevin, 2013).

Though Nigeria is a major exporter of crude oil globally, with huge revenues accruing from oil sales; the country faces the rapid depletion of her crude oil reserves and many other challenges. These problems have given rise to the need to explore and adopt alternative sources such as the use of vegetable oils from underutilized seeds to produce economic, clean, renewable and environmentally friendly biofuel. Also, existing vegetable oil used in the production of biofuel are competing with local consumption, therefore there is a need to identify non-edible oils such as luffa seed oil which can be used in place of the edible vegetable oils. Production of biodiesel will lead to the following: availability of cheap renewable source of fuel on the farmstead to power vehicles and machines; non-pollution of the environment, as biofuels is clean and environmentally friendly; Increased efficiency and lifespan of diesel engines and dependency on petroleum fuels are reduced; also costs are reduced. Thus, this study is aimed at synthesis and characterization of biodiesel from luffa seed oil

2. METHODOLOGY

2.1 Sample preparation

Matured and dry luffa sponges utilized for this study were collected from Bosso, Minna, Niger State. The sponges were opened and dry seeds of about 30 to 50 seeds per sponge were shaken out and collected in a bowl. Defective seeds and dirt were removed. The dry seed has a black coat that is difficult to break, peel off or remove.



Plate I: Showing seeds after removal from pods

2.2 Determination of Moisture Content

An empty container/crucible was weighed (W_1) and then the cleaned seed was placed in the crucible and weighed (W_2). The crucible with the seed was placed in the Electric fan-circulated Oven with a set temperature of 110°C . Heating and weighing were carried out at intervals of 6 (six) hours until a constant weight was obtained (W_3). The percentage (%) moisture content was then calculated (wet basis) using the formula:

$$\frac{(W_2 - W_3)}{(W_2 - W_1)} \times 100$$

1

2.3 Oil Extraction Process

2.3.1 Determination of Oil Yield

The cleaned seeds were ground using an electric blender to give a green coloured powder with speckles of the black coat and weighed (W_1), then loaded into a pre-weighed stapled filter paper (W_2). It was then placed

into a soxhlet extractor set up with a reflux condenser and a round bottom flask containing n-hexane (to wash off the oil from the seeds), then placed on a heating mantle for eight (8) hours. The hexane extract was filtered and evaporated to give a thick mass of oil: the oil was poured into a beaker and then placed in an oven to evaporate the residual solvent. The Luffa seed oil obtained was kept in an air tight bottle for further analysis. The residual seed was air dried and weighed (W_3). The extraction process was carried out in batches and the combined oil extract was weighed and the percentage (%) yield was calculated using the formula:

$$\% \text{ oil yield} = \frac{(W_1 - W_2)}{W_1} \times 100$$

2.3.2 Solvent Recovery Process

After each batch of extraction, the samples were removed from the soxhlet extractor but the set was left the same. As soon as the heat from the heating mantle heated up the mixture to about 40°C , n-hexane solvent vapourized and condensed into the empty soxhlet extractor. The solvent continued to build up and it was collected in a separate flask just before it filled the soxhlet extractor up to the point where it was refluxed back into the round bottom flask containing solvent plus oil mixture. This process was repeated until the rate of drop of condensation of the solvent was less than one drop in three seconds.

2.4 Esterification of Oil

200ml of luffa seed oil was prepared in a measuring cylinder and transferred into a round bottom flask then preheated to about 50°C . 0.5% of acid catalyst of catalyst was completely dissolved in 40g of methanol and then mixed with the oil. The mixture was stirred using a magnetic heater for 50 minutes keeping the temperature steady at 50°C . Afterwards, the mixture was transferred into a separating funnel and kept for an hour at which two layers was formed; glycerol at the bottom and oil at the top (Plate II). The glycerol was decanted and the oil was repeatedly washed with distilled water until a glycerol free oil was left. The esterified oil was then placed in an oven to remove excess water.



Plate II: Esterified oil in separating funnel

2.5 Transesterification of Oil

200ml of the esterified oil was prepared in a beaker. 0.7g of potassium hydroxide (KOH) was measured and dissolved in 50ml of methanol and transferred into the

beaker containing the oil. The mixture was poured in a round bottom flask and set up with a magnetic stirrer – heater for 45 minutes at 50°C. Afterwards the mixture was then transferred into a separating funnel and kept for 2 hours at which glycerol separated by gravity (Plate III). The glycerol layer was carefully decanted at the bottom. This process was performed in three separate batches A, B and C.



Plate III: Biodiesel in separating funnel

2.6 Characterization of Luffa Seed Oil and Biodiesel

2.6.1 Determination of kinematic viscosity

The viscosity test was carried out with a viscometer. The viscometer was set at 60 revolution per minutes (rpm) and rotor one (1) was used. 50ml of the sample was poured in a beaker which was placed on a heating mantle, a thermometer and the rotor extending from the viscometer was gently inserted inside the oil/biodiesel sample. The viscosity at room temperature (36.5°C) was first determined as well as the viscosity at 40°C and 100°C.

2.6.2. Determination of Specific Gravity

Specific gravity bottle of volume 50ml was used to determine the specific gravity of the oil/biodiesel. The empty bottle was first weighed (W_1). It was then filled with water and weighed again (W_2). The water was poured out and the dry bottle was refilled with oil and the weight was also taken and recorded as (W_3). The specific gravity is represented by the expression given in equation 3:

$$\text{Specific gravity} = \frac{W_3 - W_1}{W_2 - W_1} \quad 3$$

2.6.3. Determination of Density

The specific gravity bottle of volume 50ml was used. The empty bottle was first weighed and recorded as (W_1) and then filled up with oil/biodiesel and weighed again (W_2). The mass of the oil was then gotten by subtracting (W_1) from (W_2) and recorded as (M). The density is represented by the formula:

$$\text{Density} = \frac{M}{V} \quad 4$$

2.6.4 .Determination of flash point

Luffa oil/biodiesel was poured into a crucible and heat was gently applied at the bottom. A thermometer was

placed just at the surface of the oil with the aid of a retort stand to monitor the temperature of the oil. At regular intervals of 3 minutes a lighted matchstick was placed just above the surface of the sample. The temperature at which the oil/biodiesel ignites but not more than a couple of seconds was recorded as the flash point.

2.6.5 Determination of cloud point

Luffa oil/biodiesel sample was poured into a small crucible and was placed in a bowl filled with iced water. A thermometer was placed just at the surface of the sample with the aid of a retort stand to monitor the temperature of the oil. The temperature at which clouds begin to form at the surface of the oil/biodiesel was recorded as the cloud point.

2.6.6 Determination of pour point

Luffa oil/biodiesel sample was poured into a small crucible and was placed in a bowl filled with iced water. A thermometer was placed just at the surface of the sample with the aid of a retort stand to monitor the temperature of the sample. The temperature at which it does not flow or pour when the crucible was turned upside down was recorded as the pour point.

2.6.7 Determination of acid value and free fatty acid

20ml of Iso-propanol was prepared with three drops of phenolphthalein. 0.1mol of potassium hydroxide (KOH) was prepared by dissolving 0.56g of it in 100ml of water, and then used to neutralize the iso-propanol solution. 1g of oil was then added to the neutralized solution and then titrated with the 0.1mol of until there was a colour change which persisted for up to 15seconds. The titre value was then used to calculate the acid value using the formula:

$$\text{Acid value} = \frac{\text{Titre value} \times \text{conc. of molar test solution} \times \text{molar mass of KOH}}{\text{mass of oil}} \quad 5$$

Free fatty acid is simply acid value divided by two

$$\text{FFA} = \frac{\text{Acid value}}{2} \quad 6$$

2.6.8 Determination of saponification value

One gram of sample was weighed into a conical flask, 25ml of alcoholic KOH solution was added, a blank sample was also prepared. Afterwards the sample was covered to ensure the alcohol does not escape and placed inside the oven for 30 minute. A few drops of phenolphthalein was then added to the warm solution as an indicator and then titrated with 0.5mol HCL to the end point until the pink colour of the indicator just disappeared. The same procedure was conducted on the blank sample. The expression for saponification value (s.v) is:

$$\text{Saponification Value} = \frac{(B-a) \times N \times 56.10}{\text{Weight of sample (g)}} \quad 7$$

where, 'B' is blank and 'a' is titre value

2.6.9 Determination of iodine value

0.3g of the sample was weighed into a conical flask and 20ml of carbon tetroxosulphate was added to dissolve the oil/biodiesel sample. 25ml of Dam's reagent was also added to the flask using a safety pipette in fume chamber. Stopper was then inserted and content of the conical flask was vigorously swirled. The flask was then placed in the dark for 150 minutes. Afterwards, 20ml of 10% aqueous potassium iodide and 125ml of water added using a measuring cylinder which gives a yellow colour. The whole content was the titrated with 0.1mol of sodium thiosulphate solution until the yellow colour almost disappeared. 3 drops of 1% starch indicator was added (blue colour observed) and the titrated again with the sodium thiosulphate drop wise until blue coloration disappeared after vigorous shaking. A blank sample was also prepared using the same procedure. The iodine value (iv) is given by the expression:

$$i.v = \frac{12.69 (b-a)}{w} \quad 8$$

Where,

a = volume of sodium thiosulphate used for sample
 b = volume of sodium thiosulphate used for blanks
 c = concentration of sodium thiosulphate used
 w = mass of the samples

3. RESULTS AND DISCUSSION

3.1 Results

The results obtained from the study are presented in Tables 1 to 3. The oil extraction process was done in six (6) batches. Total weight of grounded seed before extraction was about 660g. Total weight of grounded seed after extraction was about 488g.

TABLE 1: PHYSIOCHEMICAL PROPERTIES OF *LUFFA CYLINDRICA* SEED OIL

Properties	Values Obtained
Colour	Dark green
Moisture content (%)	4.82 ± 0.31
Oil yield (%)	26 ± 0.64
Viscosity at 40°C (mm ² /s)	5.6±0.169967
Specific gravity	0.89±0.004714
Density	0.91±0.009428
Smoke point (°C)	165±1.247219
Flash point (°C)	173±1.184552
Acid value (mg KOH/g)	11.05±0.134484
Free fatty acid	5.525
Saponification value (mgKOH)	162±1.632993
Peroxide value (meq/kg)	292±1.632993
Iodine Value (%)	122±2.357023

TABLE 2: PERCENTAGE YIELDS OF THE DIFFERENT BATCHES OF BIODIESEL

Sample	%Yield
A	63.5
B	60
C	61

Production was done in three batches. Reaction Time was 50 minutes for each batch. Settling time was about 2 hours. Volume of methanol used = 4(oil): (methanol).

Table 3: Comparison of the results of the luffa biodiesel with the ASTM standard for diesel and biodiesel.

Properties	Luffa Biodiesel	ASTM D975 Petroleum Diesel	ASTM D6751 Biodiesel
Colour	Dark green	-	-
Viscosity at 40°C (mm ² /s)	7.0±0.525	1.9 – 4.1	1.9 - 6.0
Specific gravity	0.90±0.012	0.848	0.87 - 0.98
Cloud point	10±1.247	-15 to 5	-3 to 12
Pour point	-6±1.633	-35 to -15	-15 to 16
Density	0.96±0.012	-	0.86 – 0.98
Flash point (°C)	136±1.247	60 – 80	100 – 170
Acid value (mg KOH/g)	4.68±0.151	-	2
Free fatty acid	2.525	-	-
Cetane Number	51.630	-	47 – 65

3.2 Discussion of Results

3.2.1 Luffa seed oil

The results of the characterization of extracted luffa seed oil by solvent method of extraction are presented in Table .1. Oil yield of 26% was obtained. This low yield percentage may be due to the fact that manual removal of the seed coat was an impossible task, and so the seed was grinded together with the seed coat. However, the percentage yield compared favourably with oil content of seeds such as soya bean (11 - 25%) and castor oil (30 – 55%) which are commonly extracted by solvent method of extraction. Also the percentage yield agrees with the work of Barnwal and Sharma (2005) who reported a yield of 19 – 25% for luffa oil. Viscosity at 40°C was gotten to be 5.7mm²/s; this value is lower than the value of 43.4mm²/s at 40°C reported by Oniya (2010). Specific gravity and density values were computed to be 0.90 and 0.90g/cm³ respectively. These values compared favourably with the work of Barnwal and Sharma (2005) who reported a specific gravity and density values of luffa oil to be 0.92 and 0.91g/cm³ respectively; Oniya (2010) also reported a value of 0.88 and 0.88g/cm³ for specific gravity. Acid value and free fatty acid value result were obtained to be 11.05mgKOH/g and 5.525 respectively. The acid value is higher than the minimum standard of 2.0 set by the American Society for Testing and Materials (ASTM) 6751, there was therefore a need to esterify the oil before production of the biodiesel to reduce the acid

value to a minimum. The saponification, peroxide and iodine value obtained were 162mgKOH, 292meq/kg and 122% respectively. These values agrees with the work of Barnwal and Sharma (2005) who reported values for saponification, peroxide and iodine value of loofa oil to be 168mgKOH, 280meq/kg and 130% respectively.

3.2.2. Biodiesel

The results of the characterization of the biodiesel produced by the process of transesterification are tabulated in Tables 2 and 3. The methyl ester was produced in three (3) batches and an average yield of 61.5% was obtained. Other researchers have reported a higher yield of biodiesel such as the work of Oniya (2010) with loofah ethyl ester yield of 80%. Alamu *et al.*, (2007) worked on palm kernel oil and reported a yield of 95.80% ester and Obibuzor *et al.*, (2003) reported a yield of 88 – 97% ester from the transesterification of *raphia hookeri* mesocarp oil.

Viscosity result at 40°C was obtained to be 7.0mm²/s. This value is slightly higher than the maximum set value of 6.0mm²/s by ASTM6751 standard. However, it is significantly lower when compared with the work of Oniya (2010) who reported a value of 25mm²/s at 40°C. Also there was an increment of 22% when compared with viscosity of luffa oil (5.7mm²/s) and that of luffa methyl ester (7.0mm²/s) which does not follow the trend reported by other researchers such as palm kernel ethyl ester, rapeseed ethyl ester, loofah ethyl ester that had 85.05%, 84.38%, 42.395 decrease respectively.

Specific gravity value of 0.90 was obtained for luffa oil methyl ester. This value is close to the work of Oniya (2010) who reported a loofah ethyl ester specific gravity value of 0.88 and jatropha methyl ester specific gravity value of 0.88, and falls within the range of 0.87 – 0.98 set by ASTM6751 biodiesel standard. Also the low specific gravity value indicates a potential for good ignition property as explained by Clark (1988). Density was gotten to be 0.95g/cm³. This value falls within the range of 0.86 – 0.98g/cm³ specified by ASTM6751 standard.

Cloud point, pour point and flash point values obtained from the study were gotten to be 10°C, -6°C and 136°C respectively. These values are all in the standard range of biodiesel set by ASTM6751 biodiesel standard for cloud point (-3°C to 12°C), pour point (-15°C to 16°C) and flash point (100°C to 170°C). However, these values differ significantly for pour point and flash point but slightly for cloud point when compared with the work of Oniya (2010) who reported a cloud point, pour point and flash point of 7°C, 3°C and 86°C respectively for loofah ethyl ester.

Free fatty acid obtained was 2.53. This value is slightly higher than the recommended value of 2.0 set by ASTM6751 biodiesel standard. Also when compared with that of the luffa oil of 5.525 it is significantly lower. Similarly, this value is also lower when compared with the work of Oniya (2010) who reported a value of 5.60.

Cetane number of the luffa methyl ester was obtained to be 51.63. This value falls within the range of 47 to 65 set by ASTM6751 biodiesel standard. Similarly, it compares favourably with the findings of loofah ethyl

ester of cetane number of 51.3 as reported by Oniya (2010), close to Bamgboye and Hansen (2008) and Moreno *et al.*, (1999) for esters of soybean oil (45 to 60), rapeseed oil (44 to 59), sunflower oil (50 to 61.2), peanut oil (54) and cottonseed oil (45 to 55).

4. CONCLUSION

Oil was extracted from luffa seed and biodiesel was obtained from the oil. The obtained oil is stable during storage and has the potential to be used for powering diesel engines. The valid cetane number gave assurance of its good ignition properties and satisfactory operation of a diesel engine. Considerably high yield (61.5%) of biodiesel from the luffa seed oil showed that it is viable for biodiesel production. This also manifest from the obtained values for its physicochemical properties as it compared favourably with other seed oil for biodiesel.

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