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Characterization of Biodiesel Produced by Transesterification from Groundnut Oil

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Abstract
The need for alternative source of energy with little to no harmful effect and has economic advantage over the current sources has led to intensified efforts in harnessing natural resources to produce renewable energy. The current work characterized biodiesel produced from groundnut oil by trans-esterification. The moisture content, ash content, density, viscosity of the biodiesel produced were assessed. The FTIR spectroscopy of the feedstock and the product were also determined. Results from this work showed an ester yield of 62.5%, which is a little lower than that obtained by some researchers. The moisture content was shown to be 3.45%, the ash content was 0.2%, and the density was found to be 0.76 g /cm3. The viscosity was found to be 488.21 mm²/s at 27.3 °C. Trans-esterification method is found to be good in producing biodiesel from groundnut as corroborated by several investigations. Due to high viscosity of the biodiesel produced, it is recommended that the product should be used in blend with fossil diesel and NMR can be used for further characterization.

Key words: Biofuel, groundnut, trans-esterification, NMR, viscosity

INTRODUCTION
Crude oil, coal and gas are the main dominant resources of world energy supply (Shafiee *et al.,* 2009). However, due to the rising crude oil prices, depletion of petroleum reserves and more importantly increasing environmental concerns, such as deposition of sulphur, carbonic and nitric acids, urge researchers to search for cleaner and renewable alternate sources of energy (API, 2006).

Biodiesel can serve as an alternative source of energy. It is a pure fuel before blending with diesel fuel. The blends are denoted as, "BXX" with "XX" representing the percentage of biodiesel contained in the blend (i.e. B20 is 20% biodiesel, 80% petroleum diesel, B100 is pure biodiesel) (Coronado, 2009). Furthermore, it has some properties that are close to those of diesel fuel such as high biodegradability (European tests of rapeseed-based biodiesel showed that it is 99.6 percent biodegradable within 21 days (NBD, 1993) and emission of lower harmful substances (Canakci *et al.*, 1999). It can also be used in any diesel engine without necessary modifications. It can be mixed with fossil diesel in any proportion, without problem (Hilbert, 2007). Biodiesel has recently been described as a safe fuel, because its flash point is more than 100°F higher than that of diesel, degrades about four times faster than conventional diesel (Tyson *et al.,* 2001) and has toxicity which is at least 15 times less than that of diesel. This shows that it can be classified as a non-flammable liquid.

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Vegetable oil based biodiesel has potential to replace petroleum based fuels in the long run (Ramadhas *et al.*, 2004). Vegetable oils and animal fats contain triglyceride, which is composed of three long chains of fatty acid molecules chemically bonded together to a glycerol molecule. To make diesel, we can use fatty acid methyl esters (FAMEs) biodiesel, fatty alcohols, alkanes, and linear or cyclic isoprenoids (Lee *et al*., 2008). The idea of using vegetable oils as fuel for internal combustion engines is from 1895, when Rudolf Diesel developed his engine. In presenting the diesel engine, Mr. Diesel used peanut oil as fuel (Knothe *et al*., 2005).

Groundnut (*Arachis hypogaea*) also known as peanut is the world's fourth most important source of edible oil (Govindaraj *et al.,* 2009). It is grown in nearly 100 countries on six continents in over 23,712,204 hectares of land. Developing countries account for over 97% of the world groundnut area and 95% total production (FAOSTAT, 2014). It is the 13thmost important food crop of the world; its seeds contain high quality edible oil (50%), easily digestible protein (25%) and carbohydrates (20%) (FAO, 1994). Groundnut is mainly grown in the northern part of Nigeria; over 85% of the groundnuts produced in the country were accounted for by Kano, Kaduna, Taraba, Bauchi, Borno, and Adamawa states (Abalu *et al.,* 1979).

Studies have shown that groundnut oil is rich in potassium and a good source of calcium, phosphorus and magnesium. It also contains vitamins like vitamin E as well as elements such as selenium, zinc and an amino acid arginine (Evans *et al.,* 1966). Groundnut oil is of good quality and can tolerate higher temperatures without burning or breaking down. It is a good source of palmitic, oleic and linoleic acids. Stearic, arachidonic, eicosanoic and lignoceric acids are also found in small amount (Casini *et al.,* 2003). It is an excellent source of phytosterols (Awad *et al*., 2000), and has higher percent of Avenasterol than soybean oil. Groundnut oil was found to contain good amount of iron and cupper, which serve as cofactors for some enzymes (Asemav *et al.,* 2012).

Although an edible oil, the use of groundnut oil for biodiesel production might not affect crops cultivated for food as Nigeria is ranked the 4th in groundnut oil production (USDA, 2009).

Trans-esterification, the most used approach in producing biodiesel, is a reaction of triglyceride (e.g vegetable oil or animal fats) reaction with a short-chain monohydric alcohol (methanol or ethanol) in the presence of a catalyst at about 60^{o}C.The resulting products contain alkyl ester, unreacted starting material, residual alcohol, catalysts, and glycerol (Knothe *et al*., 2007).

The high viscosity of the oils (about 10 times more than diesel) limited their use, because this implies a poor fuel atomization and an incomplete combustion. The high flash point of vegetable oils and their tendency to oxidize thermally hindered also the use of the oil, due to the formation of deposits in the injector nozzles and a decrease in lubricity. However, an attempt was made to modify the properties of the oils to approximate those properties to those of diesel, through other methods such as dilution, microemulsion, pyrolysis, or transesterification (Agarwal, 2007). However, due to low oil prices, the fossil diesel took an important place and the use of vegetable oil was not developed as an alternative (Mittelbach *et al.*, 2004).

Groundnut oil ethyl ester was found to have better fuel quality than raw groundnut oil and it can be used successfully to fuel a diesel engine (Oniya and Bamgboye, 2014). Groundnut oil can be used perfectly for biodiesel production in country with groundnut oil without affecting the oil supply for consumption purpose. Hence, the utilization of groundnut oil for biodiesel production might not affect the supply of the vegetable oil for consumption (Hill, 2002).

Therefore, groundnut oil can be used as alternative fuel for diesel engine (Bello and Fatimehin, 2015)

MATERIALS AND METHOD

Materials and Equipment

- Erlenmayer flask
- Magnetic stirrer
- Water bath
- Ethanol
- Potassium hydroxide
- Groundnut oil
- Measuring cylinder
- Electronic scale

Production of Biodiesel from Groundnut Oil

Fresh groundnut oil was obtained from *'Yan tifa* market in Dutse, Jigawa State, Nigeria. Since groundnut oil contains low free fatty acid value (Ramadhas *et al*., 2009); acid pre-treatment to convert them to ester was not carried out. The oil was trans-esterified to produce alcohol esters using a closed reactor in a two-step trans-esterification process as developed by Peterson *et al*., (1996).

Before the reaction, groundnut oil was preheated in a water bath at 60^{o}C.Ethanol was added at a molar ratio of 5:1 (ethanol to oil). Potassium hydroxide was then added, to form potassium ethoxide, and stirred using a magnetic stirrer.

The weight of potassium hydroxide used as catalyst during trans-esterification was calculated from the following formula as recommended by Peterson *et al.* (1996) in equation 1

$$KOH = 0.013 \times GO \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (1)$$

Where: *GO = the desired volume of groundnut oil processed in litres;*

KOH = Weight of potassium hydroxide required in kilogram.

The reaction temperature of 60^{o}C was selected as suggested by Alamu *et al.* (2007).

Three operations were performed in the production of the groundnut oil ethyl ester—the trans-esterification, separation and washing processes.

After initial stirring for one hour, the mixture was allowed to settle for 48 h. After settling and completion of separation, glycerol, which is the heavier liquid, collected at the bottom, while the ester product was at the top, as shown in Figure 1.

The next step, the washing process. The method used for washing, which consists of two steps developed, by Peterson *et al*. (1996), is described as follows:

a) The glycerol layer was re-mixed with the ester layer after initial settling has occurred, then 15% water was added and the entire mixtures were re-stirred for 10 min and allowed to settle for 48 h. After settling, the glycerol layer is at the bottom of the flask—the whitish layer.

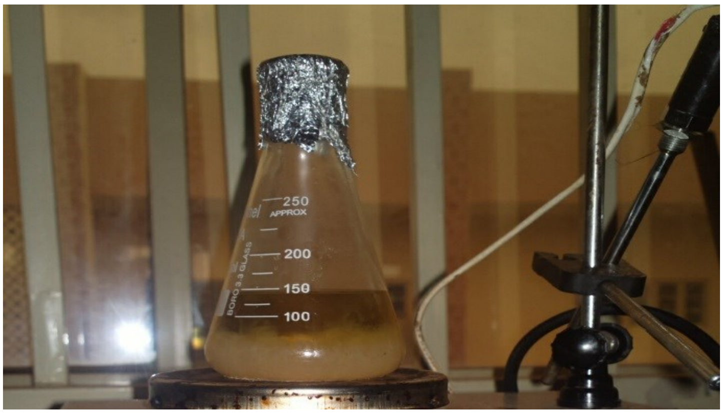


Figure 1. The ester layer at the top while the glycerol later at the bottom

Using a separating funnel, the glycerol layer was filtered away, as shown in Figure 2

Figure 2. The separating funnel was used for the separation

As shown in Figure 3, the glycerol layer settled at the bottom of the separating funnel. The switch at the lower end of the separating funnel was opened so that the ester, which is at the top, can be collected in the flask while the glycerol is left in the separating funnel.

Figure 3 shows the collection of the biodiesel in the flask.

Figure 3. The glycerol layer remains in the separating funnel during the collection

b) The ester product was washed after draining off the glycerol layer. The ester was washed with water at about 30% of the ester volume. The water was stirred into the ester with mechanical stirring using a blender. After 10 min, the stirring was stopped, and the water was allowed to settle for two days. At this point, the process was complete and the clear product was the groundnut oil ethyl ester.

Proximate Analysis

Determination of Ester Yield

The yield of the ethyl esters, Y, produced was calculated using the formula in equation 2.

$Y = Ve/Vr \text{X}\ 100\ldots$ ……………………………………………….(2)

(Oniya and Bamgboye, 2014)

Where: Y = *Yield of the ethyl esters,* V_e = *Volume ethyl esters produced in litres;* V_r = *Volume raw oil used in litres.*

Biodiesel Density Measurement

Density of biodiesel was determined by gravimetric analysis measuring a volume of 25ml biodiesel with a glass cylinder and weighting the sample on the electronic scale. The density calculated using the equation 3below:

$$p = \frac{m}{v} \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots . . (3)$$

(Ojolo *et al.*, 2012)

Where: m = weight of sample in g

v = volume of the sample in cm^3

Biodiesel Viscosity Measurement

The viscosity of biodiesel was measured using a digital viscometer as in Figure 4

Figure 4. The digital viscometer used for measuring the viscosity of the biodiesel

Unlike the Falling-ball viscometer, the Digital viscometer automatically displays the viscosity of fluids; so no further calculation was done.

RESULTS AND DISCUSSION

Trans-esterification results is summarized in table 1 below;

Table 1. Trans-esterification results

Experiments	Results
Ash content	0.2%
Moisture content	3.45%
Viscosity	308.3mPa/s
Density	0.76g/cm^3
Ester yield	62.5%

Yield of Ester

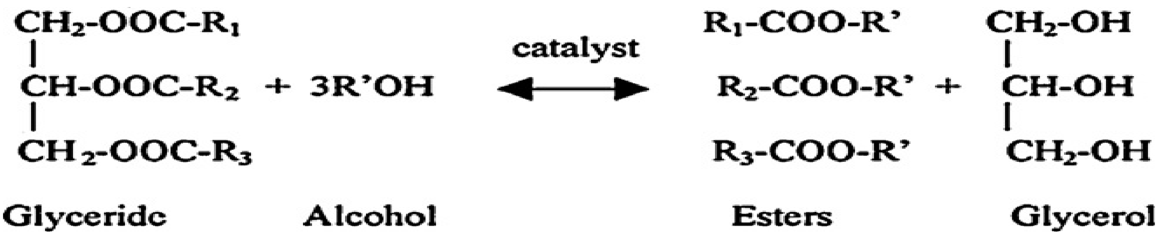
$$\text{Yield of ester (\%)} = \frac{\text{Volume of ethyl ester produced (Ve)}}{\text{Volume of raw oil used (Vr)}} \times 100$$

$$= \frac{0.15\ \text{l} \times 100}{0.24\ \text{l}}$$

$$= 62.5\%$$

The trans-esterification process yielded 62.5% groundnut oil ethyl ester on a volume basis. The yield of ethyl ester was quite lower than the value obtained by Alamu *et al.* (2007) who worked on alkali-catalysed trans-esterification of palm kernel oil, and the process yielded 95.80% palm kernel oil ethyl ester. Issariyakul *et al*. (2007) carried out a research and found that acid catalysed trans-esterification yielded more than 90% ester. While Oniya and Bamgboye (2014) obtained 86.8% groundnut oil ethyl ester on a volume basis.

Many methods such as blending, trans-esterification, cracking, micro-emulsification and pyrolysis have been used to convert vegetable oils into fuels with biodiesel properties (Sinha *et al.,* 2008). Trans-esterification is the preferred method for production of biodiesel due to better yield of the product (Yan *et al.,* 2009). The vegetable oil react with the mixed alcohol in the presence of catalyst to form crude biodiesel and crude glycerin. One molecule of vegetable oil/triglyceride reacts with three molecules of alcohol and produces three molecules of monoglyceride and one molecule of glycerol. The reaction is shown in the reaction below (Ali and Hanna, 1994; Vicente *et al.,* 2004).



Oniya & Bamgboye (2014) reported that groundnut seeds gave oil yield of 45.3%, Ibeto *et al.* (2011) obtained a yield of 79%, while Galadima *et al.*(2008) obtained 88% yield.This means that, the yield of ester depends on the feedstock used and the catalyst used for the trans-esterification reaction. The low yield obtained may be attributed to some contents like vitamins present in the groundnut oil since it was obtained commercially.

Moisture Content Determination (AOAC, 2000)

Initial weight of petri dish with sample (W_1) = 78.51 g

Weight of petri dish and lid after the drying (W_2) = 75.80 g

$$\text{Moisture content (\%)} = \frac{(W_1 - W_2)}{W_1} \times 100$$

$$= \frac{(78.51 - 75.80)\ g}{78.51\ g} \times 100$$

$$= \frac{2.71\ g \times 100}{78.51\ g}$$

$$= 3.45\%$$

Moisture content is an important factor that affect the yield of biodiesel. The moisture content obtained in this study was 3.45% which is higher than 0.05% as obtained by Jimoh *et al.* (2012), and 0.09% reported by Ibeto *et al.* (2011). The 3.45% value obtained is a bit higher than the recommended byAmerican Society for Testing and Material (ASTM-D 6571). It has been reported that moisture content greater than 3% will reduce the efficiency of the trans-esterification reaction due to possibility deactivation of catalyst active sites and soap formation (Freedman *et al.*, 1984).

According to Patricia *et al*. (2012) biodiesel has a higher affinity toward moisture content than petroleum diesel, and the water retaining capacity of biodiesel is higher than that of diesel. They also found that at constant relative humidity, biodiesel absorbed 6.5 times more moisture than diesel and temperature in the range of 283.15 to 323.15K. Therefore, the higher moisture content obtained, 3.45%, may be due high humidity and/or high temperature during the washing process.

Ash Content Determination(AOAC, 2000)

Weight of ash = 0.01 g

Weight of sample = 5 g

$$\text{Ash content (\%)} = \frac{\text{Weight of ash}}{\text{Initial weight of sample}} \times 100$$

$$= \frac{0.01\text{g} \times 100}{5\text{ g}} = 0.2\%$$

The ash content obtained in this study was 0.2% which is slightly higher than the value described by AGO—0.12% (Oniyaand Bambgboye, 2014).

Density

$$\text{Density} = \frac{\text{Weight of sample (g)}}{\text{Volume of sample (cm}^3\text{)}}$$

$$= \frac{114.58\text{ g}}{150\text{ cm}^3}$$

$= 0.76$ g/cm^3 which is the same as 0.76 g/ml

The density obtained was 0.76 g/cm^3. The value is slightly lower than the value described by Deepak *et al*. (2016) which was 0.86 g/ml. According to Blangino *et al.* (2008) density depends on upon the raw materials used for biodiesel fuel production and the biodiesel ester profile.

Viscosity

The kinematic viscosity obtained at 27.3^{o}C was 380 mPa/s which is equivalent to 488.21 mm^2/s—which is highly viscous compared to the values in the range of 3.5 to 5.0 mm^2/s. According to FEE (2019) the viscosity can be corrected by blending the biodiesel with a fuel—fossil diesel—that has a lower viscosity. Also, the density of the biodiesel affects the viscosity of the biodiesel—longer and straight chains (saturated fats) tend to have higher density than shorter and unsaturated molecules. According to Deshpande *et al.* (2012), higher viscosity is due to the presence of hydroxyl group (-OH) at the 12th carbon molecular structure. Therefore, the nature of the feedstock used can affect the viscosity of the biodiesel. At higher temperature, the viscosity tends to be lower than at lower temperature.

The Groundnut Oil

The Figure 5 shows the IR (Infrared) spectrum of the groundnut oil sample used. The result was similar to the result obtained by Neffer *et al.*(2011) as shown in Figure 6.

Absorption bands at 3009.08 cm^{-1} indicates (=C-H) stretching of the carbon-carbon double bonds; absorption bands in the range of 2926.14 cm^{-1} and 2854.77 cm^{-1} indicates (C-H) stretching of the saturated carbon-carbon bonds; 1746.62 cm^{-1} absorption indicates (C=O) stretching of the carbonyl function group; 1650.0 cm^{-1} absorption indicates (C=C) stretching of the carbon-carbon double bonds; 1163.13 cm^{-1} absorption indicates (C-O-C) stretching of the ester function group and 722.37 cm^{-1} assigned to (C-H) out of the plane stretching of the saturated carbon-carbon bonds (Neffer *et al.*, 2011).

FTIR Spectra of the Groundnut Oil

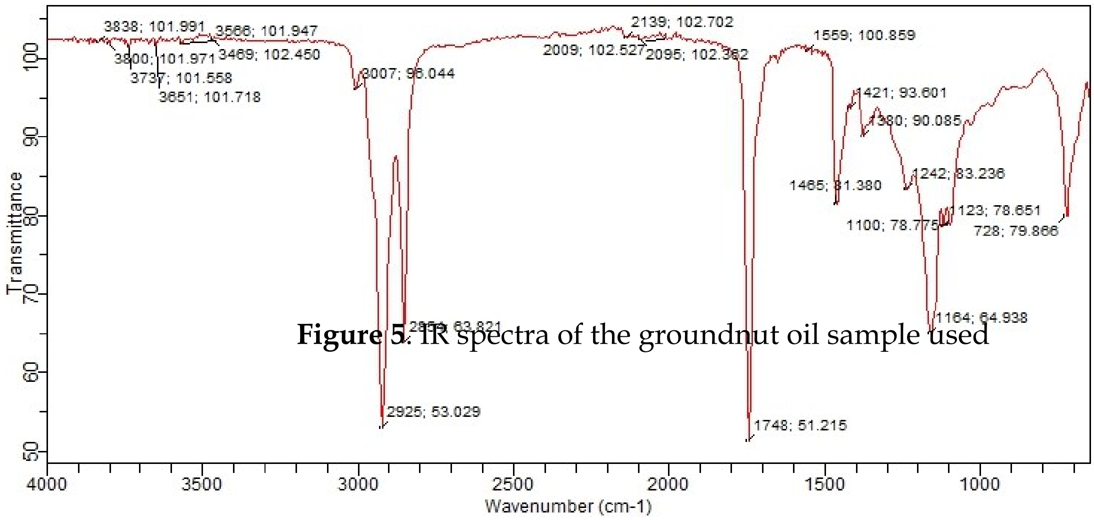
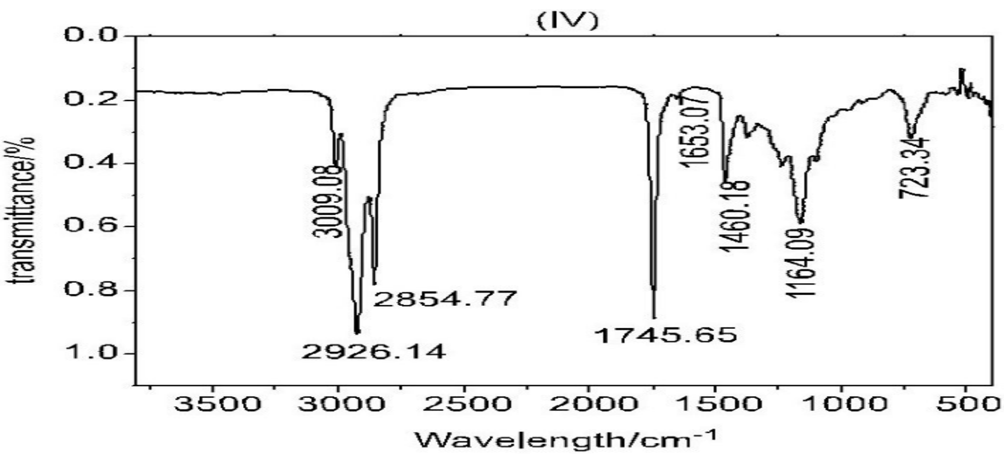


Figure 5: IR spectra of the groundnut oil sample used



(Gomez *et al.,* 2011

Figure 6 IR spectra of the groundnut oil in this Figure is similar to the one in Figure 5

FTIR Spectra of the Biodiesel produced in this study

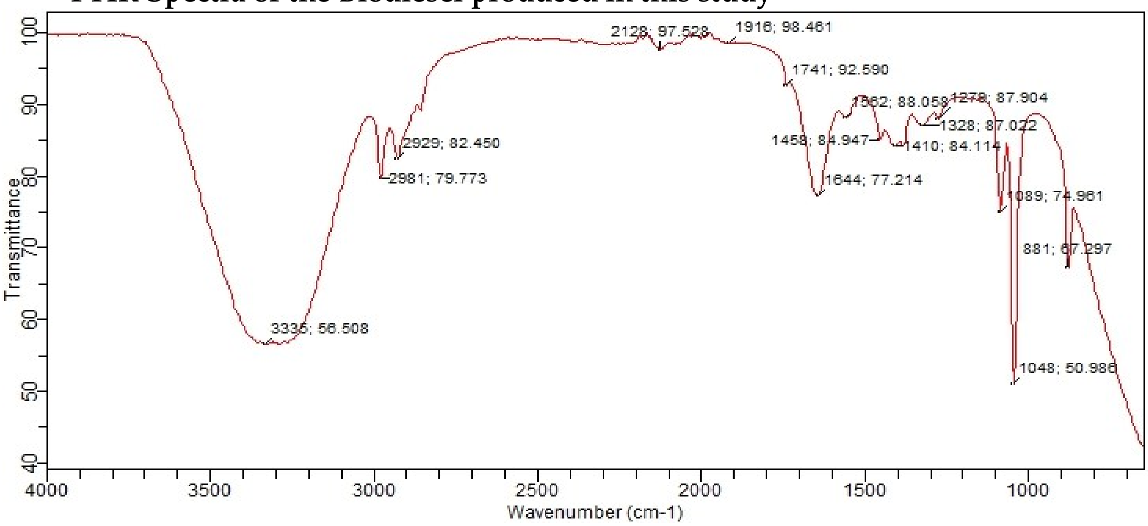
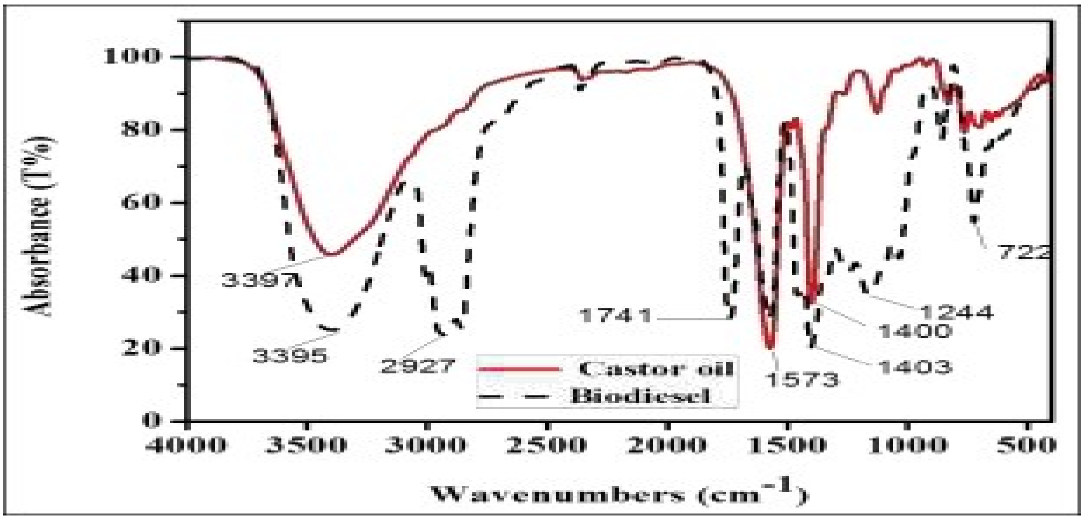


Figure 7.IR spectra of the biodiesel produced



(Rengasamy *et al.*, 2014)

Figure 8.IR spectra of biodiesel from Castor oil

IR Spectra of Biodiesel produced

Figure 7 shows the IR spectra of the biodiesel produced. The result was similar to the one in Figure 8, which is IR spectra of biodiesel from Castor oil according to Rengasamy *et al.,*(2014).The band at 3335 cm^{-1} indicates the presence hydroxyl group (OH); at 1458cm^{-1} it is an indication of the presence of C-C stretching; 1244cm^{-1} band represent the presence of C(=O)-O. Figure 9 shows details on both groundnut oil and biodiesel spectra.

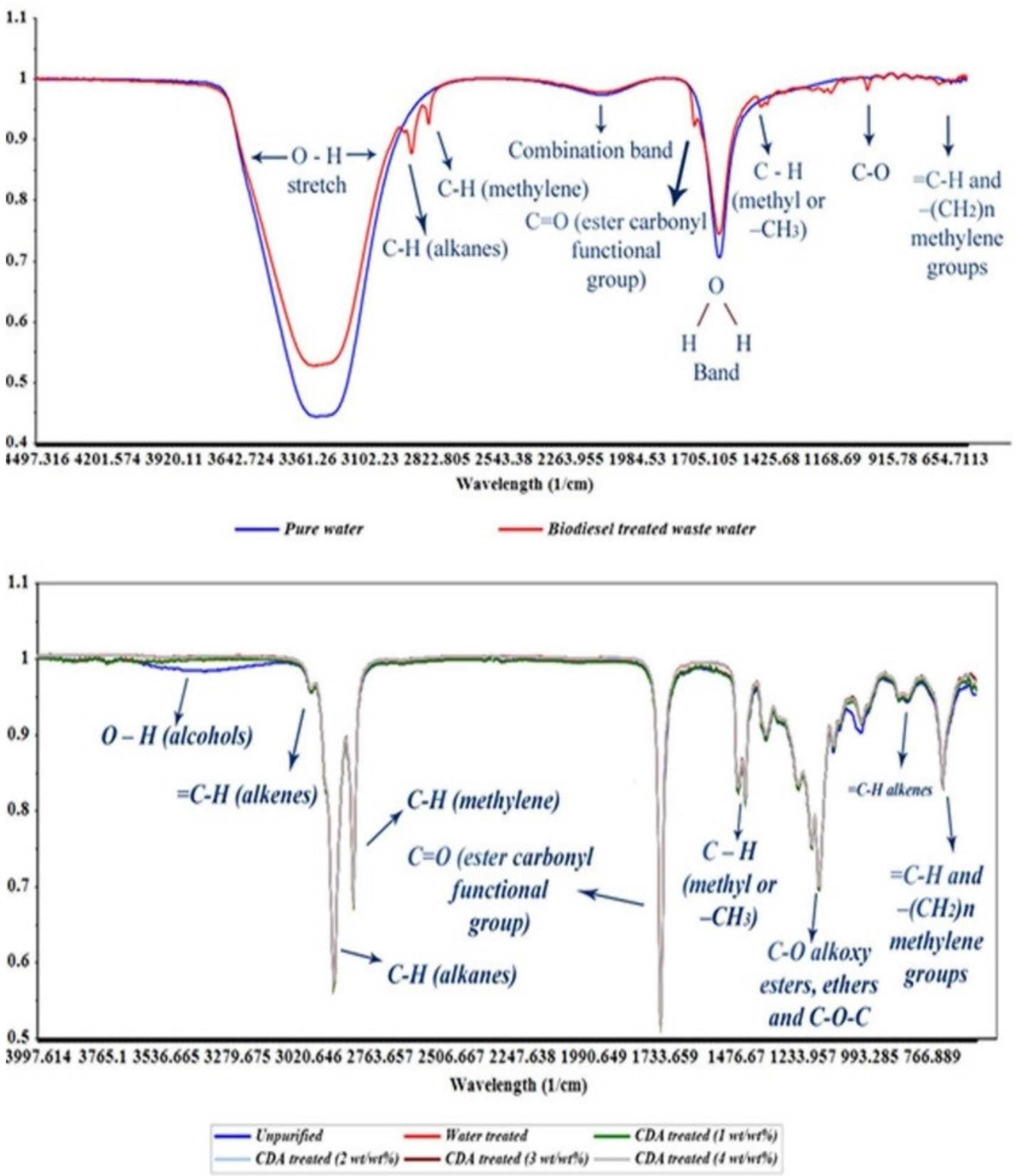


Figure 9. Spectra of biodiesel (top) and groundnut oil (bottom) (Avinash *et al*., 2017).

CONCLUSION

In this work, the percentage yield of ester was found to be 62.5%, which is a bit lower than that obtained from other experiments. The ash content, moisture content, viscosity and density were found to be 0.2%, 3.45%, 308.3 Pa/s and 0.76 g/cm^3 respectively.

The production of biodiesel from groundnut oil by trans-esterification can give a better fuel quality, ease of operation and yield, however, some factors such as cost, properties, production methodology, required equipment, etc. should be considered.

The product of this study should be blend with the fossil diesel because high viscosity can cause damage to diesel engines.

RECOMMENDATIONS

FTIR spectroscopy was carried out for the characterization of the groundnut oil sample and biodiesel produced, Nuclear Magnetic Resonance (NMR) spectroscopy is recommended for further characterization.

It is recommended that acid pre-treatment should be carried out in order to produce biodiesel with lower viscosity.

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