

Synthesis, Characterization and Properties of Trimethylolpropane Triesters from Coconut (*Cocos nucifera*) Methyl Esters.

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Abstract

In this study, the acid-alkaline transesterification of refined coconut seed oil (RCOSO) to fatty acid methyl ester was followed by the production of a trimethylolpropane-based thermosensitive biolubricant using potassium hydroxide, and its physicochemical characteristics were evaluated. The American Standard Test for Materials (ASTM) was employed to ascertain the biolubricant's pour point and index of viscosity, which were found to be $-4\text{ }^{\circ}\text{C}$ and 283.75, respectively. The opposite connection between lubricant viscosity and temperature was shown by the measured viscosities at varied transesterification to be transformed into biodiesel. Following this, a biolubricant was created by further transesterified temperature. The ester group is verified by comparing the Fourier Transfer Infra-red (FTIR) spectra of the biodiesel and the biolubricant between absorption bands ($1755.07\text{-}1737.21\text{ cm}^{-1}$). Gas chromatographic data shows that sweet almond oil contains 71.725 % unsaturated fatty acids for use as a biolubricant, 53.478 % more unsaturated fatty acids than saturated fatty acids, and 9.52 g iodine per 100 g of oil sample more than unsaturated glycerides. At 31.44 and 45.93 %, respectively, linoleic acid made up the majority among the fatty acids in the oil and synthetic biolubricant. In addition, palmitic, linoleic, and oleic acids were present in the biolubricant and coconut oil. The biolubricant's characterization results correlated highly with the ISO VG-32 standards, suggesting that it has the capacity to serve as light gear oil.

Keywords: Biolubricant, biodiesel, transesterification, vegetable oil, viscosity.

Introduction

The fast-growing energy demand resulting from an ever-increasing commercial industry, as well as the expansion of energy demands are all significantly reliant on petroleum-based energy. On one hand, there are products that are restricted, and on the other, there is a population. Industrialization is growing at a rapid pace, putting further strain on energy supplies. Products made from petroleum are fuels and lubricants are the primary sources of energy for these industries. All of these petroleum-based lubricants are hazardous and non-renewable, and they constitute the primary source of environmental pollution¹. We can expand the

number of potential sources to include a renewable and environmentally friendly alternative to petroleum-based products.

The right lubricant can help you boost the durability of mechanical parts, their usability, and their dependability. Lubricants are used in almost every industry that requires manual or automatic machining procedures in either an open or closed system application. There are benefits to using lubricants, but there are also drawbacks. These lubricants contribute to environmental pollution through lubricant loss due to evaporation and spills every year, which is significant enough that it

cannot be avoided². The discipline of bio-lubricants is among the most crucial that offers easy, toxic-free, and renewable products with the potential to serve as a substitute product to save the environment while also meeting the demands of modern industry to minimize friction and wear to increase the flexibility of components that are in close proximity to one another.

To overcome these environmental losses, if we are to survive, it is necessary to discover a practical solution for these lubricants that is biodegradable, non-harmful to the environment, and recyclable, obtained from renewable and environmentally friendly sources. Vegetable oils, for example, have suitable lubrication properties, such as a high viscosity index, corrosion resistance, oxidation stability, and so on. The molecule of triglycerides is practically all vegetable oils have the same basic makeup structure, and poly-saturated fats are all types of fat. Natural fatty acids in plants are classed as fatty acids. Vegetable oils are prepared to make them suitable for use in engines and equivalent to traditional lubricants³.

Plants and animals serve as feedstocks in the production of bio-lubricants, while vegetable oils and other sources are also used, as well as a variety of other agro-residues⁴. The viscosity index of seed oils can be quite high^{5,6}. Vegetable oils have an elevated flash point when compared to mineral oils, which is considered a significant feature.

Materials and Methods

Ingredients and Reagents Collection

We bought coconuts from Markets in Kwara State, Nigeria. Chemicals and reagents such as sodium chloride, sodium hydroxide, potassium hydroxide, chloroform, sodium thiosulphate, anhydrous methanol, hydrochloric acid, sulphuric acid, trimethylolpropane (TMP), glacial acetic acid, wj's reagent, isopropyl alcohol (IPA), phenolphthalein, activated charcoal, and sodium hydroxide were obtained from Sigma Aldrich. The instruments used were GCMS-QP2010 plus, Pour Point Analyzer (SETA – LEC Cloud and Pour Point Refrigerator), FTIR Spectrophotometer (FTIR-8400S, Shimadzu, Japan), Brookfield® (Synchro-lectric Viscometer: RVT) and Multiflash Flash Point Tester (Model: 34000), and Oil Bath (HAAKE FISON: DC3000).

Extraction from Coconut Seeds and Refinement

The husk of the coconut was removed and then it was grated to a smaller size. The coconut was

Biodegradable vegetable oils are non-toxic and sources of energy that are lessen reliance on fossil fuels and petroleum fuels from other countries are vegetable oils that provide a number of benefits. Many vegetable oils have drawbacks, such as limited oxidative stability in their natural state. They have enough oxidative stability to be used as lubricants⁷.

Due to its qualities coconut can be utilized as a lubricant and transformer oil. Due to its high viscosity, it is used as a biodiesel feedstock. Coconut trees are uncommon in dry areas because without regular irrigation, plants cannot grow. Most of these trees can be found in tropical regions.

Because of their light weight, buoyancy, and strong water resistance, coconut tree fruits have evolved to travel great distances on sea currents^{8,9}. Coconut oil doesn't oxidize readily since it contains a lot of saturated fatty acids of 91%, which makes it. Many people in Southern India use coconut oil to lubricate their scooters and rickshaws. There is evidence that using this coconut oil can increase gas mileage, engine power, and overall performance. Furthermore, when burned, coconut oil emits less smoke^{10,11}. This study aims to produce bio-lubricant utilizing a two-stage transesterification reaction from the coconut seed oil, investigate temperature effects, and ascertain the synthetic bio lubricant physical and chemical characteristics and compare them to the ISO VG standards.

soaked and the coconut milk was strained using a sieve to get as much of the shredding out as possible. The milk was frozen for 24 hours. The frozen portion of the oil was skimmed and boiled to evaporate the water content of the milk, leaving the crude coconut seed oil (COSO).

Conversion to Biodiesel

Alkali catalysis is a good method for producing the coconut seed oil methyl esters (CSMEs) based on the quantities of water and free fatty acid (FFA) in the outgoing coconut oil. A two-stage process with intermediate methanol addition was used, similar to Mustapha *et al.*^{12,13} to increase methanolysis yields. Table 1 provides a summary of the operating settings that were used for this study. The stirring parameters for the initial reaction phase and catalyst were permanently set to 500 rpm and NaOH, respectively. This process for methanolysis fulfilled the prior description. However, we were able to

meet these limits while achieving the other important methanolysis reaction parameters, including temperature. High yields and low cost biodiesel were created, depending on the ratio of methanol to oil, the mass concentration of the catalyst, and its mass fraction. It was also examined in this context whether crude glycerol waste from earlier biodiesel production cycles could be used again.

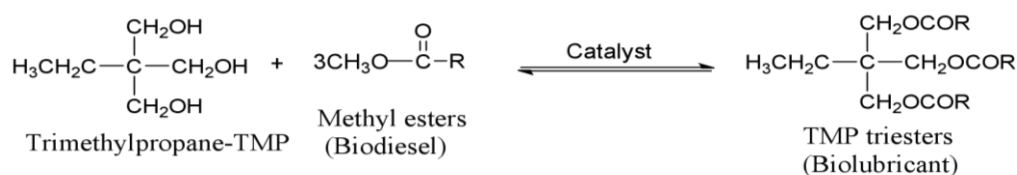
Each methanolysis process was performed in a flask with a spherical bottom with three necks that also had a magnetic stir bar, a sample outlet, a reflux condenser, and a thermometer. The 100 g of COSO was weighed and heated to the necessary temperature in the dried reaction flask. The experiment's specified amounts of methanol and sodium hydroxide (NaOH) were combined simultaneously in another flask, dissolved in 128.56 mL of methanol, and heated to the experiment's temperature prior to being included in the COSO; it is taken into consideration to have started at this point. Phase separation was then applied to the methanolysis product after it had been vigorously agitated and given the time needed for the reaction to occur at room temperature. A sufficient value for the reaction time was chosen to get the highest conversion in 50 min. Methanolysis was observed off-line at a particular sample frequency using gas chromatography-mass spectrometry (GCMS-QP2010). By using GC-mass spectrometry tools and conducting sample analyses, the COSMEs were initially found^{14,15}. The methanolysis byproduct was then put through a separating funnel following the reaction. The upper phase, which was mostly consisting of COSMEs with some residual glycerides and after the bottom phase, which included the injected and liberated glycerol with entrained COSMEs, was removed initially, some glycerol was then removed.

The leftover methanol in each phase was then evaporated at 50°C in order to further separate the glycerol from the COSMEs¹⁵⁻¹⁷. The yield of the methanolysis and separation fractions was then

calculated from the recovered COSME fractions, and the remaining fraction was used to produce biolubricants.

Synthesis of a Biolubricant

The conversion of all methyl biodiesel into biolubricant via transesterification reactions took place in a flask with a circular bottom and three necks, a temperature gauge that regulates temperature, a magnetic stirrer device and a sampling outlet. With a second thermometer on top and a Liebig condenser attached to it on the side, it was used in place of the reflux condenser. This was then linked to two flasks for collecting distilled methanol, a vacuum take-off adapter for coupling to the vacuum pump, and a three-way valve. For bulk reasons, the Liebig condenser was cooled using a recirculating silicon oil bath at 0 °C, whereas the two collection flasks were chilled with an ice bath. A dried reaction flask was filled with the refined COSMEs of 20 mL, which were then weighed into the flask and heated to the required temperature. In a separate flask, 5 mL of methanol was combined with 0.3 g of NaOH to create the catalyst's active species. This mixture was then heated to the experiment's temperature previously to being added to the COSMEs. The reactive distillation unit was then connected to the vacuum pump by switching the three-way valve. A dose of 1.0 g of trimethylolpropane (TMP) was added to the mixture. The reaction was monitored offline by GC-MS at a predetermined sampling frequency for the duration of the experiment, which was carried out at 70 °C for two hours following an initial sampling with a response time of four minutes at 10-min intervals. At this point, the response was said to have begun at time zero. Similar to COSMEs, the coconut biolubricant (COBL) was initially identified by GC-MS and FTIR, and details relevant to the apparatus and sample analysis are provided in Fig.1



Note: R = alkyl group

Figure 1. Synthesis of biolubricant

COSMEs' Purification and Biolubricant

Batch bubble-washing was used to refine the crude COBL, first with a 4 wt. % solution of citric acid and subsequently with distilled water at 35 °C and 1 atm. Washing solution and esters were put in a separating funnel, with the volume proportions being 1/3 and 2/3, respectively. Purification batches were conducted until the aqueous phase was clear.

Determination of Temperature on Biolubricant Viscosity

The viscosity of the synthetic biolubricant was determined at 30, 40, 60, 80, and 100 °C. The

sample was first transferred to a 100 mL beaker. The biolubricant's temperature was raised to the required amount by heating on a heating mantle while swirling continuously. The sample's degree of viscosity was ascertained by turning on the Brookfield® (Synchro-lectric Viscometer: RVT) and letting it run until a stable reading was achieved.

Results and Discussion

Coconut Oil's Attributes and Evaluation

Table 2 displays the values of the crude oil's characteristics. The physical-chemical characteristics of unrefined coconut oils were

comparable to the values recommended¹⁸⁻²² for rubber and linseed oils. To avoid excessive oil saponification, the FFA level in the oil must be reduced to 1% or less via esterification.

Table 2. The physical-chemical characteristics of unrefined crude and refined coconut oil

Physicochemical Properties	Coconut Oil		Ximenea americana seed oil ²⁰	Jatropha oil ²²	ASTM ²¹
	Crude Coconut Oil (COSO)	Refined Coconut Oil (RCOSO)			
Yield (%)	13.7%	72%	-	-	46-55
Colour	Gold	Colorless	Brown	Yellow	Amber
Specific gravity(g cm ⁻³ , 30 °C)	0.940	0.94	0.93	0.98	0.957–0.968
Density at 40°C (g/cm ³)	0.937	0.937			0.88
Viscosity (cp, 40 °C)	3.73	4.3		-	1.9-6.0
Acid value, (mg/KOH)	2.24	5.6 ± 0.019	19.18	1.0	0.4 – 4.0
Saponification,(mg/KOH)	230		181.14	185.0	175 – 187
Iodine value, (I ₂ /100g)	6.0	9.52 ± 0.01	136.2	188.0	82 – 88
Peroxide value, (Meq O ₂ /kg)	3.89		-	-	10
%FFA	1.13	2.82	9.54	0.5	0.3 – 1.0
Average molecular weight (gmol ⁻¹)	246.35		-	-	

Biodiesel and Biolubricant Main Performance Properties

Viscosity is a key component of lubricating oil, as demonstrated in Table 3 evaluates a material's flow resistance at a given temperature. The biolubricant's viscosity was 31.46 cSt at 40 °C and 8.88 cSt at 100°C, respectively. This figure exceeds the ISO-specified limits for the viscosity grade of light gear oil, which are 28.8 and 4.1 cSt, respectively. Previously, palm oil-based trimethylolpropane (TMP) esters were synthesized, and the effects of composition on pour points, viscosity, and viscosity index of the high oleic content palm oil-based TMP esters were also evaluated. Compared to coconut

oil-based TMP esters, there were variations in the viscosities and viscosity index values of high-oleic palm oil-based TMP, in the region of 50 cSt and 199, respectively²³. The created biolubricant seems to have a greater viscosity index (VI), suggesting that it works well over a wide temperature range. It is conceivable to connect the attraction that exists between liquid molecules with the temperature-dependent reduction in viscosity. The viscosity index, which is a dimensionless quantity, shows how kinematic viscosity and temperature interact with a product. A viscosity index of 130 is ideal for use in a variety of engines because viscosity lowers as the index rises and vice versa.

Table 3. Properties of Coconut biodiesel and biolubricant

Properties	Biodiesel	Coconut Lube	Jathropa Lube ²²	Sesame Lube ²⁴	ISO Viscosity Grade ³		
					46	32	68
Flash point (°C)	130	200	-	-	220	204	250
Pour point(°C)	-8	-6	-7	-12	-10	-6	< -10
Viscosity 40 °C, (cSt)	35.02	31.46	55.17	35.43	≥41.4	≤28.8	>61.4
Viscosity 100 °C, (cSt)	10.00	8.88	10.96	7.93	≥4.1	≥4.1	>4.10
ASTM D2270 Viscosity index	291.1	283.75	195.22	206	≥90	≥90	>198

The number that was discovered is appropriate for ISO VG220 and ISO VG46 light gear oil. Additionally, as demonstrated in Table 3, it has a greater viscosity index than biolubricants made from other vegetable oils, like sesame and jatropha. The capacity of a lubricant to be used at low temperatures is determined by its pour point. The coconut-based biolubricant has a pour point of -6 °C. This value fell under the 6 °C gear oil temperature limit of ISO VG 32 standards. This outcome illustrates that coconut biolubricant may function at lower temperatures without clogging the filter. To determine a lubricant's flammability, look at its flash point. The coconut oil based biolubricant has flash point value (200 °C) that exceeds the equivalent biodiesel's 130 °C value by a significant margin²⁵. The chemical alteration of basic oil is what causes this raised flash point. The final temperature range, which falls between 204

and 250 oC, is well within the ISO VG 68 and ISO VG 32 standards. The results are almost in line with the claimed 262 °C for the neem oil biolubricant, as reported in Table 3. Coconut oil-based lubricants were particularly safe to use and store because they weren't manufactured with methanol.

Temperature Effects on the Viscosity of Coconut Oil

The various viscosity grades that ISO demands are listed in Table 3. The main grading criteria are the pour point and viscosity index. At 30, 40, 60, 80, and 100°C, the measured viscosities were 41.69, 31.46, 22.30, 11.60, and 8.88cSt, respectively. As seen in Fig. 2, the opposite connection between lubricant viscosity and temperature was observed. The estimated viscosity index was employed to ascertain the viscosity grade

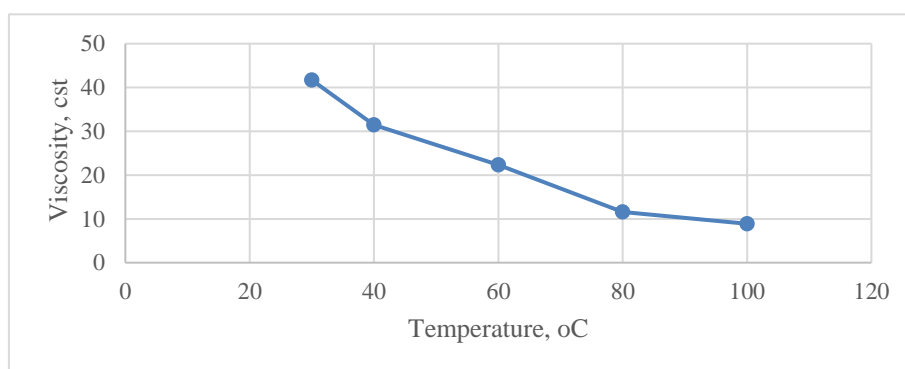


Figure 2. Illustrates the impact of temperature the COBL viscosity.

As temperature rises, a cohesive force that attracts the lubricant's molecules to one another weakens, reducing the lubricant's viscosity^{9, 26}. The minor decrease in viscosity with rising temperature may be caused by the lubricant's extraordinarily high viscosity index. This variation also reveals the amazing stability of the synthetic biolubricant over

such a large temperature range, which makes it appropriate for use as motor oil²⁷

Analyses Using Fourier Transform Infrared (FTIR)

In the transmittance versus wave number plot of the infrared radiation spectrum, the bond vibration energy varies horizontally. The peak graphs in Fig.

3 demonstrate low transmittance and strong absorption with the horizontal wave number rises as we turn to the left. Peak-less areas show that photons are not absorbed at that frequency, indicating that the molecule doesn't have that particular bond there. Findings from the FTIR analysis of the chemical composites RCOSO in Fig.

3a, COSME in Fig. 3b, and COBL in Fig. 3c are respectively shown. The crucial IR peak locations for RCOSO and COSME were revealed by the FTIR spectroscopy data, demonstrating that chemical changes were causing the fingerprints to appear between 500–4000 cm^{-1} .

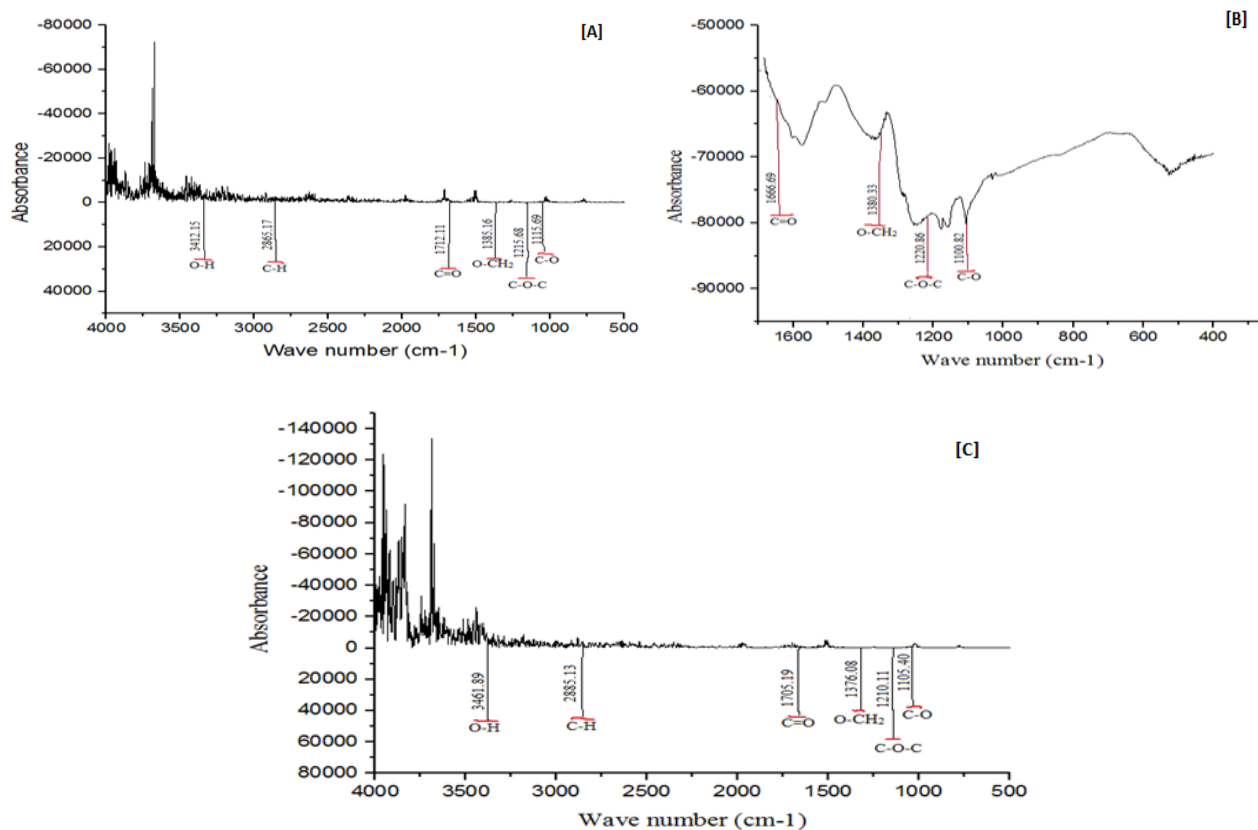


Figure 3. FTIR spectra of A: RCOSO; B: COSME; C: COBL

In 1385.16 cm^{-1} spectral, glycerol group O-CH₂ of mono, di, and triglycerides were found in Fig. 3a¹². The 3600-3200 cm^{-1} was the alcohol O-H stretching vibration at 3412.15 cm^{-1} were observed. The C-H: spectral was found at 2865.17 cm^{-1} and in a comparable FTIR observation¹², Mustapha *et. al.*¹² determined that the C-H was an aliphatic stretching vibration of 2913.91-2882.17 cm^{-1} . The FTIR bands of C=O, C-O-C and C-O were observed at 1712.11, 1215.68 and 1115.69 cm^{-1} , respectively¹². The region of the IR spectrum in COSME of 1666.69 cm^{-1} indicates the existence of an ester carbonyl group with associated C-O bend of 1100.82 cm^{-1} and C-O-C at 1220.86 cm^{-1} . The COBL sample revealed a novel ester linkage bond (C-O-C). Absorption band of C=O at 1705.19 cm^{-1} is visible in the FTIR spectra of COBL and these absorption bands were

used to show the presence of ester group in COBL with the C-O (1105.40 cm^{-1}) and C-O-C (1210.11 cm^{-1}) stretching vibrations in the ester. Furthermore, the hydroxide group peak in RCOSO was measured at 3412.15 cm^{-1} as illustrated in Table 3a was disregarded in COSME in Table 3b¹². This shows that the transesterification to fatty methyl esters were almost completed. The COBL was formed in Table 3c.

Evaluating Free Fatty Acids

To measure free fatty acids, gas chromatography mass spectrometry (GCMS) analyses were utilized. Coconut oil was chosen for GCMS analysis for fatty acid profile evaluation due to the high lubricating capabilities discovered in synthetic biolubricant research. In Table 4, coconut oil's fatty acid content

before it was esterified into a biolubricant is displayed through GC-MS analysis.

Table 4. Fatty acid and esters content in coconut oil and biolubricant in wt. %

Refined Coconut oil (RCOSO)			Coconut biolubricant (COBL)		
Fatty acid	Saturation	Area (wt. %)	Fatty esters	Saturation	Area (wt. %)
Glutamic acid	C ₅ H ₉ NO ₄	6.284	Methyl octanoate	C ₉ H ₃₈ O ₂	5.298
Luaric acid	C ₁₂ H ₂₄ O ₂	9.800	Methyl decanoate	C ₁₁ H ₂₂ O ₂	8.024
Dodecanoic acid dimethyl ester	C ₁₄ H ₂₆ O ₄	3.753	Luaric acid methyl ester	C ₁₃ H ₂₆ O ₂	23.982
Tridecanoic acid	C ₁₃ H ₂₆ O ₂	5.123	Myristic acid methyl ester	C ₁₅ H ₃₀ O ₂	16.411
Brassylic acid	C ₁₃ H ₂₄ O ₄	45.812	Palmitic acid methyl ester	C ₁₇ H ₃₄ O ₂	15.812
Oleic acid	C ₁₈ H ₃₄ O ₂	29.228	Methyl eladiate	C ₁₉ H ₃₆ O ₂	16.195
			Methyl stearate	C ₁₉ H ₃₈ O ₂	7.141
			(E)-pent-2-en-3-yl acetate	C ₇ H ₁₂ O ₂	1.570
			Others		5.566
Total fatty acids		93.716	Total Triester		94.434
Total unsaturated FFA		78.793	Total unsaturated FA		17.765

The GC-MS data shows that RCOSO includes 17.76% more unsaturated fatty acids than biolubricant of 78.193%, including dodecanoic acid dimethyl ester, brassylic acid, and oleic acid. The oil has higher iodine content because unsaturated glycerides have a reduced capacity for iodine absorption of 9.52 g of iodine per 100g of sample. This oil is appropriate for the creation of

biolubricants because most vegetable oils do not coagulate at room temperature and have a high unsaturation level²⁷. The fatty acid profiles of the RCOSO and COBL, which had percentage contents of 78.793% and 17.765%, respectively, are shown in Figs. 4a and 4b. Compositional differences were seen in comparable RCOSO and COBL variants.

Conclusion

Positive outcomes were obtained using the two pathways that were explored in this work for using COSO as biolubricant (COBLs) or an energy carrier (COSMEs) via conversion of COSMEs. The alkali technique was chosen even though it is based on homogeneous catalysis for the conversion of COSO into COSMEs, leading to the intriguing possibility of the inexpensive synthesis of a 100% renewable biodiesel. First and foremost, this alternative's biodiesel key characteristics are its straightforward operating parameters of 60 °C, atmospheric pressure, a molar ratio of 1:6 between oil and methanol, at 50-minute runtime.

Furthermore, an acceptable biodiesel content of 96.5 wt. % was achieved, and the resulting COSMEs met the fundamental biodiesel

requirements of density, color, viscosity, acid value, flash, cloud and pour points. However, eliminating unwanted species through refinement processes from the departing COSO before methanolysis was useful in order to ensure the resulting biodiesel complies with the EN 14214 standard. Reactive distillation was chosen with optimized operating conditions of 0.3 wt.% of KOH, at oil to COSMEs molar ratio, at 60°C for 120 min allowed for the production of COBL through transesterification of purified COSMEs, which resulted in a high conversion of COSMEs to COBL content of 74.8 wt.%. The lubricant's viscosity-temperature characteristics were remarkably consistent with the specifications for automobile light gear oil.

Authors' Declaration

- Conflicts of Interest: None.

- We hereby confirm that all the Figures and Tables in the manuscript are ours. Besides, the Figures and Images, that are not ours, have been given the permission for re-publication attached with the manuscript.

- Ethical Clearance: The project was approved by the local ethical committee in University of Kwara State University.

Authors' Contribution Statement

M. A. O. conceptualization, methodology design and project leadership. R. S. O. data collection and refinement of crude vegetable oil. R. M. T. data validation and curation. A. S. K. sample

preparation. B. F. P. data collection and sample physicochemical analysis. A. Y. T. biolubricant synthesis. O. D. FTIR data analysis and all authors took part in manuscript writings and revisions.

References

1. Narayana Sarma R, Vinu R. Current status and future prospects of biolubricants: Properties and applications. *Lubricants* 2022;10(4):70. <http://dx.doi.org/10.3390/lubricants10040070>
2. Dong L, Li C, Zhou F, Bai X, Gao W, Duan Z, et al. Temperature of the 45 steel in the minimum quantity lubricant milling with different biolubricants. *Int J Adv Manuf Technol.* 2021;113(9–10):2779–90. <http://dx.doi.org/10.1007/s00170-021-06708-0>
3. Hua J, Shi Y. Non-corrosive green lubricant with dissolved lignin in ionic liquids behave as ideal lubricants for steel-DLC applications. *Front Chem .* 2019;7:857. <http://dx.doi.org/10.3389/fchem.2019.00857>
4. Singh R. Progress of Environment Friendly Cutting Fluids/Solid Lubricants in Turning-A Review. *MaterToday Proc* 2021. 37:3577–80. <https://doi.org/10.1016/j.matpr.2020.09.585>
5. Zhang Y, Li HN, Li C, Huang C, Ali HM, Xu X, et al. Nano-enhanced biolubricant in sustainable manufacturing: From processability to mechanisms. *Friction.* 2022;10(6):803–41. <http://dx.doi.org/10.1007/s40544-021-0536-y>
6. Singh Y, Kumar Singh N, Sharma A, Singla A, Singh D, Abd Rahim E. Effect of ZnO nanoparticles concentration as additives to the epoxidized Euphorbia Lathyris oil and their tribological characterization. *Fuel (Lond).* 2021; 285(119148):119148. <http://dx.doi.org/10.1016/j.fuel.2020.119148>
7. Rahim SANM, Lee CS, Abnisa F, Aroua MK, Daud WAW, Cognet P, et al. A review of recent developments on kinetics parameters for glycerol electrochemical conversion – A by-product of biodiesel. *Sci Total Environ.* 2020;705(135137):135137. <http://dx.doi.org/10.1016/j.scitotenv.2019.135137>
8. Gautam R, Vinu R. Reaction engineering and kinetics of algae conversion to biofuels and chemicals via pyrolysis and hydrothermal liquefaction. *React Chem Eng.* 2020;5(8):1320–73. <http://dx.doi.org/10.1039/d0re00084a>
9. Sarma N, Vinu R. Current status and future prospects of biolubricants: Properties and applications. *Lubricants.* 2022;10. <https://doi.org/10.3390/lubricants10040070>
10. Al Hilfy AAAH, Kathiar SA, Al Shammari HI. Effects of castor oil nanoemulsion extracted by hexane on the fourth larval stage of *Culex quinquefasciatus* from Al hawizeh marsh/Iraq, and non-targeted organism. *Baghdad Sci J.* 2022 [cited 2023 Jan 22];19(6(Suppl.)):1512. <https://bsj.uobaghdad.edu.iq/index.php/BSJ/article/view/7451>
11. Cecilia JA, Ballesteros Plata D, Alves Saboya RM, Tavares de Luna FM, Cavalcante CL Jr, Rodríguez-Castellón E. An overview of the biolubricant production process: Challenges and future perspectives. *Processes (Basel).* 2020;8(3):257. <http://dx.doi.org/10.3390/pr8030257>
12. Mustapha AO, Usman KA, Zakariyah TA, Adekoya OM, Afolabi YT, Oladele J, et al. Optimization of process parameters for biodiesel production from three indigenous vegetable oils. *Ibn AL- Haitham J Pure Appl Sci.* 2022;35(3):98–109. <http://dx.doi.org/10.30526/35.3.2810>
13. Mustapha AO, Adepoju RA, Ajiboye RY, Afolabi YT, Azeez SO, Ajiboye AA. Improvement of fuel properties and fatty acid composition of biodiesel from waste cooking oil after refining processes. *Int J of Res and Sci Innov.* 2021;08(04):80–7. <http://dx.doi.org/10.51244/ijrsi.2021.8405>
14. Suriapparao DV, Vinu R, Shukla A, Haldar S. Effective deoxygenation for the production of liquid biofuels via microwave assisted co-pyrolysis of agro residues and waste plastics combined with catalytic upgradation. *Bioresour Technol.* 2020;302(122775):122775. <http://dx.doi.org/10.1016/j.biortech.2020.122775>
15. Nitiëma-Yefanova S, Tschamber V, Richard R, Thiebaud-Roux S, Bouyssiere B, Bonzi-Coulibaly

- YL, et al. Ethyl biodiesels derived from non-edible oils within the biorefinery concept – Pilot scale production & engine emissions. *Renew Energy*. 2017;109:634–45.
<http://dx.doi.org/10.1016/j.renene.2017.03.058>
16. Zhou W, Boocock DGB. Phase distributions of alcohol, glycerol, and catalyst in the transesterification of soybean oil. *J Am Oil Chem Soc*. 2006;83(12):1047–52.
<http://dx.doi.org/10.1007/s11746-006-5161-4>
 17. Egbuna SO, Nwachukwu UJ, Agu CM, Asadu CO, Okolo B. Production of biolubricant samples from palm kernel oil using different chemical modification approaches. *J Eng Rep*. 2021;3(11).
<http://dx.doi.org/10.1002/eng2.12422>
 18. Alang MB, Ndikontar MK, Sani YM, Ndifon PT. Synthesis and characterisation of a biolubricant from Cameroon palm kernel seed oil using a locally produced base catalyst from plantain peelings. *Green Sustain Chem*. 2018;08(03):275–87.
<http://dx.doi.org/10.4236/gsc.2018.83018>
 19. Perera M, Yan J, Xu L, Yang M, Yan Y. Bioprocess development for biolubricant production using non-edible oils, agro-industrial byproducts and wastes. *J Clean Prod*. 2022;357(131956):131956.
<http://dx.doi.org/10.1016/j.jclepro.2022.131956>
 20. Hamad AA, Alamer KH, Alrabie HS. The Accumulation Risk of Heavy Metals in Vegetables which Grown in Contaminated Soil. *Baghdad Sci J*. 2021;18(3):471–9.21.
<https://doi.org/10.21123/bsj.2021.18.3.0471>
 21. Testing Materials (ASTM). *ASTM Standards Methods*: ASTM pub; Philadelphia. 2003
 22. Bilal S, Nuhu M, Kasim SA. Production of biolubricant from *Jatropha curcas* seed oil. *J Chem Eng Mater Sci*. 2013;4:72–9.
<https://doi.org/10.5897/JCEMS2013.0164>
 23. Yunus R, Fakhru'l-Razi A, Ooi TL, Omar R, Idris A. Synthesis of palm oil based trimethylolpropane esters with improved pour points. *Ind Eng Chem Res*. 2005;44(22):8178–83.
<http://dx.doi.org/10.1021/ie050530+>
 24. Afifah AN, Syahrullail S, Wan Azlee NI, Rohah AM. Synthesis and tribological studies of epoxidized palm stearin methyl ester as a green lubricant. *J Clean Prod*. 2021;280(124320):124320.
<http://dx.doi.org/10.1016/j.jclepro.2020.124320>
 25. Mendes AA, Soares CMF, Tardioli PW. Recent advances and future prospects for biolubricant base stocks production using lipases as environmentally friendly catalysts: a mini-review. *World J Microbiol Biotechnol*. 2022;39(1):25.
<http://dx.doi.org/10.1007/s11274-022-03465-4>
 26. Jedrzejczyk MA, Van den Bosch S, Van Aelst J, Van Aelst K, Kouris PD, Moalin M, et al. Lignin-based additives for improved Thermo-oxidative stability of biolubricants. *ACS Sustain Chem Eng*. 2021;9(37):12548–59.
<http://dx.doi.org/10.1021/acssuschemeng.1c02799>
 27. Ahmad U, Raza Naqvi S, Ali I, Saleem F, Taqi Mehran M, Sikandar U, et al. Biolubricant production from castor oil using iron oxide nanoparticles as an additive: Experimental, modelling and tribological assessment. *Fuel (Lond)* 2022;324(124565):124565.
<http://dx.doi.org/10.1016/j.fuel.2022.124565>
 28. Appiah G, Tulashie SK, Akpari EEA, Rene ER, Dodoo D. Biolubricant production via esterification and transesterification processes: Current updates and perspectives. *Int J Energy Res*. 2022;46(4):3860–90.
<http://dx.doi.org/10.1002/er.7453>

تحضير وتشخيص ودراسة خصائص مشتق ثلاثي مثيلويل بروبان ثلاثي الاستر من زيت جوز الهند (كوكوس نوسيفيرا)

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الخلاصة

في هذه الدراسة، تمت متابعة عملية التحويل الحمضي القلوي لزيت بذور جوز الهند المكرر (RCOSO) إلى إستر مثيل الأحماض الدهنية من خلال إنتاج مادة تشحيم بيولوجية حساسة للحرارة تعتمد على ثلاثي مثيل البروبان باستخدام هيدروكسيد البوتاسيوم، وتم تقييم خصائصها الفيزيائية والكيميائية. تم استخدام الاختبار القياسي الأمريكي للمواد (ASTM) للتأكد من نقطة صب مادة التشحيم البيولوجية ومؤشر اللزوجة، والتي وجدت بـ 4-درجة مئوية و 283.75، على التوالي. تم توضيح العلاقة العكسية بين لزوجة مادة التشحيم ودرجة الحرارة من خلال اللزوجة المقاسة عند تحويل الأستر المتباعدة إلى ديزل حيوي. بعد ذلك، تم إنشاء مادة تشحيم بيولوجية عن طريق درجة حرارة الأستر. تم التحقق من مجموعة الإستر بمقارنة أطياف تحويل فوربييه بالأشعة تحت الحمراء (FTIR) لوقود الديزل الحيوي ومواد التشحيم الحيوية بين نطاقات الامتصاص (1755.07-1737.21 سم⁻¹). (1-تظهر بيانات كروماتوغرافيا الغاز أن زيت اللوز الحلو يحتوي على 71.725% أحماض دهنية غير مشبعة لاستخدامها كمادة تشحيم بيولوجية، و 53.478% أكثر من الأحماض الدهنية غير المشبعة من الأحماض الدهنية المشبعة، و 9.52 جم من اليود لكل 100 جم من عينة الزيت أكثر من الجلسريدات غير المشبعة. بنسب 31.44 و 45.93% على التوالي، شكل حمض اللينوليك الغالبية بين الأحماض الدهنية في الزيت ومواد التشحيم الحيوية الاصطناعية. بالإضافة إلى ذلك، كانت أحماض البالميتيكواللينوليكوالأوليك موجودة في زيوت التشحيم والزيت من جوز الهند. ترتبط نتائج توصيف زيوت التشحيم الحيوية ارتباطاً وثيقاً بمعايير ISO VG-32، مما يشير إلى أن لديها القدرة على العمل كزيت تروس خفيف للسيارات.

الكلمات المفتاحية: وقود الديزل الحيوي، زيوت التشحيم الحيوية، الأستر، الزيت النباتي، اللزوجة.