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A Study of Extraction Yield and Properties of Some Oil for Biodiesel Application

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Abstract : A clear pattern in economic development indicates a heavy dependence on the availability of cheap transportation fuel. Given the finite nature, escalating cost and pollution effect of fossil fuel, continuous dependence on hydrocarbon fuel for accelerated economic developmentin the near and long term particularly in developing economies is unsustainable. Biodiesel presentsa significant advantage as a viable alternative in this regard. Experimentally and analytically exploring this advantage is imperative. Soxhlet and soaking extraction processes were implemented with n-hexane, petroleum ether and distilled gasoline as solvent forJatropha and Moringa. Subsequently, a series of physio-chemical tests were conducted on the oil. A code to computethe equivalent heating value for the biodiesel/diesel blend was also executed to generate a simplified correlation for some common biodiesel. Theresultsobtained largely agreed with the existing literature and provided useful insight for future works.

Keywords: Biodiesel, Extraction, Physio-chemical, Heating value, Correlation

1.0 Introduction

Emerging trends in the global energy arena in the transportation sector over several decades have forced the convergence of opinion on the need to diversify supply sources, improve efficiency, curb emissions, and reduce the consumption of non-renewable fuel. Currently in the United States, for example, 85%of their energy comes from hydrocarbon sources including gas, petroleum and coal. Transportation accounts for a large portion of that consumption. This pattern reflects global trends. Indeed, global oil consumption is projected to increase from 162 Quads in 2003 to 239 Quads in 2030 with 60% of that increase being used for transportation (Mcllroy et al, 2006). This increasing demand for conventional oil is coming against the backdrop of dwindling global reserves. At the current consumption rate of about 18 billion barrels of crude oil per annum, these reserves would last for approximately 70 years. Added to this is the negative impact of emissions on the environment. Recent studies have shown strong correlations between atmospheric temperature and the level of greenhouseemissions (Hei, 2001). Again as supply dwindles against the backdrop of increasing demand, prices are set to increase, undercutting global economic growth which in turn exacerbates social upheaval.

There is a need to aggressively explore the potential of biofuel in meeting these challenges. Biodiesel presents an opportunity to switch dependency from finite to renewable supply sourcesreduce or eliminate impact on the environment and, build new supply architecture that could foster accelerated economic growth and improve living conditions in sub-Saharan Africa. The current global energy architecture and technology are built to run on conventional fuel. Despite their impact on the pace of development over a century, these technologies are still in need of improvement to increase efficiency and reduce emissions. For instance, while the pre-mix, spark ignition engine burns fuel more efficiently at near stoichiometric air its high-temperature combustion leads to a higher production of NOx gases but, the reverse is the case in the compression ignition engine where liquid fuel spray atomization, evaporation, mixing and combustion all takes place spontaneously in the cylinder leaving insufficient time for optimal mixing and thus producing a mix of soot and NOx. Over the last few decades, combustion modeling has played an increasing role in bringing about novel engine designs that present exciting potential to address these shortcomings. To meet the challenge of the future, combustion modeling of biodiesel will play an increasing role in the improvement of new technology that will facilitate access to an eclectic mix of new fuel supply sources. Given Africa's rapid urbanization and vast amount of margin land in the Sahel, Moringa, Jatropha, waste and other non-edible oil provide a unique niche for biodiesel production on a large scale. Investigating the comparative advantage provided by these potential fuel sources is imperative.

Traditionally, biodiesel had been sourced from edible vegetable oil, about 93% of global biodiesel is produced from edible oil and the supply sources have been mostly secured from the Agricultural industries (Gui and Bhatia, 2008). However, it is becoming increasingly clear to most stakeholders that this trend is unsustainable in the longer term because of the food versus fuel dispute ithas triggered (Rafaat, 2010). Biodiesel production used around 4.4 million hectares of arable land in the European Union. Replacing 10% of EU diesel with biodiesel would account for around 19% of world edible oil production in 2020 which means more land will be planted with crops and more land somewhere in the world will be converted into farmland, thereby releasing Green House Gas emissions. The alternative to these is non-edible or waste oil, hence the focus on Moringa, Jatrophaand other non-edibleoils.

Moringa and Jatrophatrees are both tropical and sub-tropical trees growing easily on marginal land and having an oil yield of between 40 – 60%. They can propagate easily in drought-proneareas and are highly adaptable (Divakara etal, 2010). Both plants have found multiple uses, and this is particularly so for Moringa whose leaves and other extracts serve as remedies for a diverse range of ailments in sub-Saharan Africa. Large-scale cultivation of these plants for biodiesel production will offer additional incentives to farmers. Also, waste restaurant oil is set to play an important role in biodiesel fuel sources in the coming decade on the continent. The demographic shift on the continent toward a younger more urbanized and educated population has become a dominant trend in the last few decades. This trend is set to accelerate and accompanying this, will be an increase in restaurant waste oil generation. The only economically beneficial and sustainable way of disposing of this waste is its use in the production of biodiesel.

Biodiesel fuels are mainly non-petroleum-based diesel fuels. They compriselong-chainalkyl esters produced by the transesterification of vegetable oil or animal fats (Gabrosky et al. 1998). These fuels are typically created by reacting fatty acids from many types of vegetable oil as well as animal fats with an alcohol in the presence of a catalyst to produce the diesel alkyl esters and glycerine. At the end of the reaction, the glycerine, catalyst and any remaining alcohol on fatty acids are removed from the mixture. The alcohol used is typically methanol; although ethanol and higher alcohol also have been used. Some attractive attribute of biodiesel includes lower emission of particulate matter and carbon monoxide. It also typically has improved lubricity and ignition quality because of a comparatively higher cetane number (Bamgboye and Hansen, 2008). Other advantages include ready availability, renewability, higher combustion efficiency, lower sulphur and aromatic content and higher biodegradability.

Biodiesel is not without limitations which include the fact that engine emission of nitrogen oxides (NOx) increases by approximately 1% for every 10 vol% of biodiesel blended into diesel fuel. Other disadvantages include higherviscosity, lower energy content, higher cloud point, and pour point, lower engine speed and power, injector coking, engine compatibility, high price, and higher engine wear (Demirbas, 2009).

The esters have the structure R-(C=O)-0- R^1, where R and R^1 are chains of alkyle and alkenyl groups with as many as 17-19 carbon atoms (Golovitchev and Yang, 2009). Biodiesel typically has five basic components they are; methyl palmitate ($C_{17}H_{34}O_2$), methyle stearate ($C_{18}H_{38}O_2$), methyl oleate ($C_{19}H_{36}O_2$), methyl linoleate ($C_{19}H_{34}O_2$) and methyl linolenate ($C_{17}H_{32}O_2$). The first two are saturated while the last three are unsaturated oil. The production process, as described by Prasath et al (2010), which can be optimised (Maa and Hannab, 1999, and Venkatachalam and Sampathrajan, 2005) involves using methanol in the presence of NaOH as a catalyst.

As a prelude to modeling works on the fuel source, this paper presentsthe result of extraction yield from three different solvents under two different processes and the experimental determination of some physio-chemical propertiesof the extracted oil. Analytical determination of the equivalent heating value of resulting biodiesel/diesel blends with their deduced correlation is also presented.

2.0 Experiments and Results

The required amount of Moringa and Jatropha seed were collected from three different locations in Northern Nigeria, namely, Bauchi, Makurdi and Kaduna. The kernels were manually processed, and the oil-bearingseeds were pulverized and oven-dried to prepare the oil matrix for extraction. The experiments were conducted in three different laboratories, namely, Grand Cereals Lab, Bukuru, Jos, Abubakar Tafawa Balewa University Chemistry Lab., and Federal Polytechnic, Bauchi Chemistry Lab. All in Nigeria

2.1 Oil Extraction Yield

The sample seedswereair-dried and pulverized in a clean, nonreactive vessel. Afterthis, the sample was oven-dried. Extraction was achieved via two separate parts (soaking and Soxhlet) using three different solvents, namely, petroleum ether, normal hexane (n-hexane) and distilled gasoline, for every 150g of sample 400ml of solvent was used for the soaking and 600ml for the Soxhletextraction. The extraction results obtained are given in Table 1.

Table 1 Oil Extraction yield.

Method	**Oil Extraction Yield (w/w%)**					
	Moringa			**Jatropha**		
	n-Hexane	Petroleum ether	Distilled gasoline	n- hexane	Petroleum ether	Distilled gasoline
Soxhlet	37.1	24.3	40.2	51.8	25.3	34.1
Manual soaking	13.7	14.1	20.8	26.7	17.7	23.13

2.2 Specific Gravity Determination

The specific gravity bottle was washed, cleaned, dried, cooled and weighed empty. The bottle was then filled with distilled water and weighed. The same bottle was emptied, dried, and filled with an equivalent volume of the extracted oil sample and re-weighted one after another (Jacob, 1999).

Given that:

Weight of sp. gr. Bottle --- W_0

Weight of sp. gr. Bottle + distilled water --------------------------------- W_2

Weight of sp. gr. Bottle + oil sample ------------------------------------- W_3

$$specificgravity = \left(\frac{W_3+W_0}{W_2+W_0}\right) + 0.00064(t^oC - 15.5^oC) \quad (1)$$

Where t^0C is the ambient temperature during weight determination and 0.00064 is the average coefficient of expansion for all oils.

For Moringa

$W_3 = 39.1g$

$W_6 = 10.6g$

$W_2 = 42.9g$

Hence given t=32^{0}C

Sp. gr. of Moringa oil =0.89295

Hence the density of Moringa oil=892.95kg/m^3

For Jatropha oil

W_2 = 38.7g

W_0 = 10.6g

W_2 = 42.9g

Hence sp. gr. of Jatropha oil =0.88052

Hence the density of Jatropha oil=880.52kg/m^3

2.3 Determination Of Viscosity

A standard viscometer (Ostwald tube)was used at 40^{0}C in a bath to determine the viscosity of the two-sample oil and their biodiesel derivatives and the time was logged for three trials.

Hence $viscosity = \frac{t_1+t_2}{t_2}$ (2)

Where t_1 is the trial time for oil and t_2 is the trial time for water.

The viscosity test result is therefore given in Table2

Table 2 Viscosity Test Result

Oil	Viscosity (mm/s
Moringa oil	53.90
Jatropha oil	41.45
Moringa biodiesel	29.90
Jatropha biodiesel	25.81

2.4 Determination of Pour And Cloud Point

With the aid of appropriate glassware, a thermometer and a refrigeration bath, the pour and cloud points were determined, and the results are tabulated below.

Table 3 Cloud and pour points.

Sample	Cloud Point	Pour Point
Moringa oil	13^{0}C	10^{0}C
Jatropha oil	5^{0}C	1^{0}C
Moringa biodiesel	6-4^{0}C	2^{0}C
Jatropha biodiesel	4-1^{0}C	1^{0}C

2.5 Determination Of Iodine Value

Procedure (WUS Method)

- Dissolve about 0.25 of the sample in 15ml CCl_4 in a conical flask
- Add 25ml of WUS solution
- Close the conical flask with a grand glass stopper, mix and allow

Standing at about 20^0C for 1 hour in the dark.

- Add 20ml of 10% aqueous KI solution and about 150ml $H2^0$
- Titrate the unreacted halogen with an accurately standardized thiosulphate solution (0.1m) in the presence of starch to a blue endpoint.
- Conduct two determinations and blank in the same manner as the sample but without oil (use distilled water instead of oil)

Calculation:

$$Iodinevalue = \frac{(B-S)*N*12.692}{W}(3)$$

Where B = blank titre

S = sample titre

N = normality of $Na_2S_2O_3$

W = weight of sample

The computed iodine value is shown in Table 4.

Table 4 Iodine Value

Sample Oil	Iodine Value
Moringa oil	64.84
Jatropha oil	108.40

2.6 Determination Of Refractive Index

In this determination, two methods were adopted, they are;

i. Computing from iodine value using the correlation

Refraction Index=0.0001171 x Iodine value + 1.4515 (4)

ii. Experimentally, using the Abbe refractometer (Prolabo). This was used for the determination of the refractive index. The prism of the instrument was cleaned properly, and three drops of water were used to standardize it by adjusting the setting knob until the field was demarcated at the mark “X”. The water was cleaned and three drops of oil were placed on the prism one after another. The knob of the refractometer was adjusted until the field was demarcated by a sharp line dividing the field into equal halves and also coincides with the mark “X” on the field. The reading of refractive index value was taken by viewing through the eyepiece dropper. Normally, the refractive index of oil is taken at 25^0C and 40^0 to 60^0C for high melting fats (Jacobs, 1999).
At the time of the experiment, the ambient temperature was 32^0C. The refractive index values obtained are given in Table 5.

Table 5Refractive Index

Sample Oil	Refractive Index	
	Computational method	Prolabo
Moringa oil	1.4591	1.458
Jatropher oil	1.4642	1.455

2.7 Determination Of Extracted Oil Flash Point

Using a standard heater, test tube with the appropriate seal, thermometer and flame source, the sample oil was progressively heated in the sealed test tube and at intervals of temperature increase, the flame source was brought to the vicinity of the unsealed test tube opening. At the oil flash point, a pop sound ignition will be heard. The process was repeated at a much-reduced temperature interval to obtain an accurate result. The results are given in Table 6.

Table 6 Flash Point Determination

Sample	FlashPoint in ^{0}C
Moringa Oil	215^{0}C
Jatropha Oil	195^{0}C

2.8 DETERMINATION OF FREE FATTY ACIDS

Well-mixed sample (liquid) is weighed based on the table of values for this specific purpose. 50ml of hot neutralized alcohol (methylated) is measured and added to the sample. Add a few drops of phenolphthalein and titrate with NaOH (0.10N) shaking vigorously to the appearance of the first permanent pink colour of the same intensity as that of the neutralized alcohol (methylated spirit) before the addition of the sample. The colour must persist for 30 seconds.

Calculation:

$$\text{Oleic,\% FFA} = \frac{\text{ml of Alkali x N x 28.2}}{\text{Weigh of sample}} \quad (5)$$

The results obtained are given in Table 7.

Table 7FreeFatty Acid Value

Sample	FFA (%)
Moringa oil	9.96
Jatropha oil	2.48

2.9 Determination Of Saponification Value

Procedure

- Accurately weigh about 2g of sample into 250ml quick fit flask
- Pipette 25ml of 0.5N alcoholic K0H solution (28.06g of KOH in 1000ml 95% ethanol or 7.02g of KOH in 250ml of 95% ethanol) into the flask
- Connect flask with air condenser and boil until fat is completely saponified (for about 60 min)
- When the solution is cold, titrate with 0.5N HCL using phenolphthalein as an indicator
- Conduct blank determination and titrate with 0.5N HCL

Calculation:

$$\text{Saponification value: (mg KOH/g)} = \frac{(B - S) \times N \times 56.10}{W} \quad (6)$$

Where B = Blank titre
S = Sample titre
N = Normality of HCL
W = weight of sample

The results obtained are given in Table 8.

Table 8 Results of saponification Value (mgkOH/gm)

Sample	saponification Value (mgkOH/gm)
Moringa oil	185.15
Jatropha oil	196.44

2.10 Determination Of Peroxide Value

Procedure

- Weigh about 5g of oil or fat sample in a round bottom flask.
- Add 30ml of Glacial Acetic Acid/chloroform solution (3:2).
- Swirl the content in the flask until the sample is dissolved.
- Add 0.50ml of saturated KI solution and allow to stand for 1 minute with occasional shaking.
- Add 30ml of distilled water and 0.50ml of starch indicator titrate with 0.01N $Na_2S_20_3$ until the colour changes from blue to colourless.
- Carry out blank determination analysis.

Calculation:

$$\text{PV (mEq/kg)} = \frac{(S - B) \times N \times 1000}{W} \quad (7)$$

Where S = Titration value of the sample
B = Titration value of blank
N = Normality of $Na_2S_2O_3$

(Note: Peroxide value is expressed as milli – equivalent of oxygen in 1kg of oil)

Table 9 Peroxide Value

Sample	Peroxide Value
Moringa	1.84
Jatropha	3.21

3.0 Computational Work

As part of the preparation for conducting an experimental and analytical study of biodiesel blends and ideal gas cycle in compression ignition engines using thermodynamic and ideal gas laws, acomputational scheme was executed. A code to compute the equivalent heating value for the biodiesel/diesel blend was written to obtain a correlation between the equivalent heating value, Q_{qHV} and biodiesel blend ratio, x_{rb}. Normally, to obtain an equivalent heating value for any blend, the variables needed are, diesel heating value, Q_d, biodiesel heating value Q_{bd} and biodiesel blend ratio, x_{rb}. Hence, computing for Q_{qHV} we have;

$$Q_{qHV} = x_{rb}Q_{bd} + (1 - x_{rb})Q_d (8)$$

Hence, the code simply computes for Q_{qHV} for x_{rb} =0.01, 0.02 … 0.99.

That is n = n + 0.01 in steps of 0.01 up to 0.99 with the input of Q_{bd} and Q_d. The code was written in Visual Basic with a Microsoft Excel interface that generated the chart and correlation in first-degree curve fit. Hence, a Jatropha/biodiesel blend for example produced a correlation of:

$$Q_{qHV} = 45.32 - 8.62x_{rb} \ (9)$$

Thus, reducing the number of independent variables in equation (8) from three to one in equation (9). This has the potential to make the modeling scheme more efficient when it is used.

Figure 1 gives a chart produced on the Excel interface by the code with the correlation and Table 10 gives the correlation computed for some common biodiesel.

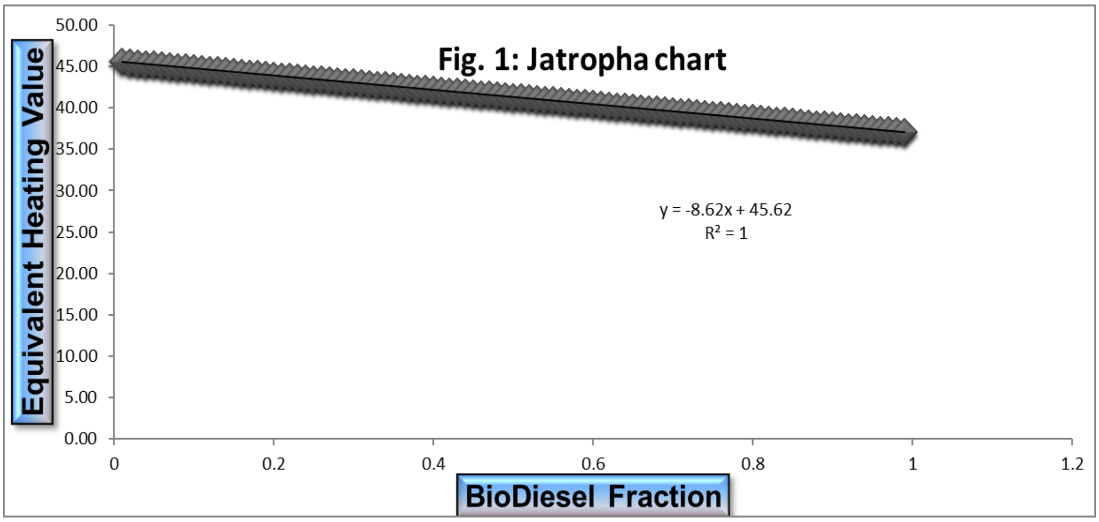

Figure 1 Jatropha Chart

Table 10. Equivalent heating value correlation for some biodiesel.

Biodiesel Sample (MJ/kg)	**Heating Value of Biodiesel**	**Equivalent Heating Value Correlation**

JatrophaBiodiesel	37.26 [25]	$Q_{qHV} = 45.62 – 8.62x_{rb}$
Karanja Biodiesel	36.12 [21]	$Q_{qHV} = 45.62 – 9.62x_{rb}$
Rice BranBiodiesel	41.38 [26]	$Q_{qHV} = 45.62 – 4.62x_{rb}$

Note commercial diesel heating value is 45.62MJ/Kg

4.0 Conclusion

Results of the extraction yield show the optimal performance of the Soxhlet process which is consistence with existing results in the literature.For solvent type, n-hexane is still the preferred choice, although distilled gasoline showed some promise, and when viewed against its ready availability and fairly cheaper cost, the result is significant. The physio-chemical data obtained for both jatropha and Moringa largely projects Moringa as the oil of choice for biodiesel application, this is more so because of other beneficial uses of the Moringa by-product.

The analytical work presented on the equivalent heating value, when validated in a life test rig will serve a useful purpose in optimizing the modeling scheme when used in a larger model where biodiesel/diesel blends are being simulated.

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