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LUBRICITY PROPERTIES OF BIOLUBRICANT PRODUCED FROM MARULA KERNEL OIL (*Sclerocarya birrea*) BLENDED WITH MINERAL OIL

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Abstract

The rising concern over the existing lubricants have driven the need for biolubricants, extensively anticipated as the most suitable and sustainable lubricating oils. In this study, a biolubricant was synthesized from Marula kernel oil (MKO) via a two-step transesterification process using trimethylolpropane (TMP), in the presence of potassium hydroxide as catalyst. The kernel oil was first extracted then characterized using standard laboratory techniques before being chemically modified to produce Marula kernel oil biolubricant (MKOB). The synthesized biolubricant and its blends with mineral oil were analyzed for their lubricity properties. The results obtained shows that the kinematic viscosity at 40 °C and 100 °C were (9.3 cSt) and (3.3 cSt) respectively, viscosity index was quite high with a value of (207) while the flash and pour point value were (215 °C) and (11 °C) respectively. The lubricity properties of the synthesized biolubricant were comparable with those of the mineral oil (SAE 20) and are also within the range of recognized testing standards such as viscosity grade 32 (ISO-VG-32). From the results obtained, it can be deduced that the blends of the MKO biolubricant with mineral oil (SAE20) in various proportions were proved to have the quality lubrication properties, making them a suitable alternative to mineral oil based lubricant, and could therefore be used to reduce the consumption of mineral oil, which will in turn help to achieve a greener future.

Keywords:
Biolubricant, Mineral Oil, Marula Kernel Oil, Lubricity, Transesterification, Blending

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Introduction

Historically, vegetables oils and animal fats have been a medium to reduce the friction and wear dates back to 1650 B.C. However, the discovery of crude oil resulted in replacement of vegetables oils and animal fats (Pawar *et al*., 2022). Currently, most lubricants are produced from mineral oil; although these lubricants meet technical requirements, their production is narrowed by the availability of oil reserves (Roberto *et al*., 2023), (Monteiro *et al*., 2023). A significant portion of the lubricants consumed worldwide ends up polluting the environment, as a result, many efforts are made to minimize these environmental impacts caused by spillages and evaporation (Mulula *et al*., 2021), (Hussein *et al*., 2023). These high lubricant losses into the environment were behind the development of environmentally friendly biolubricants (Heikal *et al*., 2017) (Ahmed *et al*., 2023). Even though some markets have been explored based on plant oil oriented products, still there are many bright possibilities of expansion in the field of vegetable oils (Salih, 2021).

The term biolubricant has been ascribed to all lubricants derived from biobased raw materials such as plant oils, animal fats, or other environmentally benign hydrocarbons. Biolubricants are intended to be biodegradable and non-toxic to humans and other living organisms, particularly in aquatic environments where the impacts are more detrimental (Oguche *et al*., 2022), (Nogales-delgado *et al*., 2023).

Chemically, VOs are natural oils that comprise triglycerides, esters of glycerol, and three straight-chained fatty acids. Fatty acids from VOs are mostly in the category of C_{12}-C_{24}. These three fatty acids are dependent on feedstocks and provide a vital function in ascertaining the properties of the oil (Buonvino *et al*., 2023). Plant oil-based biolubricants such as (Marula) oil and its derivatives have excellent lubricity and biodegradability properties for which they are being more closely examined as a base stock for lubricants and functional fluids (Salimon *et al*., 2010), (Salih *et al*., 2021). Also, these oils exhibit high viscosity (Rakesh *et al*., 2022). However, plant oils have several disadvantages including instability that limit their applications in biolubricant industries. Plant oil formulations provide many challenges such as low oxidative stability (Musa and Salisu, 2022), and poor low temperature properties ((Nogales-delgado *et al*., 2020).This is due to the presence of the weakest track of double bonds present in the unsaturated fatty acid structure and instable β-hydrogen at glycerol backbone in the oil triacylglycerol molecule structure (Ahmed *et al*., 2023), Demir and Ilker, (2023).

Sclerocarya birrea, popularly known as Marula, is an indigenous deciduous savanna tree that belongs to the Anacardiaceae family. It is widely distributed throughout sub-Saharan Africa, from the tropical, central to Southern Africa (Ejilah *et al*., 2012), (Etim *et al*., 2021). It normally grows up to 18 - 20 metres in height and well adapted to a woodland habitat on a sandy loam soil and is leafless during winter (Nndwammbi *et al*., 2018). The kernels

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oil contains a large proportion of monounsaturated fatty acids and natural antioxidants and oil content up to 56% (Ejilah *et al*., 2012).

Hence, this underutilized seed oil with high level of unsaturated fatty acids could be considered a good precursor for biolubricant production. Figure 1A through D, shows the Marula tree, fruits, nuts and kernel seeds respectively.

The present work, concerns with the production and evaluation of the lubrication properties of a biolubricant from Marula kernel oil using double step transesterification technique and comparison of its lubricity properties with those of the mineral oil.

Figure 1 A through D shows the Marula tree, fruits, nuts and kernel seeds respectively.



A B C D

Figure1. (A): Marula tree, (B): Marula fruits, (C): Marula nuts (D): Marula kernel seeds

MATERIALS AND METHODS

Plant materials

The Marula fruits were collected from surrounding areas located in Yankuzo village of Tsafe Local Government area, Zamfara State in the North-Western Nigeria. The material was authenticated at the herbarium of the department of biological Sciences, Ahmadu Bello University, Zaria, Nigeria. The fruits were manually peeled to expose the pulp (edible part) which was removed to obtain the nut. The nuts were dried under the sun for a period of one week, and subsequently crushed with the aid of hammer to obtain the Marula seed kernels. The kernels were dried under the shade for 2 days, and then grounded to powder using mortar and pestle.

SAE20 mineral oil

The SAE20 mineral oil used in this study is mono-grade engine oil, processed by Oando PLC, Nigeria.

Oil extraction process

The oil content of the kernel seed was extracted using soxhlet extraction technique, based on a study reported by Ezennajiego and Ndive, (2023), In the process, 50 g of the

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powdered kernels were placed inside the thimble of the Soxhlet extractor. Accurately, 150 ml of the n-hexane solvent was poured into round bottom flask of the extraction set up which was placed on a heating mantle. The extraction was performed for one hour at 60 °C. Upon completion of the extraction process, the solvent was recovered using a rotary evaporator. The extracted oil was then placed in an oven at 50 °C for 5 hours so as to obtain a solvent free oil sample. The weight of the oil sample was recorded so as to obtain the percent oil yield of the seed sample. The process was repeated for three different runs.

Esterification of MKO

This was carried out base on the procedure reported by Inuwa *et al*. (2020). This is required to decrease the free fatty acid (FFA) content of the oil as it may lead to high saponification.

Biolubricant Production

The method used in the production of biolubricant from MKO was based on a study by Ezennajiego & Ndive, (2023). This usually involves a two-step transesterification process which was preceded by an esterification process aimed at reducing the FFA value of the oil sample to a value below 1%. The synthetic scheme of the process is described in Fig. 2.

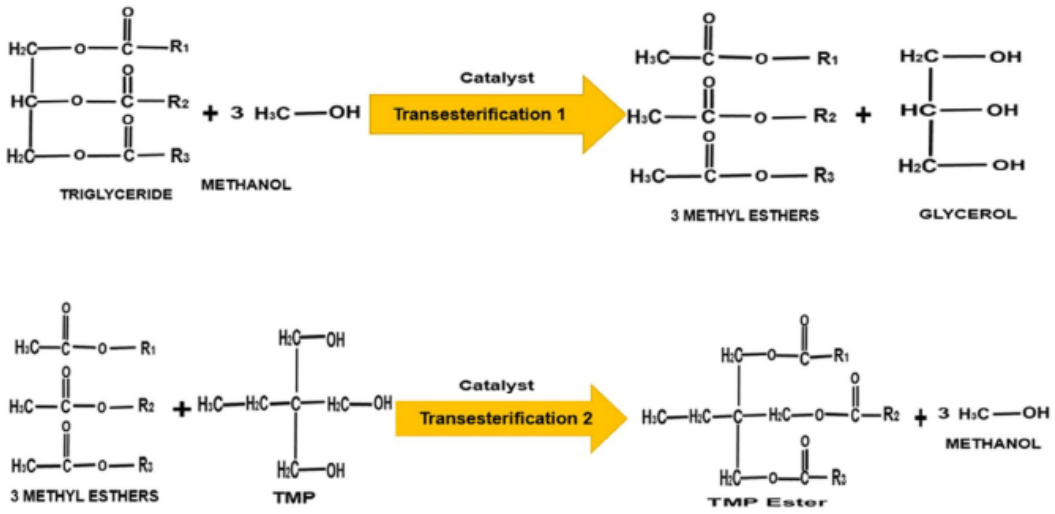

Fig 2: Synthetic scheme for biolubricant production

Table 1: Standard methods employed in characterization of MKO biolubricant

S.N.	Specifications	Methods
1	Acid value	ASTM D664
2	Kinematic viscosity	ASTM D445

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3	Pour point	ASTM D97
4	Cloud point	ASTM D2500
5	Viscosity index	ASTM D2270
6	Density	ASTM D445-12
7	Flash point	ASTM D93
8	Iodine number	AOCS Cd-92 (2017)
9	Saponification number	AOCS Cd 3-25(2017)
10		

Formulation of biolubricant/mineral oil blends

The blends were prepared by mixing 10,20, 30, 40 and 50 wt% biolubricant with 90, 80,70, 60 and 50 wt% mineral oil respectively, using magnetic stirrer for 15 min at 40 °C and 600 rpm (Owuna *et al*., 2019).]. The proportion (v/v %) of each of the components in the blends and their codes are shown in Table 2.

Table 2: Biolubricant/mineral oil proportions in the blends (v/v %)

S/N	Sample's codes	Biolubricant ratio (V/V %)	Mineral oil ratio (V/V %)
1	B100	100	0
2	B50	50	50
3	B40	40	60
4	B30	30	70
5	B20	20	80
6	B10	10	90
7	SAE20	0	100

RESULTS

Table 3: Physicochemical properties of Marula kernel oil

S/N	Specifications	values	ASTM Std.
	Yield (%)	51.32	-
1	Acid value (mgKOH/g)	6.56	0.5 max
2	Free fatty acids (%)	3.28	0.25 max
3	Saponification value (mgKOH/g)	179.81	175-187
4	Density (g/cm^3)	0.898	≤ 0.91
5	Iodine number (mgI_2/100g)	82.32	82-88
6	Pour point (°C)	11	-15 - 10
7	Flash point (°C)	239	≥ 135
8	Kinematic viscosity at 40 °C (cSt)	7.5	1.9-6.0

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9	Kinematic viscosity at 100 ℃ (cSt)	2.9	-
10	Viscosity index	227.8	-
11	colour	Pele yellow	-
12	State at room temperature	Liquid	-

Table 4: Lubricity properties of MKO biolubricant and its blends with mineral oil

S/N	Specifications	BL100	BL50	BL40	BL30	BL20	BL10	SAE20	ISO-VG32
1	Density (g/cm^3)	0.913	0.20	0.907	0.891	0.897	0.889	0.911	≤ 0.91
2	Pour point (℃)	11	7	3	0	0	-10	-13	-6
3	Flash point (℃)	215	201	201	196	191	188	188	204
4	KV at 40 ℃ (cSt)	9.3	19.7	23.8	27.3	24.6	32.1	38.5	> 28.8
5	KV at 100 ℃ (cSt)	3.3	7.2	9.0	11.1	6.4	8.0	14.2	> 4.1
6	Viscosity index	207	197	190.6	186.8	187.8	168.1	175.8	> 90

KV = Kinematic viscosity, FFA = Free fatty acids

DISCUSSION

Physiochemical Properties of Extracted Oil from Marula kernel Oil

The result of the physicochemical properties of the MKO is presented in Table 3. The result of the analysis carried out in this study showed a percentage oil yield of (51.32%). The obtained yield was close to the yield (56%) reported for marula kernel oil by Ejilah *et al*. (2012) and lower than the value (59%) by Gandure & Ketlogetswe.(2011). However, the yield obtained from the current study is higher than result of Eticha & Kiros, (2022) who reported (41.57%). This variation in the oil yield may be as a result of environmental factors that influence the growth and development of the Marula seed kernel.

The acid value and percentage FFA obtained are 6.96 mgKOH/g and 3.28% respectively. These values are slightly lower in contrast to those reported in the literature for MKO (7.52 mgKOH/g and 3.8%) by Eticha & Kiros, (2022) and also respectively higher than 4.348 mgKOH/g and 2.174% reported by Etim *et a*l. (2021). As noted, the higher the acid value of oil, the lower its storage quality and vice-versa. Free fatty acid of oil determines the suitability of the oil for industrial usage (Taiwo *et al*., 2020). This implies that the free fatty acid value of this oil (3.28%) is higher than the 1% specified limit for transesterification, hence the oil needs to be esterified. Saponification value of the extracted oil was found to be 179.81 mgKOH /g. The value is lower than that of MKO (229.015 mgKOH/g) reported in literature by Etim *et a*l. (2021). However, similar saponification number for MKO (184 mgKOH/g) was reported in the literature by Anietie *et al*. (2021). The high saponification

value of this oil is an indication of the less long chain fatty acids (found in the oil) which have a relatively higher number of carboxylic functional groups per unit mass of the oil contrasted with short chain fatty acids.

Iodine value is the proportion of the extent of unsaturated acids or fat present in vegetable oil. The iodine value of MKO from this study was determined to be 82.32 gI_2/100g. This value was slightly lower than 84.40mgI_2/100g reported for MKO by Anietie *et al*.(2021) and 87.94 g/100g reported for Castor seed oil by Omotehinse *et al*.(2019).However, the iodine value obtained from this study was also observed to be high when compared to 58.9 g/100g reported for Almond seed oil by (Akinola and Adeyinka, (2022).

Lubricity properties of MKO biolubricant and its blends with mineral oil

The MKO biolubricant (100%), mineral oil (SAE20) and their blends (BL 50, BL40, BL30, BL20 and BL10) were tested for their lubrication properties. Table 4 shows results for all the specifications tested. The kinematic viscosity of all the samples at 100℃ greatly drop compared to samples at temperature of 40℃. This might be due to the low boiling point of marula kernel oil. It can also be attributed to the fact that the heat gives the energy that breaks the reaction between the oil itself, thus resulting in lower concentration compared to samples at temperature 40℃.

The density of the biolubricant (0.913 mg/cm^3) was observed to increase when compared to the raw oil (0.898 g/cm^3 b). This may be attributed to increase in molecular complexity brought in by TMP backbone or it may be due to the presence of impurity as suggested by Alang *(et al*. (2018). Comparatively SAE20 mineral oil has a density of (0.911 g/cm^3). Furthermore; the density of all the blended samples slightly improved with respect to concentration of SAE20 oil in the blends. The densities of BL100 and BL50 are greater than (0.91 g/cm^3) (the maximum required by the ASTM standard).

Density is one of the principal fuel properties used to estimate the quantity of fuel injected by the injection systems to provide proper combustion. The density of the fuels depends on several factors like feedstock used, conversion method and methyl ester profile (Mulula & Manoka, 2021).

The kinematic viscosity of the synthesized biolubricant has significantly improved when compared to the raw marula kernel oil. The improvement in the viscosity may be due to a slight branching and mass increase in the ester structure. Decrease in viscosity was also observed due to the temperature change from 40 ℃ to 100 ℃.

The flash point value of the produced biolubricant was (215 ℃), which is lower when compared to the raw MKO with flash point at (239 ℃), this may be due to series of reactions it undergone through esterification and transesterification. Comparatively, SAE

20 oil is having very lesser flash point (188 °C) than that of the produced MKO biolubricant and thus the blends of the two oils give moderately better flash point.

The viscosity index represents how fast the viscosity change with temperature. As seen in Table 2, viscosity index of raw MKO was 227.8. The values decreased to 207 after the transesterification processes (Table 3). However, after blending of MKOB and SAE20 engine oil, a slight increment was observed with respect to viscosity index of SAE20 which increase progressively with increase in the percentage of MKOB in the blends. Among the blended samples, BL10 produced the lowest viscosity index, and the highest viscosity index was obtained from BL50. This indicates that the BL50 has less viscosity change when temperature increased thus a better resistance to thinning.

A significant change was observed in pour point of the biolubricant compared to crude MKO sample, decreasing the value from 20°C in MKO to 11°C in MKO biolubricant, while SAE20 oil is having the lowest pour point (-10°C), which may be attributed to the additive Package incorporated in to it, which may include pour point depressants. Moreover, a significant improvement was achieved with respect to the pour point of the biolubricant after blending with SAE20 mineral oil in different proportions, with BL10 having the lowest value (-13°C), followed by BL20 and BL30 with both their pour points at 0°C, BL40 with power point at 5°C and BL50 displays the highest pour point value at 8°C. The pour point of all the blended samples improved depending on the quantity of mineral oil present in the blends.

Conclusion

Marula kernel oil showed good potential as base stock for biolubricant formulation. Despite the high pour point, other lubricating properties such as Kinematic viscosity at 100 °C, viscosity index, acid value and density are comparable to SAE-20 engine oil and are within the ASTM standard limit.

The synthesized biolubricant from Marula kernel oil have higher viscosity index compared to the mineral oil based lubricant, therefore viscosity index improvers are not required, thus reducing the cost of production.

The blends of Marula kernel oil biolubricant with mineral oil in various proportions have qualities that make them suitable for any application where SAE-20 based mineral oil is applicable and could therefore be used to reduce the consumption of mineral and will thus help us to achieve greener future as they don't pollute the environment.

Conflict of Interest

The authors report no conflicts of interest. The authors alone are responsible for the content and writing of this article.

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