Production and Characterization of Biodiesel from Jatropha Oil and Neem Oil.

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ABSTRACT

Transesterification of two oil samples "Jatropha and Neem oils" in methanol was studied with the use of potassium hydroxide as a catalyst. The properties of the produced biodiesel such as flash point, density, pour point and cloud point were measured and compared to the properties of automotive gas oil. The biodiesel produced from Jatropha oil have properties within the acceptable range for automotive gas oil while the Neem biodiesel properties are not within the range. The Neem biodiesel properties obtained are flash point (160°C), density (0.64kg/m3), pour point (10°C), cloud point (15°C) while that for Jatropha biodiesel are flash point (46°C), density (0.42kg/m3), pour point (2°C) and cloud point (3°C) respectively. The fuel properties obtained for Neem biodiesel are higher in value than those obtained for Jatropha biodiesel.

Key words: Production, Characterization, Biodiesel, Jatropha Oil, Neem Oil.

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1.0 INTRODUCTION

Due to constant increase in price and non-renewability of petroleum products, which are obtained from finite sources, alternative resources of energy have started gaining attention. The alternative sources of energy especially for automobile fuel that are gaining popularity today are biofuel, hydrogen as in the case of fuel cell powered vehicle, solar energy and electrical energy. Of all the above mentioned sources, biofuel have shown more prospects. This obviously is because it is biogradeble, renewable and its sources are infinite and abundant.

Biodiesel, biogas and bioethanol are all examples of biofuels. One important feature is that they can be blended at any proportion with any fuel obtained from petroleum whose properties conform to the end use. For example, biodiesel with diesel fuel, bioethanol with gasoline can all be blended. Biodiesel is a methyl ester which can be produced from vegetable oil, animal fats etc. Vegetable oil, being one of the sources of biodiesel, is in abundant in Nigeria. This is because most seeds contain vegetable oil which can be used to make biodiesel. In this study, "Jatropha oil and Neem oil" were used as they are not part of food menu.

Jatropha is a plant which is drought resistant and can grow in gradually sandy saline soils. This plant can grow up to 8metres; it has large green to pale green leaves and can produce seeds up to 35 years. [1-5, 15].

Neem plant is an evergreen tree which is endemic to the Indian sub-continent and has been introduced to many other areas in the tropics. [5, 8-10]

With rising cost of conventional fuels and the limited reserves available, this investigation looks into how alternative renewable fuel resources could be used as substitutes and can be justified on the following:

- Conventional diesel (AGO) produced from crude oil is neither renewable nor environmentally friendly fuel.
- Jatropha oil and Neem oil are abundantly available in Nigeria but are not properly exploited.
- Biodiesel is renewable and contributes less to global warming when compared with fossil fuels due to its closed carbon cycle.
- Market for better utilization of these vegetable oils leading to more employment and less dependency on conventional fuel.
- Exhaust emissions from biodiesel are lower than with conventional diesel fuel.

1.1 **Conventional Diesel Fuel.**

Conventional diesel fuel is one of many fuel products produced from petroleum through a refining process. The primary purpose of a petroleum refinery is to separate the complex mixture of hydrocarbon into usable products.

1.2 Biodiesel.

Biodiesel is a mono- alkyl ester produced through trans-esterification processes. [1-15]. It is obtained from the transesterification of vegetable oil or animal fats. Transesterification reaction is the transformation of an ester, in this case, a triglyceride (vegetable oil) and alcohol, ethanol in this case, into another ester in the presence of acid or base as a catalyst. In the production of biodiesel, the products are mixtures of fatty esters (biodiesel) and glycerol [15-20].

2.0 METHODOLOGY.

2.1 Extraction of Jatropha Oil from it seeds.

50g of pounded seed of each sample was placed in a filter paper. The paper containing the sample was placed into a soxhlet apparatus. The extraction flask was then fitted with 250ml of petroleum ether using an electric heating mantle, the petroleum ether was gently heated. This continued for four hours after which the extraction was stopped by noticing how oil comes with the petroleum ether. The oil was separated from the solvent by evaporation. The filter paper was removed and dried. The filter paper and its content were weighed and the change in mass determined. The experiment was repeated using the same amount of the sample.

The percentage of lipid was calculated as follows:

% lipid = (weight of lipid) (weight of sample) 100%.

2.2 Characterization of Oil

The chemical and physical properties of the jatropha and Neem oil as well as the biodiesel obtained from them were determined with the view of characterizing the oil and the biodiesel. The major properties determined includes the density, specific gravity, iodine value, acid value, free fatty value, moisture content. Ash content, saponification value, flash point, cloud point, freezing point and pour point.

2.3 Determination of Density

The weight of a small empty bottle was determined using an electronic weighing balance. The bottle was then filled to the brim with the oil and the weight of the bottle and oil determined. This procedure was repeated with the biodiesel labeled as D2 and D3 respectively and the density calculated using the formula below.

Density (
$$\rho$$
) $\frac{w^2 - w^1}{v}$

W2=weight of bottle and oil.

W1=weight of bottle.

V =volume of oil.

2.4 Determination of Specific Gravity

An improvised specific gravity bottle was washed and rinsed with acetone dried in the oven. The bottle was cooled at room temperature in a desiccators and the weight of the empty bottle determined using an electronic weighing balance.

The weight of bottle filled with water was recorded, then the water was poured out and the bottle rinsed with acetone and dried in the oven. The procedure was repeated with the jatropha and Neem oil and the specific gravity calculated thus:

Specific gravity=
$$\frac{w^3 - w^2}{w^1}$$

Where

W3= weight of bottle and oil

W2= weight of empty bottle

W1= weight of equal volume of water

2.5 Saponification Value (S.V)

The method used for the determination of the saponification value was that of the British standards institute 1995.

The oil of both samples (2g) was placed in a 250ml conical flask and 25ml of 0.5M ethanol potassium hydroxide solution added, this was done separately for each of the sample.

A reflux condenser was attached and the flask content refluxed for 30min on a water bath with continuous swirling until it simmered. The excess potassium hydroxide was titrated with 0.5 hydrochloric acid using phenolphthalein indicator while still hot. A blank determination was carried out under the same condition and the S.V calculated as thus:

Saponification value (s.v) = $\frac{(B-R)*28.05}{weight of oil}$

Where B = Titre value of blank R= Titre value of real sample (oils)

2.6 Determination of Acid Value (A.V)

The method used for the determination is that of British standard institute no 684.

Each of the oil sample (4g) was placed in a 250ml conical flask and warmed. Methanol (25ml) was added with through stirring followed by two drop of phenolphthalein indicator and a drop of 0.14m sodium hydroxide solution while shaking vigorously until a permanent light pink colour, which persisted for 1 minute, was observed, recorded as end the point and used in the calculation of the FFA value as indicated below:

Acid value = %FFA (as oleic) x 1.99

2.7 Determination of Free Fatty Acid (FFA) reagent;

The method used for the determination is that of British standard institute no 684.

The 2 sample of the oil (4g) each was placed in a 250ml conical flask and warmed. Methanol (25ml) was added with thorough stirring followed by two drop of phenolphthalein indicator and a drop of 0.14m sodium hydroxide solution. The contents were then titrated with 0.14m sodium hydroxide solution while shaking vigorously until a permanent light pink colour, which persisted for 1 minute, was seen. The end point was recorded and used to calculate the FFA value as thus:

% FFA (as oleic) = $\frac{\text{Titre x N x 28.2}}{\text{Weight of sample}}$

Where N =molarities of base

2.8 Determination of Iodine Value (IV).

The oil of the 2 samples (1g) each was placed in a 250ml conical flask stopper and the content mixed and placed in the drawer for exactly 30 minutes. Potassium iodine solution (10ml of 15% W/V) was added to the flask washing down any iodine that may be found on the stopper. This was titrated against 0.14m Na₂S₂O₃ until the sodium become light yellow. Starch indicator (1%, 2ml) was added and the titration continued until the blue colours just disappeared.

A blank determination was carried out under the same conditions. The titre value was recorded and used to calculate the I.V as indicated below.

Iodine value = (B-R) x molarities of Na₂So₃ x 12.69

Weight of sample

B= Titre value of Blank

R=Titre value for real determination.

2.9 Preparation of Fatty Acid Methyl Ester (FAMES) Reagent

The method used was developed by the biodiesel team. Into 240ml of the Neem oil and jatropha oil, a mixture of methanol (96ml) and KOH (4.8g) was poured drop wise while stirring within a period of 30 minutes. Then the reaction mixture was covered and left to stir slowly (using a magnetic follower) for about 18hous until two layers were formed on setting. Care was taken not to stir vigorously in order to avoid emulsification. The mixture was poured into a separating funnel and allowed t settle for an hour. The lower layer was run off, which contains most of the glycerine that was realized during the reaction. About 10ml of 50° C distilled water was added to the crude product and swirled slowly and left for some time to stand. The lower layer was

then run off. This washing process was repeated until the product was clear. A small quantity of anhydrous magnesium sulphate was added and stirred for 5 minutes and the magnesium sulphate was allowed to settle. The biodiesel was later filtered using a filter paper to separate the biodiesel from the hydrated magnesium sulphate. The yield of the biodiesel was calculated thus:

% yield of biodiesel = _____Vol of biodiesel

Vol of each oil sample

3.0 **RESULTS AND DISCUSSION**

PARAMETER	NEEM BIODIESEL	NEEM OIL	JATROPHA BIODIESEL	JATROPHA OIL
Specific gravity (g)	-	0.318	-	0.316
Saponification value (g)	-	211.15	-	196.36
Acid value (g)	-	13.945	-	4.52
Free fatty acid value (%)	_	7.10	-	2.27
Iodine value	_	132.23	-	112.88
Flash point (^O C)	160	165	46	45
Pour point (^O C)	10	10	2	-
Cloud point (^O C)	15	15	3	2
Freezing point (^o C)	8	7	-5	-5
Density (g/ml3)	0.64	0.69	0.42	0.65

3.1 DISCUSSION OF RESULTS

Petroleum ether was used in the extraction of the oil from jatropha oil and this gave a maximum yield of 133%(w/w). This shows that jatropha is a good source of oil compared to other seeds like cotton seed, water melon seed etc which contains about (35-40)%, (25-35)%, respectively [17-25] and can easily serve as a source of raw material to industries like soap industries

From table 1.0, the acid value of jatropha oil is 4.52 while that of neem oil is 13.945, the high difference in the acid values shows the effect of solvent extraction over mechanical extraction, as the acid content of jatropha oil have been reduced during the solvent extraction [17-26]. The acid

value also gives a measure of the extent to which the constituent glyceride has been decomposed by lipase action, [17-20, 26].

Table 1.0 also shows that the saponication value of jatropha oil is 196.36 while that of neem is 211.15, this implies that the triglycerides of neem oil have higher molecular weight of fatty acids (saturated and unsaturated). The results obtained compared favorably with the saponication value of palm oil (196-205), olive oil(185-196), soy oil (193) [18-26].

Table 1.0 shows the percentage free fatty acid calculated as 7.10% and 2.27% for neem oil and jatropha oil respectively, showing that the oil is unstable and can easily degrade when they are not properly stored hence it cannot be used in formulating storage chemicals demanding low fatty acid content, [18,26]

From table 1.0, the physical analysis of jatropha oil, shows the specific gravity to be 0.316 and that of neem is 0.318 while the density of the oils (jatropha and neem) are 0,65 and 0.69 (g/ml³) respectively, while for neem biodiesels is $0.64(g/ml^3)$ and $0.42(g/ml^3)$ for jatropha biodiesel. Showing that neem biodiesel and it's oil is denser than jatropha biodiesel and oil, as it is evident from the values obtained.

Table 1.0 shows the iodine value for jatropha oil to be 112.88 and that for neem oil is 132.23; this result justifies the fact that the two oil are not edible. Iodine value for edible oil is less than 100. The high iodine value of the two samples also indicates that the oils are semi drying type. In general, the greater the iodine value, the higher the degree of unsaturation and the higher the tendency of the oil to undergo oxidative rancidity [17-26].

Also In the area of biodiesel production, the iodine value also shows that neem and jatropha oils been semi drying oils are a good source of raw material for biodiesel production, because the higher the iodine value the more the number of unsaturated double bonds present in the molecular structure and the less the viscosity of the oil [16,18-26].

Also from table 1.0, the pour point of neem oil is 10° C which is greater than the pour point of jatropha oil (2°C). Also the pour point of neem biodiesel was computed to be 10° C and still greater than that of jatropha biodiesel which is 2°C as shown on table 1.0. This gives an idea of the temperatures at which these oils seizes to flow , it therefore follows that, in a temperate climate below the pour points of these oils, they are not suitable for use as biodiesel as it can lead to Inefficient combustion in engines.[12, 20-26].

Similarly, from table 1.0, the cloud points of neem biodiesel were computed to be 15° C and that of jatropha biodiesel is 3° C, showing the temperatures at which the oil becomes cloudy.

Table 1.0 shows the flash point of the jatropha biodiesel to be 46° C, which is within the acceptable range of the flash point of automotive gas oil that is in the range of (38-55) $^{\circ}$ C. But the flash point of neem biodiesel was found to be 160° C, which is very far from the flash point of automotive gas oil.

CONCLUSION.

Biodiesel has been successfully produced from the seeds of jatropha and neem. The properties of the produced jatropha oil and neem oil determined and compared to those of automotive gas oil.

For the biodiesel obtained from neem oil, the properties differ significantly from those of automotive gas oil.

REFERENCES

- [1] Alenezi, R., Leeke, G.A., Winterbottom, J.M., Santos, R.C.D., Khan, A.R., 2010. Esterification kinetics of free fatty acids with supercritical methanol for biodiesel production. Energy Conversion and Management 51, 1055–1059.
- [2] Anandan S, Kumar GKA, Ghosh J, Ramachandra KS. Effect of different physical and chemical treatments on detoxification of ricin in castor cake. Animal Feed Science and Technology 2005;120:159–68.
- [3] Berchmans HJ, Hirata S. Biodiesel production from crude Jatropha curcas L. seed oil with a high content of free fatty acids. Bioresource Technology 2008;99:1716–21.
- [4] Berrios, M., Siles, J., Martin, M.A., Martin, A., 2007. A kinetic study of the esterification of free fatty acids (FFA) in sunflower oil. Fuel 86, 2383–2388.
- [5] Biodiesel standard EN 14214. European Standard Organization; 2003.
- [6] Caetano, C.S., Fonseca, I.M., Ramos, A.M., Vital, J., Castanherio, J.E., 2008. Esterification of free fatty acids with methanol using heteropolyacids immobilized on silica. Catalysis Communication 9, 1996–1999.
- [7] Cardoso, A.L., Neves, S.C.G., Da silva, M.J., 2008. Esterification of oleic acid for biodiesel production catalyzed by SnCl2: a kinetic investication. Energies 1, 79–92.
- [8] Carmo, A.C., de Souza, L.K.C., de Costa, C.E.F., Longo, E., Zamian, J.R., da Rocha Filho,
- [9] Coulson, J.M. and Richardson, J. E. (2002), 5th edition, vol.2, Butterworth-Heinemnn pp (502).
- [10] Chhetri AB, Tango MS, Budge SM, Watts KC, Islam MR. Non-edible plant oils as new sources for biodiesel production. Int J Mol Sci 2008;9:169–80.
- [11] G.N., 2009. Production of biodiesel by esterification of palmitic acid over mesoporous aluminosilicate Al-MCM-41. Fuel 88 (3), 461–468.
- [12] Hanumantha, J. (1957), Floral of west Africa, 2nd edition, Part1&2, Vol.1,pp(366,397,410).
- [13] Heller ,M .(2008),Investigating the biodiesel stability characteristics, B.Eng Thesis Department of Chemical Engineering, Ahmadu Bello University Zaria pp(8-10).
- [14] Henning, (1996), Extraction of black date oil with hexane; B.Eng Thesis, Department of Chemical Engineering Ahmadu Bello University, Zaria pp(25-28).

- [15] Keith, (2009), Encyclopedia of Chemical Technology, 2nd edition vol8 pg (778-840).
- [16] Knothe G. Fuel properties. In: Knothe G, Gerpen JV, Krahl J, editors. The biodiesel handbook. Champaign, Illinois, USA: AOCS Press; 2005.
- [17] Kumar Tiwari A, Kumar A, Raheman H. Biodiesel production from jatropha oil (Jatropha curcas) with high free fatty acids: an optimized process. Biomass and Bioenergy 2007;31: 569–75.
- [18] Pa quart and Pearson (1981), Synthesis and kinetic study of biodiesel from shear oil under varying condition, 2nd edition, university of London press Ltd,pp (93-98,109).
- [19] Pearson .D. (1981), The Chemical analysis of food, 7th edition, Church hill Livingstone, Edinburgh.pp505-530.
- [20] Martı'nez-Herrera J, Siddhuraju P, Francis G, Davila-Ortiz G, Becker K. Chemical composition, toxic/antimetabolic constituents, and effects of different treatments on their levels, in four provenances of Jatropha curcas L. from Mexico. Food Chemistry 2006;96:80–9.
- [21] Mittelbach M, Remschmidt C. Biodiesel: the comprehensive handbook. Graz, Austria: Martin Mittelbach Publisher; 2004. p. 24.
- [22] MoP (Ministry of Petroleum and Natural Gas). Basic statistics; 2009. http://petroleum.nic.in/petstat.pdf.
- [23] MNRE (Ministry of New and Renewable Energy). Annual report 2009–10 chapter 3: renewable energy for rural applications; 2010.
- [24] Schuchard U., J. Braz. (1998) Chem., soc., vol.9, No.1, pp (199-210)
- [25] Sunday, D.G. (1998), Extraction and characterization of oil from bagaruwa seed; B.Eng Thesis, Department of chemical engineering, Ahmadu Bello University, Zaria pp(16-25).
- [26] Trabi M, Gu" bitz GM, Steiner W, Foidl N. Toxicity of Jatropha curcas seeds. In: Proceedings of the symposium "Jatropha 97" (biofuels and industrial products from Jatropha curcas), Managua, Nicaragua, February 23–27; 1997.