PRODUCTION OF BIODIESEL FROM WILD GRAPE'S SEED

(Lannea Microcarpa, Local Name: Faru)

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ABSTRACT

Biodiesel is becoming an increasingly important alternative source of energy in the world. The use of biodiesel will reduce the use of fossil fuels, thereby minimizing the emission of green house gases. The use of biodiesel will also lead to environmentally friendly, sustainable and viable sources of energy, which will reduce the dependency on depleting fossil fuels. Oil from wild grape's seed (lannea microcarpa, local name: Faru) was extracted using soxhlet extractor with normal hexane as solvent. The oil was transesterified with methanollic using sodium hydroxide as catalyst. The percentage yield of the oil was 41% and that of the biodiesel obtained was 79.80% .The fuel properties of the oil and its methyl ester were evaluated for flow and heat characteristics. The results shows that, the moisture content, ash content, acid value, saponification value, pour point, density and viscosity of the biodiesel obtained are; ***1.0, 1.5, 5.61, Nil, 0-7, 0.88, 12.6*** *respectively. These were compared with ASTM (American Society for Testing Material) and USNSDB (United State National Soya-Diesel Board) standards. The result obtained from GC/MS analysis of the biodiesel showed that, the predominant fatty acid methyl esters were oleic acid methyl ester, hexadecanoic acid methyl ester, eicasonoic acid methyl ester and butanoic acid-3-hydroxy ethyl ester. The physico-chemical properties were very close to those of diesel fuel and hence can be used as alternative fuel for diesel engines and thus it is recommended that more research effort should be directed to improve on this research.*

Keywords: Acid Value, Biodiesel, Moisture Content, Renewable Energy, Saponification, Transesterification

INTRODUCTION;

Energy from fossil fuel play very important role in our lives, but such important roles is being clouded due to the environmental hazards from fossil fuel emissions (amazon.com 2010). This has led to the new dimension in energy production and utilization known as renewable sources of energy. As a substitute for the fast depleting fossil fuel

(Sambo, 2010), biodiesel which is an alternative for or an additive to diesel fuel that is derived from the oils and fats of plants, like Sunflower, Canola or Jatropha had come to stay (Jatrophabiodiesel, 2010). It should be able to reduce and maintain the price of automobile fuel and drastically minimized the environmental unfriendliness of the hydrocarbons based fuel (Jatrophabiodiesel,2010).The under exploited and unexploited edible and non edible seeds of many plants are good sources of Biodiesel (Jatrophabiodiesel,2010). Our country is endowed with many of such plants. Research is being carried out now to convert also vegetable oils into Biodiesel through biotechnological processes (Jatrophabio diesel, 2010). With a concentrated and coordinated effort, wide use of Biodiesel in our country is going to be a reality in the days to come. A national energy master plan and renewable energy master plan drafted by Energy Commission of Nigeria encouraged Bio-Diesel production which covers all aspects of plantation, procurement of seed, extraction of oil, trans-esterificatation, blending and trade, as well as research and development. As at 2008, diesel forms about 11% of the energy consumed in the form of hydrocarbon fuels, and its demand was estimated at 1,517,496.64 million litres (NNPC ASB, 2008).

Therefore, blending becomes necessary to minimize environmental pollution and also saves the country's exchequer (Jetrophabiodiesel, 2010). Nigeria has vast stretches of degraded land, mostly in areas with adverse agro-climatic conditions, where species of Jatropha, Soya beans, Wild grapes, Sunflour and so on can be grown easily.

Even 30 million hectares planted for bio- diesel can completely replace the current use of fossil fuels. The production of Biodiesel will also boost the rural economy and more enthusiasm in more than one billion lives in the area and generates thousands of jobs for both rural and urban areas (Jatrophabio diesel, 2010).

What is Biodiesel?

Biodiesel can be defined as simple alkyl esters of fatty acids produced from vegetable oils and animal fats. And it also refers to a non-petroleum-based diesel fuel consisting of long-chain alkyl (methyl, ethyl or propyl) esters. Thus, the word "bio" refers to a biological source in contrast to the petroleum-based-fuel. Biodiesel is made by chemically-reacting lipids, typically vegetable oil or animal fat (tallow), and alcohol. It is a clean burning renewable fuel made through a chemical process which converts oils and fats of natural origin into fatty acid and methyl esters (FAME) (Smart Fuels LLC, 2009).

Types of Biodiesel

1) Methyl Palmitate/ Methyl Hexadecanoate

2 Methyl Stearate/Methyl Octadecanoate

3 Methyl Oleate/Methyl 9(z)- Octadecenoate

4 Methyl Linoleate/Methyl 9(z), 12(z)-Octade Cadienoate

5) Methyl Linoleate/Methyl 9(z), 12(z), 15(z)-Octadecadienoate

Biodiesel Production

Biodiesel is produced from vegetable oil or animal fats. It is an alternative fuel formulated exclusively for diesel engines. With fuel prices increasing and growing environmental awareness, the need to consider alternative energy and fuel sources is becoming a necessity. One alternative is the use of biodiesel fuel which is becoming more and more popular (Haresh K., 2008). In the United States, much of the impetus for the development of biodiesel had come from soybean farmers who saw it as a potential new market for soybean oil. In March 1992, a trade group of soybean farmers formed the National Soyfuels Advisory Committee to investigate whether a potential market existed for biodiesel. The committee formed the National Soydiesel Development Board (NSDB) in October 1992 which was later changed to National Biodiesel Board (NBB). Hence, the NBB coordinated and financed much of the biodiesel related research during the 1990's (NBF, 1994).

Types of Substrate for Biodiesel Production

A variety of substrates could be used to produce biodiesel such as:

(i) Virgin oil feedstock; rapeseed and soybean oils: soybean oil alone accounts for about 90% of all biofuels stocks in the US. (Adams C., *et al*, 1983).

(ii) Field pennycress, Jatropha and such crops like mustard, flax, sunflower, palm oil, and Wild grape. The biodiesel from Jatropha seed gave one of the best properties of biodiesel. From a research conducted on production of Biodiesel from Jatropha seeds in S. Traore, Polytechnic Institute, Department of Chemical Engineering, University of Conakry, its physico-chemical property values were reported as: viscosity 4.8 mm^2/s; density $0.87 g/cm^2$; free fatty acid 0.25 mgKOH/g; and acid value 0.49mgKOH/g. These properties are all within the ASTM biodiesel standard (Ma P., et al, 1998).

(iii) Waste Vegetable Oil (WVO). This is defined as any non-petroleum-based or synthetic oil that, through use or handling, has become unsuitable for its original purpose due to the presence of impurities or loss of original properties. For instance groundnut oil which has been used to fry meat and bean cake repeatedly (Andrew Stevens et al, 2005).

(iv) Animal fats including tallow, lard, yellow grease, chicken fat, and the by-products of the production of Omega-3 fatty acids from fish oil (Kiong E, 2006).

(v) Algae, which can be grown using waste materials such as sewage without displacing land currently used for food production (Leonard and Christopher, 2007).

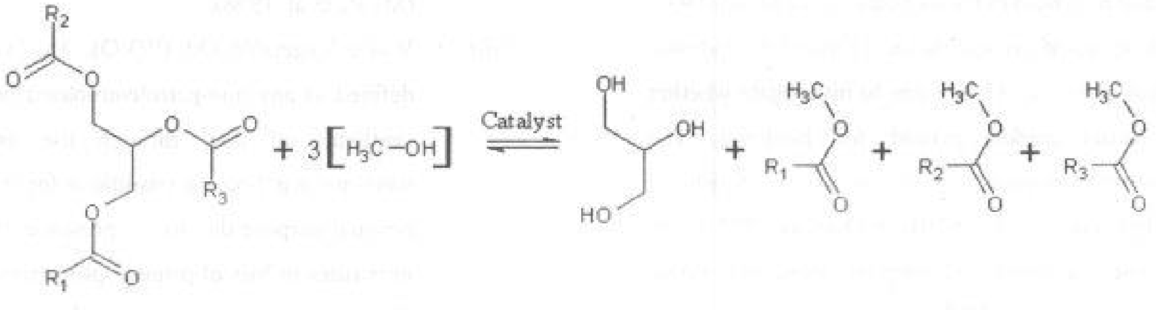
(vi) Oil from halophytes such as *Salicornia bigelovii*, which can be grown using saltwater in coastal areas where conventional crops cannot grown with yields equal to the yields of soybeans and other oilseeds grown using fresh water irrigation (Glenn Edward P ., *et al*, 1998).

Production of Biodiesel from *Lannea Microcarpa* (Wild Grape's Seeds)

This research paper used wild grape's seed which is botanically known as *lannea microcarpa* as the substrate utilized to produce bio-diesel. So far, there is no known literature on the production of biodiesel from lannea microcarpa seeds, and the exploration of its potential utilization in bio-diesel production.

Transesterification Process

Transesterification is the process of reacting triglycerides with alcohols (methanol or ethanol) in the presence of a catalyst (NaOH, KOH or CH_3ONa) to produce biodiesel. The process breaks down the molecules of vegetable oils into constituent molecules forming biodiesel as the main product and glycerin as the by product. The chemical name for biodiesel is fatty acid alkyl esters (Haresh K., 2008).The chemical equation for the reaction is given as shown in figure I.



Triglyceride Methanol (3) Glycerol Methyl Esters

Figure 1:

Source: Wikipedia (2009).

The catalyst is usually sodium or potassium hydroxide that has already been mixed with the methanol. R_1, R_2 and R_3 indicate the fatty acid chains associated with the oil or fat which are largely palmitic, stearic, oleic and linoleic acids for naturally occurring oils and fats (NBB, 2007, Wikipedia, 2009).

The catalyzed based production of biodiesel generally occurs using the following steps:

(i)Mixing of Alcohol with Catalyst: The catalyst is typically sodium hydroxide (caustic soda) or potassium hydroxide (potash). It is dissolved in the alcohol using a standard agitator or mixer (NBB, 2007).

(ii)Reaction: The alcohol/catalyst mixture is then put into a closed vessel and the oil or fat is added. The system from here on is totally closed to the atmosphere to prevent the loss of alcohol for a recommended time of 1 -8 hours. The reaction mix was kept just above the boiling point of alcohol (around 160^0F; 71^oC) to speed up the reaction (NBB, 2007).

(iii) Separation: Once the reaction is complete, two major products exist: glycerin and biodiesel. The glycerin phase is denser than the biodiesel phase and the two can be gravity separated with glycerin simply drawn off the bottom of the settling vessel. In some cases a centrifuge is used to separate the two materials faster (NBB, 2007).

(iv) Alcohol Removal: Once the glycerin and biodiesel phases have been separated, the excess alcohol in each phase is removed with a flash evaporation process or by distillation (NBB, 2007).

(v) Glycerin Neutralization: The glycerin by-product contains unused catalyst and soaps that are neutralized with an acid and sent to storage as a crude glycerin. In more sophisticated operations, the glycerin is distilled to 99% or higher purity and sold into the cosmetic and pharmaceutical markets (NBB, 2007).

(vi) Methyl Ester Wash: Once separated from the glycerin, the biodiesel is sometimes purified by washing gently with warm water to remove residual catalyst or soaps, dried and sent to storage. This is normally the end production process, resulting in a clear amber yellow liquid with a viscosity similar to petro-diesel (NBB, 2007).

Chemical Component of Biodiesel

As earlier mentioned, biodiesel is derived from vegetable oils. The major components of vegetable oils are triglycerides which are esters of glycerol with long-chain acids commonly called fatty acids (Gerpen J.V et al, 2004).

Vegetable oil comprises about 98% triglycerides and small amount of monoglycerides and diglycerides. The fatty acid varies with the number of carbon atoms and in each the first carbon carries the functional group (Cebula D.J., and Smith K.W., 1991). Table 1 below lists the most common fatty acids and their corresponding methyl esters.

Table 1: ***Prospective Biodiesel from Vegetable Oils***

S/No	**Fatty Acid**	**Name (Trivial name)**	**Structure**	**Formula**	**Methyl Ester**	**Name (Trivial name)**
1	Palmitic Acid	Hexadecanoic Acid	$R - (CH_2)_{14} - CH_3$	$C_{16}H_{32}O_2$	Methyl palmitate	methyl hexadecanoate
2	Stearic Acid	octadecenoic acid	$R - (CH_2)_{16} - CH_3$	$C_{18}H_{36}O_2$	Methyl stearate	methyl octadecanoate

\3	Oleic acid	9(z)- octadecenoic acid	$R - (CH_2)_7 - CH = CH - (CH_2)_7 - CH_3$	$C_{18}H_{34}O_2$	Methyl oleate	9(z)- octadecenoate
4	Linoleic cid	9(z octadecadienoic acid), 12(z)	$R - (CH_2)_7 - CH = CH - CH_2 - CH{=}CH{-}(CH_2)_4{-}CH_3$	$C_{18}H_{34}O_2$	Methyl linoleate	methyl 9(z), 12(z)-octade cadienoate
5	Linolenic acid	9(z), 12(z), 15(z)- octadecatrienoic acid	$R - (CH_2)_7 - (CH = CH - CH_2)_3 - CH_3$	$C_{18}H_{30}O_2$	Methyl linolenate	methyl 9(z), 12(z), 15(z)- octadecadienoate.

Source:Gerpen J.V., *et al.*, 2004.

MATERIALS AND METHODS

(I) Extraction of Oil from Wild Grape's Seed

A given mass (80.00g) of the powdered sample was placed into a thimble and covered with clean wool. The thimble was inserted into the soxhlet extractor which was greased and fitted into a round bottom flask containing 250cm^3 of n-hexane. The round bottom flask was placed into a heating mantle and the set-up was clamped to a retort stand. A reflux condenser was then fitted to the top of the soxhlet extraction column and the heating mantle was switched on, the tap connected to the reflux condenser was also turned on. When the n-hexane began boiling, the vapor passes through the condenser which condenses it to liquid and then drops into the sample until the thimble was filled. As the liquid drops into the sample, it extracts the oil and when the thimble was filled up the oil extracted together with the n-hexane was emptied into the round bottom flask. This was repeated several times until the oil was extracted completely and this took about 7 hours. Thereafter, the whole setup was removed leaving only the round bottom flask on the heating mantle. The content heated at 40^{o}C for 10minutes with the round bottom flask placed on a water bath and the heating continued until the excess solvent was expelled and only the oil was left. The oil was then placed in a vacuum oven at 40^{o}C for about 30 minutes to further dry it. The volume and mass of oil extracted were determined.

(II)Transesterification of the Oil

A given mass (0.40g) of NaOH was added to 40cm^3 of methanol and stirred until it was completely dissolved. 100.00g of the oil was placed in a flask and the methanolic NaOH solution was added to it. The mixture was stirred for 20minutes at 25^{0}C and then poured into a separating funnel (Dalai A.K 2004). After about an hour no separation occurred and the mixture was left to stand in the separating funnel for 24 hours, but there was still no separation then, a solution of 1% sulphuric acid was then added and the mixture was stirred and poured into a separating funnel, forming three layers of water, mixture of soap solution and biodiesel. The water was at the bottom, the middle layer was soap and the top layer is the biodiesel. This is common with fatty acids such as oleic acid (Gerpen J.V., *et al.*, 2004) and the acid is present in the faru oil used for the transesterification. The soap solution and water were collected leaving the biodiesel. It is important to mention that glycerin was not observed in the mixture.

The methyl ester was heated on the heating mantle at low temperature and then washed by adding silica gel, stirring the mixture for 20minutes and then filtered. Sodium sulphate crystals were added to a vacuum filtration funnel and the methyl ester was filtered through to remove any trace of water. The dried methyl ester was then bottled and kept for further analysis as recommended by Dalai A.K (2004)

RESULTS AND DISCUSSIONS

RESULTS: The results of the analysis carried out for both the oil and the biodiesel are given in Tables 2, 3 and 4:

Table 2: Physicochemical Parameters for Oil and Biodiesel

S/No	Parameter	Values	
		Oil	Diesel
1	Percentage yield (%)	41	79.80
2	Moisture content (%)	5	1.0
3	Ash content (%)	2.50	1.5
4	Acid Value (mg KOH/g)	11.22	5.61
5	Saponification value (mg KOH/g)	112.2	---
6	Pour point (°C)	-9	0 - 7

7	**Density (g/cm^3)**	0.9255	0.88
8	**Viscosity (mpas)**	36.6	12.6

Table 3: GC/MS Identification of Fatty Acids in Oil

S/No	Fatty Acid Type	Molecular Formula	RT
1	**Oleic Acid**	$C_{18}H_{34}O_2$	29.48
2	n-hexadecanoic Acid	$C_{16}H_{32}O_2$	28.29
3	Butanoic Acid	$C_4H_8O_2$	8.30

Table 4; GC/MS Identification of Fatty Acid Methyl Esters in Biodiesel

	Methyl Ester	Molecular Formula	RT
1	Hexadecanoic acid methyl ester	$C_{17}H_{34}O_2$	28.00
2	Butanoic acid-3-hydroxy ethyl-ester	$C_{19}H_{12}O_3$	8.31
3	Oleic acid methyl ester	$C_{19}H_{36}O_2$	29.08
4	Eicasonoic acid methyl ester	$C_{21}H_{42}O_2$	30.48

DISCUSSION

Ali (2008) studied the production of biodiesel from calabash, water melon and pumpkin and found that the calabash seeds gave highest yield (54.1%) of oil extract followed by watermelon (49.4%) and pumpkin with 48.9%. However, in the case of bio-diesel, pumpkin, calabash and water melon seed's oil gave a yield of 57.80%, .98% and 40.20 respectively. The oil obtained from the *Lannea microcarpa seeds* was dark brown in colour with a percentage yield of 41%. Although this was higher than the oil content of between 10.9 to 30.9% reported for twelve pumpkin tubers by Stevenson *et al.*, (2005); it was lower than the oil yields obtained by Ali (2008) in respect of calabash, watermelon and pumpkin seeds as shown above. However, the bio-diesel produced from the oil of the *Lannea microcarpa seeds* gave a yield of 79.80%, which was substantially higher than biodiesel yield from the oils extracted from calabash, watermelon and pumpkin. Therefore, the bio-diesel obtained was economically viable in terms of biodiesel yields.

Moisture Content

This method is usually applied in the determination of moisture to measure the weight of sample loss due to drying (Gunstune F.P *et al.*, 1986). The moisture content of the extracted oil was 5% and that of the bio-diesel was 1%. The moisture content of the bio-diesel was therefore a little above the ASTM standard of bio-diesel which was less than 0.08%. This will enhance microbial growth in the biodiesel and encourage the reversal of biodiesel into free fatty acids (ASAE, 1996).

Ash Content

The ash content of oils and bio-diesels is a measure of their metallic constituents; it is also an indication of the mineral elements contained in the oil which is a reflection of the nutritional value of the oil (Ali, 2008, Didem O. and Filiz K., 2003). However, in bio-diesel, high ash content is detrimental because it can cause corrosion problems and residues (Didem O. and Filiz K.,2000). The ash content of the oil extracted was found to be 2.5% and that of the bio-diesel was 1.5% which was higher than threshold values of USA bio-diesel standard of 0.02%. The ash content was high and therefore could cause corrosion problems and engine deposit on combustion (ASAE, 1996).

Acid Value

The acid content of the oil can be defined as the percentage of free fatty acid present in the oil. The acid value is the number of milligram (mg) of potassium hydroxide required to neutralize 1.0g of oil (Ali, 2008). The Acid Value (mgKOH/g) of the oil extracted in this research was 11.22 and for the bio-diesel were 5.61. The acid value of the bio-diesel obtained from wild grape's seed was above the standard limit but lower than that of the oil extracted from thesame seed. High acid value in the biodiesel causes corrosion and engine deposits that affect the performance of the engine (Mahajan S., et al, 2005).

Saponification Value

Saponification value is the number of milligram of potassium hydroxide (mgKOH) which is required to saponify 1g of oil; that is, to neutralize the free fatty acids and the fatty acids combined as acylglycerol (Gunstone F.P., *et al.*, 1994). In other words, it is the amount of alkali necessary to saponify a definite quantity of the sample. It has an inverse relationship to the Molar mass of the fatty acids in oils or fats; and can be used to estimate the potential or the ability of the oil for soap manufacturing. The saponification values reported by Ali (2008) were 140.23, 210.38 and 252.45 mgKOH for pumpkin, water melon and calabash respectively. He concluded that the values indicated the presence of high molecular weight fatty acids in pumpkin seed oil which may make it less suitable for soap and paint industries compared with calabash seed oil which contained low molecular weight fatty acid.

The saponification value of the oil obtained in this research work was 112.20 mgKOH which was much lower than the values reported by Ali (2008) for pumpkin, water melon and calabash. This suggests that the oil in *Lannea microcarpa seeds* has higher molecular weight fatty acids than the oils in pumpkin, water melon and calabash.

Pour Point

The pour point of a liquid is the lowest temperature at which it will pour or flow under prescribed conditions. It can also be defined as the minimum temperature of a liquid, particularly a lubricant, after which the liquid ceases to flow on increasing the temperature (Wikipedia, 2009). Its significance is to indicate the lowest temperature at which particular product will not move when it is held horizontally. It is therefore an important parameter to consider in handling, storage and transportation of oils (Didem and Filiz, 2003). Ali (2008) reported that the pour point of pumpkin oil was -11°C which was lower than that of watermelon (-9 °C) and calabash (-7 °C). He also reported that the pour point of the bio-diesel obtained from the three seed oils were -7, -5 and -4°C for pumpkin, calabash and water melon, respectively. These are all below 0.02°C recommended by the USA Bio-diesel Standard. He concluded that the bio-diesel could be stored and transported very easily and that it will not freeze in temperate regions.

The pour point of the oil was found to be -9°C and that of the bio-diesel was -7 °C. The values showed that pour point for the oil and bio-diesel obtained in this work were in the range with those obtained by Ali (2008) for calabash, water melon and pumpkin. They were much lower than 0.02°C recommended by the USA bio-diesel standard. Therefore, it can be concluded that the biodiesel can be easily stored and it will not freeze in temperate regions.

Density: Density is also an important parameter used to check the purity of bio-diesel and suggest the likely influence of the oil to the performance of pumps (Ali, 2008). High density of biodiesel affect the volume of oil delivered into the combustion chamber of the diesel engine (Chris Sharp, 1997). The density of the oil was 0.93(g/cm^3) and that of the bio-diesel was 0.88(g/cm^3). This is also within the recommended range of 0.87 to 0.98 for biodiesel (USA Biodiesel Standard, 2005).

Viscosity is a resistance of a liquid to flow or it's thickness; it describes a fluids internal resistance to flow and may be thought of as a measure of fluid friction. Thus water is "thin" having a lower viscosity while vegetable oils were "thick" having a higher viscosity (Wikipedia, 2009).The higher the viscosity the less readily the liquid flows and it causes the injectors not to work properly (Allen C.A.W., et all ,1999).Ali (2008) reported viscosity values of 101.5,86.8 and 72.8 mm2/s for the oil extracted from pumpkin, calabash and watermelon respectively. He also reported the viscosities of the biodiesel produced from three oils as 19.6, 21.0 and 22.4 mm2/s for pumpkin, calabash and watermelon respectively. The viscosity of the wild grape's oil extracted was 36.6mm2/s while that of biodiesel obtained from the oil extracted from the wild grape's seed was 12.6mm2/s . This shows that the viscosity of the biodiesel obtained from wild grape's seed was higher then the maximum value of 6.0mm2/s recommended in USA standard for biodiesel. Hence it will have less adverse effect on the injectors of engines.

GC/MS Analysis

Gas chromatography is a common confirmatory test which separates all the components in a sample and provides a representative spectral output. Mass spectroscopy identifies these components by electrically charging the spacemen molecules, accelerating them through a magnetic field, breaking the molecules into a charge fragments and detecting the different charges. The GC/MS analysis result shows that the main fatty acids in the oil are oleic acid, n-hexandecanoic acid and butanoic acid. While the main methyl-esters are hexadecanoic acid methyl ester, oleic acid methyl ester, butanoic acid -3-hydroxy methyl ester and eicasanoic acid methyl ester .

Conclusion

The increase cost of conventional fuel necessities the exploration of other sources of energy. The under exploited and unexploited edible and non edible seeds of many plants are good sources of biodiesel. Raw materials for Biodiesel production are abundant throughout Nigeria especially in rural areas. Biodiesel can be produced from plant seeds to substitute for fossil fuel. The search for alternative energy sources like Biodiesel should be intensified so that wastage of non edible plant's seed can be arrested. The results of the GC/MS studies conducted on the products of the esterification of oil extracted from Lannea Microcarpa seeds shows that it contains methyl esters such as hexadecanoic acid methyl ester, oleic acid methyl ester, butanoic acid -3- hydroxyl methyl ester and eicasanoic methyl ester. This indicated that biodiesel could be obtained from Lannea Microcarpa seeds. Some of the physico-chemical properties of biodiesel determined revealed that the quality of the biodiesel obtained from Lannea Microcarpa seeds was good.

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