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PHYSICOCHEMICAL ANALYSIS AND CHARACTERIZATION OF BIODIESEL PRODUCTION FROM *RICINUS COMMUNIS* SEED OIL

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ABSTRACT

The global energy concern with the availability of recoverable fossil fuel reserves and the environmental problems caused by those fossil fuels, considerable attention has been given to biodiesel production as an alternative to petro diesel worldwide. In this research, the seed oil of *Ricinus communis* (castor bean) was investigated for its viability as a feedstock for biodiesel production. The oil was extracted from the seed using petroleum ether .The oil quality characteristics of the seed oil were; Acid values for both *Ricinus communis* oil and biodiesel, 4.208 and3.93, and free fatty acids for both *Ricinus communis* and biodiesel were 1.2408 and 2.508, saponification value (SV) for both *Ricinus communis* and biodiesel,, 185.0 and 173.16, specific gravity (S.G) for both *Ricinus communis* and biodiesel, 0.976g/ml and 0.935g/ml. the PH value for both *Ricinus communis* and biodiesel, 6.67 and 6.8. Refractive index for both *Ricinus communis* and biodiesel, 1.37 and 1.191, moisture content of seed, 0.052. Colour for both and *Ricinus communis* oil and biodiesel is brown colour and amber colour, respectively. The oil was transesterified using methanol and concentrated sulphuric acid. And with other investigated oils from the literature and were found to fall within acceptable limits, this implies that *Ricinus communis* seed oil could be used as diesel in combustion engines in tropical climates like Nigeria.

Keywords: Biodiesel; *Ricinus communis,* FTIR*;* Petroleum ether and Transesterification.

INTRODUCTION

Biodiesel is an alternative fuel for diesel engines that is produced by chemically reacting vegetable oil or animal fat with alcohol and catalysts. Biodiesel is miscible with diesel fuel and can be easily blended with diesel fuel with minor or no modifications to the engine and fuel system. According to EU guidelines, the consumption of biofuels for road transportation should represent 20% of the total fuel consumption by 2020, and the use of biofuels will be stimulated by environmental aspects. For these reasons, biodiesel has become a popular topic in energy sources (Zhang *et al*., 2003).

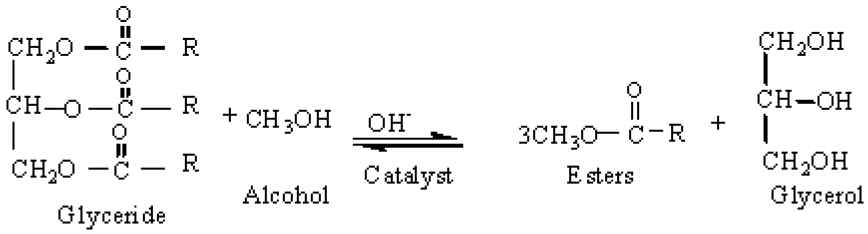
Biodiesel is implied to be operated in excellent diesel engines and is separated from the vegetable and waste oils operated in fuel converted diesel engines. Methyl ester biodiesel can operate alone and or combined with petroleum diesel in any quantity and can also be operated as heating oil (Omidvarborna; *et al*., 2014).

Biodiesel is a nondepleted energy source that enhances greenhouse gas emissions, GHG reduction, and decreases the carbon footmark in agriculture. Biodiesel also provides a very lower global warming because the carbon in the fuel is detached from the air by the plant raw material (Sheehan *et al.*, 1998).

The most common way to produce biodiesel is the trans-esterification method, which refers to a catalyzed chemical reaction involving vegetable oil and alcohol to yield fatty acid alkyl esters (i.e., biodiesel) and glycerol. The reaction requires a catalyst, usually a strong base, such as sodium and potassium hydroxide or sodium methylate. A catalyst is usually used to improve the reaction rate and yield. Since the reaction is reversible, excess alcohol is used to shift the equilibrium to the product side. Especially methanol is used as alcohol because of its low cost and its physical and chemical advantages. Methanol can quickly react with vegetable oil and NaOH can easily dissolve in it (Sheehan *et al.*, 1998).

The Transesterification process is a method whereby the reaction of a triglyceride of fat and oil with alcohol either methanol or ethanol, is used to form esters and glycerol. A triglyceride contains a glycerin molecule as its base with three long chain fatty acids attached to it. The presence of fat is determined by the nature of the fatty acids attached to the glycerin. The nature of the fatty acids can in turn affect the features of biodiesel. Throughout the esterification procedure, the triglyceride is reacted with alcohol in the presence of a catalyst, usually a strong alkaline like sodium hydroxide or potassium hydroxide. The alcohol then counters with fatty acids to form the mono-alkyl ester, or biodiesel and some crude glycerol (Knothe *et al.* 1997).

The chemical route for biodiesel are given in Figure 1 below;



Scheme 1: Chemical routes for Biodiesel (Zhang *et al*., 2003).

Among the methods, the transesterification procedure is most suitable for industrial manufacture of biodiesel. Biodiesel can also be obtained from alcohols other than oil of vegetables and fats from animals, which can be used in compression ignition engines or mixed with normal diesel oil. The ASTM International identified this fuel as a combination of long chain monoalkylic esters with fatty acids found in renewable resources to be performed in compression ignition engines (Knothe *et al.* 1997).

MATERIALS AND METHODS

SAMPLING

The castor fruits were collected from State Low-cost Damaturu, Yobe State. It was identified by a Taxonomist Salihu Abdullahi in the Department of Biological Science Yobe State University, Damaturu. The samples were dried in the sun for 24 hours and separated the beans from the seed shell. The dried seeds were ground into powdered form by using a simple grinder machine and stored in a cool dry place.

SOXHLET EXTRACTION

The soxhlet extractions were performed using distilled water, petroleum ether, hexane, and ethanol. The solvents chosen for the present study are normally used to extract oil from *Ricinus communis* seeds. 150 mL of solvent was poured into a round-bottom flask and 10 g seeds were added. The flask was placed inside the heating mantle. Extractor was connected to the flask and then the condenser was connected to the extractor. The solvent was boiled and evaporated, the vapour was condensed and dropped into the extractor. The solvent dripped back into the round-bottom flask. This evaporation-condensation process continued for 3 to 6 hours. Then the apparatus was allowed to cool. The cold solvent-seed oil mixture was transferred into a beaker and oven heated at 700C to remove solvent. Soxhlet extraction of *Ricinus communis* oil from seeds was carried out.

DETERMINATION OF MOISTURE CONTENTS OF THE SEEDS

40 g of the cleaned sample was weighed and dried in an oven at 80°C for 7 hrs and the weight was taken after every 2 hrs. The procedure was repeated many times until a constant weight was obtained. After each 2 hours, the sample was taken out from the oven and placed in the desiccator for 30 minutes to cool. It was then taken out and reweighed. The moisture percentage in the seed were calculated by the formula,

% moisture = 100% (W1-W2)/W2

Where W1 = original weight of the sample before drying;

W2 = weight of the sample after drying.

DETERMINATION OF FREE FATTY ACID VALUE:

25 ml of diethyl ether and 25 ml of ethanol were mixed in a 250 ml beaker. The mixture was added to 10 g of oil in a 250 ml conical flask and few drops of phenolphthalein were added to it. Then this mixture was titrated with 0.1M NaOH to the end point. Free fatty Acid (FFA) was calculated by the formula, FFA = Vo/Wo ×2.8×100

Where, 100 ml of 0.l M NaOH = 2.83 g of free fatty acid, Wo = sample weight;

Then acid value = FFT×2

DETERMINATION OF PH VALUE

Samples of 2gm was each poured into a 25ml glass beaker, 13ml of hot distilled water was added to each sample and stirred slowly. The mixtures were then cooled in a cold-water bath to 25°C. The pH meter electrode was standardized with buffer solution and then immersed into the sample and the pH value was measured (Akpan *et al.,* 2006).

DETERMINATION OF SPECIFIC GRAVITY

Density bottle of 5ml capacity was weighed (W0), filled with oil, then the stopper inserted and reweighed (W1). The oil was substituted with water after washing and drying the bottle and weighed (W2). The expression for specific gravity is:

Sp.gr = (W1-W0)/ (W2-W0) = Mass of the substance / Mass of an equal volume of water (Akpan *et al.,* 2006).

DETERMINATION OF REFRACTIVE INDEX

Few drops of oil samples were transferred to the glass slides of the refractometer (ATAGO Co., Ltd. Japan). Through the eyepiece of the refractometer, the dark portion viewed was adjusted to be in line with the intersection of the cross, in this case the pointer on the scale pointed to the refractive index and values were recorded (Akpan *et al.,* 2006).

DETERMINATION OF ACID VALUE

25ml of each of diethyl ether and ethanol was mixed in a 250ml beaker, then was added to 10gm of oil contained in a 250ml conical flask and a few drops of phenolphthalein were added. The mixture was titrated with 0.1M NaOH to the end point with consistent shaking, a dark pink color was observed, and the volume of 0.1M NaOH (V0) was recorded.

Free Fatty Acid (FFA) was calculated (Akpan *et al.,* 2006; Kyari, 2008) as below.

V0/W0·2.82·100

100ml of 0.lM NaOH = 2.83gm of oleic acid

W0 = sample weight

Then, acid Value = FFA.2

DETERMINATION OF SAPONIFICATION VALUE

Two grams of oil sample was weighed into a conical flask and 25ml of 0.1N ethanolic potassium hydroxide (KOH) was added. The mixture was constantly stirred and allowed to boil gently for 60min. A reflux condenser was placed on the flask containing the mixture. Few drops of phenolphthalein indicator

Were added to the warm solution and titrated with 0.5M HCl to the end point until the pink color of the indicator disappeared. The same procedure was used for other samples and blanks. Saponification value was calculated (Akpan *et al.,* 2006; Kyari, 2008) as below:

S.V = 56.1 N (V0-V)/M

V0 = volume of the solution used for the blank test

VI = volume of the solution used for determination

N = Actual normality of the HCl used

M = Mass of the sample indicator method

TRANS ESTERIFICATION

The following basic step were followed (Penugonda and Venkata, 2012) to Produce Biodiesel from castor oil

1 25ml of castor oil were taken.
2. Add 7.5 ml of methanol & 0.25ml of concentrated sulphuric acid to oil.
3. The temperature of this mixture is to be set at 65-70ºC and to be maintained for about 6hrs. With continuous stirring.
4. The mixture was allowed to settle for 8 hours for complete end reaction.
5. The settled reactant mixture would consist of two layers, the upper layer as biodiesel and traces of glycerin, etc. and the bottom layer as glycerin and gum etc.
6. Glycerin was removed from the biodiesel preparation unit by opening the tap provided on the bottom and this pre-washed biodiesel is transferred to a separating funnel.
7. Add 200 ml of hot water at approximately 40º C per liter of crude biodiesel with shaking and allowed to settle to separate the two layers for nearly 7-8 hrs.
8. Repeat the above step at least three times to eliminate traces of glycerin and soap from the biodiesel yield.

FTIR SPECTROMETRY

The sample was placed in contact with sodium chloride screen and FT-IR spectra were collected in frequency 4500-400cm-1 by coadding 32 scans and at a resolution of 4cm-1. All spectra were rationed against a background spectrum. In each scan, a new reference background spectrum was detected. These spectra were recorded as absorbance values at each data point in triplicate.

RESULTS AND DISCUSSION

Physicochemical properties of *Ricinus communis* seed oil

The physicochemical properties are important parameters that give significant information as well as the applications of the oil. These properties comprise of acid value, saponification value, PH, free fatty acids, specific gravity, refractive index, castor, moisture content, and colour. The physicochemical properties of *Ricinus communis* oil and refined oil

Table 1, Physicochemical properties of *Ricinus communis* oil

S/N	PARAMETER	VALUES
1	ACID VALUE	4.208
2	FREE FATTY AACID	1.2404
3	SAPONIFICATION VALUE	185.04
4	PH	6.67
5	SPECIFIC GRAVITY	0.976
6	REFRACTIVE INDEX	1.37
7	MOISTURE CONTENT	0.052
8	COLOUR	BROWN

Table 2: physicochemical properties of biodiesel

S/N1	PARAMETER	VALUES
1	ACID VALUE	3.93
2	FREE FATTY AACID	2.508
3	SAPONIFICATION VALUE	173.16
4	PH	6.8
5	SPECIFIC GRAVITY	0.935
6	REFRACTIVE INDEX	1.191
8	COLOUR	AMBER COLOUR

TABLE 3: COMPARATIVE PHYSICOCHEMICAL PROPERTIES OF BOTH *RICINUS COMMUNIS* OIL AND BIODISEL

PARAMETERS	*R. COMMUNIS* OIL	BIODIESEL	ASTM 6751
ACID VALUE	4.208	3.93	
FREE FATTY AACID	1.2408	2.508	
SAPONIFICATION VALUE	185.04	173.16	< 200
PH	6.67	6.8	
SPECIFIC GRAVITY	0.976	0.935	0.888
REFRACTIVE INDEX	1.37	1.191	1.476-1.479
MOISTURE CONTENT	0.052	_	
COLOUR	Brown	Amber Colour	

MOISTURE CONTENT

The obtained results of moisture content were varied from those recorded by other researchers' variation from the literatures (Salunke and Desai, 1941), which was between 5 to 7 % In these studies the moisture content of the *Ricinus communis* oil was 0.052%, indicating the low moisture or volatile content might be as a result of effectiveness of the distillation apparatus used for oil recovery. Again, the low moisture or volatile content is an indication of good shelf life characteristics.

Specific gravity

The specific gravity is a key fuel property, which affects the mass of fuel injected into the combustion chamber. This property directly affects the engine performance characteristics because the fuel injection pump meter works on by volume, not by mass (Rengasamy *et al.,* (2014). The specific gravity of *Ricinus communis* oil was found to be 0.976 and significantly decreased to 0.935 for biodiesel. Similar result 0.961 of castor oil was obtained by Encinar *et al.,* (2011).

After the transesterification process, the specific gravity of biodiesel as presented in Table 2 was reduced to 0.935. The obtained value is slightly greater than the specification of biodiesel ASTM D6751 standard. A similar result was observed by Encinar et al., (2011) which found that the specific gravity of castor oil biodiesel was 0.917 with 1% of potassium methoxide as a catalyst. The specific gravity of biodiesel was observed to be slightly higher than that of the conventional diesel. This result indicates that the slightly greater mass of biodiesel may be delivered into the diesel engine

Moreover the specific gravity of oil is an indication of the density of the oil and the density in turn determines the energy content of the oil. Denser oils burn slowly and gives less energy compared to less dense oils. As such, the value obtained for both castor oil and biodiesel in this research indicates that the oil can serve as a good feedstock for biodiesel production.

The specific gravity was 0.976 for castor oil and 0.935 for biodiesel, this was in line with 0.9587 reported by Salunke (1992). This density can further be reduced by esterification to 0.85 to meet the biodiesel energy application (Bello and Makanju, 2011).

pH

The pH of the sample was 6.67 for *Ricinus communis* oil and 6.8 for biodiesel, which is close to the neutral, good indicator of the advantageous utilization of the oil in soap making.

Acid value

Acid value (AV) is a common parameter in the specification of fats and oils. The high acid value implies that the oil has high susceptibility to decomposition (Omohu and Omale, 2017). Excess or higher acid and free fatty acid values of oil greater than 5% were not suitable for base catalyzed transesterification reaction (Rakesh *et al.,* 2014). The acid value of *Ricinus communis* oil obtained is 4.208 mgKOH/g and 3.93 mgKOH/g for biodisel which is lower than 15.57 mgKOH/g obtained by Omohu and Omale (2017) and higher than the maximum value specified by ASTM of 2 mgKOH/g. Therefore, direct use of the oil may cause corrosion in the engine fuel system and the biodiesel may decompose into different products. Hence, the oil should be neutralized prior to storage or use in the diesel engine. Several studies showed that the fresh oil acid value should be less than 1.0mgKOH/g and that all raw materials should be anhydrous. If the above requirements are not met, it is still possible to produce biodiesel, but the overall yield of the reaction is significantly reduced due to the deactivation of the catalyst and the formation of soaps. High acid values may be corrected by the addition of sodium hydroxide or by the heterogeneous acid catalyst. The acid value obtained in this research is high. This may account for the reduced biodiesel during transesterification and would have been overcome if sodium hydroxide had been used as the catalyst.

Saponification Value

This is the measure of the total free and combined acid in the oil expressed as the number of milligrams of potassium hydroxide required for complete saponification on 1 g of oil (Omahu and Omale, 2017). Saponification value is related to the average molecular weight of the sample. The molecular weight is inversely proportional to the saponification value. Saponification values increased with the yield of methyl ester (Encinar *et al.,* 2005). The saponification of *Ricinus communis* oil decreases from 185.04 mgKOH/g to 173.16 mgKOH/g for biodiesel. A comparative result was obtained by Nakarmi and Joshi (2014); after their refining of oil, the *Ricinus communis* oil saponification value decreases from 79.159 to 76.258 mgKOH/g. Decreases in the saponification value may be due to the fact that certain fatty acids have been neutralized during the refining process. Higher saponification value would lead to the formation of more soap during transesterification with basic catalysts, which would hinder the biodiesel formation and affect the quality of biodiesel produced (Lam *et al.,* 2010). Hence, refined castor with lower saponification value is preferable for biodiesel production.

The saponification value of the biodiesel at optimum yield as presented in Table 1 is 185.04 which is comparable with the result (182.40 mgKOH/g) for castor oil biodiesel obtained by Encinar *et al.,* (2010) The saponification is useful for the detection of oil or fat with high proportion of the lower fatty acid (Mohibbe *et al.*, 2005). Ester value is a measure of the amount of saponifiable glyceride in the oil (Omohu and Omale, 2017). The ester value was determined by subtracting the acid value from the saponification value. Furthermore the saponification value is a measure of the average molecular weight of triacylglycerol in a sample. The smaller the saponification number, the larger the average molecular weight of the triacylglycerol present and vice versa. Values of 200 meqKg-1 and above indicate fatty acids of low molecular weight; while values less than 100 meqKg-1 indicate fatty acids of high molecular weight. The value obtained for both *Ricinus communis* oil and biodiesel indicates a moderate average molecular weight for the fatty acids, which is acceptable for feed stocks meant for biodiesel production.

Refractive index

This is a measure of the extent to which radiation is refracted on passing through the interface between two media. It indicates the clarity of the oil. The refractive index analysis showed a significant difference between the value 1.37 of *Ricinus communis* oil and 1.191 of biodiesel, this may be attributed to the fact that some impurities and other components were removed during refining Akpan *et al.,*(2006). In addition, Akpan *et al.,* (2006) observed difference between the value obtained for *Ricinus communis* oil 1.37 and that of biodiesel after washing was 1.191.

The refractive index of the *Ricinus communis* oil obtained is 1.37, and similar result was obtained by Nakarmi and Joshi (2014). The refractive index was determined to be 1.37 for *Ricinus communis* oil and 1.191 for biodiesel after washing. This value is an indication of the level of saturation of both oil. The level of saturation based on the refractive index of *Ricinus communis* oil is higher than the biodiesel, and this as a result 0f the transesterification that occur.

Determination of Functional Groups using FTIR

The most convenient and effective method of studying the structural characteristics of *Ricinus communis* is by FTIR Spectroscopy. The following FTIR spectra from Figures 1- 2 depict the spectrum of *Ricinus communis*.

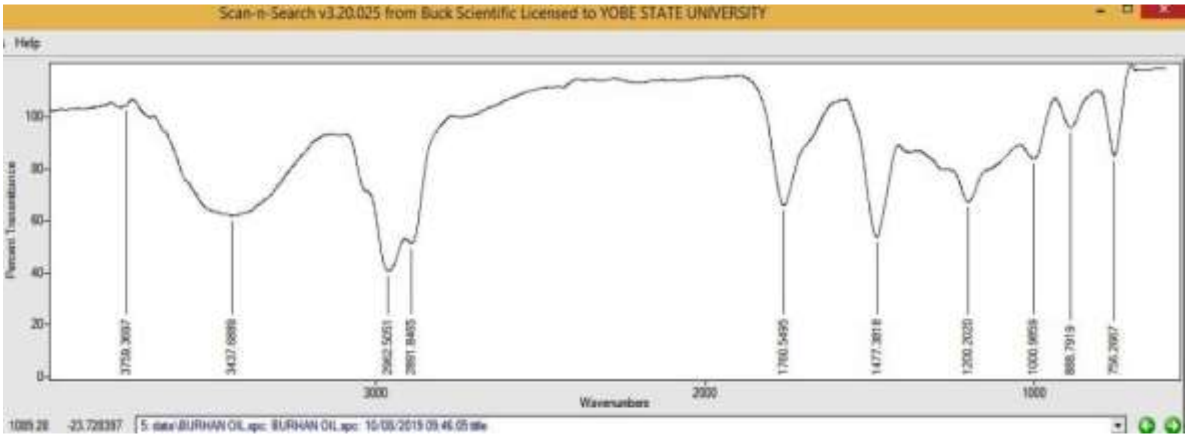


Figure 1 FTIR spectroscopic analysis of *Ricinus communis* seed oil

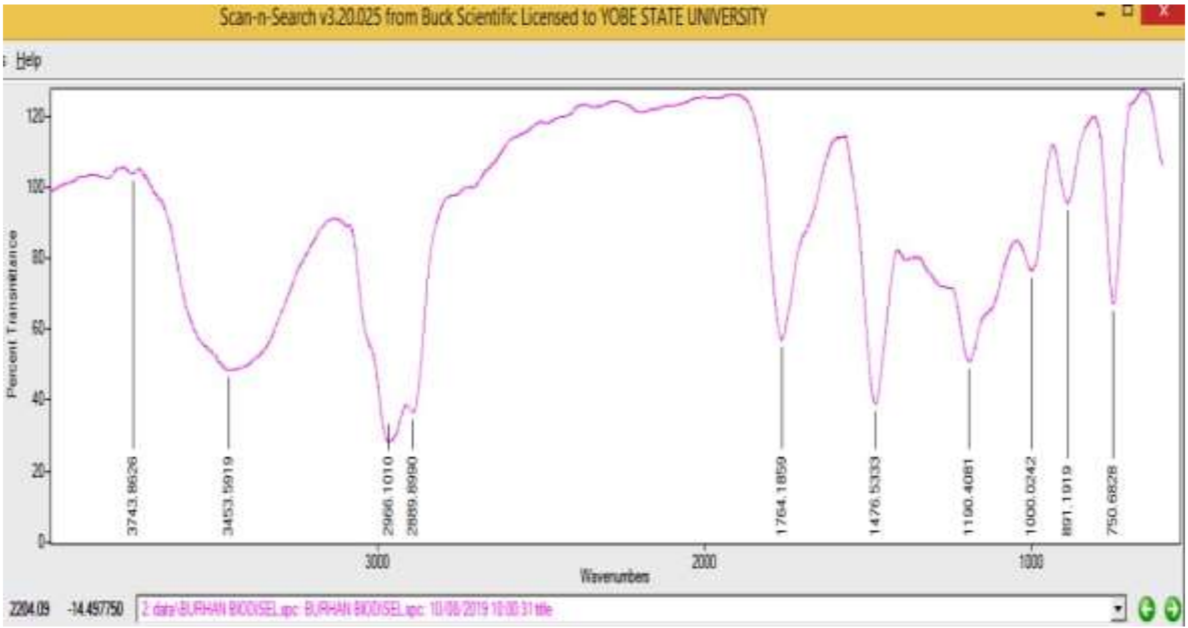


Figure 2 FTIR spectroscopic analysis for biodiesel

Table 3: Frequency, functional group, vibration mode, and intensity of FTIR Spectra.

S/N	Frequency	Bond	Functional group
1	3743.8626	C=C-H	Alkene
2	3453.5919[m]	O-H	Alcohols
3	2966.1010[m]	C-H	Alkane
4	2889.8990[m]	C-H	Alkane
5	1764.1859[s]	C=O	Carbonyl
6	1476.5333[m]	C-H bend	Alkane
7	1190.4081[m]	C-N stretch	Aliphatic amines
8	1000.0242[s]	=C-H bend stretch	Alkenes
9	891.1919	C-H ‘oop’	Aromatics
10	750.6828	C-Cl stretch	Alkyl halide

From figures 1 and 2 above, the FTIR spectrum of *Ricinus communis* oil and biodiesel range between (4500–400 cm−1), using a 100 μm BaF2 transmission cell. This spectrum illustrates the dominant spectral features associated with the oil absorption regions. The peaks at 2962 and 2966 $cm^{-}1$ correspond to **C-H**$_{sp3}$ stretching absorption and 2891 and 2889 $cm^{-}1$ correspond to **C-H**$_{sp2}$. The OH stretching absorption of an alcohol function has very characteristics shape absorption range from (3650- 3250 cm-1), often 3437 cm -1 and 3453 cm -1 for oil and biodiesel. In the region from 1800-1700 cm-1, often 1760 $cm^{-}1$ and 1764 cm -1, the peak can be assigned to the stretching of Carbonyl, i.e., **C=O**, of typical esters, and thus are common in Fatty Acid Methyl Esters (FAME). The main spectrum region that allows for chemical discrimination between FAMES is in the range 1500-1000 $cm^{-}1$, known as "fingerprint" region. (Soares *et al*., 2008). FTIR determined the absorption of triglyceryl as (edible fat and oil) due to the presence of ester molecules present, castor oil also possess's hydrogen bond due to the presence of OH group. And the type of vibration mode that was present in the oil.

CONCLUSION

The *Ricinus communis* oil content was classified as economically viable for biodiesel production. From the experimental result, it could be concluded that the percentage oil content of *Ricinus communis* seed is found to be 39.33 %, As such a satisfactory result can be gotten by solvent extraction process by laboratory Soxhlet apparatus. *Ricinus communis* seed oil and methyl ester (biodiesel) products produced in this research work were examined for pH, moisture content, specific gravity, refractive index, acid value, saponification value, and identification of their functional groups respectively. Since the results of the seed oil and biodiesel were conformed to the standard specified by USA (ASTM D6751) and European organization (EN 14214) and have met the specified standard recommendation. It can be confirmed that the biodiesel production from the *Ricinus communis* seed oil is suitable for use in diesel engines while the production and effective usage of biodiesel will help to reduce the cost of protecting the atmosphere from the hazards of using fossil diesel and hence will boost the economy of the country.

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