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ORIGINAL ARTICLE

Synthesis and Analysis of Biodiesel from Oils of *Luffa* cylindrical Seeds, *Hyphaene thebaica* Seeds and Palm Kernel

Adedoyinsola Adebayo,*Linus N. Okoro, Bolade Agboola, Wan Jin Jahng, Muhammad Yahya
Department of Petroleum Chemistry and Engineering, School of Arts and Sciences, American University of Nigeria
Lamido Zubairu Way, Yola By-Pass. P.M.B 2250 Yola, Adamawa State, Nigeria.
E-Mail: linus.okoro@aun.edu.ng

ABSTRACT
The prospects of biofuels as an alternative fuel to petroluem sources of fuel cannot be overemphasised, partly due to their renewable nature, relative low or absence of sulphur and partly due to cheap feedstock used for their production. This present study investigates the potentials of oils extracted from Luffa cylindrical(LC) seeds, Hyphaene thebaica seeds and Palm kernel respectively in the production of biodiesel. The oils were first extracted by soxhlet method using n-hexane as the solvent; two-steps were utilized in the production, firstly, esterification process using concentrated sulphuric acid to reduce the free fatty acids in the oils and then transesterification process with potassuim methoxide as catalyst at a reaction temperature of 75^{0}C, time of 120min., and constant stirring rate of 350rpm. Quality analysis of the biodiesel produced was also carried out. 90.20%, 57.17% and 30.40% yields of biodiesel were obtained from the oils of Palm kernel, Luffa cylindrical seeds and Hyphaene thebaica seeds respectively. The qualitative analysis showed that the products were comparable to standard quality.
Keywords *: Luffa cylindrical, Hyphaene thebaica, Biodiesel, Palm kernel,*

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INTRODUCTION

All countries are at present heavily dependent on petroleum sources of fuels for transportation and agricultural machinery. The fact that few nations in the world are the producers of the bulk of petroleum has led to high price fluctuations and uncertainties in supply for the consuming nations which are in the majority. This in turn has led to the search for alternative sources of fuels [1](Raja et al., 2011). More so, going by the 2050 prediction when the world energy demand is expected to double its present status, which will indeed exact great pressure on the conventional petroleum sources of energy, efforts are therefore being intensified by researchers all over the world in the search for alternate energy sources [2]. In addition, the current world mainstay of fuel energy is non-renewable and thus liable to exhaustion sometime in the future. This further poses some serious threat to our continued energy sustainability, thus the need for alternatives.

Several alternatives being considered are solar cells, hydrogen cells, nuclear power system and biofuels. Although biofuels had been known in the early 1900s, more attention has been drawn to it as an alternative source of energy to petroleum in more recent times, because of the increasing risk of environmental pollution and ozone layer depletion, high oil prices and depleting world petroleum reserve [3]. However, biofuels has proven to be the most feasible, economic and environmental friendly energy sources in the world partly due to their renewable nature as well as its cheap feedstocks.

Biofuel synthesis results in either the production of bioethanol as an alternative to gasoline or biodiesel as an alternative to petro-diesel. The former involves the conversion of sugar rich biomass such as cereals (sorghum, maize, and corn), and sugar cane into bioethanol through fermentation and distillation. Biodiesel on the other hand is an alternative fuel made from renewable biological sources such as vegetable oils (both edible and non-edible oil) and animal fats. This is commonly made from a chemical process known as transesterification, which involves the conversion of one ester to another ester via the use of an alcohol in the presence of a catalyst which could be organic or inorganic [4].

Biodiesel according to ASTM international is a fuel composed of monoalkyl esters of long chain fatty acid derived from renewable vegetable oils or animal fats, for use in combustion ignition diesel engines (ASTM, 1995).

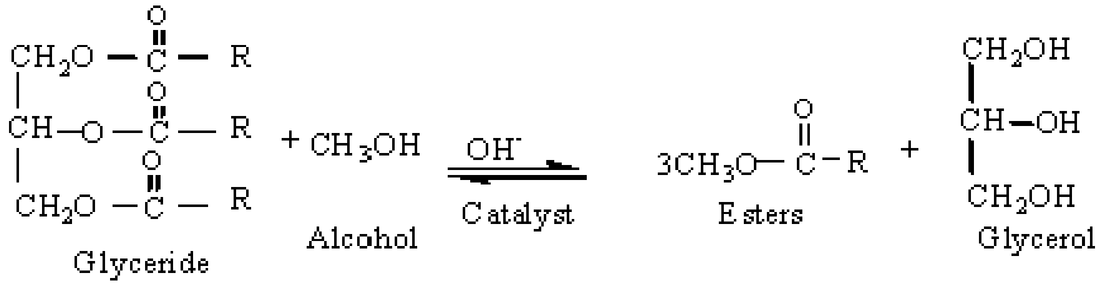

Figure 1: simplified reactions of biodiesel production

Many common and mostly edible vegetable oils have been utilized as feedstocks in biodiesel production of which concerns has been raised over possible sustainability of such usage especially with rise in human population and demand for these oils for consumption and other industrial uses. It follows therefore that researches are needed to be conducted on the wild and non-edible sources of oil to ascertain their potentials for oil generation and subsequent use as feedstocks for bodiesel production. This is the central aim of this research study.

Luffa cylindrica is a family of Curcurbitaceae, found mostly in the sub-tropics, and requires warm summer temperatures with long frost-free growing season when grown in temperate regions. Iqbal [5] asserts that its popularity is most pronounced in Asia and Africa, with major commercial production countries being China, Korea, India, Japan and Central America [6]. In Nigeria, Ndukwe *et al* [7] pointed that this plant grows mostly in the wild and on abandoned building structures and fenced walls in towns and villages. *Luffa cylindrica* is an annual climber which produces smooth and cylindrically-shaped fruits containing fibrous vascular system and seeds. Two known species, includes, *Luffa cylindrica* (LC.) Roem syn *L. aegyptiaca* Mill, popularly called vegetable sponge, sponge gourd, loofa, bath sponge or dish cloth gourd, and ribbed or ridge gourd [*L. acutangula* (L.) Roxb] are domesticated in many villages [8].

The fruits are harvested before maturity, and may be eaten like vegetables. The ripe fruit when dried is a good source of the *luffa* or plant sponge. The sponge most times contains at least about 30 seeds; some may produce more of which most times are discarded. The fibres however contain 60% cellulose, 30% hemicelluloses and 10% lignin [9].

[A] [B]

Figure 2: Typical *Luffa* Sponge and *thebaica* Seeds respectively

Doum palm fruit (*Hyphaene thebaica*) is a known desert palm tree with an oval-like fruit which is edible. The plant is originally native to the Nile valley; however it grows very well in the northern part of Nigeria. It belongs to the palm family, Arecaceae. The trunk of this small palm commonly branches into two like Y and often each branch divides again in a Y form, giving the tree a very distinctive appearance; it is dichotomous and arborescent in nature. It is listed as one of the useful plants of the world [10]. Its fiber and leaflets are used by people along the Nile to weave baskets. Doum palm fruit is also a source of potent antioxidants [11]. The fruit has a brown outer fibrous flesh which is normally chewed and spewed out. Doum palm kernel is edible when it is unripe but hard when it is ripe. The foliage is used to make mats, ropes, baskets, and hats while the stem with the leaves are used for construction purpose [12]. Furthermore, the hard seed inside the fruit, known as 'vegetable ivory', is used to treat sore eyes in livestock using charcoal from the seed kernel as well as making buttons and small carvings, and artificial pearls [13]. There is paucity of literature on the extraction of oil from those seeds and virtually known on the use of the oil in biodiesel production.

The Palm kernel is a seed of the oil palm tree. The fruit yield two different type of oil, palm oil is derived from the outer parts of the fruits and palm kernel oil is derived from the kernel.

MATERIALS AND METHODS

The materials used for this research include the following: thermometer, retort stand, pipette, separating funnel, magnetic stirrer, stop watch, measuring cylinder, conical flask, electronic weighing balance (Adam- pw254), hot plate, distilled water, potassium hydroxide, methanol, petro diesel, bomb calorimeter (Ecobomb), viscometer (Rheotek), GC-MS (Agilent-7890A), IR Spectroscopy (Nicolet IR-100 FT), hexane, sodium hydroxide, sulfuric acid. All the reagents used were of standard analytical grade.

Preparation of Samples For Extraction Process: The seeds and kernel of the samples were harvested, dried and ground to reduce surface area for optimum extraction.

Extraction Process: Soxhlet method of extraction was used in the extraction of the oils from the ground samples using n-hexane as the solvent. Rotary evaporator (Buchi-rotavapor R-210) was later used to obtain the oil from a mixture of oil and the solvent.

Two step-production process: Firstly, esterification was carried out in order to reduce the free fatty acids (FFA) content of the oils as suggested by Ibeto *et al* [14] for *Luffa* oil. The extracted oils were heated up at 65^{0}C for 20 minutes in separate beakers. The acid was then mixed with the methanol to give dimethyl sulphate and further transferred into the boiling oils. The reaction time was set for 2 hours at constant temperature of 75^{0}C, after which, the solutions were allowed to cool for an hour before transesterification process. For the transesterification process; 1.73g of potassium hydroxide was dissolved in 15mL of methanol to give potassium methoxide. The dissolved potassium methoxide solution was transferred into the products of the esterification process already in different round bottom flasks respectively. A reflux was devised to produce a close system in order to avoid the evaporation of the methanol at above 65˚C temperature. The reaction temperature was set at 75˚C and the process allowed for two hours; after which, the process was stopped and the mixture allowed to settle and to separate into two, the biodiesel appearing at the upper layer and the glycerol settling in the solution. The glycerol was then descanted from the solution. The biodiesel was washed with warm water to remove soap, excess glycerol, methanol and potassium methoxide.

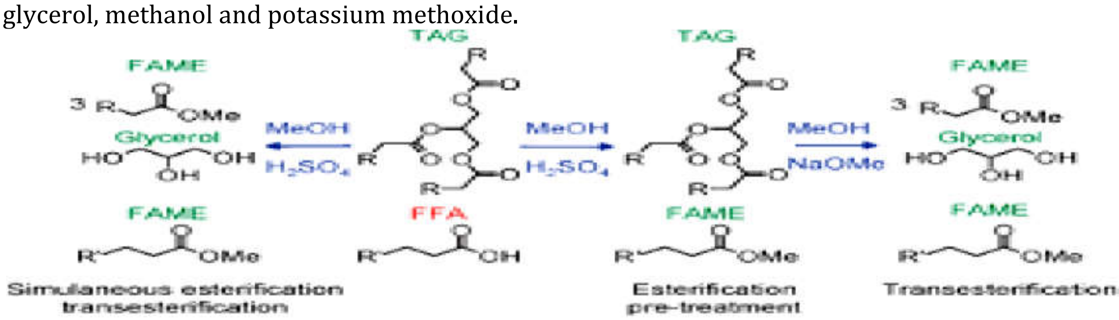

Figure 3: A scheme of the two-step production process

Measurement of Viscosity

Part 1: At Constant Temperature of 40˚C

Kinematic viscosity was evaluated using 3C viscometer. About 17ml of each sample to be analyzed was added through the open end. The viscometer's temperature was set at 40^{0}C. Pipette filler was then placed at an open end and then the end was closed; oil was sucked up above the upper boundary line. The oil was further allowed to fall to the upper mark where the stop watch starts to count. The watch is stopped when the liquid sample reaches the lower boundary. The time it took the liquid to move from one boundary to the other was multiplied against the calibration constant of the viscometer to obtain the viscosity in mm^2/s. This was done for each of the oil sample.

Part 2: Varying Temperatures

About 17ml of each sample to be analyzed was added. The temperature was increased from 40˚C to 70˚C in steps of 10^{0}C. The samples used were pure biodiesel, B20 and petrol diesel. The temperature was increased to know at what temperature there will be high viscosity or low viscosity.

IR Spectroscopy: To ascertain that the real reaction has taken place and that the biodiesel has been formed, the functional groups must correspond with the known functional groups of methyl esters. The Nicolet IR 100 FT—IR machine was used for this purpose. The machine was properly cleaned. Few drops of the samples were dropped on the sample holder and the peaks were collected. Functional Groups in Methyl Ester are C—H Peaks between **2930—2960c**m^{-1} C—O Peak at **1100** cm^{-1} C=O Peaks between **1720—1750**cm^{-1}

Heat Value: There are two major types of heat value. Gross heat and Net heat value. The gross heat value is the amount of heat produced by the complete combustion of a fuel. The gross heat value is obtained when all products of the combustion are cooled down to the temperature before the combustion and also when all the water vapor formed during combustion is condensed. The net heat value is obtained by subtracting the latent heat of vaporization of the water vapor formed by the combustion from the gross heat value.

Common units used for heating Value are

1 Btu/Ib =2,326.1 J/Kg = 0.55556Kcal/Kg

1 J/Kg = 0.00043 Btu/Ib = 0.000239Kcal/Kg

1 Kcal/Kg = 1.80 Btu/Ib = 4,187 J/Kg

RESULTS AND DISCUSSION

Table 1.0 Showing the various samples used, their weights, volume of oil extracted, percentage yields of the oil, percentage yields of biodiesel and specific gravity of the biodiesel

		Percentage yields		
Sample	Volume of oil extracted (mL)	Oil	Biodiesel	Specific gravity of biodiesel
Luffa Seeds	100	15.57	57.17	0.82
(ground 600 seeds)				
Thebaica seeds	40	8.09	30.40	0.62
(Ground 300 seeds)				
Palm Kernel	200		90.20	0.87

Table 1.0 shows the percentage (%) *yield* of the oils. The value of 15.57% yield for *luffa* seeds was comparable with the yield of 12.30% and14.08% reported by Ibeto *et al* [14] and Audu *et al* [15] for the same seed. This makes the seeds a good source of non-edible oil for industrial processes. Its percentage biodiesel yield of 57.17% is high and comparable to most researched feedstocks for biodiesel production such as Jatropha. The percentage oil yield value for *thebaica* 8.09% is low though may increase with the use of other extraction methods. Its percentage biodiesel yield of 30.40% relative to the low oil yield was quite surprising and thus further research on the optimization parameters could help to ascertain the maximum yield parameters. Palm kernel oil yielded the highest percentage of biodiesel. However the enourmous industrial uses of palm kernel oil poses a great challenge in its utilization in biodiesel production on a large scale range. All their biodiesel specific gravity apart from *thebaica* fell within the standard range 0.87-0.90 for biodiesel [16]. Further viscometric and calometric analysis of the biodiesels produced all showed great performances with high proximity to the standard values.

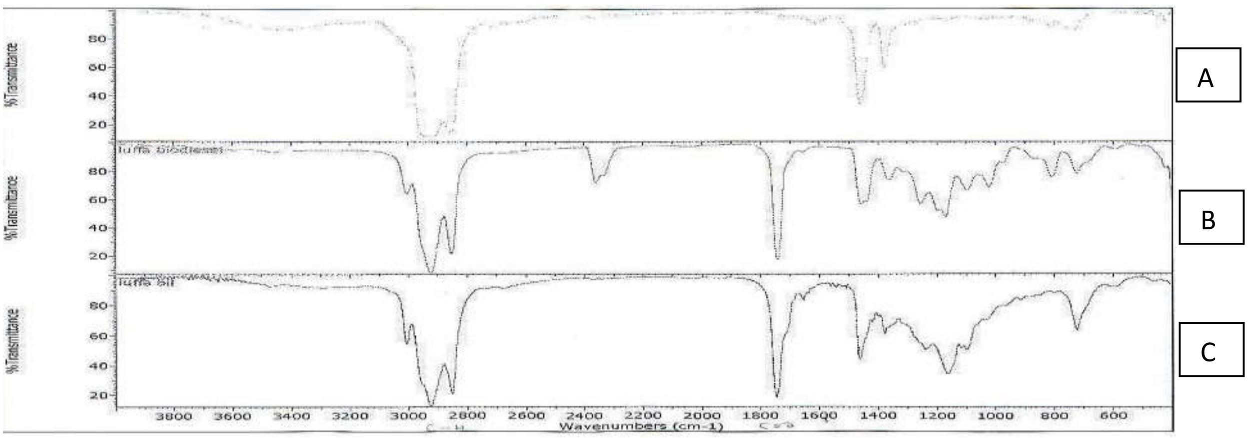

Figure 4: Infrared Spectroscopy of Petro Diesel (A), *Luffa* Biodiesel (B) and *Luffa* Oil (C)

Figure 4 shows the IR for the *luffa* biodiesel, *luffa* oil and the petrol diesel. Figure 4B shows the IR for the *luffa* biodiesel. The peaks in the figure corresponded with the actual peaks of methyl ester compound.

IR Peaks for Luffa Biodiesel

2850-2960cm^{-1} ---Sp^3 Hybridized Bond; **1720-1750cm^{-1} ---**C=O Ester

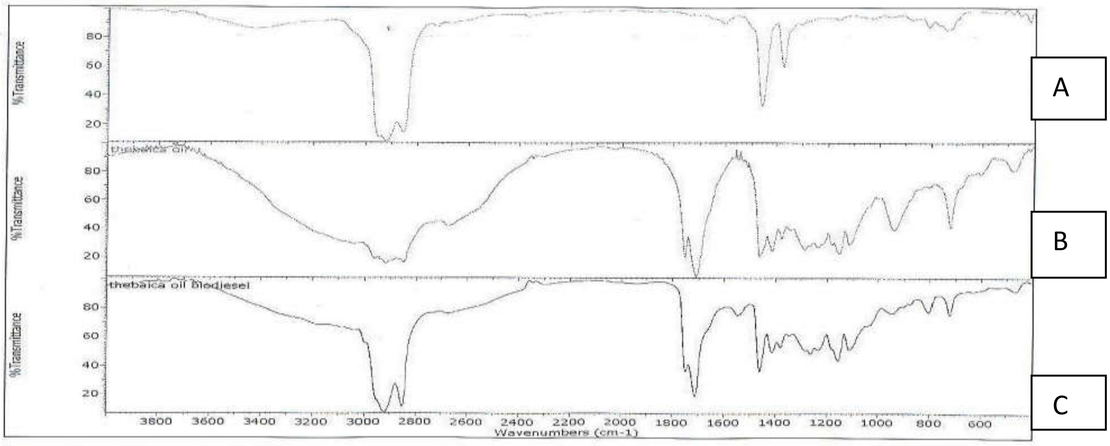

Figure 5: Infrared Spectroscopy of Petrol Diesel (A), *Thebaica* Oil (B) and *Thebaica* Biodiesel (C)

From figure 5, figure 5B shows the IR peaks for the *Thebaica* oil, it is observed that there is a dragging of the peaks from about 3400-3000cm^{-1} due to some impurities or water in the oil. Figure 5C shows the peaks for the *Thebaica* biodiesel which also corresponds with the functional groups in methyl ester compound.

IR peaks for *Thebaica* Biodiesel

2850-2960cm^{-1}--- C-H Bond; **1720-1750cm^{-1}—** C=O Ester

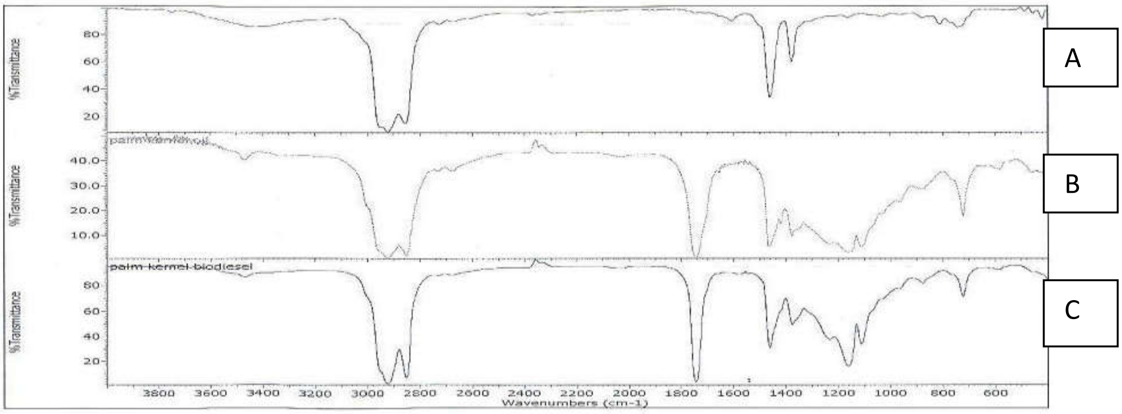

Figure 6: Infrared Spectroscopy of Petrol Diesel (A), Palm Kernel Oil (B) and Palm Kernel Biodiesel (C)

In figure 6, it can be observed that figures 6B and 6C have small sharp peaks at 3500cm-1 which explains that there is a trace of OH during the formation of the biodiesel. Figure 6C shows the peaks of palm kernel biodiesel which also corresponds with the actual peaks of methyl esters.

IR Peaks for Palm Kernel Biodiesel

2850-2965cm^{-1} --- C-H Bond; **1720-1755cm^{-1}** –C=O Ester; **1100-1100cm^{-1}** –C-O ether

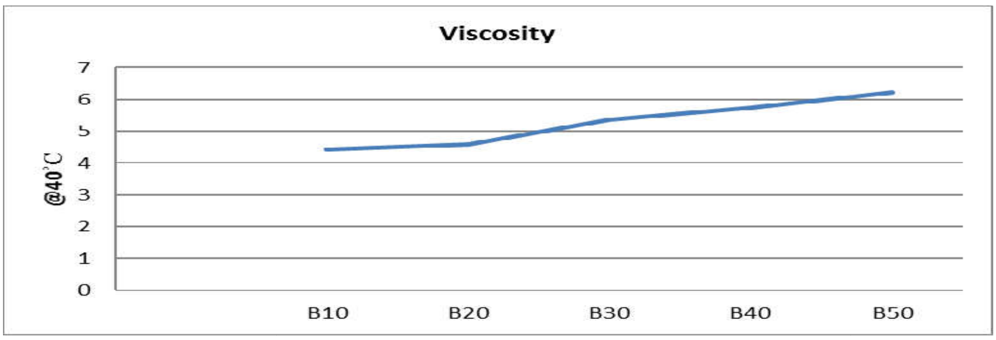

Figure 7: Viscosity at Constant Temperature for *Luffa* Biodiesel

The acceptable viscosity value for biodiesel at 40˚C is from a range of 1.9- 6.0mm²/s. Blending of biodiesel with petrol diesel is carried out to know at what point of the blend, the viscosity will be close to the acceptable value and from B10 till B50 the values correspond with the standard value.

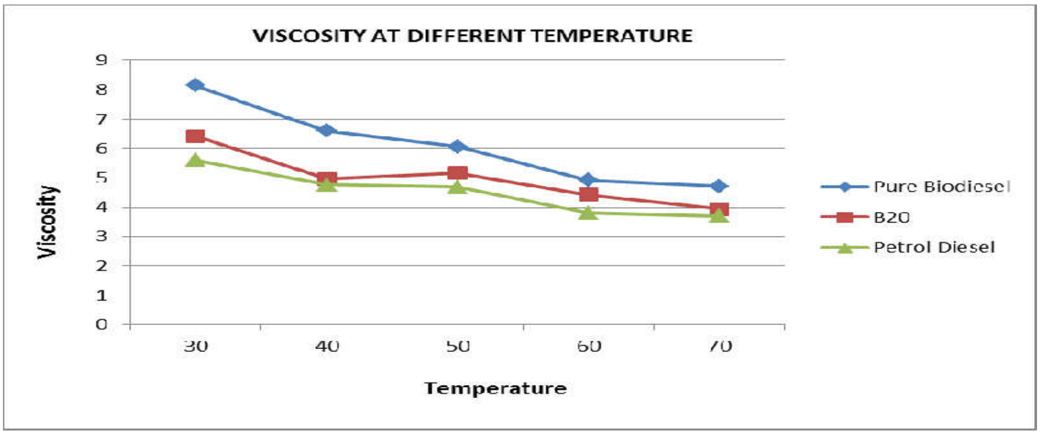

Figure 8: Viscosity at Different Temperature for *Luffa* Biodiesel

In figure 8, as the temperature increases, the viscosity decreases. The pure biodiesel can be used at a temperature from 50˚C and above because at any temperature lower than 50˚C, the viscosity will be high. The B20 blend from the figure above can be used from a temperature of 35˚C and above.

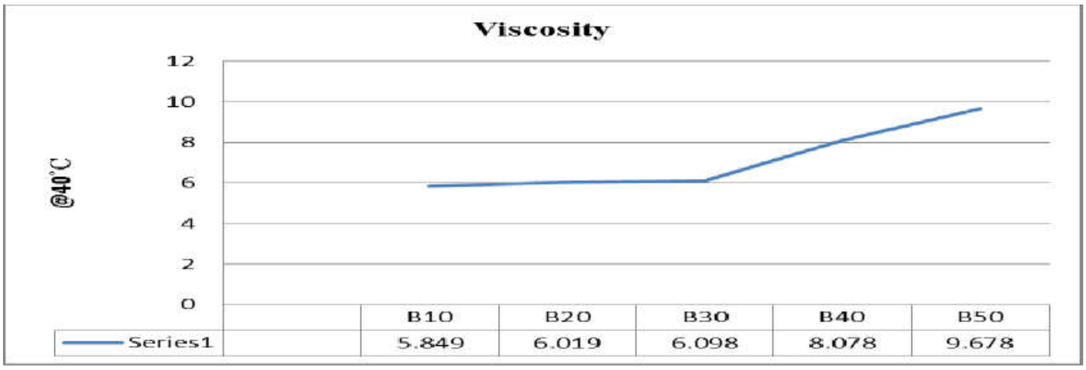

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Figure 9: Graph of Viscosity at Constant Temperature for Palm Kernel Biodiesel.

In figure 9, the viscosity for the pure biodiesel does not meet the acceptable value for biodiesel viscosity but B10, B20, and B30 viscosity values correspond with the standard value.

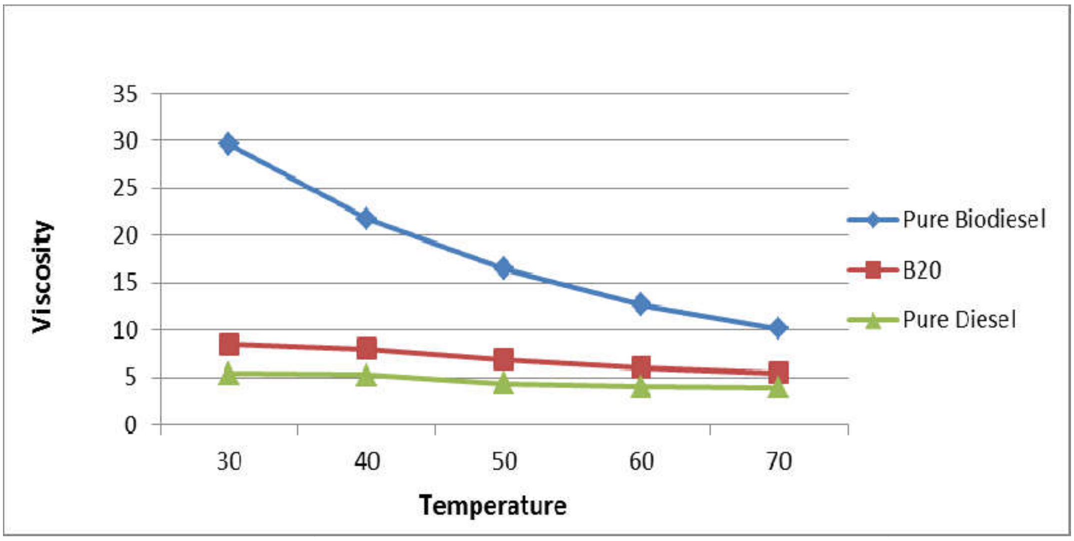

Figure 10: Graph of Viscosity at Different Temperature for Palm Kernel Biodiesel.

The result obtained in figure 10 shows that palm kernel biodiesel cannot be used where the temperature is low. The B20 blending can be used in a geological area where the temperature is from 60˚C and above.

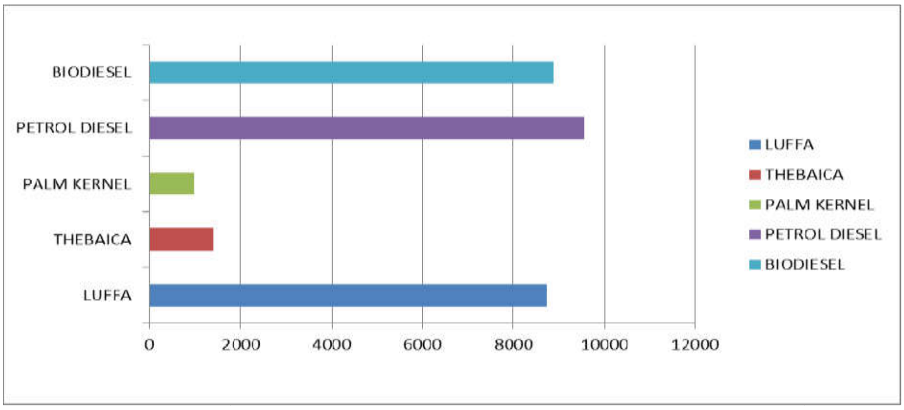

Figure 11: Gross Heat Value for *Luffa*, *Thebaica*, Palm Kernel Biodiesel, Standard Biodiesel and Petrol Diesel

From figure 11, *Luffa* biodiesel has the highest heat value (comparable with petro diesel and pure biodiesel) than biodiesel of the other two oils. Therefore *Luffa* biodiesel has a higher combustion rate than the other biodiesel samples followed by *thebaica* and the lowest being palm kernel biodiesel.

GC-MS Analysis

Table 2: Percentage compositions of fatty Acids in the methyl esters (biodiesel) of the samples

Common name	IUPAC name	*Luffa* (%)	*Thebaica* (%)	Palm kernel (%)
C6:0	Hexanoic Acid	-	3.005	-
Hexanoic Acid				
C8:0	Octanoic acid	4.24	-	2.84
Caprylic acid				
C10:0	Decanoic acid	-	-	4.652
Capric Acid				
C12:0	Dodecanoic acid,	-	9.147	7.958
Lauric acid				
C16:0	Hexadecanoic	9.462	-	-
Palmitic acid				
C18:1	Trans-octadecenonic acid,	16.521	-	26.425
Oleic Acid				

CONCLUSION

It is imperative that because of the non-renewable nature of petroleum resources, alternative fuel production is fast becoming a pressing need of the world energy demand and one of the promising alternatives is biodiesel produced from plants or vegetable oil and animal fats. This study thus unveiled the possibility of using some non-edible and wild oil sources for the production of this promising alternative especially on the recent concern about the use of conventional edible and industrial based oils. However more researches will be needed to be carried out on the areas of optimization and blends to ascertain the optimization parameters and best performing blend for larger scale industrial production.

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