Review of *Sclerocarya birrea* seed oil extracted as a bioenergy resource for compression ignition engines

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Abstract: *Sclerocarya birrea* (Marula) seed oil was extracted and characterized for its physico-chemical properties and fatty acid compositions, respectively, by using standardized laboratory methods of the Association of Official and Analytical Chemist (AOAC). The fuel and lubrication properties of marula oil were also determined by using the ASTM methods, and the oil was evaluated in terms of its antiwear, viscometrics, volatility, stability, environmental compatibility properties and energy content. It was found that the high percentage of mono-unsaturated oleic acid (73.6%) provided the oiliness that makes marula oil a natural alternative to genetically modify high oleic acid sunflower oil used in biodiesel production. The aggregate properties of seed oiliness as exemplified by the high oleic acid content, high saponification value (178.6 mg/KOH) and viscosity (41 mm^2/s) makes marula oil to be prospective based oil for engine crank case biolubricants with antiwear and friction reduction properties. However, the higher oil viscosity exhibited by marula seed oil in comparison to diesel could pose some durability problems to compression ignition engines, when used directly as fuel. Nonetheless, the reduction of oil viscosity would be required by heating, blending with diesel fuel, or by transesterification to forestall the risk of engine failure resulting from the use of unmodified marula oil. The flash point of marula oil (235℃) is somewhat close to that of monograde SAE 40 mineral oil (240℃), and appreciably higher than that of diesel fuel (52℃). The high flash point makes the seed oil less flammable and ensures safer handling and transportation. While, the low pour point (-13.7℃) ensures the oil usability for engines at cold start and under low load conditions. The oxidation stability of marula oil is ascribed to the traces of natural antioxidants presented in the oil and improves the oil's shelf life, notwithstanding the high peroxide value (4.58 mequiv/kg), and linolenic acid content (0.3%), which ought to have been the culprit for lipolytic hydrolysis and rancidity. Furthermore, marula seed oil is more biodegradable and environmentally friendly than oils derived from petroleum crude. The closely related cetane number (47.8) and heating values (38.2 mJ/kg) of marula oil to diesel fuel would undeniably sustain the combustion efficiency of diesel fuel and also supply a comparable engine performance output in compression ignition engines. The candidacy of marula seed oil, as a bioenergy resource for alternative fuel, fuel additives and lubricants, will no doubt expand the energy supply mix, conserve fossil fuel reserves and mitigate environmental contamination.

Keywords: marula seed oil, high oleic acid, oxidation stability, cetane number, heating value, bioenergy resource, compression ignition engine

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1 Introduction

In recent years, the fluctuating price of crude oil, depleting fossil fuel resources, and the growing concerns over environmental pollution has stimulated renewed interest in seed oils as a substitute for petroleum crude for reasons as following: they exhibit high calorific value, they have superior environmental compatibility, they are portable, non-toxic and non-flammable and safely handled, they contain low sulphur and aromatic content, and they are available from renewable sources[1]. Nonetheless, some engine durability problems occurred in longer term usage, such as clogging of fuel filter, coking of injector tips and trumpet formation on the injector, more carbon deposits, oil ring sticking, and thickening and gathering of the lubricant oil. These problems are evident in direct use of neat seed oils[2-11]. The overwhelming challenge posed by the direct use of neat seed oil as fuel in a long term basis could be evaluated in terms of its high fuel viscosity in compression ignition engines, lower volatility and higher reactivity due to the presence of unsaturated hydrocarbon chain. However, to surmount these shortcomings, seed oils must either be heated, blended with diesel fuel or chemically altered (by alcoholysis) to prevent premature engine failure[12]. Alternatively, an engine modification designed to accommodate the conditions of use and the oil involved has also proved to be helpful[13].

For the purpose of this study, *Sclerocarya birrea -sbsp caffra* (marula) oil was extracted from the kernels of the marula tree of the *Anacardiaceae* family. The plant occurs through west, north-east and east tropical Africa across a range of vegetation types, principally wooded grassland and dry savannah of the northern tropical Africa and sahelian region[14], and this include northern Nigeria. The fruit is round or oval drupe, usually wide with a diameter of 30-40 mm. The shape and number of nuts per stone determine the final shape of the fruit. Marula fruit has a thick, soft leathery exocarp with tiny, round or oval spots, enclosing a juicy, mucilaginous flesh that adheres tightly to the stone and can be removed only by sucking. The flesh tastes tart, sweet and refreshing, although the fruit has a slight turpentine-like aroma and can give off a very unpleasant smell when decaying. Each fruit contains an exceedingly hard seed, which is covered by fibrous matter. The nut comprises an average of 90% of shell and only 10% of kernel. According to Quin[15] and Shon[16], the seeds inside the stone can also be eaten and they have a delicate nutty taste, and a high nutritive value and high (up to 56%) oil content per kernel. The energy value of the kernel is approximately 2 699 to 2 703 kJ per 100 g kernel[16]. There are more than 350 oil bearing crops identified, among which are sunflower, safflower, soybean, cottonseed, rapeseed and peanut oils, which are considered as potential alternative fuels for Diesel engines[17]. Even though, marula oil is traditionally used in cosmetics, in food as cooking oil and as a meat preservative and to treat leather against spoilage, so far no published results have shown marula oil's suitability as biodiesel and lubricant for compression ignition engines. Hence, the objective of this study is to characterize marula oil obtained from marula tree in northern Nigeria, and comparatively evaluate its bioenergy potentials in comparison to conventional mineral oil based lubricants and diesel fuel used in compression ignition engines.

2 Materials and methods

2.1 Sample collection.

The marula plant was identified in the Herbarium of Abubakar Tafawa Balewa University in Nigeria. Ripened fresh fruits were collected from marula trees within the premises of Federal Polytechnic and Abubakar Tafawa Balewa University in Bauchi.

2.2 Oil extraction

The nuts were sun dried in yards and mechanical decorticated to expose the kernels. The marula kernels were roasted at temperatures between 45℃ and 47℃ to make oil extraction easier and reduce the moisture content[18]. Oil from marula kernels is extracted by

mechanical expression using manual ram presses[19]. The expressed oil is collected for analysis and the residual marula seed cake that remains after oil extraction was disposed as feed for ruminants animals.

2.3 Physico-chemical characterization

The physical and chemical properties of marula oil were determined according to the standard procedure recommended by AOAC[20], Pearson[21], and Pa Qurt[22]. The oil properties, including specific gravity, saponification, peroxide, iodine, and free fatty acid values, were analyzed.

2.4 Fatty acid determination

The fatty acid of marula oil sample was determined in accordance to the method described by Atasie et al.[23]. In this case, about two grams of the oil sample was weighed, in a small beaker and dissolved in 50 mL of chloroform, transferred into a hundred volumetric flasks and diluted to the mark with chloroform. One milliliter of the marula oil sample was transferred into a 10 mL screw top culture tube with a Teflon liner. Exactly 1.00 mL of a standard solution of 0.814 mg/mL pentadecanoic acid was then added. The glyceride in the oil sample was esterified as well as the pentadecanoic acid standard. The efficiency for the esterification of the standards is the same as that of the glycerides. Also, the response of the detector of each of the fatty acid methyl ester with the internal standard was the same. Hence, the amount of each ester in the fat was determined by comparing the integrated areas with the known concentration of the standard. Most of the chloroform was then evaporated under a stream of nitrogen until 100 μL of the solution remained. One milliliter of interesterification reagent (25% volume of a 12% BF3 methanol solution, 20% volume of benzene and 55% volume methanol) was added. The tube was flushed with nitrogen, sealed and heated in a 100℃ water bath for 30 min, after which the methyl esters was extracted with hexane and water, the final mixture of the reagent, hexane and water were in the ratio of 1:1:1 (adding 1.00 mL each of hexane and water to the reaction mixture). The mixture was shaken thoroughly for two minutes. A stable emulsion was formed which was broken by centrifugation. Half of the top hexane phase was transferred into a small test tube for injection.

2.5 Lubrication analysis of marula oil and mineral oil

The lubrication analyses of seed oils in comparison to mineral oil based lubricants (i.e. SAE 40 and SAE 20w50) were carried out in accordance with the American Society of Testing and Materials (ASTM) standard test method[24]. In this regard, ASTM D445-88 was used to determine the kinematic viscosity: ASTM D2270-86, viscosity index (VI); ASTM D4052-91, relative density; ASTM D97-87, pour point; ASTM D93-90, flash point; ASTM D2015-85, higher heating value; and ASTM D482-91, ash content tests.

2.6 Determination of fuel properties

The fuel properties of marula oil and diesel fuel were carried out in accordance with standardized ASTM test procedures and they include: ASTM D97-93, density; ASTM D2015-85, higher heating value; ASTM D93-94, flash point; ASTM D613, octane number; and ASTM D445, kinematic viscosity[24].

3 Results and discussion

3.1 Physico-chemical properties of marula oil

It could be seen from the results of physic-chemical characteristics of marula oil in Table 1 that the refractive index of crude marula oil (1.46) is comparable to castor oil[25]. The viscosity of crude marula seed oil (41 mm^2/s**)** is lower than that of castor oil (98.62 mm^2/s), but higher than that of palm oil (34.7 mm^2/s), soya oil (25.0 mm^2/s), groundnut oil (24.7 mm^2/s), cotton oil (18 mm^2/s) and much higher than that of petroleum diesel (3.6 mm^2/s)[25]. It could also be seen from Table 2 that the viscosity of crude marula oil is higher than that of petroleum diesel (3.6 mm^2/s or cSt). The obvious difference between the viscous behaviour and marula seed oil could be attributed to higher “van der waals” forces acting among oil molecules, molecular weight of the fatty acid and the types of carbon chain presented in the oils[26]. It has been observed that fuel viscosity has impacts on fuel injection and combustion. The use of unrefined seed oil leads to poor atomization due to high oil fuel viscosity. Hence, if fuel viscosity is high, the injection pump will be

unable to sufficiently pump fuel into the combustion chamber and consequently leads to power loss in engines due to poor combustion[27,28]. From the foregoing, there is need to chemically modify the high viscosity profile of crude marula seed oil to overcome any foreseeable engine durability problems that would arise when in use as fuel for compression ignition engines. Conversely too, the high viscosity and the marula seed oil could also be exploited for developing engine crank case lubricants.

The result in Table 1 revealed that marula seed oil also exhibited a much higher acid value (41.4 mg KOH/g) than palm oil (2.46 mg KOH/g), groundnut oil (0.159 mg KOH/g), cotton oil (0.159 mg KOH/g), soya oil (0.139 mg KOH/g), and castor oil (1.05 mg KOH/g) acid values[25]. The acid value is a measure of the extent to which the constituent glycerides have been decomposed by lipase action. The edibility of oils is evaluated on the basis of the oils acid value[20,21]. Hence, oils with lower acid values are more likely to be used for food. The high acid value of marula oil also confirms the presence of high free fatty acid (i.e. 20.7%) in the oil, and its susceptibility to oxidation[23].

Table 1 Physio-chemical properties of marula oil

Characteristics	Description
Colour	Light yellow
Odour	Nuttish
Refractive index	1.46
Kinematic viscosity (mm^2/s)	41
Specific gravity at 15^o	0.88
Acid value (mg KOH/g)	41.4
Peroxide value (mequiv/kg)	4.58
Saponification value (mg KOH/g)	178.6
Iodine value (g/100 g)	100.34
Free fatty acid (%)	20.7
Unsaponified matter (%)	3.06

Table 2 Fuel properties of marula oil in comparison with diesel fuel

Characteristics	Diesel oil	Marula oil
Specific gravity at 31℃	0.820	0.989
Viscosity (cSt)	3.6	41
Cetane number	47.8	47.8
Flash point (℃)	52	235
Heating value (mJ/kg)	45.45	38.2

The peroxide value of marula seed oil of 4.58 mequiv/kg is higher than that of cotton seed oil of 2.5 mequiv/kg[29]. The higher peroxidation value predisposes the oil to lipolytic hydrolysis and oxidation deterioration (i.e. rancidity) with a consequent shorter shelf life. The saponification value is an indication of the average molecular mass of fatty acids presented in marula seed oil. It could be seen from Table 1 that the seed oil had a high saponification value of 178.6 (mg KOH/g), suggested the presence of fatty acids of high molecular mass. It is worthy to mention that higher saponification levels and oleic acid content improves the lubrication property of the oil, it reduces engine wear and hence, extends the operational life of diesel fuel pumps and injectors[27,30].

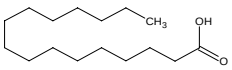
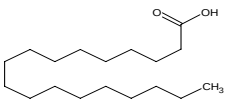
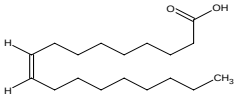
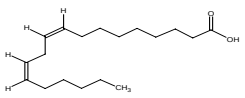
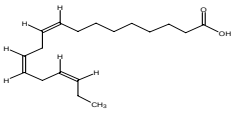
Iodine value is used to measure the degree of unsaturation of oils, and also offers a scale for oil classification. According to Plummer[31] and Lambert and Muir[32], oils with iodine values ranging from 130 to 200 are regarded as drying oils; those with iodine value between 90 and 130 are semidrying oils. While seed oils with iodine values below 90 are considered as non-drying oils. The iodine value of marula seed oil is 100.33 g/100 g. However, this value is lower than that of soya oil (127.1 g/100 g) and cotton oil (114.86 g/100 g), respectively[25]. The iodine value of marula seed oil indicates semi-drying oil with a high degree of unsaturation. The value of the unsaponifiable matter is 3.06 which is significantly high and suggests the presence of squalene, waxes, sterols, phospholids, vitamins, terpenes, steroids, and some hydrocarbon[33].

The result of the low temperature property of marula oil is also presented as pour point (-13.75℃). The pour point is the temperature at which the oil containing so many agglomerated crystals and gels will cease to flow. It is an indicator of whether the fuel can be pumped, even if it would not be suitable for use without heating[34]. If the fuel begins to gel at low temperature conditions, it could clog fuel filters of dispensing equipment and make oil pumping difficult. It is important to note that low temperature behavior is required for engine at cold start and low load conditions.

3.2 Lubricant properties of marula oil

Most lubrication oils are formulated with 70%-94% base oil. These base oils come from three primary sources: crude oil, chemical synthesis, and natural resources, such as fats, waxes, and vegetables[36]. However, four stroke cycle engine oils must have good low- and high-temperature viscosity, low volatility (i.e. high flash point), and good thermal and oxidative stability, and biological base stocks derived from seed oils are ideal base stocks with respect to these properties[37]. In the same vein, the properties of marula oil could be evaluated in terms of its antiwear performance, viscosity, volatility, stability, environmental compatibility properties and energy content, respectively. It is evident from Table 3 that marula oil contains a large proportion of monounsaturated fatty acids. The result presented the fatty acid composition of marula oil as mono-unsaturated oleic acid (73.6%), poly-unsaturated linoleic acid (6.1%), linolenic acid (0.3%), saturated palmitic acid (12.8%), and stearic acid (7.2%). It is, however, important to note that the high percentage of mono-unsaturated oleic acid

Table 3 Fatty acid composition

Fatty acid	Composition (%)	Saturation level	Chemical structure
Palmitic	12.8	Saturated	
Stearic	7.2	Saturated	
Oleic	73.6	Mono-unsaturated	
Linoleic	6.1	Poly-unsaturated	
Linolenic	0.3	Poly-unsaturated	

provides a high degree of oiliness. The combination of the oiliness property, the natural propensity for soap formation (i.e. high saponification tendency), greater affinity towards metal surfaces, and fairly high viscosity could be exploited to minimize metal to metal contact, control temperature, improve the lubricity and avoid wear of vital engine components under boundary and hydrodynamic lubrication conditions. Hence, this property of marula oil makes it suitable as a biolubricant resource.

Table 4 also shows that marula oil exhibited higher VI in comparison to monograde SAE 40 mineral oil lubricant. The VI is the measure of a loss of viscosity with increasing temperature, and is a function of the degree and the type of chain branching in the oil molecule. In general, the more linear the carbon chain length becomes, the higher the VI exhibited[37]. Hence, the comparatively better viscometric property of marula oil can be attributed to the influence of "van der waals" forces acting among oil molecules, the molecular weight of the fatty acid and the types of carbon chain present in the oils[26]. The flash point of marula seed oil is 235℃ (Table 4). This value is somewhat close to that of monograde SAE 40 mineral oil (240℃), but appreciably higher than that of diesel fuel (i.e. 52℃). It is important to note that the flash point is the measure of oil volatility, and also serves as an indicator of flammability for oil stored under ambient conditions. A fuel becomes difficult to handle if the flash point falls below 130℃ limit set by ASTM standards[24]. Hence, the high flash point of marula seed oil makes transportation and handling safer.

The shelf life of lubricant can be measured by the oil oxidative and thermal stability. However, because oils are derived from agricultural feed stocks, storage ability is of vital concern. A study conducted by Burger et al.[42] had analyzed the oxidation stability of marula oil in comparison to other seed oils using the accelerated Rancimat oxidation test. The oil samples were heated to 120℃ with an air flow of 20 L/h. The results showed a higher induction period (and by implication a higher thermo-oxidative stability) of marula oil in comparison to other seed oils (Figure 1). The stability behavior of marula oil could be attributed to the presence of fewer numbers of unsaturated bonds which are prone to oxidation by oxygen from air. Hence, the impressive stability of marula oil could be ascribed to the presence of the high level of saturated and monounsaturated fatty acids; and also the low level of polyunsaturated linoleic and linolenic acid[42-45]. Burger et al.[42] ascribed the resistance of marula oil to oxidation to its fatty acid

constituents. However, more recent understanding of the role of fatty acid compositions to triglyceride stability suggests that this may not be the case[46]. Berthiaume[47] observed that the presence of antioxidants prevents the formation of radicals, and hence slows down oil degradation despite its exposure to oxygen, light, and impurities, such as residual free fatty acids, peroxides, hydro peroxides, and short chain organic acids. Furthermore, Hore[48] and Mariod et al.[49] observed that traces of tocopherols, sterols and flavonoids, procianidine, galattotannin and catechins provide antioxidant action and make the storage and preservation of the marula oil for longer periods possible. Hence, oils with high oxidation stability (longer induction time) have a longer shelf life[34]. However, high oxidative stability is also an essential requirement for biolubricant products.

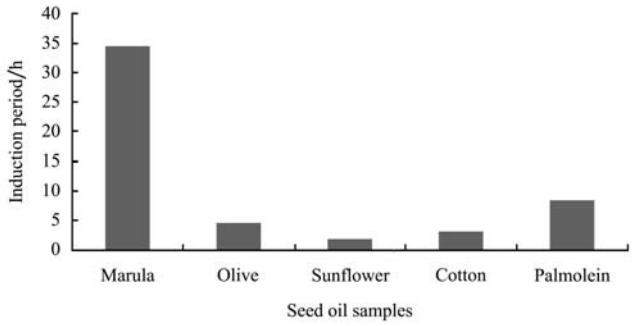


Figure 1 Oxidative stability of marula oil in comparison with other seed oils[42]

In terms of environmental compatibility, lubricants are labeled in terms of their biodegradability, aquatic toxicity, health harzardness, and renewability predispositions. According to Kitamura[50], vegetable (or seed) oils and fatty acid ester are more biodegradable than polyethylene glycol, paraffinic mineral oil, naphthenic mineral oil and polyethylene/propylene glycol. It was also established that vegetable oils (triglycerides), diesters, polyol esters exhibit better biodegradability (60%-100%) than mineral oil. Oil biodegradability decreases for carbon number less than C_4 or greater than $C_{25,}$ and also with increase in chain branching. When the oil degrade as esters, their degradation products are also more soluble in water, although their effects on the ecosystem and ground water are yet unknown[37,51]. Therefore, marula oil with carbon number ranging from C_{14} to C_{24} is no doubt biodegradable and environmentally friendlier than oils derived from petroleum crude[51]. Hence, disposal of used lubricants containing marula oil will pose less environmental risk.

3.3 Fuel properties of marula oil

It could be seen from Table 2 that the cetane number of marula oil is 47.8 and is similar to diesel fuel. The cetane number is a measure of the ignition delay of fuels. The higher the cetane number, the shorter the ignition delay time becomes. According to Demirbas[35], longer fatty acid carbon chains with more saturated molecules are more likely to produce higher cetane numbers and higher combustion efficiency in compression ignition engines[27]. Hence, highly saturated feedstocks (such as lard, tallow, and used cooking oils) can have a cetane number of 60 or higher. According to ASTM D6751 specifications, oils predominated with mono-unsaturated fatty acids such as soy oil, sunflower oil, corn oil, and canola oil-based biodiesels, exhibited cetane numbers closer to the minimum standard of 47 for biodiesel requirement[24,34]. It was also shown in Table 3 that marula oil with a high composition of mono-unsaturated fatty acid also falls within this category.

The high mono-unsaturated oleic acid (73.6%) composition of marula oil makes it serve as a natural alternative to genetically modified high oleic acid sunflower oil used as feedstock for biodiesel production[38]. In comparison, the higher viscosity values of marula oil than diesel fuel could constitute some engine durability challenges when it is used directly as fuel. A minimum viscosity is required for some engines because of the occurrence of power losses caused by injection pump and injector leakage. Higher viscosity fuels could lead to poor fuel combustion, and engine deposits formation. Furthermore, higher fuel viscosity adversely affects fuel atomization, emission levels and fuel consumption[40]. The maximum allowable viscosity in ASTM D975 for No. 2 diesel is 4.1 mm^2/s at 40℃ or slightly higher[34]. Therefore, a significant modification of marula oil viscosity could be achieved by heating, blending with diesel fuel, or by transesterification to reduce the possibility of engine failure occasioned by the high oil viscosity. The volatility of marula oil could be

explained in terms of its flash point. The flash point of marula oil (235℃) is appreciably higher than that of diesel fuel (52℃).

According to NREL[34], the term stability in fuels refers to either long-term storage stability (i.e. aging) or stability at elevated temperatures (or pressures) of fuel reticulating through the engine's fuel system. In petroleum diesel fuel, long-term storage stability is commonly used in place of oxidative stability. Nonetheless, thermal stability is the commonly used term for stability of fuels at elevated fuel system temperatures. In biodiesel, if fuel aging and oxidation stability exceed the ASTM D6751 limits, it leads to high acid numbers, high viscosity, higher oil density, and the formation of gums and sediments that clog filters and constitute durability problems[24,40,41]. It could also be seen from Tables 2 and 4 that marula oil displayed energy content (about 84%) close to diesel fuel but higher than mineral oil based lubricants. It is on record that the inherent energy content of conventional diesel fuel is the largest factor affecting fuel economy, torque, and horsepower delivered by the fuel[34]. If this information is anything to go by, then the relatively high heating value of marula oil is likely to improve the performance output of compression ignition engines, and hence makes it a suitable candidate for fuel additives and biodiesel production.

Table 4 Lubrication properties of marula oil in comparison with mineral oils

Characteristics	Mineral oils		Marula oil
	SAE 40	SAE 20w50	
Specific gravity at 31℃	0.895	0.890	0.989
Kinematic viscosity at 40℃	149	170	41
Kinematic viscosity at 100℃	4.5	18.0	6.3
Viscosity index	95	122	100.5
Flash point (℃)	240	250	235
Pour point (℃)	-15	-27	-13.75
Heating value (mJ/kg)	7.15	7.15	38.2
Ash residue (%)	1.4	1.4	-

4 Conclusions

In this study, marula oil was characterized and the following bioenergy potentials were identified:

1) The high viscosity, saponification value and oleic acid content of marula oil could favorably enhance the lubricity of engine parts and mitigate friction and wear. The reduction of the viscosity profile of marula oil as fuel is required to overcome likely engine durability problems.

2) The high cetane number is comparable to diesel fuel and would no doubt support efficient fuel combustion and engine performance.

3) The heating value of marula oil is slightly lower than diesel fuel, and could slightly affect the engine power output.

4) The high flash point of marula seed oil used as biolubricants and biodiesel could make ransportation and handling safer. Conversely, low pour point is suitable for engines at cold start and under low load condition.

5) The high oxidative stability of marula oil will improve the shelf life of its lubricant and fuel products.

6) The biodegradable property of marula oil makes its lubricant and fuel less harmful to the environment.

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