

Production of Biodiesel and its Physiochemical Properties Produced from *Ricinus communis* Seeds by Trans-Esterification Process

Iwuzor Kingsley Ogemdi^{1,*}, Ayofe Isreal Ibraheem²

¹Department of Pure and Industrial Chemistry, Nnamdi Azikiwe University, Awka, Nigeria

²Department of Chemistry, Federal University of Technology, Akure, Nigeria

Email address:

kingsleyiwuzor5@gmail.com (I. K. Ogemdi)

*Corresponding author

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Abstract: In the wake of rising prices and unstable supply besides environmental issues, renewed attention has been paid to shifting away from the use of petroleum based fuels. The world's energy demand is commencing its dependency on alternative fuels. Such alternative fuels in use today consist of bio-alcohols (such as ethanol), biomass, and natural oil/fat-derived fuels. In search for new energy sources, much attention is focused on biodiesel as a reliable and renewable resource that is to satisfy a significant part of the energy demands. The oil extracted from the dried seeds of *Ricinus communis* was used in the production of biodiesel under the condition of 3:1 methanol/oil molar ratio for 1 hour at 65°C. The biodiesel produced was subjected to physiochemical analysis. The methyl esters produced were found to meet the ASTM standards for biodiesel. The results obtained from analysis of biodiesel from *Ricinus communis* include free fatty acid: 0.312%; acid value: 0.628mgKOH/g; specific gravity: 0.8937; kinematic viscosity: 7.435Cst; refractive index: 1.6254; flash point: 155°C; fire point: 132°C; water content: 3.24%. Thus, the values obtained met with the ASTM standard making the sample a promising potential for biodiesel production. From the result of this work, the oil extracted from the *Ricinus communis* seed is a potential feedstock for the production of quality biodiesel since almost all the fuel characteristics met the ASTM standards for biodiesel.

Keywords: Bio-Diesel, *Ricinus communis*, Physiochemical Analysis, Specific Gravity, Water Content, Biofuel

1. Introduction

In the wake of rising prices and unstable supply besides environmental issues, renewed attention has been paid to shifting away from the use of petroleum based fuels. The world's energy demand is commencing its dependency on alternative fuels. Such alternative fuels in use today consist of bio-alcohols (such as ethanol), biomass, and natural oil/fat-derived fuels. In search for new energy sources, much attention is focused on biodiesel as a reliable and renewable resource that is to satisfy a significant part of the energy demands [1].

Currently, biodiesel is considered a promising alternative due to its renewability, better gas emissions, non toxicity and its biodegradability [2]. Biodiesel is defined as mono alkyl esters of long chain fatty acids derived from vegetable oils or

animal fats [3]. The term 'mono alkyl esters' indicates that biodiesel contains only one ester linkage in each molecule. Plant oils and animal fats (triglycerides) contain three ester linkages between fatty acids and glycerol which makes them more viscous.

Generally, it has been observed that trans-esterification of triglycerides to alkyl esters (biodiesel) generates a mixture that approximates the properties and performance of petroleum diesel fuel, which allows it to be used directly as substitute fuel without modifications or as blending agents for diesel fuel [4].

Various vegetable oils are potential feedstock for the production of fatty methyl esters or biodiesel but the quality of the fuel is affected by the oil composition [6]. Research results abound in literature on the production of biodiesel through trans-esterification of edible and non edible oil from

different parts of the world [7]. The production of biodiesel from edible vegetable oils has progressively stressed food uses, price, production and availability of oils. New oil-seed crops that do not compete with traditional food crops are needed to meet existing energy demand. In Nigeria, there is an abundance of oil seeds that are relatively unexplored, with no competing food uses.

The recent research developments in the exploitation of biodiesel provide a reliable platform for adoption of biodiesel as an alternative energy source. The following could be key reasons to adopt and promote biodiesel production and research;

1.1. Availability of Feed-Stocks

The availability of vast biodiesel resources which include crude oil from avocado pear fruit, Beni seed, soybean, castor seed, cotton seed etc has a reliable potential for production of biodiesel that will immensely help in its utilization an alternative energy source.

1.2. Global Warming

Another key justifiable reason for embracing and promoting the use of biodiesel is Global warming. This is the increase in the average temperature of the atmosphere, oceans and land mass of the earth.

Environment Researchers have found out that global warming is humanly induced. Its chief cause includes burning of fossil fuels such as coal, oil and natural gas by automobiles which continually release carbon dioxide, oxides of nitrogen etc into the atmosphere. According to UNDP 2007/2008 Human Development Report, the world temperature has increased around 0.7°C since the advent of industrialization and the rate is skyrocketing yearly. It is argued that bio-fuel is environment friendly because carbon dioxide released from burning bio-fuels is balanced by carbon dioxide intake by growing plants from where bio-fuels are made.

1.3. Greenhouse Effect

Biodiesel has the ability to reduce green house gas emissions when compared to those of fossil fuels. Greenhouse effect is the process that occurs when atmospheric greenhouse gases absorb thermal radiation and re-radiates in all direction, leading to the average increase in the surface temperature. Carbon dioxide emitted by engines is the primary source of greenhouse gas emissions. Burning of biodiesel produces carbon dioxide just as fossil fuels but the former is more advantageous as the carbon dioxide released from burning biodiesel is balanced by carbon dioxide intake by growing plants from where biodiesel are made through the process of photosynthesis.

1.4. Pollution

Biodiesel has a higher cetane rating than fossil fuels do. As a result, biodiesel has a higher performance and clean up emissions. When compared to petro diesel, biodiesel contains

fewer aromatic hydrocarbons. It has it capacity of reducing direct tailpipe emission of particulates to the environment.

1.5. Safety and Stability

Biodiesel is safer to handle than petroleum diesel fuel because of its low volatility. Due to the high energy content of all liquid fuels, there is a danger of accidental ignition when the fuel is being stored, transported or transferred. The possibility of having an accidental ignition is related in part to the temperature at which the fuel will create enough vapors to ignite, known as the flash point temperature. The lower the flash point of a fuel, the lower the temperature at which it will form a combustible mixture. For example, petroleum diesel has a flash point of 64°C, which means that it can form a combustible mixture at temperature as low as 64°C. Biodiesel on the other hand has a flash point of over 150°C, meaning it cannot form a combustible mixture until it is heated well above the boiling point of water. It is rare that biodiesel fuel is subjected to these types of condition, making biodiesel quite safe to store, handle and transport. Biodiesel is therefore classified as a non-flammable liquid.

Although the advantages make biodiesel seem very appealing, there are also some disadvantages when using biodiesel. Due to the high oxygen content, it releases relatively high NO_x levels during combustion. But this can be reduced to below petroleum diesel levels by adjusting engine timing and using a catalytic converter. Storage conditions of biodiesel must be monitored strictly as biodiesel has a lower oxidative stability. Biodiesel has lower temperature flow properties than petroleum diesel which means it will crystallize into a gel at low temperatures when used in its pure form. Biodiesel is also more susceptible to degradation, which is promoted by the presence of oxygen, high temperatures, and the presence of certain metals.

Ricinus communis, the castor bean or castor oil plant, is a species of perennial flowering plant in the spurge family, Euphorbiaceae. It is the sole species in the monotypic genus, *Ricinus*, and subtribe, Riciniinae. The evolution of castor and its relation to other species are currently being studied using modern genetic tools. It reproduces with a mixed pollination system which favors selfing by geitonogamy but at the same time can be an out-crosser by anemophily (wind pollination) or entomophily (insect pollination) [9]. Its seed is the castor bean, which, despite its name, is not a true bean. Castor is indigenous to the southeastern Mediterranean Basin, Eastern Africa, and India, but is widespread throughout tropical regions (and widely grown elsewhere as an ornamental plant). [10]

Contribution of petroleum sourced fuel to accumulation of carbon dioxide in the environment necessitated an alternative renewable, carbon neutral transport fuel. Raw and spent vegetable oils have attracted attention as a potential renewable resource for the production of biodiesel. This has led to serious research worldwide. As observed by Nezahat *et al.*, the canola oil methyl ester produced from canola oil over an alumina based solid catalyst can safely be used as an alternate diesel fuel. Their research investigated the trans-

esterification process used in the production of canola oil methyl ester from canola oil and methanol over synthesized $\text{KF/AL}_2\text{O}_3$ catalyst. Different amount of KF solution (15-65wt%) were impregnated into alumina loading 35wt% of KF into an alumina catalyst gave the maximum yield of 99.6% under the following reaction conditions; 60°C, 8hrs reaction time, 15:1 molar ratio of methanol/canola oil and 3wt% of catalyst. They tested for the fuel properties and obtained the following results; cetane number, 62.8, pours point, -10°C and freezing point, -12.9°C [12].

Oghenejoboh and Umukoro, did a comparative analysis of fuel characteristic of biodiesel produced from selected two non-edible oils (jatropha and rubber seed oils) and two edible oils (palm oil and palm kernel oil). They compared their fuel characteristic as well as their 10:90 and 20:80 blend with those of petroleum based diesel. They concluded that the non-availability of arable land for the cultivation compete with the cultivation of foods crops which are of paramount importance to the populace though their fuel characteristic showed that they are good for engines. They also concluded that production of biodiesel from palm oil and palm kernel oil is not feasible since Nigeria cannot even meet the food requirement of this oil [13].

Anitha and Dawn produced biodiesel from waste groundnut oil using supported heteropoly acids and the performance characteristics of biodiesel was checked. From the analysis, he discovered the composite loaded catalyst system a maximum yield of 90.50% for 0.3% catalyst concentration, 6:1 oil to methanol molar ratio for 1hour reaction time and temperature of 60°C. The methyl esters formed were formed to meet the American standard for testing materials for biodiesel. They checked the performance characteristics of biodiesel with diesel on four stroke diesel engine under loaded conditions: mechanical (67.79%), brake and indicated thermal efficiencies are better for biodiesel obtained from diesel (64.3%) thus making it a promising candidate as an alternate fuel [14].

Another research was carried out on avocado seed oil by Radichmoellah *et al.*, The purpose of their work was to produce biodiesel and study the effect of washing method to reach the highest methyl content. Trans-esterification process was run for 60mins with sodium hydroxide as base catalyst concentration of 1% by weight. It was found that the avocado seed oil contained less than 2% free fatty acid so that the trans-esterification process can be carried out with no addition step to convert free fatty acid content. Traces of impurities were removed by washing. Here, they compared dry and water washing. They reported that the dry washing method is technically feasible with less complexity than water washing method. From their research, they concluded that the characteristic of biodiesel from avocado seed oil is acceptable for alternative fuel. The optimum condition was obtained at molar ratio 1:6 alcohol to oil ratio and temp 60°C. Water washing method resulted to methyl ester content of 82.9% while dry washing method gave 84.5678%. Therefore, the best washing method is dry washing with difference of 1.8559% [15].

Xiaohu, *et al.*, produced biodiesel from crude cotton seed oil using response surface methodology. A second order mathematical order was obtained to predict the yield as a function of methanol/oil molar ratio, catalyst concentration, reaction temperature and rate of mixing. They carried out the analysis under the practical optima condition for the production of biodiesel as follows: methanol/oil; 7:9, temperature 53°C, time 45mins, catalyst concentration 1.0% and the rate of mixing is 268rpm. This gave them biodiesel yield of 97% which was confirmed that triglycerides of cotton seed oil were almost completely converted to fatty acid methyl ester [22].

The aim of this study was to investigate the properties of oil methyl esters produced from *Ricinus communis* (Castor) seeds by trans-esterification process. The specific objectives include:

- i. The extraction of oil from *Ricinuscommunis* seeds using n-hexane as solvent.
- ii. Trans-esterification of *Ricinuscommunis* seed oil through methanolysis with a base catalyzed esterification.
- iv. Physicochemical characterization of *Ricinuscommunis* seed oil alkyl ester

2. Method

2.1. Sample Collection

The *Ricinuscommunis* (Castor) nuts were locally obtained and identified. The nuts were cleaned of adhering soil and unwanted materials were handpicked. The nuts were broken and the seeds removed. The seeds were ground with a blender into fine particles and were ready for oil extraction.

2.2. Preparation of Materials

2.2.1. Extraction of Oil from the *Ricinuscommunis* Seed

The extraction of oil was done using soxhlet extraction technique (hot method). The powdered seeds were packed into the extraction chamber and normal hexane poured into the round bottom flask of the soxhlet extractor. The mantle heater was set at about 65°C and the oil in the seeds was leached for 24 – 48 hours in each case until all the powdered seed was extracted. An exhaustive oil extraction was considered to be achieved when no more oil was obtained. The extracted oil was oven dried at 40°C for 48 hours. The seed oil was filtered through a filter paper to remove foreign particles. The pure oil preserved in cold storage. The percentage oil yield was calculated as follows:

$$\text{Percentage oil yield} = \frac{\text{Weight of oil obtained in gm} \times 100}{\text{Weight of seed taken in gm}} \quad (1)$$

2.2.2. Biodiesel Production (Trans-Esterification)

The oil produced was transferred to a round bottom flask and pre-heated to a temperature of about 65°C. Sodium hydroxide concentration of 3g was dissolved in 30ml of methanol of 3:1 methanol oil mole ratio. The mixture of sodium hydroxide in methanol is added to the oil in the round bottom flask, while stirring. The mixture was allowed to

stand for 1hr by maintaining a constant temperature to ensure homogeneity. After 1hr the mixture was poured in a separating funnel and was allowed to stand for 10mins to enable separation of glycerol from the biodiesel (methyl ester). Two layers were observed of which the glycerol is the bottom layer while the diesel is the upper layer. The glycerol is discarded while the diesel was washed with hot water for about 7times to enable complete removal of the catalyst, unreacted methanol and soap. Then the washed biodiesel was heated in an oven to dry off the water in it at about 60°C temperature. The percentage conversion as calculated is

$$\% \text{conversion} = \text{volume of biodiesel} \div \text{volume of oil} \times 100 \quad (2)$$

2.3. Physiochemical Properties of Fatty Methyl Esters (Biodiesel)

2.3.1. Determination of Acid Value

The acid value was determined by American standard for testing material (ASTM) method. About 2g of the diesel produced was weighed into a conical flask and 25ml of methanol was added and 25ml of diethylether. The mixture was titrated against 0.1M of NaOH in the presence of 1ml of phenolphthalein indicator and a titre value was obtained. Then, the titre value obtained is used to calculate for both acid value and free fatty acid.

Observation: pink colouration was observed.

$$\text{Acid value} = \frac{\text{titre value} \times 5.61}{\text{wt of sample}} \quad (3)$$

$$\text{Free fatty acid} = \frac{\text{titre value} \times 0.0282}{\text{wt of sample}} \quad (4)$$

2.3.2. Determination of Specific Gravity

To determine the specific gravity of the biodiesel produced. A clean and dry cylinder of 25ml was weighed (w_0) and filled with water and reweighed (w_1). Thus, specific gravity is calculated as,

$$\frac{W_2 - W_0}{W_1 - W_0} \quad (5)$$

W_2 = weight of cylinder + diesel

W_1 = weight of cylinder + water

W_0 = weight of empty cylinder

2.3.3. Determination of Moisture Content

To determine the water content in the biodiesel produced from the *Ricinus communis* seed.

Procedure: weight of dried empty crucible was weighed as W_1 , 2g of the sample was weighed into the crucible using a spatula and the weight recorded as W_2 . The crucible containing the sample was placed in an oven at a temperature of 100 for 3hrs, after which it was cooled and reweighed as W_3 .

Calculation;

$$\frac{W_2 - W_3}{W_2 - W_1} \quad (6)$$

W_1 = weight of empty crucible

W_2 = weight of empty crucible and sample before drying

W_3 = weight of empty crucible and sample after drying

2.3.4. Determination of Saponification Value

American standard for testing material (ASTM) is used for the determining the saponification value of the biodiesel produced. 2g of the biodiesel was weighed into a conical flask containing 25ml of 12% alcohol potassium hydroxide. Attach a reflux condenser and heat the flask in boiling water for 60mins; shaking frequently. The resulting solution was subsequently titrated against 0.5M of HCl with phenolphthalein as an indicator, using same procedure for the blank. The titration continues until the pink colouration turns colourless.

Thus, it was calculated as;

$$\frac{(B - A) \times M}{\text{weight of sample}} \quad (7)$$

B = ml of HCl required by blank

A = ml of HCl required by sample

M = Molarity of HCl

2.3.5. Determination of Iodine Value

About 0.3g of methyl ester produced was weighed into a conical flask and 20ml of carbon tetrachloride was added to dissolve the oil, 10ml of wigg reagent was added and left for 30mins, a stopper was inserted and vigorously swirled. At the end, 15ml of 10% of KI and 100ml of distilled water was added. And the content was titrated with 0.1M sodium thiosulphate using few drops of 1% starch indicator, in which the sodium thiosulphate was added drop by drop until colouration disappears completely after vigorously shaking. Same procedure was used for blank;

$$\frac{(B - A) \times 1.269}{\text{weight of sample}} \quad (8)$$

B = volume of sodium thiosulphate used for blank

A = volume of sodium thiosulphate used for sample

2.3.6. Determination of Refractive Index

Refractive index of the biodiesel produced was determined using the value from iodine value, saponification value and acid value.

Calculation;

$$\frac{1.4643 - 0.0000665 - 0.0096A}{S + 0.0001171I} \quad (9)$$

2.3.7. Determination of Kinematics Viscosity

The sample was charged in to the viscometer at 40°C and 100°C respectively and its dynamic viscosity was recorded.

$$\frac{\text{Dynamic viscosity}}{\text{density of sample}} \quad (10)$$

2.3.8. Determination of Flash Point

The flash point was determined using a crucible, thermometer and hot plate. About 5ml of the methyl ester produced was poured in the crucible and placed uncovered on the hot plate. The thermometer was inserted into the

sample and the temperature rise was observed carefully. The temperature at which the diesel started to burn while on the red-hot filament of the hot plate was immediately recorded and taken as the flash point. This process was done twice and its average was recorded.

2.3.9. Determination of Fire Point

This was also done using similar method to flash point and the temperature at which the ester gives off enough vapor

which ignites and burns continuously for at least 5sec was recorded as the fire point.

3. Result

The Experimental result of the Biodiesel produced in relation to the ASTM standard is given below;

Table 1. Table showing the parameters of *Ricinus communis* oil and the standard.

S/N	PARAMETERS	UNIT	ASTM D6751-10	RICINUS COMMUNIS
1	Free fatty acid	%	0.5 max	0.312
2	Acid value	mgKOH/g	0.8 max	0.628
3	Specific gravity	-	0.87-0.9	0.8937
4	Kinematic viscosity	Cst	1.9-6.0	7.435
5	Refractive index	-	1.4665	1.6254
6	Flash point	°C	130 min	155
7	Fire point	°C	53 min	132
8	Water content	%	0.03 max	3.24
9	Saponification value	mgKOH/kg	-	168.32
10	Iodine value	mgKOH/kg	-	161.21

The Experimental result of the Biodiesel produced in relation to the Petroleum and Biodiesel standards are given below;

Table 2. Table showing the comparison of the results of the properties of biodiesel and that of petroleum diesel.

S/N	PARAMETER	PETROLEUM DIESEL	BIODIESEL	RICINUS COMMUNIS
1	Flash point	52-96	100-170	155
2	Water content	0.05 max	0.03 max	3.24
3	Kinematic viscosity	1.9-4.1	1.9-6.0	7.435
4	Specific gravity	0.81-0.89	0.87-0.90	0.8937

4. Discussion

The percentage yield of biodiesel produced from the *Ricinus communis* seed oil was calculated, which yielded 61.982% biodiesel. This high percentage conversion of the *Ricinus communis* seed oil to biodiesel makes it a good feedstock for biodiesel production. The trans-esterification reaction was carried under the following conditions; 3:1 methanol/oil molar ratio; 65°C temperature, 1hour reaction time, 3g NaOH. Prior to production of biodiesel from its oil, free fatty acid was (FFA) composition was checked since higher amount of FFA in the feedstock can directly react with the sodium hydroxide catalyst to form soaps which are subjected to form stable emulsions and thus prevent separation of the biodiesel from the glycerol fraction and decrease the yield [21]. Therefore it is better to select oils with low free fatty acids composition or remove FFA from the oil to an acceptable level by means of acid esterification during their production of biodiesel from feedstock that have high FFA composition (FFA greater than 4%). Hence, FFA greater than 4% needs to be esterified. Nevertheless, the FFA content of the *Ricinus communis* seed used only 1.628% which was in the allowed level for being directly used for reaction with the NaOH catalyst to produce biodiesel.

Table 2 represents the comparison of the results of the properties of the biodiesel produced and that of petroleum

diesel. It can be observed that from table 2 that the standard limits of petroleum diesel and biodiesel differ. The flash point of the biodiesel produced from the *Ricinuscommunis* seed was 155°C which fell in the standard range for biodiesel (130°C minimum). The value obtained is higher than that of petroleum diesel and this makes it more stable to fire. Since flash points do not anything to do with engine performance but rather fuel handling and storage and safety of the fuel [6]. The flash point of the *Ricinuscommunis* seed reduced after trans-esterification into methyl ester. This observation is due to the increase in volatile components in the fuel. The acid value measure the content of FFAs in the sample. The suggested ASTM standard for pure biodiesel sets the maximum FFA values 0.8 max mgKOH/g to control the level of free fatty acid. Analysis of *Ricinuscommunis* biodiesel gave the Acid value value of 0.628% which fell within the 0.8max biodiesel standard. This is good as the result is not above the specified limit. The sample contains 3.24% of water as shown in the table 1 above. Therefore, the product is not 100% good for engine because due to the presence of water it can cause engine breakdown.

Kinematics viscosity is the ratio of the dynamic viscosity of the biodiesel produced to its density. The kinematic viscosity of the *Ricinuscommunis* biodiesel is 7.435 cst (mm²/s). Viscosity is very much related to the chemical structure of the fuel. Its viscosity does not fall within the range of the standard specification ranging from 1.9-6.0. Due

to its high viscosity it can lead to negative impacts on fuel injection performance, hence not a good product for biodiesel production. The specific gravity obtained for the biodiesel produced was 0.8937g/ml which was within the standard range of 0.87-0.90. Densities and other gravities are important parameters for diesel fuel injection systems. The values must be maintained within tolerable limits to allow optimum air to fuel ratios for complete combustion. High-density biodiesel can lead to incomplete combustion and particulate matter emissions [14]. The iodine value for the biodiesel produced was 161.21 mgKOH/g higher than the standard iodine value for biodiesel of 120 by Europe's EN 14214 specification [16]. Iodine value is the measure of unsaturation; therefore the high iodine value indicates high unsaturation of fat and oil [9]. Note that iodine value does not necessarily make the best measurement for stability as it does not take into account the positions of the double bonds available for oxidation, so its value can be misleading. The saponification value for the biodiesel produced was 168.32mgKOH/kg. This measures the average molecular weight of fatty acid and expressed in milligrams of potassium hydroxide. High saponification value indicates the presence of fatty acids which can lead to soap formation. [16], hence the separation of product will be exceedingly difficult. This could account for low yield of biodiesel product.

Generally, the values obtained for the fuel characteristics of the biodiesel from *Ricinus communis* seed falls within the recommended specified limits for biodiesel except for the high values of its kinematic viscosity and moisture content and for this reason doesn't 100% qualifies it to be a good feedstock for biodiesel production unless further analysis is carried out on the seed.

5. Conclusion

The reaction conditions used for the trans-esterification reaction; 1:3 oil/methanol ratio; 65°C reaction temperature; 1hour reaction time; 0.3g NaOH for *Ricinus communis* seed oil, was successfully accomplished and was validated with the biodiesel yield of 61.982%. The biodiesel produced was analyzed for the physical and chemical properties. From the result of this work, the oil extracted from the *Ricinus communis* seed is a potential feedstock for the production of quality biodiesel since almost all the fuel characteristics met the ASTM standards for biodiesel.

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