Potential of Magnetic Sand as Catalyst for the Heterogeneous Transesterification of Luffa (*Luffa cylindrica*) Seed Oil to Biodiesel

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Authors' contributions

This work was carried out in collaboration between all authors. Author FJT designed the study, performed the statistical analysis, wrote the protocol and wrote the first draft of the manuscript. Authors JTB and MEK managed the analyses of the study. Author AA managed the literature searches. All authors read and approved the final manuscript.

ABSTRACT

The catalytic activity of crude Magnetic sand obtained from Michika Local Government Area of Adamawa State, Nigeria, in converting luffa oil to its methylester was investigated. The X-ray diffraction spectra have confirmed the sample to be crystalline phase of magnetite Fe_2O_3. X-ray fluorescent analysis revealed that the sample contains several impurities in various minute quantities which act as dopants substituting some parts of Fe in the spinel structure without forming any other individual phase. The crystallite size was found to be 78.10 nm. Fe_3O_4 was used as heterogeneous catalyst for the conversion of Luffa oil to its methyl ester. The effect of methanol/oil molar ratio, reaction time, catalyst amount and reaction temperature were investigated. The optimum yield of 34.1% of Luffa methyl ester was obtained at 60°C (temperature), 15:1 methanol/oil molar ratio, 5 h reaction time and 5wt% catalyst amount. Fuel properties of Luffa

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methyl ester were also investigated; Viscosity at 40°C is 6.7mm^2/s, Flash point 81°C, Pour point 4°C, Ash content 0.03%, Density 0.88 g/cm^3, and Specific gravity at 15°C being 0.82. Functional characteristics of Luffa methyl ester was obtained using Fourier transform infra- red spectroscopy (FT-IR). The fuel viscosity and flash points are indicative of the viability of this fuel in diesel engines.

1. INTRODUCTION

The search for an alternative energy source continues to increase owing to factors ranging from unsustainability, to negative environmental effects that are associated with petro based fuels [1]. Biofuels have over the years been an area of interest to many researchers due to their wider potentials as a future substitute to the conventional petro fuels [2]. Effort has been made in various capacities to utilize the available green resources that have the potential to be used in their crude or refined form as fuels.

Biodiesel obtained from vegetable oil has proven to be one of the frontiers of green energy sources that could be used directly or blended with petro diesel without major modification of the diesel engine because of their similar properties [3]. However, the cost of raw materials particularly vegetable oil and catalyst is one of the major impediments to the general commercialization of biodiesel in some parts of the world [4]. Vegetable oil utilized for this purpose could be edible or non-edible. Using edible oil for biodiesel production is bound to create competition with edible oil and this will ultimately lead to scarcity and high cost to consumers. Therefore, it is necessary to find new feedstock suitable for biodiesel production from non-edible oils [5]. Thus, the use of nonedible, luffa, which grows in tropical and subtropical cllimates across the developing world [6]. In Nigeria, it is known as '*soso*' in Hausa, '*kankan*' in Yoruba and '*asisa*' in Igbo languages [7].

However, Luffa oils have high Free Fatty Acid (FFA) content which can corrode engine parts if used directly. High viscosity associated with, also creates poor fuel atomization, incomplete combustion, carbon deposition on the injector and it builds up in the lubricant resulting in serious engine fouling [1] therefore the reduction or elimination of FFAs and improvement in viscosity of the bio-oil is necessary.

Demirbas [8], reported a satisfactory performance for several alcoholysis reactions achieved with calcium carbonate catalysts at higher temperatures, typically greater than 475 K. The selection of catalyst depends on the amount of free fatty acid (FFA) present in the oil. Generally, the catalysts are bases, acids, or enzymes. However pioneering work by [9], suggested that for triglycerides stock having lower amount of FFAs, base-catalyzed reaction gives a better conversion in a relatively short time while for higher FFAs containing stock, acid catalyzed esterification followed by transesterification is suitable.

Possible treatments employed to improve oil FFAs includes acid esterification followed by transesterification and direct transesterification using heterogeneous catalysts. Two steps transesterification processes are not cost effective compared to heterogeneously catalyzed processes which are reported to be much less dependent on FFA content of the oil and ultimately do not form soap [4,10].

On the basis of cost and availability of the vast deposit of untapped magnetic sand found in Michika Local Government Area of Adamawa State, North-Eastern Nigeria, the sands viability as heterogeneous catalyst in tranesterifying luffa seed oil was investigated.

2. MATERIALS AND METHODS

Five litres of Luffa seed oil was purchased from a commercial oil producer in Mubi metropolis of Adamawa State. Methanol 99% purity (analytical grade) was obtained from Northern scientific (Nigeria). 500 g magnetic sand was collected directly from its deposit with the aid of a magnet bar to minimize impurities. The samples were packed into plastic containers prior to analysis.

2.1 X-Ray Diffraction

The X-ray diffraction (XRD) characterization of magnetic sand was performed on an Empyrean (panalytical model) based generator X-ray diffractometer. The analyzed material was finely

ground to pass through 63 microns, homogenized, and average bulk composition was determined. 1 g of the powdered sample was then prepared using the sample preparation block and compressed in the flat sample holder to create a flat, smooth surface that was later mounted on the sample stage in the XRD cabinet.

The sample was analyzed using the reflection-transmission spinner stage using the Theta-Theta settings. 2θ starting position was 0.00483 and ends at 75.000 with a 2θ step of 0.026 at 3.57 seconds per step. Tube current was 40mA and the tension was 45VA. Fixed Divergent Slit size of 1° was used and the goinometer radius was 240 mm.

2.2 Crystallite Size Determination

Fityk 0.9.8 version software was used to obtain the Full Width at Half Maximum (FWHM). The average crystallite size of catalysts was calculated from the line broadening or FWHM of corresponding peaks (the most intense peaks), using Modified Scherrer formula which decreases errors by obtaining the average value of crystallite size through all the peaks (or any number of selected peaks) using least squares method [5]. The basic Scherrer formula is written as:

$$\beta = \frac{K\lambda}{L.Cos\theta} = \frac{K\lambda}{L} \cdot \frac{1}{Cos\theta} \qquad (1)$$

Where L is the crystallite size for (hkl) phase (nm), λ is the x-ray wavelength of radiation for Cuk_α, β_{hkl} is the Full Width at Half Maximum (FWHM) at (hkl) in radian and θ is the diffraction angle for (hkl) phase. $K = 0.89$ and λ ($\lambda Cuk\alpha 1 = 0.15405$ nm).

2.3 Analysis of Bulk Chemical Composition of Magnetic Sand Using X-Ray Fluorescence (XRF)

Magnetic sand sample was prepared by reducing the particle size to less than 63 microns using a Tema vibrating mill. This was done by the Agate Moter in the mill crushing the sample before sieving through 63 microns. Beads used for the major elemental analysis expressed in oxide weight percent were prepared by first drying 10 g of the sample powder in an oven at 110℃ for 24 h to remove the moisture in the sample. 5.0 g of dry sample powder was weighed in the silica crucible and then ignited in the furnace at 1000℃ for 2 to 3 h for the calcinations of

impurities in the sample. The sample was then removed from the furnace and allowed to cool to room temperature in desiccators. The ignited sample powder was then weighed again to determine the weight of the calcinated impurities which were H_2O and CO_2. 1.0 g of the stored ignited sample powder was weighed and exactly 5 times of flux [X-ray Flux-Type 66:34% (66.0% Lithium Tetraborate: 34% Lithium metaborate)] was added to lower the verification temperature of the sample powder. It was then mixed properly in a platinum dish and ignited in the pre-set furnace (Eggon 2 Automatic fuse bead maker) at 1500℃ for 10 minutes to form a glass bead. The glass bead was then slotted into the computerized XRF (Epsilon 5 Panalytical model) for major elemental analysis.

Trace elemental analysis was carried out using compressed powder pellets. These pellets were prepared by weighing 3.0 g of oven dried powdered sample and 3.0 g flux (cellulose-powder) added as a binder and dispersive agent, and shaking in small plastic containers for 12 minutes. The well mixed sample was then compressed by applying pressure of 1500 Kg/m^2 using both manual and electronic compressors. The pellets were placed in the computer-programmed XRF and the conditions for trace elemental set to give the result.

2.4 Catalyst Testing

The transesterification reaction was carried out in a 150 mL two-neck round bottomed flask equipped with an external rotary stirrer at one neck and a reflux condenser at the other. Various amount (1-5wt%) of magnetic sand was suspended in a required volume of methanol. The temperature of the mixture was controlled at varying degrees ranging from 45℃ to 60℃ by a water bath. Oil was then added into the mixture under vigorous stirring. The methanol/oil molar ratio was varied between 3:1 to 15:1. At the end of the reaction (1 to 5 h), the catalyst was separated via centrifugation and the reaction mixture was loaded into a rotary evaporator (NYC R-205D) at 35℃ to remove excess methanol. Since the transesterification of triglycerides can produce methyl ester and glycerol, the conversion of oil was calculated based on the amount of recovered methyl ester.

2.5 Biodiesel Characterization

Fuel properties such as viscosity, flash point, cloud point, specific gravity, acid value and free

fatty acid were determined according to American Society for Testing and Materials (ASTM) standards. The biodiesel product was analysed by Fourier Transform Infra-Red spectrometer (Nicolet 5700).

3. RESULTS AND DISCUSION

The XRF result of crude magnetic sand (Table 1) revealed that it contains 56.24% Fe_2O_3, 27.04% SiO_2, 6.3% Al_2O_3, 2.32%K_2O, 1.71%CaO and other impurities such as Na_2O and MnO in minute quantities. The result indicated that the catalyst used is virtually impure having its major component to be magnetite. CaO and K_2O have been reported to have catalytic property in converting vegetable oils to biodiesel [5]. Therefore the presence of these impurities could have positively altered the catalytic behavior of magnetic sand by acting as dopants substituting some part of the Fe in the structure without forming any other individual phase [11].

The XRD patterns of magnetic sand in the range of 210° to 105° are shown in Fig. 1. The lattice parameters including values of the distances between adjacent planes in the miller indices (hkl calculated from Braggs equation $\lambda=2dsin\theta$), lattice constants a, b, and c, interplanner angles and unit cell volumes are recorded in Table 2.

Table 1. XRF result of the chemical composition of magnetic sand (%)

Oxide	% composition
Fe_2O_3	56.24
SiO_2	27.04
Al_2O_3	6.30
TiO_2	2.58
K_2O	2.32
CaO	1.71
LOI	1.57
Na_2O	1.02
MnO	0.81
P_2O_5	0.41

X-ray diffractogram of crude magnetic sand gave intensified and narrowed peaks at 2θ of 30.02°, 35.36°, 56.82°, 62.59° for Fe_3O_4; 20.82°, 26.60°, 50.07° (JCPDS File No. 5-490) for SiO_2; 10.92°, 21.94°, 27.39°, 27.95° for CuS; 8.67°, 17.56°, and 26.78° (JCPDS File No. 2-462) for Crystalillite.

Crystallite size of catalyst was calculated from the line broadening (Fig. 1) of the most intense peaks. The crystallite size of catalyst was in agreement with the line widths of the peaks in which decrease of FWHM correspond to increment of the crystallite size. The average crystallite size of magnetic sand was found to be 78.10 nm.
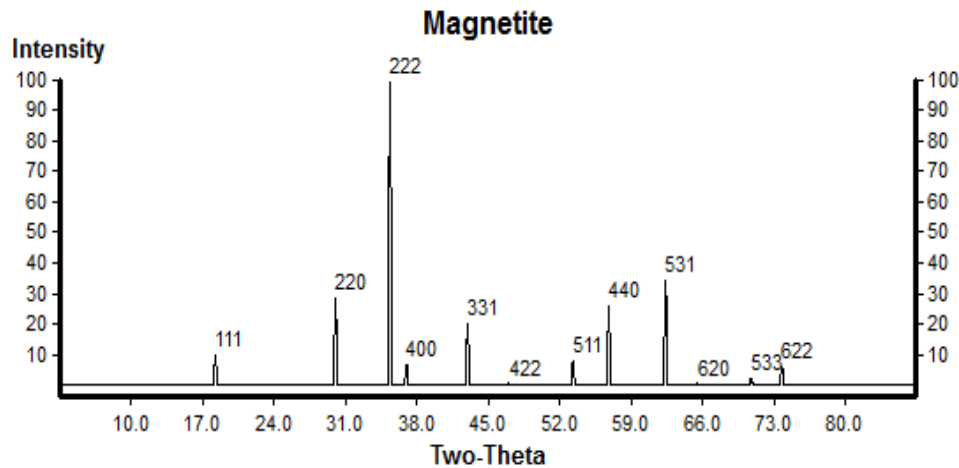

Fig. 1. XRD pattern of magnetic sand (magnetite)

Table 2. Values of FWHM (β) and the diffraction angle (θ) of the most intense peaks

hkl	d_{hkl}	β(rad)	lnβ(rad)	θ	cosθ	1/cosθ	ln(1/cosθ)
220	2.9743	0.00635	-5.0590	0.233	0.974	1.027	0.027
222	2.4285	0.00581	-5.1329	0.244	0.970	1.021	0.021
531	1.4220	0.0227	-3.7859	0.309	0.953	1.049	0.048

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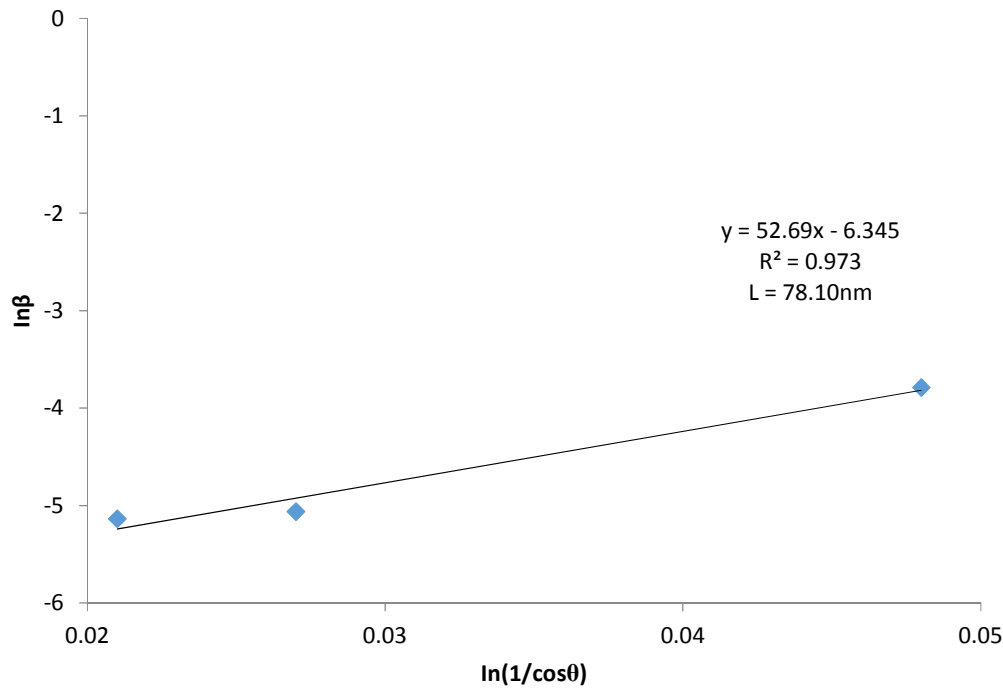
y = 52.69x - 6.345
R² = 0.973
L = 78.10nm

Fig. 2. Magnetic sand crystallite size

Table 3. The structure parameters of magnetic sand and magnesium oxide

Sample	2θ	hkl	d_{hkl} (A°)	Crystal system	Crystallite size (nm)	Lattice constants (A)	Interplaner angle (°)	Unit cell volume ($10^6 pm^3$)
Magnetic sand	30.02	(220)	2.974	Cubic	78.10	a = 8.4	α = 90	595.40
	36.98	(222)	2.428			b = 8.4	β = 90	
						c = 8.4	Υ = 90	

(hkl= Miller indices; d_{hkf}=d-spacing; A^0= Angstrom)

Fig. 3 shows the influence of magnetic sand catalyst on the conversion of triglycerides to methyl ester at reaction condition: 60℃, 5 h, 15:1 methanol/oil molar ratio. The catalyst amount was varied in the values of 1.0, 2.0, 3.0, 4.0 and 5.0 wt/wt% oil. At catalyst amount of 1wt%, 2wt% and 3wt% there was no any good methyl ester yield. However, a significant increase of up to 34.1% methyl ester yield was recorded at 5wt/wt% oil which in comparison is more than the yield (10%) obtained by [5], from MgO catalyst using *Jatropha curcas* oil.

3.1 Reaction Time

According to Fig. 4, there was no detectable oil conversion to methyl ester in the first 3 hours. However, conversion was increased up to 28.1% yield after 4 h and gradually increases to 32.2% after 5 h of reaction time. The long time required

to record an appreciable yield of luffa methyl ester can be attributed to the presence of heterogeneous mass transfer systems [5].

3.2 Methanol/Oil Molar Ratio

Transesterification reaction is a reversible one. Although the stoichiometric molar ratio of methanol to triglyceride for transesterification is 3:1, higher molar ratios are used to enhance the solubility and to increase the contact between the triglyceride and alcohol molecules [12]. Higher molar ratios result in greater ester conversion in a shorter time. From Fig. 5, the conversion of *luffa* oil in the presence of magnetic sand exhibited a strong dependence on the methanol to oil ratio. Methanol/oil ratio was varied in the values of 3:1, 6:1, 9:1, 12:1 and 15:1. Between 3:1 and 9:1 methanol/oil combination, there was a gradual increase in the methyl ester yield of

31.10% to 16.33% respectively. When the methanol loading amount was further increased, the conversion was increased considerably. The maximum conversion (29.23%) was obtained at a molar ratio of 15:1. Similar trend was observed by [13] who investigated the effect of molar ratio on the transesterification of sunflower oil with methanol. They obtained the results for transesterification of sunflower oil, in which the molar ratio varied from 6:1 to 1:1 and concluded that 98% conversion to ester was obtained at a molar ratio of 6.1.

3.3 Reaction Temperature

A minimum yield of 13.1% which gradually increased to 15.9% then to 16.33% at 40℃, 45℃ and 50℃ respectively was observed as shown in Fig. 6. Significant increase of up to 29.23% was recorded at 60℃. These results show that methyl ester yield is more pronounced at higher temperatures.

3.3.1 Viscosity

The result (Fig. 7) agrees with theoretical claims which opine that viscosity decreases with an increase in temperature. It shows a steep drop in viscosity from 23.74 mm/s^2 at 30℃ to 8.72 mm/s^2 as the temperature was increased to 70℃ which was slightly above the limit (2-6 mm^2/s) specified by the American Society for Testing and Materials Standards and slightly above the viscosity (7.1 mm^2/s) for a B10 blend of luffa raw oil and AGO as recorded by [6]. This indicates that the biodiesel produced from *luffa* oil under the above conditions had enhanced fluidity as fuel for diesel engines. If viscosity is too high the injectors do not perform properly. The figure showed that *luffa* methyl esters produced has viscosity of 8.72 mm^2/s at 70℃, a value very close to the limit specified by ASTM.

3.3.2 Density

The density of diesel oil is important because it gives an indication of the delay between the injection and combustion of the fuel in a diesel engine (ignition quality) and the energy per unit mass [2]. The density of Luffa methyl ester, at 15℃ was found to be 0.88 g/ml, which is closer to the density of diesel (0.83 g/ml). This shows the potential use of luffa biodiesel as an alternative fuel.
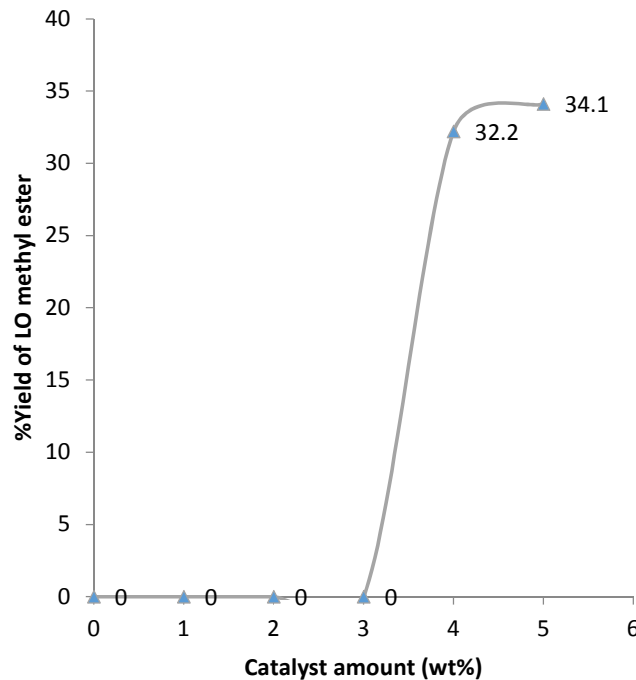

Fig. 3. Influence of catalyst amount on the conversion of luffa seed oil. Reaction condition: Luffa oil 44.2 g, reaction time 5 h, reaction temperature 60℃ and methanol/oil molar ratio 15:1
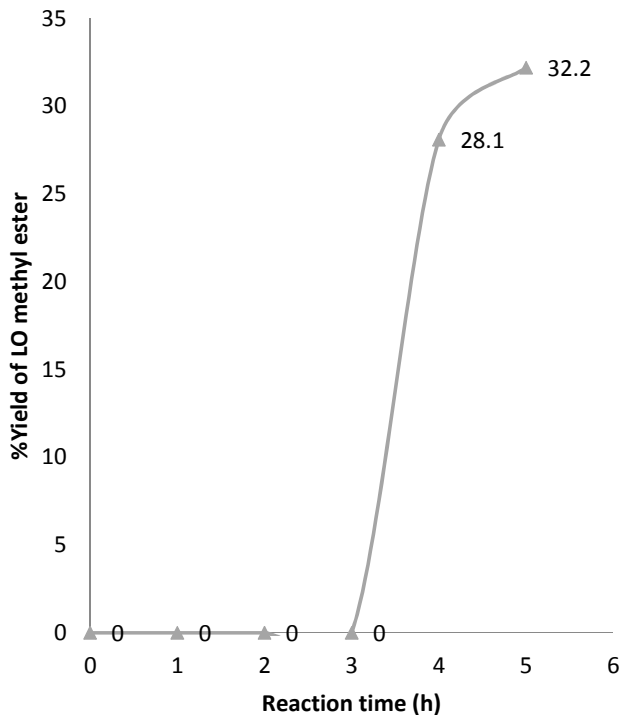
Fig. 4. Influence of reaction time on the conversion of luffa oil by magnetic sand catalyst. Reaction condition: Luffa oil 44.2 g, catalyst amount 5 wt%, reaction temperature 60℃ and methanol/oil molar ratio 15:1
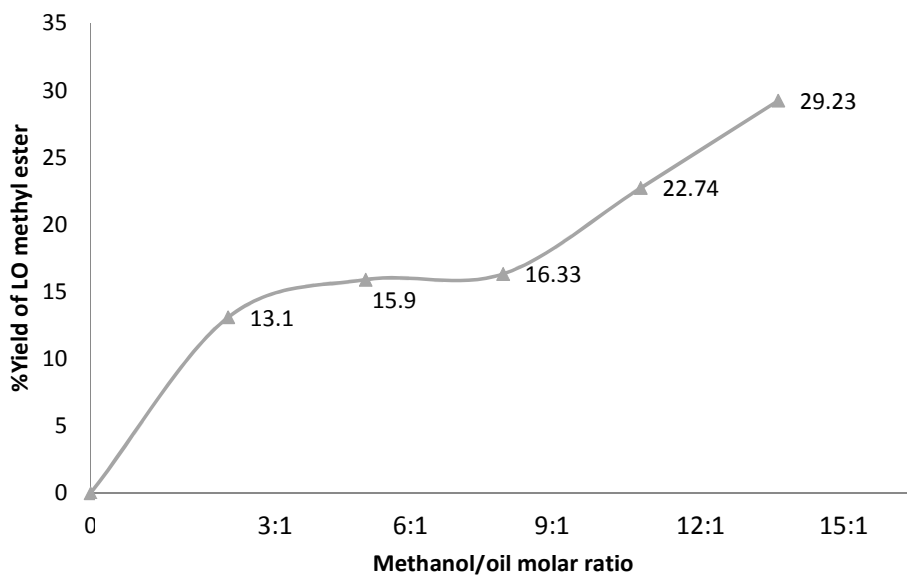

Fig. 5. Influence of methanol/oil molar ratio on the conversion of luffa seed oil. Reaction condition: luffa oil 44.2 g, catalyst amount 5 wt.% oil, reaction time 5 h and reaction temperature 60℃
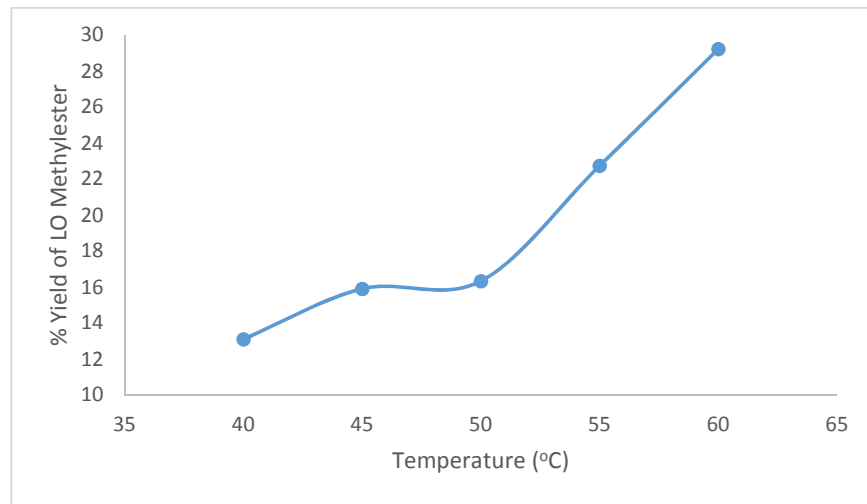
Fig. 6. Influence of reaction temperature on the conversion of luffa seed oil. Reaction condition: Luffa oil 44.2 g, catalyst amount 5 wt.%, reaction time 5 h and methanol/oil ratio 15:1
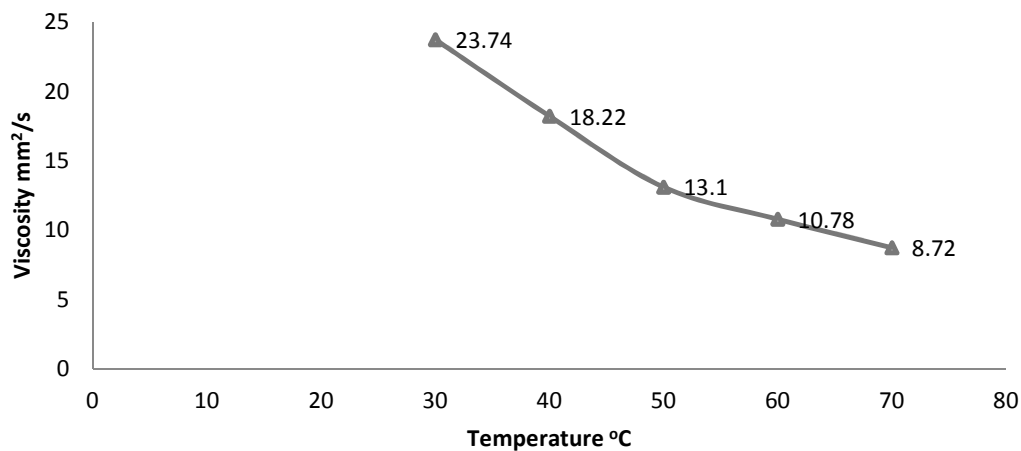

Fig. 7. Variation of viscosity of luffa oil methyl ester with temperature

3.3.3 Ash content

The ash content describes the amount of inorganic contaminants such as abrasive solids and catalyst residues, and the concentration of soluble metal soaps contained in a fuel sample [14]. The biodiesel from luffa oil had ash content of 0.03% and is lower compared to that of the crude oil which was reported by [2] to be 0.078%. The measure of the amount of metal contained in the fuel is the ash content. Therefore, this result showed that the use of *Luffa* biodiesel as a fuel would reduce injector nozzle clogging, combustion deposits and injector system wear compared to the crude *Luffa* oil and Automated Gas Oil (AGO) which had higher ash content of 0.12% [6].

3.3.4 Flash point

One of the most important characteristics of any fuel is its flash point. Flash point is the temperature that indicates the overall flammability hazards in the presence of air; higher flash points produce safer handling and storage of biodiesel [16]. The Flash Point of biodiesel produced was 81℃ which in comparison is higher than the standard diesel flash point of about 74℃ [6]. This makes the biodiesel sample safe for use and storage [17]. Fuels with lower flash point which tend to ignite at lower temperatures are highly dangerous if not stored and used properly. Most non-edible based seeds oils flash point are higher than fossil diesel [18] and Luffa seed oil methyl ester is not an exception.
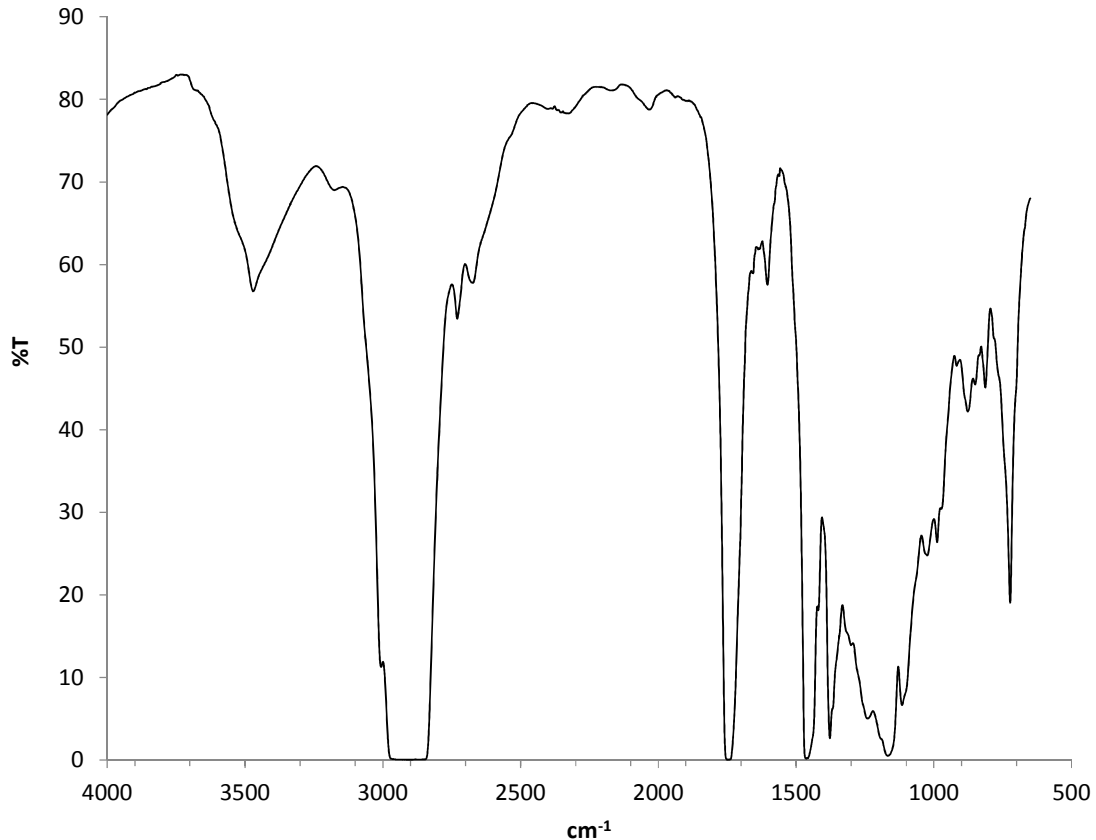
Fig. 8. FT-IR spectrum of Luffa Methyl ester

Table 4. Comparison of the fuel properties of Luffa Methyl ester, petrodiesel and American standard and testing material (ASTM)

Properties	ASTM	Limit	LME	Petro-diesel
Viscosity at 40℃ (mm ^{2}/s)	D445	1.9-6	6.7	1.9 - 6.0
Specific gravity at 15℃	D1298	0.95max	0.82	0.86 - 0. 90
pH	-	-	6.99	-
Pour point(℃)	D975	-35 to -16	4	-10
Flash point (℃)	D93	100 - 170	81	74
Ash content (%)	D482	0.1max	0.03	-
Density (g/cm ^{3})	-	-	0.88	-

[LME= Luffa Methyl ester, 2,6,15]

3.3.5 Pour point

The pour point is the lowest temperature at which the oil/fuel sample can flow. This property is related to the use of biodiesel in colder region [3]. Luffa oil methyl ester has its pour point at 4℃. The value clearly indicates that the use of luffa oil methyl ester in colder regions is limited. However, this value is also indicative of the high potential of this fuel as biodiesel particularly in

Northern Nigeria where temperature is always above 20℃, a temperature at which the oil is fluid.

3.4 FT-IR Analysis for Luffa Oil and Its Fatty Acid Methyl Ester

The FT-IR spectra in the mid-infrared region have been used to identify functional groups and the bands corresponding to various stretching

and bending vibrations in the sample of biodiesel. The position of carbonyl group in FT-IR is sensitive to substituent effects and to the structure of the molecule [19]. According to Fig. 8, the characteristic stretching absorption peaks of O–H, C=O, =C-H and C-O were observed at 3471, 1746, 988 and 1115 cm^{-1}, respectively. The asymmetric and symmetric stretching vibration peak of $–CH_2$ group was located at 2901 cm^{-1}. The anti-symmetric stretching vibration absorption peak of C–O–C was found at 1239.9 cm^{-1}.

The result indicated that we could get biodiesel sample including the entire groups which we needed from luffa seed oil using magnetic sand catalyst.

4. CONCLUSIONS

In this research, catalyst characterization by XRF and XRD analysis, Transesterification of *luffa* oil and Characterization of *luffa* oil methyl ester using FT-IR technique have been studied. The results indicated that biodiesel could be produced via transesterification of luffa oil using a crude magnetic sand solid catalyst as heterogeneous catalyst. Soap formation was not observed in this research, which reduces the cost of production of biodiesel. The 5wt% catalyst amount of crude Fe_2O_3 was sufficient for producing methyl ester content with promising yield. The 5 hours of reaction time gave the highest methyl ester yield and conversion of oil to biodiesel at 60℃ using 15:1 methanol/oil molar ratio.

The results are consistent with those of other researchers and agree with international standards.

5. RECOMMENDATIONS

We recommend that other plant of the same family could be used to ascertain their biodiesel status.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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