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Characteristics of a Typical Nigerian *Jatropha curcas* oil Seeds for Biodiesel Production

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Abstract

The cost of biodiesel production is the major hurdle towards its commercialization. Biodiesel production is considered to be economically viable only if price compete favorably with petroleum diesel. But biodiesel from edible oil may be too expensive and bring about food crisis. One viable way of ensuring commercial availability of biodiesel is to use less expensive inedible plant oil. This paper presents the result of characterization of typical Nigerian Jatropha curcas oil as a potential feedstock for biodiesel production. The Jatropha curcas oil was extracted at 70 ^{0}C using n-hexane as solvent and at a particle size of 0.7 mm. The resultant oil was analyzed for its physical and chemical properties such as density, viscosity, specific gravity, refractive index, acid value, saponification value, iodine value, peroxide value and percentage free fatty acid. The fatty acid composition was revealed using a gas chromatography. It was found that the oleic and linoliec were the principal fatty acids, while the saturated fatty acids were palmitic acid and stearic acid. The oil low peroxide value and high iodine value is a strong indication of its stability to oxidation. The oil yield was high and its exhibit excellent properties that make it an exciting proposition as the most economically viable feedstock for biodiesel production in Nigeria.

Keywords: *Jatropha Curcas*, vegetable oil, characteristics, biodiesel.

Introduction

The energy commission of Nigeria's long term (2016-2025) plan on the nation's energy requirements is completely non-fossil. This is borne out of the global depletion of the non renewable energy sources, its attendant negative environmental impact and the intent to utilize the neglected abundant renewable resources in Nigeria. Vegetable oil is one important renewable feedstock in the long-term (2016-2025) vision of providing secure, abundant, cost effective and clean source of energy for the nation[1]. Various vegetable oil such as palm oil, soybean oil, sunflower oil, rapeseed oil and canola oil have been used for biodiesel production[2].

Biodiesel is a fuel made from plant or animal oils which can be used in conventional diesel engines to serve as a substitute for petro diesel or blended with petro diesel to reduce emissions. It is oxygenated, sulphur free, biodegradable, non-toxic, and environmentally friendly alternative automotive fuel[3]. Biodiesel production from edible oil seeds grown for traditional market may prove too expensive for use as fuel and may bring about rising cost of food[4]. So, there is the need for an alternative biodiesel feedstock that is in expensive, inedible and meets all criteria for biodiesel feedstock[5] .It had been reported that approximately 70–95 % of the total cost of biodiesel production arises from the cost of the raw material i.e. vegetable oils or animal fats[6]. One way of reducing the biodiesel production cost is to use less expensive feedstock containing fatty acids such as inedible oils, animal fats, waste food oil, and by products of vegetable oil refining[7]. Fortunately, inedible vegetable oils, mostly produced by seed-bearing trees and shrubs can provide an alternative. With no competing food uses, this characteristic turns attention to *Jatropha curcas* plant. *Jatropha curcas* is a multipurpose bush/small tree belonging to the family of *Euphorbiaceae*. It is a plant with many attributes, multiple uses and considerable potential. It is a native of tropical America, but now thrives in many parts of the tropics and sub-tropics in Africa/Asia. The wood and fruit of *Jatropha* can be used for numerous purposes including fuel[8].

Jatropha curcas has the potential to become one of the world's key energy crops. At present it is globally taking the centre stage as the oil seed of choice in biodiesel production. This plant grows in tropical and subtropical climates across the developing world. In Nigeria Jatropha can grow very well and already been planted by farmers but mainly for border demarcation of small farm land holdings[9]. It is widely cultivated in the tropics as a live fence (hedge) around farm lands since the toxins (curcins) in the plant deters animals[10].The tree have a life span of up to 30-40 years and can grow on a wide range of land types, including non arable, marginal and waste land, and need not to compete with vital food crops for agricultural land. Crude Jatropha oil is not edible and its price is not distorted by competing food uses[9]. The oil content of the seed ranges between 50-60 %[11]. A hectare of Jatropha produces about 2000 liters of fuel. When compared to oil commonly used such as palm oil (yield 5,950 liter/ha), Avocado (yield 2,638 liters/ha), castor (yield 1,413 liters/ha); and soybean oil (446 liters/ha) for biodiesel production, Jatropha may not be the most attractive. However, because of the consideration of competition with food, and low input in production Jatropha stand as the most economically viable feedstock[9].

With the increasing global acceptance of *Jatropha curcas oil* as a non edible, inexpensive feedstock for biodiesel production, characterization of the *Jatropha oil* originating from Nigeria is essential.

This objective of this study is to carry out the characterization of a typical Nigerian *Jatropha curcas* oil seeds specifically from Niger State to validate their suitability for the production of biodiesel. As previous research on Nigerian Jatropha curcas oil[12] was limited to seeds obtained from Ilorin town in Kwara State. This research will fill the gap currently existing in literature on the physico-chemical properties of *Jatropha curcas* oil obtained from Nigerian soil.

Material and Methods

Preparation of Seed: Indigenous *Jatropha* Curcas seeds were collected within Mokwa town in Niger State of Nigeria. The damaged seeds were discarded and the seed in good condition were cleaned, dehulled, dried and the hulls were later separated by winnowing. The whitish seeds were ground prior to oil extraction using soxhlet apparatus and n- hexane as solvent. All reagent used were of analytical grade.

Oil Extraction: The whitish seeds were ground prior to oil extraction, soxhlet apparatus was used for the extraction and n-hexane as solvent at 70^0C at a particle size of 0.7 mm using a slid to solute ratio of 6:1. After 8 hrs the extraction mixture was cooled and filtered to get rid of the solid from the solvent. The filtrate was concentrated under vacuum in a rotary evaporator. The calorific value was evaluated in a calorimeter bomb Parr 1241 using oxygen at a pressure of 3.0 Mega Pascal (MPa) according to the standard method of the American Society for Testing and Materials (ASTM) D240 and the Flash point was determined using Automatic Pensky Martens Tester by ASTMD-93 Method.

Gas chromatography (GC) and Mass Spectrometry (MS) Analysis: *Jatropha* oil sample was subjected to GC–MS analysis using a Gas Chromatograph (Model: QP2010 Plus, Shimadzu, Japan at NARICT, Basawa, Zaria) attached with quadruple Mass Spectrometer (Model: HP 5973) with a capillary column of length = 30m, inner diameter = 0.25mm and film thickness = 0.25µm). About 0.1 ml oil was converted to methyl ester using 1ml NaOMe (1 M) in 1ml hexane before being injected into the GC. The injector column temperature was maintained at 250°C and the oven temperature was programmed linearly. Injection mode was split type at 250°C; total and column flow were 40.8 ml/min and 1.80 ml/min respectively at a linear velocity of 46 .3cm/sec., purge flow of 3.0 ml/min, ion source temperature 200°C and split ratio of 20. The identification of the peaks was achieved by retention times by means of comparing them with authentic standards analyzed under the same conditions. Computer matching was done with the mass spectral libraries (NIST05s.LIB) provided with the computer controlling the GC–MS System.

Specific Gravity Determination: The specific gravity bottle was cleaned with acetone, ether and dried in an oven at 60°C, the weight of the empty bottle was taken, after which the bottle was filled with the oil sample and properly covered, the weight was then taken using a weighing balance, after which the sample was removed from the bottle, the bottle was properly washed and filled with distilled water, after which the weight was taken and finally, the specific gravity was computed using the relationship below.

$$\text{Specific Gravity} = \frac{W_o - W}{W_1 - W} \quad (1)$$

Where, W = weight of empty bottle, W_o = weight of the bottle and oil content, W_1 = Weight of bottle and water content.

Viscosity Determination: 400 cm^3 of oil sample was poured into the cup of "Clandom Viscometer, Model VT – 03 Viscometer", the lowest number spindle was selected and screwed into the underside of the viscometer, the cup containing sample was carefully locked into position so that the spindle cone would be completely immersed in the sample, the machine was switched on and pointed deflection on the machine scale was observed for about ten seconds and allowed to stabilize, after which the position of the pointer on the scale was read off, this gives the value of viscosity of the oil sample in centipoises.

Refractive index Determination: The refractive index was determined using Abbey refractometer, Number 00836, the glass prism of the refractometer was thoroughly cleaned with alcohol to ensure that it is free from dust, a drop of oil sample was placed on the lower prism and smeared, then closed with the other covering prism, the light source of the refractometer was switched on, while viewing through the telescope, the coarse adjustment knob was rotated until the black shadow appears central in the cross wire indicator, while still viewing through the telescope, the fine knob adjustment was moved until the rainbow-colored fringe which appeared on the black dividing line disappeared, the coarse knob was rotated to give fine adjustment and make the black shadow appear exactly central in the cross wire indicator, the reading under the telescope and that of the fine adjustment knob were noted and divided by 10,000, this value was then added to the value obtained through the telescope field to give the value of the refractive index of the oil at room temperature.

Acid Value Determination: Two grams of sample was dissolved in 50 cm^3 of mixed neutral solvent (25 cm^3 diethyl ether with 25 cm^3 ethanol carefully neutralized with 0.1M NaOH using 1 % phenolphthalein solution), the mixture was titrated with 0.1M NaOH aqueous solution with constant shaken to faint pink color.

$$\text{Acid value} = \frac{\text{Titre value} \times 5 \times 61 \times 0.00282}{\text{Weight of sample(g)}} = \text{mgKOH/g} \quad (2)$$

Free Fatty Acid Determination: The amount of free fatty acid (FFA) was calculated as being equivalent to half the value of acid value, that is,

$$\text{FFA} = \frac{\text{Acid value}}{2} = \text{mgKOH/g} \quad (3)$$

Saponification Value Determination: 0.5 M KOH was prepared in 95 % ethanol, 2g of oil sample was weighed and 25 cm of the KOH was added, 25 cm^3 of the blank solution was also measured into a conical flask, the two sample were then connected to a reflux apparatus and allowed to boil for an hour until the reflux is completed, 1 cm^3 of phenolphthalein was added to the mixture and the resulting mixture was titrated while hot against 0.5 M HCl acid solution, the volume of the acid used to attained the end point was recorded, the blank determination was carried out using the same procedure described above until the color changes from blue to transparent white, then the volume of acid used was noted, the Saponification value was determined using the relationship below.

$$\text{Saponification value, (S.V)} = \frac{56.1 \times T(V_o - V_1)}{M} \quad (4)$$

Where, T= molarity of the standard KOH solution used, V_o= volume of acid used for the first titration with oil sample, V_1= volume of acid used for the second titration of the blank solution, M= mass of the oil sample used.

Peroxide Value Determination: Two grams of sample was weighed into clean dried boiling tube, 1 gram of potassium iodine (KI) powder was added to the liquid oil and 20 cm^3 of the solvent mixture (i.e, glacia acetic acid and chloroform in the ratio 2:1), then the boiling tube was placed in boiling water bath so that the liquid mixture boils within 30 seconds and allowed to boil vigorously for not more than 30 second, the content after boiling was quickly poured into a flask containing 20 cm^3 of 5 % potassium iodine, (KI) solution and the tube was washed out twice with 25 cm^3 of water, then the mixture was titrated with 0.002 M sodium thiosulphate using fresh 1 % starch solution, a blank titration was carried out at the sample time, the peroxide value was calculated using the relationship below.

$$\text{Peroxide value} = \frac{T \times M \times 1000}{\text{weight of sample (g)}} \quad (5)$$

Where: T = titre value of $Na_2S_2O_3$ = sample titre – blank titre M = molarity of $Na_2S_2O_3$

Results and Discussion

Physio-Chemical Characterisation: Table 1 summarizes the result of characterization of the oil. It is shown that the oil yield of indigenous *Jatropha curcas* oil is 52.75 %. This values was higher than 30 – 40% reported [13] but fell within the value of 25 – 60 % reported by[14,15] who stated that in most cases, yield of *Jatropha* seed are within 40 – 60 %. This oil content was also found to be higher than linseed oil, soybean and palm kernel oil which are 33.33 %, 18.35 % and 44.6 % respectively[16]. Although the yield from this work is lower than the literature value of 63.16 % reported by[16]. The difference in oil yield could be attributed to variation in genes, climate and plant species. Soil condition and improper processing techniques such as prolong exposure of harvested seeds to sunlight can impair the oil yield considerably[17]. The high oil content is one plausible reason why *Jatropha curcas* oil is a suitable non-edible vegetable oil in oleo chemical industries (biodiesel, fatty acids, soap, fatty nitrogen derivatives, surfactants and detergents e.t.c)[18].

Moisture is a chemical contaminant which is usually well mixed with lubricating oil like *Jatropha oil*[12]. This study reveals a value of 0.18 %. This value shows appreciable consistency with the literature[12]. This low value is an indication of high quality possessed by the oil. Low moisture in oil implies that the oil cannot easily be subjected to rancidity[19]. The presence of much water (moisture) in biodiesel feedstock has negative impacts on transesterification process and the effective cost of the feedstock[20]. This is because it supports microbial growth.

The specific gravity of the oil was 0.913. This value shows an appreciable consistency with report of [12, 21, 22]. It is important to note that density of oil decreases with molecular weight, yet increase with unsaturation level[22].

Viscosity is the measure of material resistance to flow, higher viscosity materials flows with great difficulty and a material with less viscosity flow more easily.Viscosity is important to diesels and biodiesels because it has impacts on the operation of some engine components such as the fuel pump[23]. The viscosity of the oil was found to be 36.2 mm^2/sec. This value was higher than 20.49 mm^2/sec reported by[12] but however shows close proximity to 38.8 – 40.4 mm^2/sec in the literature [24]. The more viscous oil is, the better its use as a lubricant; hence *Jatropha curcas* oil will have high lubricating properties[25]. The viscosity of the *Jatropha* must be reduced for biodiesel application because high viscosity of vegetable oil is not suitable. If it is used directly as engine fuel, often it results in operational problem such as carbon deposit, oil ring sticking, thickening and gelling of lubricating oil. Different methods such as pre heating, blending, ultrasonically assisted methanol transesterification, supercritical methanol transesterification and acid or base catalyzed transesterification are being used to reduce the viscosity and make them suitable for engine application[26].

Iodine value is the measure of degree of unsaturation of the oil[27]. Higher iodine value indicated higher unsaturation of fats and oils. The limitation of unsaturation is necessary due to the fact that heating higher unsaturated fatty acid results in polymerization of glycosides. This can lead to the formation of deposits or to deterioration of the lubricating properties[16]. The iodine value for this study was determined to be 105 g I_2/100 g. Vegetable oil with iodine value between 100 and 130 belongs to the groups of semi-drying oil. This group of oil absorbs atmospheric oxygen slowly; produce only soft film after prolonged exposure to air. The iodine value of *Jatropha* curcas seed oil suggests their use in production of alkyl resin, shoe polish, varnishes etc.[16].

Peroxide value is a valuable measure of oil quality. This value is an indicator of the oil stability to oxidation. Crude Jatropha curcas oil has a peroxide value of 2.0 Meq/kg, this low value attest to the oxidative stability of the oil.

Saponification value reported for this study was 190. This high value was consistent with the findings in literatures[15,16,17] who reported that Jatropha curcas oil is usually associated with high saponification value. Such high value establishes the fact that Jatropha curcas oil contains normal triglycerides[16].

An acid value is indication of the age and quality of the oil or fat. The acid value was determined to be 36.2 mg KOH/g which implies high fatty acid content, however the results from this study was quite consistent with values obtained by previous researchers[12,16]. The Free fatty acid (FFA) and moisture have significant effects on the transesterification of glyceride with alcohol using catalyst[20]. High FFA (%wt) cause soap formation during alcoholysis process and lead to difficulties in separation of biodiesel from it's by product; as a result it reduced the biodiesel yield [28]. However, if the vegetable oil has a higher free fatty acid content or more water, acid-catalyzed transesterification is more preferable to the base transesterification[20]. But the liquid acid catalyzed transesterification process does not enjoy the same popularity in commercial application as the base catalyzed process because it is about 4000 times slower[29].

Composition of fatty acid: Fatty acid is a term used to describe any aliphatic acid usually with a chain of ten or more carbon atom's which occur naturally in fat, oils and related (compounds) lipid and also some other acid closely related structure[30]. The properties of the triglyceride and the biodiesel fuel are determined by the amount of each fatty acid that is present in the molecule. Chain length and the number of double bonds determine the physical characteristics of both fatty acids and triglyceride[16].

Transesterification does not alter the fatty acid composition of the feedstock's and this composition plays an important role in some critical parameter of biodiesel such as cetane number and the cold flow properties[16]. There are three main types of fatty acids present in a triglyceride: Saturated (CN:0), monounsaturated (CN:1) and polyunsaturated (CN:2,3) and an ideal vegetable oil as a potential feedstock for biodiesel production should have low saturated and polyunsaturated fatty acid and be high in monounsaturated fatty acid[30]

The result of this study was in accordance with this literature[30]. The Jatropha curcas consist of 28.7 % saturated fatty acid, 39.78 % monounsaturated fatty acid and 31.49% polyunsaturated fatty acid. Vegetable oils that are rich in polyunsaturated fatty acids such as linoliec and linolenic acids, tend to give methyl ester (biodiesel) with poor oxidation stability and high freezing points. Experimental result from this work and other researchers[16, 27] revealed that the monounsaturated fatty acid of Jatropha curcas seed oil is higher than other vegetable oil such as palm kernel (15.4 %), sunflower (21.1%), and soybean oil (23.4 %).

Table 2 show that the predominant fatty acid present in Jatropha curcas oil were oleic, linoliec, palmitic and stearic acid whose percentage composition were 39.74 %, 31.49 %, 19.23 % and 9.03 % respectively. The high content of oleic and linoliec acid is one plausible reason why Jatropha curcas seed oil can be classified as oleic-linolenic oil. The linoliec acid in the Jatropha curcas oil studied was 0 and much lower than European upper limit standard of 12 %.

Table-1
Physicochemical Properties of Jatropha curcas Oil

Composition	This work	1	Reported 2	3
Moisture Content (%,)	1.8	-	-	-
Oil Content (w/w%)	52.75	63.16	25 – 60	-
Specific Gravity at 25°c	0.913	-	-	0.917
Refractive Index	1.466	-	-	1.470
Viscosity @25C (cst)	40	42.88	-	52
Ph	5.69	-	-	-
Acid Value (mg kOH/g)	36.2	-	0.92 – 6.16	1.0-38.2
% Free Fatty Acid	18.1	2.23	-	-
Peroxide Value (Meq/kg)	2.0	1.93	-	-
Saponification Value(mgKOH/g)	190	193.55	188 – 209	188 – 198
Iodine Value (g I_2/100g)	105	103.62	89 -112	90.8 – 112.5
Density (@20 C)	-	0.90317	0.86 – 0.933	-
Calorific Value(MJ/kg)	42	-	-	9470 kcal/kg
Pour point (^{0}C)	-	-	-	8
Flash point (^{0}C)	108	-	-	110
Colour	Golden colour	-	-	Golden Yellow

SOURCE: 1.Akbar *et al.,* 2009, 2. Maricela *et al.,*2010 3.plant.in/…/chemical-analysis-of- Jatropha- Curcas.html

Table-2
Relative Composition of Fatty Acid in Jatropha curcas Oil

Fatty acid	**This work**		**Reported values**	
		1	**2**	**3**
Lauric (C2:00)	-	-	-	5.9
Myristic C14:0)	0.09	-	0.1	2.7
Palmitic (C16:0)	19.23	15.6	14.2	13.5
Palmitoliec (C16:1)	1.40	1.0	0.7	6.1
Margaric (C17:0)	-	-	0.1	-
Stearic (C18:0)	9.03	5.8	7.0	-
Oleic (C18:1)	39.78	40.1	44.7	21.8
Linoleic (C18:2)	31.49	37.6	32.8	47.8
Linolenic (C18:3)	-	-	0.2	-
Arachidic (C20:0)	0.39	-	0.2	-
Other	-	-	-	-
Saturated (CN:0)	28.74	21.4	21.6	22.1
Monounsaturated (CN:1)	39.78	41.1	45.4	27.9
Polyunsaturated (CN:2,3)	31.49	37.6	33.0	47.4

Source: 1. Nzikou *et al.,* 2010, 2. Akbar *et al.,* 2009, 3.Jefferson *et al.,* 2009.

Table-3
Comparison of Profile of Fatty Acid Types in Vegetable Oil

Types of fatty acid	**Palm oil**	**Palm kernel oil**	**Sunflower**	**Soybean**
Saturated (CN:0)	49.9	82.1	11.3	15.1
Monounsaturated (CN:1)	39.2	15.4	21.1	23.4
Polyunsaturated (CN:2,3)	10.5	2.4	66.2	61.0

Source: Akbar *et al.,* 2009

Conclusion

Based on the experimental investigation, the high oil yield shows that Jatropha curcas oil seed is actually a viable feedstock for biodiesel production in Nigeria. This was clearly attested to by its excellent physicochemical properties and its superior fatty acid composition when compared with other edible vegetable oil. The fact that Jatropha cannot be used for nutritional purposes and can grow on waste land make it a viable feedstock for competitive commercial biodiesel production in Nigeria as the nation has abundant land mass specifically within the Northern part in which commercial plantation of Jatropha can be cultivated for oil production and its subsequent conversion to biodiesel.

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