# Assessment of Biodiesel Potential from Jatropha Oil using Lubricity Strength, Gas Chromatograph-Mass Spectroscopy (GC-MS) and Fourier Transform Infrared Spectrophotometer (FTIR) Analyses

Abdulkabir Aliyu, Member IAENG, Gary Leeke and Vasilije Manovic

**ABSTRACT-Biodiesel from jatropha oil was manufactured and the strength in lubricity, percentages of methyl esters as well as the functional groups present were analysed using high frequency reciprocating rig (HFRR), gas chromatograph-mass spectroscopy (GC-MS) and Fourier transform infrared spectrophotometer (FTIR) techniques respectively. The results of the analysis indicated that manufactured biodiesel has the average wear scan value of 239 µm which is in line with the acceptable limit wear scan value stated in petroleum diesel standards EN 590 and ASTM D975. The percentages of methyl esters before and after manufactured biodiesel were in total of 10.6% and 96.2% respectively. The 96.2% content of methyl ester was also in line with the acceptable EN biodiesel standards. The physico-chemical properties of manufactured biodiesel was also carried out. The results were in agreement with the American Society for Testing and Materials (ASTM) Standard for sulphur content, viscosity, cetane number, acid value, colour and specific gravity.**

**Keywords. Functional groups, jatropha biodiesel, lubricity, methyl ester, physico-chemical properties.**

## I. INTRODUCTION

Non-edible vegetable oils are one of the promising alternatives to fossil fuel due to their benefits ranging from improved quality of exhaust emissions to some extents and environmental benefit [6], [3], [7] and [10]. Biodiesel from these sources do not produce as much carbon dioxide as compared to the petroleum-based-diesel, if the life cycle is considered. The studies have shown that one of the main consumers in the energy sector is transportation sector. About 600 million cars consumed energy worldwide [25] primarily run on diesel engines. A high viscosity and FFA (free fatty acid) content and the accumulation of carbon deposits within the engine are some of the challenges associated with the direct use of vegetable oils in Compression-ignition engines. [6], [19].Biodiesel can be produced from a feedstock that can be reproduced, regrown and reused. Jatropha biodiesel originates from non-edible vegetable oils has being categorised as second generation feedstocks [2], [18].

The application of second generation feedstocks was identified as potential to overcome the problems associated with first generation, however such feedstocks need to be converted to biodiesel by the application of appropriate technologies [6], [1], [8]. The use of oils from second generation feedstocks such as jatropha, neem and pongamia in developing countries is gaining acceptability as a potential fuels due to its low price and being unfit for human consumption [28].

Biodiesel can be produced by reacting vegetable oils, animal fats or recycled cooking oils with alcohol (methanol or ethanol), and the catalysts such as potassium hydroxide and sodium hydroxide or acid catalysts are added for the process of transesterification. Provided that the technology of conversion into biofuels is adequately adapted at the right commercial scale. The feedstocks from such non-edible vegetable oils may become the basis for the economic boost of many countries, [14], [27].

Previous research work shows that the stability of biodiesel fuel can be affected by the profile of the fatty acid present in the vegetable oil feedstock used for its production [13]. In addition, the presence of higher concentration of unsaturated methyl esters in the biodiesel fuels made it more prone to oxidation. This also has negative impact on the stability of biodiesel.

The present research examines the lubricity strength, percentages of methyl esters, functional groups and physico-chemical properties of jatropha oil in order to qualify its manufacture to biodiesel for use as a fuel in compression ignition engines.

## II. MATERIALS AND METHOD

Sample of jatropha oil was procured from National Research Institute for Chemical Technology (NARICT), Zaria, Nigeria. GC-MS analysis was conducted on raw oil before manufactured into biodiesel. Reagents such as methanol, sodium hydroxide, sulphuric acid were used as

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procured. The manufacture of biodiesel was performed in the Chemical Engineering laboratory, Ahmadu Bello University, Zaria, Nigeria. Biodiesel was manufactured from a sample of procured jatropha oil through alkaline transesterification reaction. 1g of sodium hydroxide was added to 25ml of methanol and stirred until it was completely dissolved. 100 ml of jatropha seed oil was placed in a flask and the methanolic sodium hydroxide solution was added to it. The mixture was stirred for 60 minutes and then poured into a separating funnel. After about an hour and complete separation of the products, the mixture was left to stand in the separating funnel for about 24 hours, two layers were formed: glycerol and biodiesel [20]. The dried methyl ester was stored and kept for further analysis.

### *A. Lubricity Strength*

PCS instruments High Frequency Reciprocating Rig was used for this studies and ISO12156 Method was adopted. A volume of 2 ml of the test sample was placed in the test reservoir of the HFRR test rig and the temperature was adjusted to 60°C and allowed to thermally equilibrate. At the end of the test duration, the ball was removed from the vibrator arm and the test specimens and hardware were cleaned as per the method requirements. The dimension of the minor and major axes of the wear scar formed on the ball were measured with the head of a micron-graduated magnifying optical device.

### *B. GC-MS Analysis*

The analysis of biodiesel through gas chromatography-mass spectroscopy (GC-MS) was carried out for determination of percentages of methyl ester present before and after the manufactured of the biodiesel. It was performed on raw oil of the jatropha feedstocks and the manufactured biodiesel. An ultra-inert HP-5ms (30 m×250 µm×0.25 µm) column was used and was programmed from 200 to 325 °C at the rate of 3 °C/min [26]. The temperature of both the injector and detector was set at 200 °C. A sample volume of 3 µL of the methyl ester was injected using a rip mode, with a split ratio of 5:1. The mass spectrometer was set to scan in the range of m/z 46-600 with 18 as the number of ions. The major peaks were observed in the total ion chromatogram. Each peak was traced to the corresponding fatty acid methyl ester content of each of the samples. These were identified from the library software Mass Hunter (NO. NIST14.L). Identified FAMEs with respect to corresponding retention times were verified by running the standards with reference to similar experimental conditions [23]. The respective retention time data confirmed by mass spectrometric analysis were compared. Percentage certainty was also carried out for the validation of results.

### *C. FTIR Spectrometry of Jatropha Biodiesel*

Shimadzu FTIR-8400S Fourier Transform Infrared Spectrophotometer was use for the analysis of the vibrational motion of the atoms and molecules present in jatropha biodiesel. Qualification of the compounds was performed by the application of the Fourier transformation technique. This was carry out through their corresponding band width intensities. The spectra were taken between 4000 and 650 $cm^{-1}$ with a resolution of 2.0 $cm^{-1}$. 10 scans were collected per spectrum at the rate 10 scans per minute.

### *D. Physico-Chemical Properties*

The physico-chemical properties of manufactured biodiesel were determined and the results were compared with the American Society of Testing and Materials (ASTM) standard. The tested properties includes sulphur content, viscosity, cetane number, calorific value, flash point, cloud point, pour point, acid values, colour and specific gravity.

## III. RESULTS AND DISCUSSION

### *A. Lubricity Strength*

Lubricity was tested using ISO 12156 method at a temperature of 60 $^{O}$C. Table I shows the wear scar values of the manufactured biodiesel and that of reference fuel (petroleum based diesel). From the results obtained it shows that the jatropha biodiesel possessed an average wear scan of 239 µm as compared to petroleum-based-diesel with an average of 366 µm. The process of hydro-treating/ hydrodesulphurization not only removes the sulphur content from diesel fuel to meet the standards of the emission during the combustion but also removes other compounds like nitrogen and oxygen based polar substances which are responsible for the lubricity of the diesel fuel [11]. This indicated that the reason for poor lubricity of petroleum-based-diesel is not mainly because of the sulphur removal. A smaller wear scar value signifies greater lubricity that ensures the effectiveness of interfacial lubricant fuel film on the separating action of surfaces [9]. The test in lubricity strength show that the jatropha biodiesel has the best strength in lubricity as compared to reference fuel (petroleum-based-diesel). This agreed with the acceptable limit wear scan value of 460 µm and 520 µm stated in petroleum diesel standards EN 590 and ASTM D975 respectively [15]. This also agreed with the finding of [16], which stated that wear scars of not more than 460 µm at 60 $^{0}$C indicated fuels with good lubricity strength and can be used in diesel engine.

TABLE I
WEAR SCAR (µm) AT 60 $^{O}$C VALUE OF JATROPHA AND REFERENCE FUEL

| **Wear Scar (µm) at 60 $^{O}$C** | | | | |
|---|---|---|---|---|
| **Material** | X-Axis | Y-Axis | Average | WS1.4 |
| **Jatropha biodiesel** | 292 | 186 | 239 | 241 |
| **Petroleum based diesel** | 394 | 337 | 366 | 373 |

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*B. GC-MS Results*

The percentage of methyl esters contents of manufactured biodiesel and that of raw oils were analysed using Gas Chromatography - Mass Spectroscopy. These are presented in fig. 1 and 2 for jatropha oil and jatropha biodiesel respectively. The interpretation of the peaks of the chromatogram were given with reference to contents of methyl esters in all cases as shown in Tables II and III

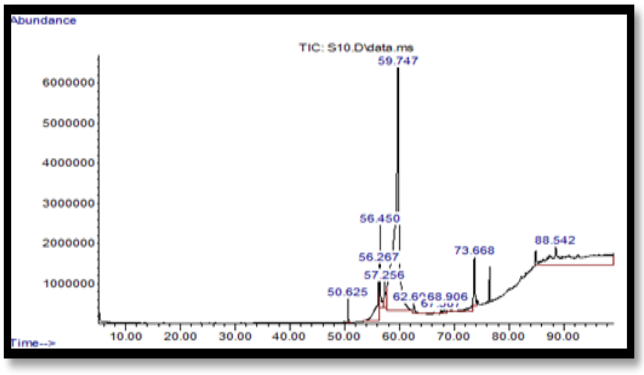


Fig. 1. GC-MS Chromatogram of Jatropha Oil

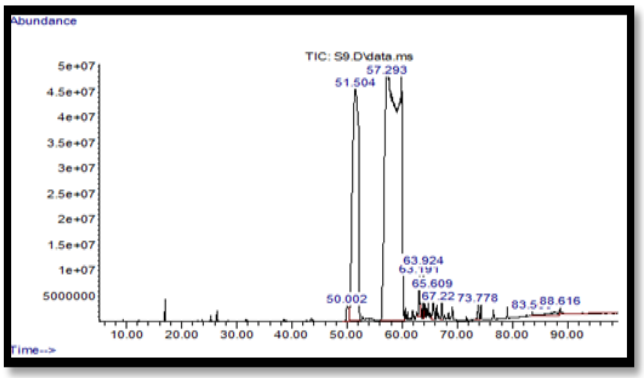


Fig. 2. GC-MS Chromatogram of Jatropha Biodiesel

Based on the chromatogram in fig. 2 as presented in Table III, manufactured biodiesel from jatropha oil has 43.40% each of 10, 13-Octadecadienoic acid methyl esters ($C_{19}H_{34}O_2$) and Hexadecanoic acid methyl esters ($C_{18}H_{36}O_2$), making a total of 86.8% for the two mono-saturated compounds. The total percentages of methyl esters present in the manufactured jatropha biodiesel was 96.2%. The balance was from Octacosanoic acid, methyl ester ($C_{29}H_{58}O_2$) with 2.05%, a branched hexadecanoic acid methyl esters ($C_{17}H_{32}O_2$) presented as 7-hexadecanoic acid methyl esters with total of 4.39%. The manufactured jatropha biodiesel is suitable source of fuel for compression ignition engine owing to the high percentage of mono-saturated methyl esters. This result is in agreement with the finding of [24], [4]. The result indicates that the available methyl esters present in the jatropha biodiesel are mono-saturated compounds and have prospects of bringing stability during combustion in diesel engine.

TABLE II
GC-MS RESULTS FOR JATROPHA OIL

| RETENTION TIME (MIN) | COMPOUND (METHYL ESTER) | AREA (%) | PERCENTAGE CERTAINTY | RETENTION TIME (MIN) | OTHER COMPOUNDS | AREA (%) | PERCENTAGE CERTAINTY |
|---|---|---|---|---|---|---|---|
| 50.62 | Hexadecanoic acid, methyl ester $C_{18}H_{36}O_2$ | 0.72 | 99 | 57.25 | Methyl stearate $C_{19}H_{38}O_2$ | 2.76 | 93 |
| 56.27 | 9,12-Octadecadienoic acid, methyl ester $C_{19}H_{34}O_2$ | 6.52 | 99 | 59.75 | 9,12-Octadecadienoic acid (Z,Z)- $C_{18}H_{32}O_2$ | 51.04 | 98 |
| 56.45 | 9-Octadecenoic acid (Z)-, methyl ester $C_{19}H_{36}O_2$ | 3.43 | 99 | 62.60 | 9-Tetradecenal, (Z)- $C_{14}H_{26}O$ | 0.61 | 58 |
| | | | | 67.59 | 9,12-Octadecadienoic acid (Z,Z)- $C_{18}H_{32}O_2$ | 0.70 | 99 |
| | | | | 68.91 | Oleoyl chloride $C_{18}H_{33}ClO$ | 1.53 | 91 |
| | | | | 73.67 | 9,17-Octadecadienal, (Z)- $C_{18}H_{32}O$ | 3.18 | 99 |
| | | | | 88.54 | 7-Pentadecyne $C_{15}H_{28}$ | 29.51 | 83 |
| | Total | 10.67 | | | | 89.33 | |

TABLE III
GC-MS RESULTS FOR JATROPHA METHYL ESTER

| RETENTION TIME (MIN) | COMPOUND (METHYL ESTER) | AREA (%) | PERCENTAGE CERTAINTY | RETENTION TIME (MIN) | OTHER COMPOUNDS | AREA (%) | PERCENTAGE CERTAINTY |
|---|---|---|---|---|---|---|---|
| 50.00 | 7-Hexadecenoic acid, methyl ester, (Z)- $C_{17}H_{32}O_2$ | 2.26 | 99 | 63.61 | 9-Tetradecenal, (Z)- $C_{14}H_{26}O$ | 1.62 | 35 |
| 51.50 | Hexadecanoic acid, methyl ester $C_{18}H_{36}O_2$ | 43.40 | 99 | 73.78 | 9,17-Octadecadienal, (Z)- $C_{18}H_{32}O$ | 0.92 | 99 |
| 57.29 | 10,13-Octadecadienoic acid, methyl ester $C_{19}H_{34}O_2$ | 43.40 | 99 | 88.61 | .gamma.-Sitosterol $C_{29}H_{50}O$ | 1.25 | 62 |
| 63.19 | 7-Hexadecenoic acid, methyl ester, (Z)- $C_{17}H_{32}O_2$ | 2.13 | 60 | | | | |
| 83.56 | Octacosanoic acid, methyl ester $C_{29}H_{58}O_2$ | 2.05 | 95 | | | | |
| 63.92 | Methyl 18-methylnonadecanoate $C_{21}H_{42}O_2$ | 1.99 | 99 | | | | |
| 67.22 | Methyl 2-octylcyclopropene-1-octanoate $C_{20}H_{36}O_2$ | 0.99 | 60 | | | | |
| | Total | 96.22 | | | | 3.79 | |

*C. FTIR of Jatropha Biodiesel*

FTIR analysis was used to determine the functional groups present in the manufactured biodiesel sample. Fig. 3 shows the FTIR spectra of jatropha biodiesel. The absorption peaks with different bond types, vibrations and intensity of biodiesel sample are discussed below.

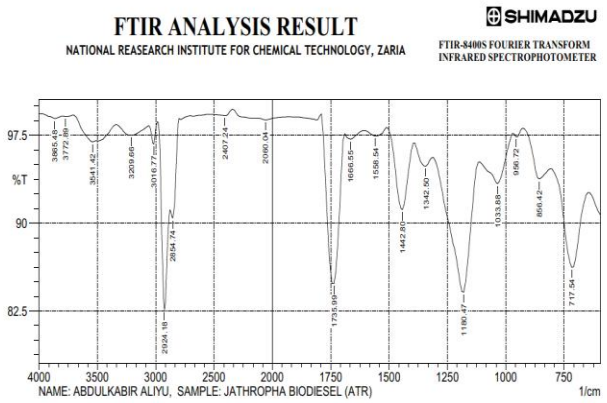


Fig. 3. FTIR Spectra of Jatropha Biodiesel

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The absorption peak at 3541.42 $cm^{-1}$ revealed the occurrence of alcohol group with stretch vibration strong intensity. The peak at 2924.18 and 2854.74 $cm^{-1}$ shows the presence of alkane compounds with both having stretch vibration and strong intensity. The peaks at 1735.99 $cm^{-1}$ indicates the presence of an ester with stretch vibration and strong intensity. The crest at 1666.55 $cm^{-1}$ shows the presence of alkene with stretch vibration and variable intensity. The absorption peaks at 1558.54 $cm^{-1}$ revealed the presence of aromatic with stretch and variable absorption. The peak 1442.80 $cm^{-1}$ shows the presence of alkane compounds with bend vibration and strong intensity while at 1342.50, 1180.47 and 1033.88 $cm^{-1}$ all indicated the presence of aromatic compounds. The peaks at 956.72 and 856.42 $cm^{-1}$ all indicated the presence of alkene with bend vibration and strong intensity. The bands at 717.54 $cm^{-1}$ shows the presence of aromatic compounds in the biodiesel with bend vibration and variable absorptions. The ATR-FTIR results of jatropha biodiesel indicated the presence of alkane, aromatic and ester groups. Thus, the presence of alkanes and ester confirmed their roles as functional groups.

### *D. Physico-Chemical Properties Of Jatropha Biodiesel*

Physico-chemical properties of jatropha biodiesel are presented in Table IV, specific gravity, viscosity, calorific value, cetane number, flash point, pour point, cloud point sulphur content colour index and acid value were measured and compared as shown below;

TABLE IV
FUEL PROPERTIES OF JATROPHA BIODIESEL AND ASTM STANDARD

| *Sample* | *Properties* | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | Specific gravity | Viscosity | Calorific value | Cetane number | Flash point | Pour point | Cloud point | Sulphur content | Colour index | Acid value |
| *Jatropha biodiesel* | 0.876 | 4.467 | 44.250 | 99.500 | 143 | -1.500 | 7.800 | 0.010 | 1.000 | 0.470 |
| *ASTM standard* | 0.900 | 1.900 to 6.000 | ASTM min: 42 | 47 min ASTM D613 | 170$^{0}$C ASTM max | -15 to 10 | | 0.050 (% wt) | ASTM min: 1.000; ASTM max: 3.500 | 0.500 mgKOH/g ASTM D3242 |

The properties obtained in this study conform to ASTM standard values when compared. This qualified it to be potential suitability as a fuel for use in a diesel engine.

Table V. shows fuel properties of some biodiesels and the corresponding ASTM Standard.

TABLE V
FUEL PROPERTIES OF SOME BIODIESELS AND ASTM STANDARD

| **Property** | **KOME** | **MOME** | **LOME** | **ROME** | **COME** | **ASTM*** |
|---|---|---|---|---|---|---|
| Density kg/m$^3$, 40 $^0$C | 865–898 | 828–865 | 874–920 | 858–900 | 872–885 | 870–900 |
| Viscosity (mm$^2$/s, 40$^0$C) | 3.8–9.6 | 2.7–6.2 | 3.36–8.91 | 1.9–6.0 | 3.6–5.94 | 1.9–6.0 |
| Flash point ($^0$C) | 110–187 | 56–208 | 161–181 | 130–174 | 70–200 | >130 |
| Pour point ($^0$C) | -6 to 14 | 1–6 | -18 to 14 | -15 to 10 | -15 to 6.0 | -15 to 10 |
| Cloud point ($^0$C) | -2 to 24 | 3–5 | -3.5 | -3 to 12 | – | – |
| Cetane number | 36–61 | 47–51 | 48–59 | 49–57 | 45–60 | 47 min |
| Calorific value (MJ/kg) | 36.0–42.1 | 36.8–43.0 | 37.5–42.2 | 36.5–42.1 | 40..1–40.8 | – |

Key codes: KOME= karanja oil methyl ester; MOME= mauha oil methyl ester; LOME= linseed oil methyl ester; ROME= rubber oil methyl ester; COME= cottonseed oil methyl ester.

Table V shows that the fuel properties of the selected methyl esters obtained from non-edible vegetable oils is subject to structure and nature of the fatty acid esters present [21], [22]. It is also subject to the nature of the vegetable oil used for the transesterification process [5]. It was observed that most of the fuel properties are within the set standard of American Society of Testing and Material (ASTM). It can be deduced that some biodiesel can be applied directly in CI engines without major modification.

## IV. CONCLUSION

The results of analysis revealed that jatropha oil is a suitable feedstock for conversion into biodiesel and has the potential to partially contribute to global energy demand.

The test in lubricity strength show the jatropha biodiesel has better strength in lubricity as compared to reference fuel (petroleum-based-diesel). This agreed with the acceptable limit wear scan value of 460 µm and 520 µm stated in petroleum diesel standards EN 590 and ASTM D975 respectively. This indicated fuel with good lubricity strength and is potentially suitable for use in a diesel engine.

The GC-MS analysis shows the percentages of methyl esters before and after manufacture of jatropha biodiesel were 10.6% and 96.2% respectively. The dominant esters are methyl octadecanoate and methyl hexadecanoate.

The presence of alkanes and ester functional groups from the FTIR results affirms the use of non-edible neem oil methyl esters as an alternative fuel to petroleum-based-diesel.

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