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Physicochemical Composition and Functional Properties of Jatropha Seed Oil and Jatropha Biodiesel: An Agro-Renewable Product

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Abstract

The composition of jatropha oil and biodiesel has attracted extensive research. However, fewer studies have been reported on the functional properties of the oil and biodiesel. In this research, the functional properties and physicochemical composition of jatropha oil and biodiesel were investigated. The functional properties were studied using spectroscopic method while the oil and biodiesel were subjected to various physicochemical tests. The results obtained revealed high levels of unsaturated fatty acid with linoleic and oleic acids accounting for 75.92 %. The free fatty acid values (%) were 0.454 and 0.347 for the oil and biodiesel respectively. The spectroscopic analysis showed that the dominant functional groups were =C-H, C-O-C, RO-H, R-OH and C=O for the biodiesel and =C-H, CH_2, C=O and O-H groups for oil. The frequencies ranges were from 1200 cm^{-1} to 3600 cm^{-1} and different vibrational types were observed to cause the molecular infrared absorbance of both the oil and the biodiesel. The spectroscopic analysis gave positive identification of the oil and biodiesel. This provided another form of qualitative analysis in terms of defining the character of the oil and biodiesel.

1. Introduction

Energy needs of mankind continuously increase as population of humans grows. The need for research into new sources of energy that are renewable and sustainable increases due to population growth and increasing damage to the environment [1, 2]. The production of biodiesel from edible and non-edible oil bearing materials had gained wide acceptability due to the unstable nature of fossil fuel demand and supply [2, 3]. Various technologies have been developed for the production and extraction of oil from oil-bearing seeds. The oils extracted contain certain properties which are peculiar to the type of oil-bearing seeds and its composition must be ascertained to determine its various applications.

Biodiesel is commonly produced by the transesterification reaction of vegetable oils or animal fats with methanol or ethanol. This is usually catalyzed by either an acid or a base catalyst. The transesterification reaction products are fatty acid methyl (alkyl) esters (FAME, or FAAE) and glycerol [4-7].

Jatropha curcas seeds are non-edible oil bearing seeds containing various toxic components with an average oil content of 37% by weight. According to different studies [8, 9], jatropha seeds contain

28% - 40% oil. The seeds of *jatropha curcas* tree provides important source of non-edible oil which can be used for biodiesel production.

The physicochemical properties of most biodiesels are similar to those of petrol diesel and could therefore be used directly on diesel engines with little or no modifications [7, 10, 11]. The physicochemical properties of oils and their corresponding biodiesels are important for assessment of the quality and purity as well as for their identification. The properties give specific measurements of the characteristics of both the oils and biodiesels. These properties are important parameters used to identify any particular oil and biodiesel.

Chemical insight is gained by analyzing the interactions of matter and electromagnetic radiation characterized by the wavelength, wave number or frequency and this provides the spectroscopic analysis of the oils and biodiesels. The measurement methods could be by core electrons (X-rays), electronic transitions (UV/VIS), radiation vibration (IR) and/or nuclear spin (RF) in which the energy of radiation is quantized in photons which excites the appropriate molecules to higher energy state. Fourier transform infrared (FTIR) is a unique and preferred method of infrared spectroscopy in which infrared radiation is passed through a sample. Some of the infrared radiation is absorbed by the sample and while some of it passed through (transmitted). The resulting spectrum represents the molecular absorption and transmission thereby creating a molecular fingerprint of the sample. No two unique molecular structures produce the same infrared spectrum [12, 13]. FTIR can be used to identify unknown materials; determine the quality or consistency of a sample and determine the amount of components in a mixture [14, 15].

Several researchers have extracted oil and produced biodiesel from jatropha seeds and also undertook the physicochemical analysis of the oil/biodiesel [2, 5, 8, 9, 17-20]. However, some salient parameters were not evaluated and there were little or no detailed spectroscopic (functional) analysis of the oil and biodiesel using Fourier infrared spectroscopy or any known spectroscopy techniques.

This study is undertaken to fill the identified knowledge gap. This research undertook the production, physicochemical analysis, fatty acid analysis and functional properties (spectroscopic) analysis of jatropha seed oil (JASO) and jatropha oil biodiesel (JAB). It fills an existing knowledge gap in terms of providing a comparative basis for in-depth understanding of the interactions of molecules and electromagnetic radiation in both the oil and the biodiesel. This will help to achieve sustainability and profitability enhancement in production and utilization.

2.0 Methodology

2.1 Materials

The material used in this study is *jatropha curcas* seed. Jatropha is an oil tree that belongs to the family *Euphorbiaceae*. Jatropha seeds look like black beans and are on average 18 mm long and 12 mm wide and 10 mm thick. The seeds consist of a hard shell that makes up around 37% by weight on average and soft white kernel that makes up 63% by weight. The dry seeds have a moisture content of about 7% and contain between 32% and 40% of oil, with an average of 34%. Virtually all the oil is present in the kernel. The oil from jatropha seeds is used for making biodiesel fuel and the seeds are a good source of oil [9, 17, 18].

2.2 Method

2.2.1 Oil extraction

Jatropha seed oil (JASO) was obtained by grinding the jatropha seeds obtained from the *jatropha curcas* tree. The hydraulic press machine was used to press the grinded jatropha seeds in a baft material against a rigid surface with small openings to separate the oil and the solid residue. Extraction was done at room condition to avoid altering the chemical properties of the oil if done at elevated temperature. The grinded jatropha seed was squeezed under high pressure in batches. The extraction was based on a screw that presses the grinded jatropha seed materials against the walls

of the metallic non-corrosive cylinder by which the oil was recovered through a mesh that allows only the passage of oil. Immediately after oil extraction, the oil was centrifuged at 1200 rpm for 15min. to remove any debris present in the extracted oil. Figure 1 shows the various stages in the jatropha seed oil production process.

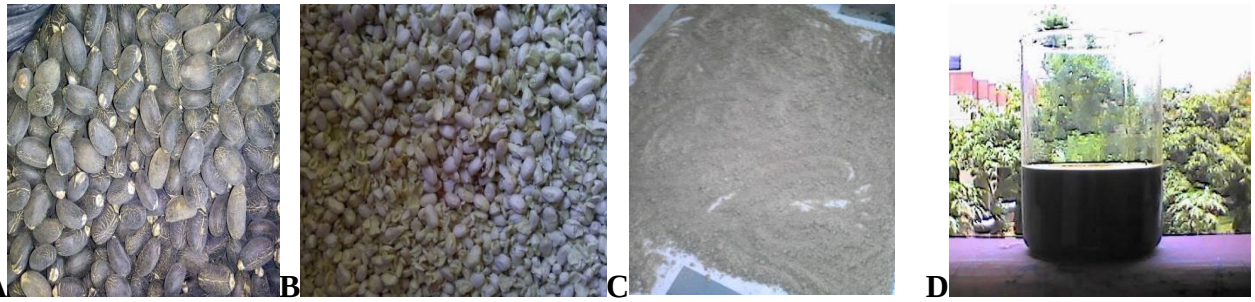

Figure 1: (A) Jatropha seeds, (B) Dried and crushed seeds, (C) Grinded seeds and (D) Jatropha seed oil

2.2.2 Biodiesel production

Jatropha seed oil (JASO) was used in the production of jatropha oil biodiesel (JAB). The reaction mechanism employed was transesterification reaction; a reaction between triglycerides and alcohols to produce biodiesel in the presence of a catalyst. Jatropha oil biodiesel (JAB) was produced in a batch glass reactor (500 ml) from jatropha seed oil (JASO) at a molar ratio of 6:1 for methanol and oil respectively. The amount of catalyst used was 5% w/v. The catalyst used was potassium hydroxide. The methanol and potassium hydroxide catalyst used in the experiment were of high analytical grades. The jatropha seed oil (JASO) was firstly introduced into the reaction chamber and heated to the desired temperature (60 °C). Thereafter, the catalyst/methanol mixture was added to the heated jatropha seed oil (JASO) to attain the reaction temperatures. The temperature was maintained constant at a particular value (60 °C) with the stirrer speed set at 1200 rpm. Constant agitation was maintained throughout the reaction. The biodiesel obtained after separation from the glycerol layer was thoroughly washed with warm distilled water to remove all traces of impurities and thereafter dried in a Thermotech TIC – 4000E oven with temperature controller to remove water and methanol still present by evaporation. The physicochemical analysis as well as the spectroscopic analysis of the obtained biodiesel was thereafter undertaken to determine its properties.

2.2.3 Characterization of the produced oil and biodiesel

The jatropha seed oil (JASO) and jatropha oil biodiesel were subjected to various physicochemical analysis which included free fatty acid value, peroxide value, saponification value, iodine value, moisture content determination, specific gravity, refractive index, viscosity, density, ash content, smoke point, fire point, flash point, cloud point, fatty acid analysis (via gas chromatographic (GC) method) and spectroscopic analysis using Fourier transform infrared (FTIR) spectroscopy.

In the GC method, 5g of the oil/biodiesel sample was dissolved in methanol overnight. The solution was then concentrated by bubbling nitrogen gas into it. GC analysis of the oil and biodiesel were performed using a Trace GC Ultra, Thermo-scientific 3000 Auto - Sampler system interfaced to a mass spectrometer equipped with Elite – 5MS fused silica capillary column. For the GC detection, an electron ionization system with ionization energy of 70eV was used. Mass spectra were taken at 70eV, a scan interval of 0.5sec and fragments from 45 to 450 Da. The relative percentage amount of each component was calculated by comparing its average peak area to the total area. Software adapted to handle mass spectra and chromatograms was a Turbo-Mass Version 5.2.0. Interpretation on mass-spectrum was conducted using the database of National Institute of Standard and Technology (NIST) having more than 62,000 patterns. The spectrum of the unknown components was compared with the spectrum of known components stored in the NIST library and the molecular

weight and structure of the compound. The gas chromatography analysis involved the separation of the sample according to their molecular mass and thus, provided the necessary data of the sample composition in all cases.

FTIR analysis of the oil and biodiesel were performed using a Shimadzu IR Affinity-1, instrument. 0.4 g of KBr was weighed and ground to powder. 0.001 g of jatropha oil/jatropha biodiesel was weighed into the ground KBr and both were thoroughly mixed together and mounted into a disc. The disc was then inserted into the sample compartment of the FTIR instrument. The scan button was pressed and the IR spectrum generated. The sample was scanned from 1200 cm^{-1} to 3600 cm^{-1}. The sample analysis process included the source, the interferometer, the sample, the detector and the computer which generated the final infrared spectrum for interpretation. The FTIR was used to identify the characteristic functional groups in the oil and biodiesel.

3.0 Results and Discussion

Table 1: Physicochemical analysis of jatropha seed oil (JASO)

Parameters	Results
Colour	Black or dark brown
Density (g/cm^3)	0.887
Moisture content (%)	0.196
Saponification value (mgKOH/gOil)	196.15
Iodine value	108.92
Acid value (%)	0.988
Free fatty acid (%)	0.454
Viscosity (cp)	158.00
Ash content	0.10
Refractive index	1.4603
Peroxide value (Meq/kg)	6.83
Smoke point (°C)	130
Flash point (°C)	185
Fire point (°C)	210
Cloud point (°C)	-3
Pour point (°C)	21
Titre (°C)	-8
Yield (%)	51.723

Table 1 presents the results of the analysis of the jatropha seed oil (JASO) in terms of its physicochemical properties. From the results of the table, the values of the properties fall within the range of values for oils used in similar applications with reference to various standards. These properties are used to establish the identity of the particular oil and were chosen to measure specific characteristics of the oil. Most of the properties are used to specify the nature of the oil while others are empirical in nature though they also give useful guidance in identifying the oil.

Table 2: Physicochemical analysis of jatropha oil biodiesel (JAB)

Parameters	Results
Specific gravity	0.857
Viscosity (mm^2/s)	1.24
Caloric value (mj/kg)	32,969.85
Acid value	0.693
Pour point (°C)	0.00
Flash point (°C)	125.00
Cloud point (°C)	-2.00
Total glycerol (%w/w)	0.00
Moisture content (%)	0.034
Density (g/cm^3)	0.857

Saponification value	134.57
Iodine value (Wiji's)	76.14
Free fatty acid (%)	0.347
Fire point (°C)	28
Ash content (%)	0.69
Conductivity (us/cm)	230
Refractive index	1.4463
Peroxide value (Meq/Kg)	5.030
Smoke point (°C)	15
Pour point (°C)	0

Table 2 presents the results of the physicochemical analysis of the jatropha oil biodiesel (JAB). The result showed good correlation as regards to important parameters of consideration in the use of oil and their associated biodiesels in diesel fuel injection and combustion system. The various values obtained were within the standard limits of the various standards applicable in the industry. The values were also observed to be in agreement with those reported for jatropha biodiesel by different authors [18, 20, 21].

Table 3: Fatty acid analysis of jatropha seed oil (JASO)

Parameters	Results (%)
Palmitic ($C_{16:0}$)	15.76
Myristic ($C_{14:0}$)	0.27
Linoleic ($C_{18:2}$)	37.24
Palmitoleic ($C1_{6:1}$)	0.58
Oleic ($C_{18:1}$)	37.14
Stearic ($C_{18:0}$)	7.97
Linolenic ($C_{18:3}$)	0.12
Arachidic ($C_{20:0}$)	0.08
Dieicosadienoic ($C_{20:2}$)	0.84
Total saturated acids ($C_{n:0}$)	24.08
Total unsaturated acids	75.92
Total monounsaturated acids ($C_{n:1}$)	37.14
Total polyunsaturated acids ($Cn:_{2,3}$)	38.78
Molecular weight (g/mol.)	1103.81
Number of fatty acids	9

The fatty acid composition of jatropha seed oil (JASO) is presented in Table 3. From the results, it is observed that the oil had high levels of unsaturation. The major fatty acid constituent of the oil was both linoleic acid and oleic acid which had influence on the character of the oil. The fatty acid analysis of the oil was in agreement with those reported by other researchers [2, 17-20] on the production of biodiesel from jatropha curcas seed oil. According to [22] and [23], fatty acids with one double bond were considered to be the best choice for biodiesel and most fatty acid constituent of oils and biodiesels remained practically the same even after the oil had undergone transesterification process [22].

Table 4: Fatty acid analysis of jatropha oil biodiesel (JAB)

Parameters	Results (%)
Methyl myristate (myristic) ($C_{14:0}$)	0.17
Methyl palmitoleate (palmitoleic) ($C_{16:1}$)	2.83
Methyl palmitate (palmitic) ($C_{16:0}$)	20.19
Methyl linoleate (linoleic) ($C_{18:2}$)	45.30
Methyl oleate (oleic) ($C_{18:1}$)	17.86
Methyl stearate (stearic) ($C_{18:0}$)	11.51

Methyl ricinoleate (ricinoleic) ($C_{18:1}$, OH)	0.72
Methyl arachidate (arachidic) ($C_{20:0}$)	0.79
Methyl stearate diglycerine (stearic) glycerin	0.32
Methyl docosanoate (docosanoic)	0.12
Methyl lignocerate (lignoceric)	0.18

The result of the fatty acid analysis of the jatropha oil biodiesel (JAB) is presented in Table 4. The result showed that the major methyl ester that influenced the character of the biodiesel (JAB) was methyl linoleate which was the same as the corresponding oil (JASO). The fatty acid analysis composition was observed to be in agreement with reports by several researchers [2, 17-20]. Also, the major fatty acid constituent of both the oil and the biodiesel were the same. The fatty acid methyl linoleate accounted for more than 70% of the unsaturated fatty acid component of the biodiesel and so was the major influence on the character of the biodiesel produced. Thus, the fatty acid components of the oil and biodiesel produced remained the same even after transesterification as reported by [22].

The Fourier transform infrared (FTIR) spectrum (spectroscopic analysis) of the jatropha seed oil (JASO) and jatropha oil biodiesel (JAB) are presented as Figures 2 and 3 respectively.

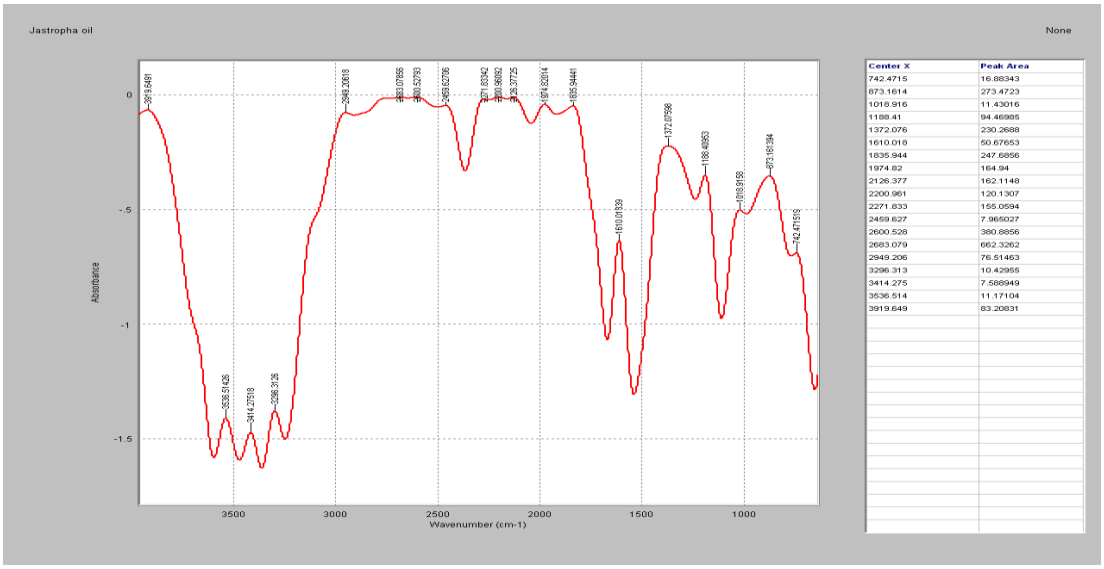

Figure 2: FTIR spectrum (spectroscopic analysis) of the jatropha seed oil (JASO)

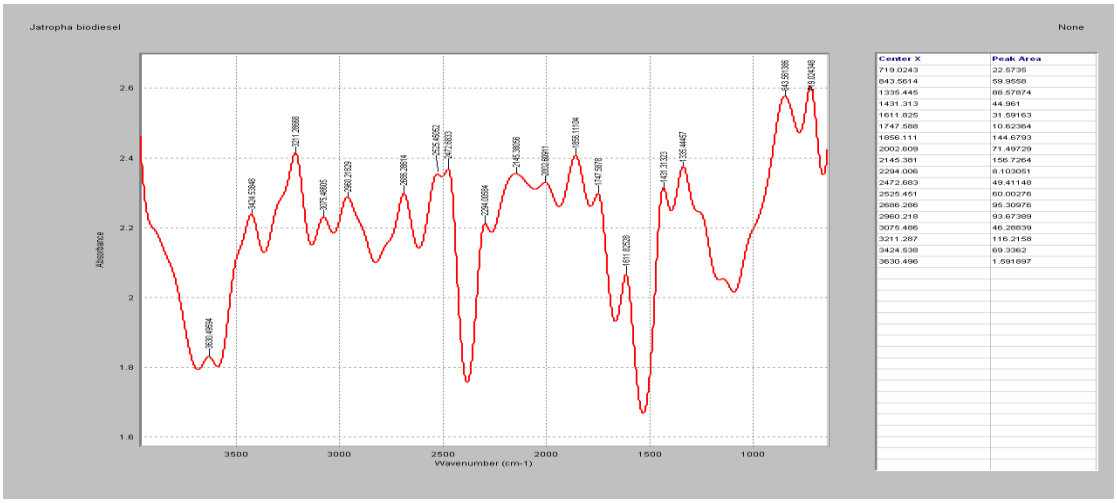

Figure 3: FTIR spectrum (spectroscopic analysis) of the jatropha oil biodiesel (JAB)

Figures 2 and 3 shows the FTIR spectra of jatropha seed oil (JASO) and jatropha oil biodiesel (JAB) respectively. The results indicated the presence of several functional groups. From both figures, the dominant functional groups of the biodiesel and oil were =C-H, C-O-C, RO-H, R-OH and C=O for the biodiesel and =C-H, CH_2, C=O and O-H groups for oil. The frequencies range from 1200 cm^{-1}, 1650 cm^{-1}, 1700 cm^{-1}, 220 cm^{-1}, 2900 cm^{-1} to 3600 cm^{-1}. However, other functional groups present are CH_2, C≡O, -C≡C-, >C=C<, RC≡N and OH in both the oil and biodiesel which had minimal influence on the oil and biodiesel to absorb (infrared) IR radiation at the investigated absorbance range (cm^{-1}). From the figures of the FT-IR spectrum of the JASO and JAB, the type of vibration causing IR absorbance in both the oil and biodiesel were symmetric stretching, asymmetric stretching, an in-plane bending vibration (scissoring), an out-of- plane bending vibration (twisting), split rocking, bending/rocking and symmetrical/stretching respectively. Isotopic effects were observed to be minimal with little effect on spring constant thereby shifting the vibrational frequencies with changes in the reduced mass due to isotopic substitution. The highest absorption band of the jatropha seed oil (JASO) was centered at 3919.649 cm^{-1} with peak area of 83.20831 at absorbance of -1.83. This represented the lowest transmittance associated with the sample at that particular condition. The lowest absorbance were at peak positions of 7.5889 (3414.2752 abs), 11.1710 (3536.5143 abs) and 10.4296 (3296.3126 abs) respectively. These were all slightly above absorbance of -1.5 and at wave number (cm^{-1}) between 3700 cm^{-1} and 3250 cm^{-1} [14, 24].

Figure 2 also shows the molecular identity of the jatropha seed oil (JASO) which was unique only to the oil, hence creating the desired identity for the jatropha seed oil in the present research. From Figure 3, the FTIR spectrum of the jatropha oil biodiesel (JAB) showed that the highest absorption of IR radiation were achieved at center points of 3211.28668 at absorbance of 2.42 abs, 1856.1110 (2.41 abs), 843.5614 (2.58 abs) and 719.0243 (2.62 abs) respectively. These points represented the lowest transmittance of the IR radiation on the jatropha oil biodiesel. The peak areas at these points were 116.2158, 144.6793, 59.9558 and 22.5735 respectively. The jatropha oil biodiesel (JAB) generally showed high absorbance of the IR radiation as shown in the FTIR spectrum hence a minimum transmittance was observed since most of the infrared radiations were virtually absorbed at the different wave numbers or frequencies. The highest transmittance was achieved at peak position 3630.49594 (1.82 abs). The absorbance at that point was far greater than the transmittance and thus prevailed over the transmittance resulting in virtually all the radiation being absorbed. Figure 3 shows the molecular fingerprint of the jatropha oil biodiesel produced in the present study which was unique only to the biodiesel sample (JAB) [13, 14].

The oil and biodiesel each had a unique combination of atoms which validated the fact that no two compounds can have or produce the exact same infrared spectrum. Using infrared spectroscopy

analysis, the oil and biodiesel were positively identified (a form of qualitative analysis) with the size of the peak in the spectra giving a direct indication of the amount of material present in addition to the different kinds of atom in the compounds. The use of infrared spectroscopy in the analysis of the oil and biodiesel was based on the principle that almost all molecules absorb infrared light at those frequencies where the infrared light affects the dipolar moment of the molecule. In the oil and biodiesel molecule studied, the differences of charges in the electronic fields of their atoms produce the dipolar moment of the molecules. The molecules with dipolar moments allowed infrared photons to interact with other molecules causing excitations to higher vibrational states.

4.0 Conclusion

Physicochemical composition and functional (spectroscopic) properties analysis of jatropha seed oil and biodiesel were evaluated in this paper. Spectroscopic analysis of jatropha seed oil and biodiesel using Fourier infrared spectroscopy presented a spectrum or fingerprint of the oil and biodiesel with absorption peaks. The peaks corresponded to the frequencies of vibrations between the bonds of the atoms making up the oil and biodiesel. The spectra produce were unique to the oil and biodiesel and served as the molecular fingerprint for further sample identification, characterization and profiling. The size of the peaks, identified by the peak area indicated the amount of functional groups present and defined the character of the oil and biodiesel. This additional analysis method provided by infrared spectroscopy further deepened the study/research on the nature and composition of the oil and biodiesel for greater usage and development. It provided a means of determining the quality and consistency of the oil and biodiesel and the amount of components in each mixture in addition to the physicochemical composition.

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