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ASSESSMENT OF THE CONVERSION OF LUFFA CYLINDRICAL SEED OIL INTO BIODIESEL USING CAO/METAKAOLIN AS HETEROGENOUS CATALYST

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Abstract

The efficiency of calcium oxide supported metakaolin catalyst in the transesterification of Luffa cylindrical seed oil into biodiesel was studied using standard analytical techniques. Some physicochemical properties of the seed oil were determined and the fuel quality parameters of its fatty acid methyl ester (Biodiesel) were investigated. The results obtained were: colour (reddish-brown), yield (39.23 %), specific gravity (0.90 g/cm^3), kinematic viscosity (17.23 mm^2/s), pH (4.72), refractive index (1.466), acid value (22.58 mgKOH/g), free fatty acid (11.29 mgKOH/g), saponification value (115.005 mgKOH/g), iodine value (39.300 mgI$_2$/100 g) while the biodiesel properties were: colour (reddish-brown), biodiesel yield (92.29 %), API gravity (22^o), refractive index (1.470), specific gravity (0.880 g/cm^3), kinematic viscosity (5.62 mm^2/s), acid value (4.21 mgKOH/g), cloud point (7 ^{o}C), pour point (4 ^{o}C) and flash point (170 ^{o}C). The values obtained indicates that the oil may not be suitable for edible purposes due to high acid value but may be employed industrially in the manufacturing of products such as liquid soaps, paints, shampoos and biodiesel. CaO/Kaolin catalyst was found to be effective in the transesterification of the seed oil and the fatty acid methyl ester (FAMEs) produced was also found to meet most of the standards for biodiesel set by ASTM (American Society for Testing and Materials) and EN (European Norm).

***Keywords**: Luffa cylindrical, calcium oxide, kaolin, transesterification, biodiesel, ASTM, EN*

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INTRODUCTION

Energy is said to be a critical factor in the advancement of socioeconomic, political, educational and technological development of any nation. Its stability and uninterrupted supply are key to the attraction and retention of private sector in the economic growth of a country. In Nigeria for example, its inadequacy in supply has limits our economic prosperity, affect quality of life, and prevent direct foreign investments. This is because a number of services that promotes our standard of living are directly hinged on energy; hence their efficient performance can only be reached via adequate energy supply (Sokoto *et al*., 2011). The period of human exclusive reliance on nonrenewable resources will soon come to past in this century. Besides the sustainability challenge, another driven force for paradigm shift from nonrenewable resources is the pollution. Hence there is the need for alternative feedstocks to substitute the nonrenewable resources (Rattan, 2017). The major problems associated with petroleum derived diesel are; depletion of natural resources, CO_2 and SO_2 emissions and contribution to photochemical smog. Biodiesel production is therefore a promising endeavor. As the name implies, biodiesel oil is a fuel derived from cultivated plant seed oil. Most studies on biodiesel emphasizes on vegetable oils from both edible and nonedible oils (Rattan, 2017). The essential component of vegetable oil is triglycerides, which are esters of glycerol having long- chain saturated and unsaturated fatty acids. The transformation of raw oil into biodiesel is usually done through transesterification process. The oil is treated with a low molecular weight alcohol majorly methanol under the influence of a catalyst to form fatty ester and glycerol. The ester is then separated from the glycerol and used as biodiesel, while the glycerol is used as a raw material for fine chemical production (Rattan, 2017). Presently, homogeneous catalysis is commonly mostly used in the transesterification of vegetable oil in biodiesel. This process is however, associated with some drawbacks that limit its application; this includes the use enormous quantity of unsustainable catalyst, waste treatment and purification of glycerol (Daud *et al*., 2015 Muhammad *et al*., 2015). The use of heterogeneous catalysts is now gaining attention and interest as they are being found to be environmentally safe and sustainable and reduce the cost of biodiesel production. In recent time, a number of findings have been carried out on biodiesel production using heterogeneous catalysts such as supported CaO (Kouzu and Hidaka, 2012; Yan *et al*., 2008), $(CH_3CH_2O)_2Ca$, MgO–functionalized mesoporous catalyst (Li and Rudolph, 2007), MgO loaded KOH (Il gen and Akin, 2009), zeolite (Bristo *et al*., 2007) and CaO supported kaolin (Muhammad et *al*., 2015). This study was conducted to assess the conversion of *Luffa cylindrical Linn*. seed oil into biodiesel using CaO/metakaolin as heterogeneous catalyst.

MATERIALS AND METHODS

Materials

Sampling and sample preparation: *Luffa cylindrica* (dry and matured) fruits were collected from small bushes around the walls fencing the Bosso Campus of the Federal University of Technology, Minna, Niger State. The sample was identified and authenticated by a Botanist at the Department of Biological Sciences, Federal University of Technology, Minna. The seeds were removed from the fruits by dissecting; defective seeds and dirt were separated out. The dry black coated seed were then sundried for one (1) week at ambient temperature for easy removal of shells and to prevent them from getting muddy. The seeds were dehulled mechanically to enable separation of seed coat from the cotyledon; the dehulled seeds were then further dried in the oven (60 ºC, 2 h 40 min) to reduce the moisture content and then ground into powder. Kaolin was obtained from a Kaolin Quarry site at Lemu Village in Gbako Local Government Area of Niger State. The kaolin was then ground into fine powder.

Experimental Methods

Preparation of Catalyst

Kaolin pre-treatment: Kaolin (~1000.0 g) was calcined (1000 °C, 5 h) in a muffle furnace. After cooling, the metakaolin was extracted with *n*-hexane using Soxhlet extractor (60 °C, 8 h), to remove soluble organic matter and dried (100 °C, 5 h).

Preparation of CaO Supported on metakaolin (CaO/metakaolin): Metakkaolin (40.0 g) was measured into a beaker containing Na_2CO_3 solution (1 M, 100 mL). The mixture was agitated for 30 min and $CaCl_2$ solution (1 M, 100 mL) was introduced slowly while stirring. The mixture was filtered and the residue ($CaCO_3$ on metakaolin) washed repeatedly with distilled water until the washed water became colourless, dried (110 °C, 5 h) and then calcined (780 °C, 8 h) (Muhammad *et al.,* 2015).

Oil Extraction: Oil from the powdered seed was extracted using Soxhlet extraction method with *n*-hexane (60 °C, 6 h) (Krishnan *et al*., 2015). The percentage oil yield was determined from **Equation 1** as shown below.

$$\text{Crude Lipid (\%)} = \frac{\text{mass of the extracted oil}}{\text{mass of the powdered sample}} \times 100 \quad \text{(I)}$$

Physicochemical Analysis of the Extracted Oil

The extracted oil was analysed for properties such as colour, pH, refractive index, kinematic viscosity, specific gravity, acid value, free fatty acids, iodine value and saponification values based on the methods adopted by Ogungbele and Omosola (2015) and AOAC (1975), respectively.

Biodiesel Production

Standard method was used in the production of biodiesel from *Luffa cyllindica* seed oil. Crude *Luffa cylindrica* seed oil (50.0 mL) in a 250 mL conical flask was preheated (60 ºC) while stirring gradually on a magnetic stirrer to properly homogenize the oil. The CPK (0.90 g) catalyst was mixed with methanol (27.0 mL) and then transferred into the oil. The mixture was then stirred at constant speed for a period of (60 min). At the end of the reaction time the mixture was then transferred into a separating funnel and allowed separate overnight under gravity the biodiesel at the uppermost layer was recovered leaving the spent catalyst and glycerol. The unrefined biodiesel in evaporating dish was heated on a water bath (90 ºC, 30 min) to remove remaining methanol. Further purification of the biodiesel was done by neutralizing with dilute phosphoric acid and washing with hot distilled water to remove residual acid and glycerol until the washed water had a pH of 7.0 residual water was removed by drying at 100 ºC over anhydrous Na_2SO_4 (Muhammad *et al.,* 2015). Biodiesel yield was calculated as shown from the equation below.

Biodiesel yield = $\frac{wieght\ of\ biodiesel}{wieght\ of\ oil} \times 100$……………………….……..………(II)

Determination of Fuel Properties of Biodiesel from *Luffa cylindrica* Seed Oil

The fuel properties of biodiesel produced was determined by Ogungbele and Omosola (2015), AOAC (1975), Ogungbele and Omosola (2015) and AOAC (1975), respectively. These properties include colour, refractive index, kinematic viscosity, specific gravity, acid value, flash point, cloud point and pour points.

RESULTS AND DISCUSSION

Results

Table 1: Physicochemical parameters of *Luffa cylindrica* seed oil

Parameters	Values
Percentage yield (%)	39.23
Moisture (%)	2.1
Colour	Reddish-brown
pH	4.72
Relative Density (g/cm^3)	0.90
Kinematic Viscosity 40ºC (mm^2/s)	17.23
Refractive Index 30ºC	1.466
Acid Value (mgKOH/g)	22.58
Free Fatty Acid (%)	11.29
Iodine Value (mgI$_2$/100g)	39.300
Saponification value (mgKOH/g)	115.005

Table 2: Fuel quality parameters of biodiesel produced from *Luffa cylindrical* seed oil

Parameters	Results
Percentage Biodiesel Yield (%)	92.12
API Gravity (º)	22
Colour	Reddish-brown
Relative Density (g/cm^3)	0.88
Kinematic Viscosity 40ºC (mm^2/s)	5.62
Refractive Index 30ºC	1.470
Acid Value (mgKOH/g)	4.21
Flash Point (ºC)	170
Cloud Point (ºC)	7
Pour Point (ºC)	4

Table 3: Comparison of fuel quality properties of *Luffa cylindrica* seed oil biodiesel with international biodiesel and petrodiesel (ASTM & EN Standards)

Parameters	***L. cylindrica* Biodiesel**	**ASTM Biodiesel Standard**	**EN Biodiesel Standard**	**Petro-diesel**
Colour	Reddish brown	-	-	-
Refractive Index	1470	1.245-1.675	-	1.48
Relative Density (g/cm^3)	0.88	0.86-0.90	0.86 – 0.90	0.85
Kinematic Viscosity @ 40ºC (mm^2/s)	5.62	1.9-6.0	3.50.5.00	1.3-4.1
Acid Value (mgKOH/g)	4.21	0.80 max	0.60	-
Flash Point (ºC)	170	130 min	120	60-80
Cloud Point (ºC)	7	-3 to 12	-	-16
Pour Point (ºC)	4	-15-to-10	-	-12

DISCUSSION

Physcochemical Parameters of *Luffa cylindrica* Seed Oil

The results of some physicochemical properties of *Luffa cylindrica* seed oil are presented in **Table 1.**

Percentage Yield

The oil yield of *Luffa cylindrica* seed was 39.23 %. This was quite high but comparable with 36.32 % as reported by Ozulu (2015). The result of the oil yield showed the seed is economical for biodiesel production.

Moisture Content

The moisture content of *luffa cylindrica* seed obtained was 2.1 %. Moisture when present in any material degrades such material. Excessive seed moisture leads to reduced extraction efficiency, seed cake storage problems and high oil moisture contents. Moisture alongside FFA are key properties in regulating the viability of oils for transesterification. Water causes hydrolysis of some of the ester produced with resulting soap formation. Saponification decreases catalyst concentration, increases viscosity which promotes gel formation making it difficult to separate glycerol (Peterson *et al.,* 1993; Hanna *et al*., 2005; Abayeh *et al.,* 2012).

Colour

The colour of the oil was reddish-brown in nature.

pH

The oil sample was found to have a pH of 4.72. The pH value of *Luffa cylindrica* seed oil was lower compared to 5.59 obtained by (Ibeto *et al*., 2012).

It shows a relatively high acidity which was in correlation with the high acid value obtained and acid value is an indicator of inedibility of oil and its suitability for industrial applications (Abayeh *et a*l., 2013).

Relative Density

The oil sample density was 0.90 g/cm^3 indicating that the oil was less dense than water. Density determines the energy content of the oil, denser oils burn slowly and gives less energy compared to less dense oils (Peterson *et al*, 1993; Abayeh *et al*., 2012). The value obtained for *Luffa cylindrica* oil indicates the oil could serve as a viable feedstock for biodiesel.

Kinematic Viscosity

Kinematic viscosity refers to the thickness of the oil. It is an important parameter in checking the viability of a feedstock for transesterification into biodiesel. Results obtained for this study indicates that the kinematic viscosity of *Luffa cylindrica* seed oil was 17.23 mm^2/s. Result is higher than 15.30 mm^2/s obtained by Ozulu (2015) but much lower compared to *Jatropha curcas* oil 34.2 mm^2/s (Edward, 2010) and neem oil 44.00 mm^2/s (Sekhar *et al*., 2009). The purpose of transesterifying fats and oils into their alkyl esters is to reduce viscosity because high viscosity leads to formation of deposits on injector which can cause incomplete combustion and increase exhaust smoke emissions (Ibeto *et al*., 2012).

Refractive Index

The refractive index is a dimensionless parameter commonly used to determine the purity and level of contamination in liquid samples. Refractive index of *luffa cylindrica* seed oil at 30 ^oC was 1.466. This value was comparable to 1.465 reported by Ozulu (2015) and *Jatropha curcas* oil 1.465 (Edward, 2010). Results obtained for this study indicates it is within ASTM specification (1.245-1.675).

Acid Value and Free Fatty Acid (%)

Acid value measures the extent of corrosive free fatty acids and oxidation products. This is an important variable in considering the quality of oil for biodiesel production because the lower the value free fatty acid, the better the quality of oil (Bailey, 1982). The acid value *of Luffa cylindrica* seed oil was 22.58 mgKOH/g. The acid value was higher 34.15 mgKOH/g as reported by Ozulu (2015). But much lower compared to *Jatropha curcas* oil 9.52 mgKOH/g (Edward, 2010). The high acid value shown by *Luffa cylindrica* seed oil indicates that the oil may not be suitable as edible oil, but rather may be useful industrially for the soaps, surface coating and biodiesel production industries. The percentage free fatty acids concentration of *Luffa cylindrica* seed oil 11.29% was higher than the maximum limit of 2.0 % (Codex Alimentairus Commission, 1993). Vegetable oils containing high free fatty acids have significant effects on the transesterification with methanol using alkaline catalyst. It impedes the separation of fatty acid ester from glycerol's leading to significant reduction in biodiesel yield (Ma and Marcus, 1999).

Iodine Value

Iodine value is used to measures the degree of unsaturation in fat and oils. High iodine value of oil and fat indicates high unsaturation and hence susceptibility to oxidative rancidity. The iodine value of *Luffa cylindrica* seed oil was 39.300 mgI_2/100g, this means the oil belongs to a class of non-drying oil. A non-drying oil has iodine value lower than 100 (Asuquo *et al*., 2012). The value obtained in this present study was much lower compared to *Luffa cylindrica* 86.29 mgI_2/100g as reported by Ozulu (2015) and *Jatropha curcas* oil 93.0 mgI_2/100g (Edward, 2010). This means that the oil cannot be stored for a longer period of time. (Asuquo *et al.,* 2012).

Saponification Value

Saponification value is a measure of the average molecular weight of the triacylglycerol (all fatty acids present) in the oil sample. The saponification value *of Luffa cylindrica* seed oil was 115.005 mgKOH/g. The saponification value obtained for *Luffa cylindrica* seed oil is lower than similar nonedible seeds such as *Jatropha. carcus* 202.60 mgKOH/g (Edward, 2010), linseed oil 195 mgKOH/g (Singh and Siroj, 2009), but higher than that of Pumkin seed oil 44.88 mgKOH/g, African bean oil (28.05 mgKOH/g) and Sesame oil (98.56 mgKOH/g) (Eze, 2012). The high saponification value indicates the presence of high percentage of free fatty acids (triacylglycerol) in the oil and therefore, implies the possible tendency of soap formation and difficulties in separation of products (glycerol and mono alkyl ester). This means it could be useful in soap making (Abayeh *et al.,* 2012).

Fuel Quality Parameters of Biodiesel Produced from *Luffa cylindrica* Seed Oil

Some fuel quality properties of biodiesel produced from Luffa cylindrica seed oil is appears as depicted in **Table 4.2.**

Percentage Biodiesel Yield

The fatty acid methyl ester yield of *Luffa cylindrica* seed oil obtained was 92.12% which is slightly higher compared with 92.06% obtained by Ozulu (2015). Biodiesel yield was higher when matched with *Jatropha carcus* FAME 80.2% (Adebayo *et al*., 2011) and soybean fatty acid ethyl esters (FAEE) 71.2 % (Hossain *et al.,* 2010). The high percentage yield of biodiesel implies that CaO/Kaolin was effective in the transesterifying *Luffa cylindrica* seed oil into biodiesel.

Colour

Biodiesel showed a reddish-brown coloration. The colour of biodiesel has no effect on its performance.

Relative Density

The relative density of *Luffa cylindrica* seed oil biodiesel (0.88 g/cm^3) is within the specified value set by ASTM (0.86 - 0.90) for biodiesel. The relative density is the ratio of the density of a fluid to the density of water. Density is a critical property of biodiesel on the grounds that fuel injection pump equipment operates on a volume metering framework. Consequently, a higher density of biodiesel results in the delivery of a slightly higher mass of fuel. Along this line, changes in the fuel density will influence engine output power due to a different mass of fuel injected (Ozulu, 2015).

Kinematic Viscosity

The kinematic viscosity of *Luffa cylindrica* seed biodiesel was 5.62 mm^2/s observed to be within the limits set by ASTM D6751 (1.9 - 6.0 mm^2/s) biodiesel standard. The conversion of *Luffa cylindrica* seed oil into biodiesel decreased the viscosity of the oil from 17.23 to 5.62 mm^2/s. The kinematic viscosity measures the resistance to flow of a fluid under gravity. Viscosity is the most essential property of biodiesel since it influences the smooth operation ability of fuel injector system, especially at low temperatures when the increase in viscosity affects the fluidity of the fuel. High viscosity prompts incomplete atomization of the fuel spray and less precise operation of the fuel injectors (Ozulu, 2015).

Refractive Index

The refractive index *of Luffa cylindrica* seed biodiesel was 1.470 which conforms to the ASTM value of 1.479 maximum. It is lower than the refractive index of petrodiesel (1.483). The refractive index which is the proportion of the speed of light in vacuum to the speed of light in a medium it is an indication of the level of saturation of the biodiesel (Oderinde *et al.,* 2009). As chain length of fatty acid and level of unsaturation builds up, the refractive index increases. Refractive index is broadly used in quality control to check for the purity and contamination of greasy materials (Hoffmann, 1986).

Acid/Free Fatty Acid Value

The acid value of *Luffa cylindrica* seed oil biodiesel was 4.21 mgKOH/g. It is slightly higher than 0.52 mgKOH/g obtained by Ozulu. (2015), sunflower oil 0.40 mgKOH/g and jatropha oil 0.4 mgKOH/g. Acid value estimates the corrosive tendencies of the fuel in the injector system of diesel engines (Wilson, 2010). Free fatty acid content present in triglycerides (fat or oil) is also one of the major difficulties in biodiesel production. High FFA content in oil when using homogeneous alkali catalyst will lead to soap formation. This side reaction will consequently lead to low biodiesel yield. Heterogeneous catalyst such as CaO/kaolin used in this work will not negatively affect biodiesel yield because they are capable of esterifying the FFA since the catalyst is in different phase with no occurrence of side reactions (Tan *et al*., 2011).

Flash Point

Flash point of a fuel is the temperature at which it ignites when exposed to a flame or spark. The flash points of *Luffa cylindrica* seed oil biodiesel (170°C) was observed to be above the minimum value (120 °C) of the EN 14214 biodiesel fuel standard and the minimum value (130 °C) of the ASTM D6751 biodiesel fuel standard. This value was however, higher than that of neem seed oil biodiesel (120 °C) and more importantly, extremely higher than that of petrodiesel (74 °C). The relatively higher flash point value of *Luffa cylindrica* seed biodiesel indicates that it is free from residual methanol contaminants which would have lowered the flash point and it is plays a crucial significance for storage and transportation safety of the fuel (Ozulu, 2015).

Cloud Point

The cloud point is a low temperature operability property of fuels. Value obtained *Luffa cylindrica* seed biodiesel determined was

7 ºC. The cloud point of *Luffa cylindrica* seed biodiesel was higher than that specified by ASTM D6751 (-1 °C maximum) biodiesel fuel standard and also, higher than petrodiesel (-16 °C). The pour point of *Luffa cylindrica* seed biodiesel is higher those stipulated by ASTM D97 (-15 °C to -10 °C) for biodiesel and that of petrodiesel (-12 °C). Since the biodiesel produced from *Luffa cylindrica* seed has high cloud point and pour point, it will possess poor cold flow characteristics in extremely cold weather. The cloud point is the temperature at which the first solid (cloud) become noticeable when cooling the diesel fuel (Encinar, 2005; Ozulu, 2015).

Pour Point

The pour point is also a low temperature operability property of fuel. Result determined for *Luffa cylindrica* seed biodiesel showed 4 °C, respectively. The pour point of *Luffa cylindrica* seed biodiesel is higher than those stipulated by ASTM D97 (-15 °C to -10 °C) for biodiesel and that of petrodiesel (-12 °C). Since the biodiesel produced from *Luffa cylindrica* seed has high cloud point and pour point, it will possess poor cold flow characteristics in extremely cold weather. The pour point is the temperature at which the fuel ceases to flow. These parameters are related to the cold engine start and should be sufficiently low since when the biodiesel freezes, the engine won't start (Encinar, 2005; Ozulu, 2015).

CONCLUSION

In this present study, the physiochemical characterizations of *Luffa cylindrica* oil fatty acid methyl ester has highlighted its potentials as a very useful resource for biodiesel production. The oil yield of the seed which was quite high when compared with other oil yielding seeds can be advantageous when used in the production of biodiesel. *Luffa cylindrica* seed oil has high saponification value which shows it could serve as a valuable feedstock for the oleo chemical industries. Other features of the oil investigated such as relative density, kinematic viscosity, refractive index fell within minimum limits while higher value of acid value, pH and percentage free fatty acid indicates oil may be inedible but may be suitable for industrial applications like biodiesel production. Low iodine value obtained indicates lower degree of unsaturation of oil and can act as a feedstock in the production of biodiesel. Some of the fuel properties explored had shown, to a reasonable extent, that quality biodiesel can be produced from *Luffa cylindrica* seed. Henceforth, its full potential can be harnessed by improved cultivation. Furthermore, CaO/Kaolin used for the conversion of *Luffa cylindrica* oil into biodiesel. Properties of biodiesel produced using the catalyst was found to conform to majority of the ASTM and E N specifications for biodiesel. In general, CaO/kaolin has been observed to be a promising catalyst for heterogeneous catalyst of transesterification of vegetable oils into biodiesel.

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