Quality Assessment of Biodiesels from *Lophira Lanceolata* and *Ziziphus Mauritiana Seed* Oils

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ABSTRACT

The purpose of this study was to produce and characterize biodiesels obtained from seeds of Lophira Lanceolata and Ziziphus Mauritiana. Oils were extracted from the seeds using soxhlet extractor with petroleum ether, transesterified using alkali hydrolysis (methanol/ethanol) to obtain biodiesels. Lophira and Ziziphus oils were transesterified under the following conditions: ethanol/methanol/oil molar ratio (6:1), potassium hydroxide catalyst (1.0%wt), temperature (55^{oC}) and mixing intensity of 300rpm.The oils and alkyl esters produced were studied for physicochemical and fuel properties. Oil yields were determined and alkyl ester yields were measured using GC-MS method. The results showed percentage oil yields of 34.0±0.33% and 36.0±0.44% for Ziziphus and Lophira respectively. Some important fuel parameters investigated include; kinematic viscosity, acid value, pour point, ash content, sulphur content as well as some of crude lipid's properties determined complied with ASTM standard specifications. Analysis of alkyl esters showed that methyl-9-octadecenoate (72.26%) and methyl eicosanoate (13.02%) as the predominant esters, while methyl hexadecanoate (78.34%) and methyl docosanoate (10.0%) are the most dominant esters for Lophira and Ziziphus biodiesels respectively. The results suggest that, oils from seeds of Lophira and Ziziphus exhibited some properties that make them suitable for biodiesel production.

Keywords: *Lophira lanceolata* and *Ziziphus mauritiana* Seed Oils, Lipid Yield, Transesterification,

INTRODUCTION

Energy is an essential driving force towards socioeconomic development of a society. Its impact touches every aspect of human endeavours such as health, transportation, agriculture and many more. Fossil fuels are the major source of energy today in most parts of the world. Its combustion generates emissions which are hazardous to the environment and adversely affect human health as reported elsewhere. It was established that these emissions are carcinogenic. However, finite nature of fossil fuels as observed in consumption of fossil fuels far outstrips the discovery of new

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reserves and climate change has redirected research interest to renewable energy resources[1]. Biofuels has therefore recently attracted renewed interest as a possible solution to reduce the menace of global warming effect and greenhouse emissions that are related to the wide use of fossil fuels. In respect of this issue, green fuel sources could represent a valuable alternative to petroleum-derived fuels due to its renewability, biodegradability, sustainability, its substantially reduced net carbon dioxide emission as well as its competitiveness to fossil fuels and food supply [2, 3]. Toxicity level of biodiesel is very low as compared to fossil diesel; this is because biodiesel burns relatively clean with reduction in all type of pollutants that contribute negatively to the environment [4]. Alternative fuels to petro diesel must be technically feasible, economically competitive, environmentally acceptable, and easily available. These are some of the advantages of using biodiesel as a replacement for diesel fuel [5, 6].

Increased interest in the use biodiesel necessitates the search for oil feedstock especially from plant seeds. Production of biodiesel from vegetable oils is widely researched; however it is uneconomical process when valuable oil seeds are used. Thus, use of waste oil or waste seeds oil (thrown-away seeds) became imperative and is recommended as raw material for biodiesel production. *Lophira lanceolata (Lophira)* commonly known as false Shea or iron wood in English and Namijin kadanya in Hausa and *Ziziphus mauritiana* (*Ziziphus*) also known as dunks or desert apple in English and Kurna in Hausa; are two wild oil seed plants that could be exploited for that purpose. Both are promising plants found in vast quantity in most tropical regions of West Africa particularly Nigeria. *Lophira* grows to about 12 m with twisted short branches. Its fruits develop between February and April in which tough reddish elongated seeds are found.

Ziziphus mauritiana is a spiny, small tree up to 12 m high which is evergreen, with trunk 40 cm or more in diameter; spreading crown spines and many drooping branches. When climatic conditions are severe, it becomes a compact shrub only 3-4 m tall. The Fruit a drupe, up to 5 x 4 cm, usually smaller when wild; yellowish to reddish blackish when ripe. Seed a tuberculate and irregularly furrowed stone, containing 1-2 elliptic brown kernels each around 4 mm long [7]. Fruits contain seeds that are grown wild or could be cultivated which are reach in oil. This research work therefore, aims at studying the potentials of these plants seeds for biodiesel production.

MATERIALS AND METHODS

Fresh fruits of *Lophira lanceolata* (false Shea) and *Ziziphus mauritiana* (dunks or desert apple) were collected respectively from Sangere town in Adamawa and Maiduguri Monday market in Borno, both states in the North-Eastern part of Nigeria. They were respectively identified at the Herbarium of the Department of Botany, Usmanu Danfodiyo University; Sokoto.The seeds were separated manually, washed with water for any adhering flesh; and dried at 50°C for 48 h. The dried seeds were crushed to powder using mortar and pestle. The powdered sample seeds were extracted using Soxhlet apparatus on a water bath with petroleum ether as the

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extracting solvent. Oils extracted were conserved in a dry place for analysis.

Analysis of *Lophira lanceolata* and *Ziziphus mauritiana* Seed Oils

The crude seed oils extracted were analyzed for kinematic viscosity, Iodine value, free fatty acids content and saponification value in accordance with AOCS, (1997) methods [8].

Transesterification reaction

The transesterification was carried out using ethanol/methanol and potassium hydroxide catalyst for the alkyl esters production. 100g of Ziziphus oil was measured and placed in a 500cm^3 round bottom flask and pre-heated at 50^{o}C. A 6:1 molar ratio ethanol to oil was prepared by dissolving 1g of KOH in 90cm^3 of ethanol. This was mixed with the hot oil in the flask which was fitted with a cork. The mixture was stirred with a magnetic stirrer for 10 minutes and then heated on a water bath at a temperature of 55^{o}C for 90 minutes for completion of transesterification reaction. The reaction mixture was transferred into a separating funnel. Two separate layers were observed, the upper phase consist of alkyl esters (biodiesels) with minute amounts of impurities like residual alcohol and glycerol, with the lower phase being mainly remains of the catalyst, and glycerol. The upper alkyl esters collected were placed in an evaporating dish and heated in water bath at 80^{o}C to distil residual methanol, then neutralized with dilute acid solution. The alkyl esters were washed with hot water several times then dried over heated anhydrous sodium sulphate, followed by filtration using Whatman filter paper (No.42). The steps described above were repeated for the transesterification of Lophira oil. The percentage yields of alkyl esters were calculated using the following equation:

$$Yield\ of\ alkyl\ esters(\%) = \frac{grams\ of\ the\ alkyl\ esters}{grams\ of\ the\ oil\ used\ in\ reaction} X\ 100 \qquad ....1$$

Fuel Properties of Lophira and Ziziphus alkyl esters

The fuel properties of alkyl esters (biodiesels) of *Lophira* and *Ziziphus* produced were determined according to ASTM (1985 and 2003) standard methods [9,10]. The fuel properties determined include kinematic viscosity, acid value, cloud point, pour point, ash content, flash point and sulphur content were carried out in accordance with ASTM D 445, ASTM D 664, ASTM D 2500, ASTM D 97, ASTM D874,
ASTM D 2622 and ASTM D 93, methods respectively.

Analysis of acquired alkyl esters' profile using GC-MS

The analysis of alkyl esters of Lophira and Ziziphus were accomplished using 6890N series model, AGILENT GC-MS machine. One microlitre of the alkyl ester sample was introduced into the gas chromatograph at injector temperature of 250^{O}C. The column temperature was programmed from 120^{O}C to 250^{O}C at a linear flow rate of 5^{O}C/min, hold 5 min at 260^{O}C. The chromatographs obtained were scanned and the biodiesels components were identified based on software matching with mass spectra.

RESULTS AND DISCUSSION

The percentage yield oils of the plant species under study are depicted in Table 1.0. Ziziphus has a higher oil content of 37.0 ±

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0.33% when compared to 34.0±0.45% of Lophira lanceolata. Oil content of a plant seed is an important economic variable that could be considered before a plant would be regarded as feedstock for biodiesel production as reported elsewhere [1]. Bugaje and Idris [11], reported oil yields for *Lannea microcarpa* seed as 41.0%, while that of *Vitex doniana* seed as 53.0%.Similarly, Wiesman *et al.*, [12] and Deshmukh *et al.,* [13], also reported highly stable oil yields for *Balanites aegyptiaca* at 46.0% and 45.0% respectively. Comparison with values by the above authors indicates that the oil yields obtained is lower than what was reported yet, the yields fall within the 30 to 40% oil; reported for *Jatropha* seed elsewhere [14]. The amounts of oil from seeds of these plants are appreciable and could be exploited for biodiesel production. The duo is shown to content saponification values of 140±8.20 and 223.0±2.53 for *Ziziphus* and *Lophira* respectively. A higher SV value indicates oils to be normal triglycerides which could be useful in industries for liquid soap production. The average molecular weight of oil can be estimated from the inverse of its saponification value as reported [15]. The SV values recorded agree with those reported for Canola, Palm and Neem seed oils [16.]

Table 1.0: Characterization of Oils Extracted from *Ziziphus mauritiana* and *Lophira lanceolata* Seeds

Parameter	***Ziziphus mauritiana***	***Lophira lanceolata***
Lipid Yield (%)	37.0 ± 0.33	34.0 ± 0.45
Kin. viscosity at 40^{oC} (cSt)	31.40±0.10	33.20±0.33
Density g/cm^3	0.920±0.00	0.90±0.01
SV(mgKOH)*	140±8.20	223.0±2.53
FFA (%)*	3.02±0.22	2.44±0.14
Iodine value (gI_2/100g)	38.0±1.11	61.0±1.33

***FFA=Free Fatty Acid Content, SV= Saponification Value**

Table 1.0 depicts Iodine values of the seed oils under investigation with *Ziziphus* and *Lophira* showing 38.0±1.11 and 61.0±1.33 respectively. Iodine value is a measure of the average amount of unsaturation of fats and oils. High Iodine value indicates high unsaturation level of fats and oils. Oils exhibit high iodine value due to high content of unsaturated fatty acids. Iodine value is used in assessing oxidative stability of biodiesels and has direct relation with the number of double bonds in alkyl chain of fatty acids [17]. The iodine values recorded are low compared to rapeseed and soybean oils, indicating the oils under study to consist of highly saturated fatty acids. Low iodine value implies high stability of the oils. However, the values agree with that of Palm and Tallow oils as reported elsewhere [18]. The oils can be classified under non-drying oils which have low level of

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unsaturation. They could be utilised as raw material in industries for the manufacture of soap and vegetable-oil based ice-cream [18].

Table 2.0: Methyl Fatty Acid Esters (approximate wt %) of Biodiesels produced from *Ziziphus* and *Lophira* Seed Oils

Methyl esters	Molecular formula	*Ziziphus m.*	*Lophira l.*
Methyl hexadecanoate	$C_{17}H_{34}O_2$	78.34	7.32
Methyl eicosanoate	$C_{21}H_{42}O_2$	4.43	13.02
Methyl-9-octadecenoate	$C_{19}H_{36}O_2$	ND	72.26
Methyl docosanoate	$C_{23}H_{46}O_2$	10.0	0.23
Methyl-9Z,12Z-Octadecadienoate	$C_{19}H_{34}O_2$	2.09	ND
Methyl octadecanoate	$C_{17}H_{38}O_2$	ND	4.02
Other non-fatty acid detected	-	5.14	3.15

ND=Not detected

The profile of fatty acids methyl ester composition of *Ziziphus* oil is presented in Table 2.0, suggest that methyl-hexadecanoate (Palmitic acid ester) is the dominant ester, followed by methyl docosanoate (Behenic acid ester). Both esters are saturated and would exhibit high level of stability with respect to per oxidation. The biodiesel from this oil feedstock would be at operational disadvantage under cold weather conditions due to high concentration of saturated fatty acids. The biodiesel of this oil could be stored for a long time but cold flow effect could be a serious challenge [19].

Table 2.0 showed the profile of *Lophira lanceolata* methyl esters, indicates methyl-9-octadecenoate (Oleic acid ester) as the dominant ester followed by methyl eicosanoate (Arachidic acid ester) and methyl hexadecanoate esters (Palmitic acid ester) as the dominant in that oil. Presence of oleic acid ester with low unsaturation level in the oil would promote operational functionality of its biodiesel. Here cold flow effects would be minimal as it would be countered by the presence of the duo of arachidic and Palmitic acid esters present. Biodiesel of *Lophira* contain high amount of unsaturated fatty acid methyl ester as compared to Ziziphus biodiesel, which may allow free flow of the fuel. Presence of unsaturation in the alkyl ester makes it suitable for practical engine application particularly at moderate temperature operations [20]. However, high profile of unsaturated fatty acid methyl esters in *Lophira oil* would cause easy oxidation of the fuel as well as affect fuel storage stability and thus may require anti-oxidant.

Table 3.0: Fuel Properties of Methyl esters produced from *Ziziphus mauritiana* and *Lophira lanceolata*

Fuel Parameter	***Ziziphus m.***	***Lophira l.***	**ASTM Limits***
Methyl ester yield (%)	92.0±2.45	86.0±2.15	NS
K.viscosity at 40^{oC}(cSt)	4.8±0.12	6.1±0.11	1.9 to 7.0
Acid value ($mgKOHg^{-1}$)	0.64±0.05	0.62±0.03	0.80max
Cloud Point (^{O}C)	11±1.02	10±1.21	-3 to 12
Pour point (^{O}C)	3.0±0.42	4.01±0.02	-5 to 10
Ash content (%)	0.04±0.001	0.044±0.001	0.02max
Sulphur content (%)	0.024±0.01	0.041±0.00	0.05max
Flash point (^{O}C)	150.0±5.01	170.0±2.00	130 min

NS= Not specified

Tocopherol being a natural anti-oxidant in all seed bearing plants helps in maintaining certain level of stability in biodiesel during storage; high level of unsaturated fatty acid methyl ester content makes it difficult to improve and maintain maximum oxidation stability during storage [21, 22]. The anti-oxidant is a chemical that delays the start or slows down the rate of oil oxidation by inhibiting the formation of free radical and hence contributes to the stabilization of the oil [23].

Table 4.0: Fuel Properties of Ethyl esters produced from *Z.mauritiana* and *L.lanceolata*

Fuel Parameter	***Ziziphus M.***	***Lophira l.***	**ASTM Limits***
Ethyl ester yield (%)	78.0±2.14	76.0±3.11	NS

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K.viscosity at 40^{oC}(cSt)	5.2±0.31	6.4±0.21	1.9 to 7.0
Acid value ($mgKOHg^{-1}$)	1.22±0.05	1.40±0.04	0.80max
Cloud Point (^{O}C)	14±2.01	10±2.11	-3 to 12
Pour point (^{O}C)	3±0.01	5±0.42	-5 to 10
Ash content (%)	0.05±0.00	0.08±0.00	0.02max
Sulphur content (%)	0.034±0.00	0.03±0.00	0.05max
Flash point (^{O}C)	155.0±2.03	161.0±3.00	

Values are mean ±SD triplicate analysis of sample

Source:* [24].

Fuel Properties of alkyl esters produced

The results obtained in this study show an improvement in biodiesel properties compared to those of their parent oil feedstock (Tables 3& 4). The transesterification reaction has separated out undesirable substances along with glycerol and hence improved the fuel properties of the oils. Fuel properties of biodiesels studied include among others: acid value, cloud point, pour point, ash content, flash point and sulphur content. Properties of biodiesel fuel depend on the nature and chemical composition of oil feed stocks as each feed stock has a unique chemical composition that directly influences the properties of biodiesel fuel produced. Acid value in biodiesel estimates level of free fatty acids that may be present in an alkyl ester and it is a quality yardstick. Free fatty acids can lead to corrosion and could be a symptom of water in the fuel [24] . The acid values for methyl esters of both *Ziziphus* and *Lophira* stand at 0.64±0.05 ($mgKOHg^{-1}$) and 0.62±0.03 ($mgKOHg^{-1}$) respectively; and are within the limits of ASTM standard specification.

The cloud point (CP) and pour point (PP)values of Ziziphus and Lophira biodiesels are observed as 11±1.02 ^{O}C, 10±1.21 ^{O}C and 3.0±0.42 ^{O}C, 4.01±0.02 ^{O}C respectively. At low temperature biodiesel becomes less appropriate to use than petro-diesel. The tests for CP and PP are essential to indicate the first wax appearance and non-flow temperatures for the fuel. The recorded values (Table 3.0) are not as high as for methyl tallowate CP 17^{O}C, PP 15^{O}C but are comparable to yellow grease methyl esters CP 10^{O}C, PP 6^{O}C [25]. The low temperature behaviour of biodiesel is significantly influenced by its molecular structure and the level of saturation of the ester, however, the effect of unsaturated ester composition can be negligible [26].

Sulphur contents results for both methyl and ethyl esters for *Ziziphus and Lophira* alkyl esters as depicted in Tables 3.0 & 4.0 shows that methyl ester values range from 0.024 to 0.041% while that of ethyl ester range from 0.034 to 0.031% respectively. The sulphur contents were analyzed using Energy Dispersive X-ray Fluorescence Spectrometry machine. Presence of sulphur in biodiesels

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may have originated from the phospholipids present in vegetable oils. In both cases, values obtained are within the limits of standards specified [9]. This result is consistent with that of Vicente *et al.,*[27] on vegetable oils.

Ash contents of biodiesels are indicative of the amount of inorganic matter, catalyst residues and other impurities [28]. High concentration of these materials in fuels could lead to injector tip plugging, combustion deposits and injection system wear [29]. The results of ash content for both methyl ester and ethyl esters for *Ziziphus* and *Lophira* range from 0.05 to 0.08% (ethyl ester), while that of methyl ester are 0.041 to 0.044% (Tables 3.0 & 4.0) respectively. However, the values observed here are higher than what was stipulated by the standards. As reported elsewhere [29], ash content is important for the heating values of biodiesels. Heating value increases with increasing ash content, therefore; provides a measure of stability of a product for a given application.

Flash point (FP) values of both alkyl esters range (Tables 3.0 &4.0) from 150 to 170 ^{O}C which is moderately good. FP is a parameter that could be influenced by the production methods or purification steps; therefore it corresponds to the content of methanol in a given biodiesel. The values derived from *Lophira* and *Ziziphus* alkyl esters are higher than diesel fuel (60 to 80) ^{O}C, but within the range of biodiesel fuel (100 to 170) ^{O}C [30]. Achten *et al.,*[31] reported FP value for *Jatropha curcas* methyl ester at 170^{O}C, soybean, castor and neem seed oil methyl esters were reported elsewhere as 182, 177 and 183 ^{O}C respectively [32]. It is a safety guide, the higher the value; the less susceptible the biodiesel obtained will be to flammability hazard. FP for biodiesel has been set at 93^{O}C (200^{O}F) minimum, which suggest that; biodiesels reported are not flammable but would require safety precautions like any fuel during usage, storage and transportation [33].

CONCLUSION

Oils extracted from seeds of *Lophira* and *Ziziphus* were transesterified to methyl and ethyl esters of fatty acids using ethanol, methanol and sodium hydroxide. The results indicates that; the seeds contained appreciable amount of oil and analysis of their methyl and ethyl esters revealed that; these biodiesels possess parameters that are within the acceptable limits of ASTM standard specification for alkyl esters (biodiesels). Therefore, Seeds of *Ziziphus mauritiana* and *Lophira lanceolata* are recommended as feedstocks for biodiesel production.

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