Production of biolubricant samples from palm kernel oil using different chemical modification approaches

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**Abstract**

This work focused on the chemical synthesis and characterization of palm kernel oil (P KO) for biolubricant production using transesterification of palm kernel methyl ester with trimethylolpropane (TMP) and epoxidation-esterification methods. The PKO was extracted using solvent extraction method. The physicochemical characteristics of the PKO and produced biolubricant samples were determined using standard methods. Fourier transform infrared (FTIR) spectrometry and gas chromatographic (GC) analyses were used to determine the predominant functional groups and fatty acids of PKO and the produced biolubricant samples, respectively. The kernel oil yield of 49.82% (by weight) was obtained at 55°C, 150 min and 0.5 mm particle size. The viscosities at 40°C, 100°C, viscosity index, pour and flash points of the biolubricants produced by transesterification with TMP (PKBL T) and epoxidation-esterification (PKBL E) methods, were (42.53 cSt, 10.65 cSt, 139, −11°C, 235°C) and (44.69 cSt, 11.42 cSt, 132, −12°C, 240°C), respectively. Time, molar ratio, and temperature effects were the main factors that significantly influenced the transesterification and epoxidation processes. The obtained physicochemical properties of PKBL E and PKBL T samples showed conformity with ISO VG 32 standard, hence their possible application as biolubricant basestock. Furthermore, the presences of the C–H, OH functional groups in the FTIR results, indicate the biodegradability of the produced biolubricant samples; while the GC analyses indicate that the samples were mainly saturated fatty acids.

**KEYWORDS**

biolubricant, epoxidation-esterification, palm kernel, transesterification, trimethylolpropane

1 **INTRODUCTION**

Globally, crude oil reserves have dropped significantly due to high consumption, as well as slow natural mineral deposit formation. There is therefore a possible impeding energy crisis if not quickly addressed. To avert this, scientists and researchers have sought for alternative energy sources using bio-based oil materials. As such, alternative oil sources for...
chemical process industries, which are renewable, biodegradable, and eco-friendly, are currently being researched. Significantly, vegetable oils obtained from seeds and nuts are often used as a suitable substitute to mineral oil obtained from petroleum,\(^2\) hence there is a need for the development/modification of the vegetable oils, into industrial products such as biodiesel,\(^3\) biolubricants,\(^4\),\(^5\) transformer fluid,\(^2\) and other vital fuels that could be used as replacement for conventional mineral base fuels/Fluids.

In Nigeria, there is a huge availability of crude oil reserves estimated at about 37.4 billion barrels,\(^6\) with a reasonable degree of production, exploration, and refining of petroleum into products such as, petrol, kerosene, and diesel. However, our interest lies in the production of petrochemical products such as biolubricants.\(^4\) This is because of the environmental friendliness of the biolubricants, when compared with its petroleum-based counterpart.\(^7\) Recently, researchers have successfully synthesized biolubricants from different vegetable oil sources, to be used as environmentally friendly lubricant. Some of these include but are not limited to \textit{Jatropha curcas} oil,\(^4,\)\(^8\) palm kernel oil (PKO),\(^1\) palm oil,\(^9\) fluted pumpkin seed oil,\(^10\) and castor bean biodiesel.\(^11\) On this premise, this work seeks to synthesize biolubricant from PKO using different process routes in order to evaluate the method that produces better sample with lubricant properties.

PKO is an edible oil which is yellowish in color and it is obtained from the kernel of palm fruit, scientifically known as \textit{Elaeis guineensis}.\(^12\),\(^13\) Its tree plant (palm tree) originates from tropical Africa and some part of Asia. Nigeria is recognized to be the fifth largest palm oil cultivator globally.\(^14\) Palm kernels are essential by-products obtained during the palm oil milling and processing. It makes up about 45%–48% (by weight) of the palm nut. The oil yield of palm kernel is about 47%–59% by weight.\(^13\) Lauric acid being the predominant fatty acid in PKO, makes up about 48.53% of the fatty acid composition.\(^15\) PKO is rich in saturated fatty acid (SFA) with a proportion of 79.91%.\(^15\) Due to the relatively high oil yield of PKO, a number of studies have been carried out on its utilization in the production of biodiesel,\(^14\) as well as in biolubricant production.\(^1,\)\(^16\) Noting this, the authors extend this present study to the use of PKO (because of its high oil yield, availability, as well as biodegradability), in biolubricant production, using modified process routes, for the purpose of comparison, since limited or no studies, have been channeled toward this direction.

A number of studies on biolubricant production using vegetable oils as a potential alternative to mineral-based lubricant, utilizing the two stage transesterification and epoxidation-esterification methods, have been previously reported. In this case, two stage transesterification using methanol as alkali catalyst and subsequently trimethylolpropane (TMP), have been extensively reported in the literature. For instance, Heikal et al.,\(^17\) Menkiti et al.,\(^4\) Sharma and Sachan,\(^18\) Encinar et al.,\(^19\) Cavalcanti et al.,\(^19\) Sarno et al.,\(^20\) and Shote et al.,\(^21\) successfully used this approach for the production of biolubricants using palm oil, \textit{J. curcas} oil, Karanja oil, castor bean biodiesel, soybean oil, waste cooking oil, and PKO, respectively. In this method, low alkyl ester yield and product separation difficulty (due to soap formation), are the main drawbacks of its straight alkali catalyzed transesterification stage (without initial esterification pretreatment stage).\(^2\) Hence, in this work, successive two-step conversion process: an acid-catalyzed esterification (aimed at lowering the FFA content), followed by the alkali-catalyzed transesterification (aimed at improving the alky ester yield and purity), prior to the final stage of transesterification with TMP, was used. The essence was to eliminate the aforementioned short-comings and to ensure that a high grade biolubricant with high purity level and yield was produced.\(^4\)

On the other hand, the epoxidation-esterification procedure is often considered because of its advantage of better thermooxidative stability, as well as its associated low temperature properties of the final product.\(^5\) Epoxidation reaction is a reaction of the double bonds and peracetic acid to produce epoxy ring.\(^2,\)\(^22,\)\(^23\) It is worth mentioning that in this work, the obtained alkyl ester from the alkali transesterification was used instead of PKO to react with peracetic acid in order to get the desired epoxy methyl ester. This decision was based on the improved thermooxidative stability and lower pour point of the final desired product.\(^2,\)\(^22\)

Therefore, to the best knowledge of the authors, there is no existing published work that compares biolubricants produced from PKO using these two approaches. Though, the production of biolubricant from PKO using transesterification with TMP alone has been reported by several authors,\(^1,\)\(^12,\)\(^16\) but not compared with its production using epoxidation-esterification method. It is therefore necessary to close this knowledge gap in the area of biolubricant production from PKO, as well as to determine the better production method to be adopted.

This study focuses on biolubricant production from PKO, using alkali-catalyzed transesterification, followed by transesterification with TMP and transesterification-epoxidation-esterification methods, for comparison purpose. This is the justification for the present study. The qualities of the produced biolubricants were evaluated on the basis of the two methods used and on the referenced standards. Furthermore, the physicochemical properties of the PKO and the PKO biolubricants samples produced were evaluated using standard procedures. In addition, the prevalent functional groups, and the fatty acid compositions of the PKO and the PKO biolubricant samples, were determined using Fourier transform infrared (FTIR), and gas chromatography (GC), respectively.
2 | MATERIALS AND METHODS

2.1 | Materials

Palm kernels were obtained from Aguneze, Ahiazu-Mbaise, in Imo State, Nigeria. TMP was purchased from Sigma Aldrich, Germany, while methanol, sulfuric acid (H₂SO₄), orto-phosphoric acid (H₃PO₄), analytical grade n-hexane, and other reagents were purchased from Conraws, Presidential Road, Enugu. The reagents had purity levels of above 99%, and they were used without further purification.

2.2 | Extraction of PKO

Oil extraction from the milled PKO sample was carried out according to the Association of Official Analytical Chemists (AOAC) 963.15 method, using soxhlet extractor unit with particle size of 0.5 mm. To enhance the solubility of the palm kernel sample in the chosen solvents, Soxhlet extractor was chosen. Milled kernels of 0.5 mm particle size (15 g) were packed in a thimble (Hawach Scientific Co. Ltd; SLGET2870 Super-fine Glass Micro-fiber Extraction Thimbles, 28 mm ID × 70 mm H) of the soxhlet extractor which was filled with 150 ml of n-hexane. Oil extraction was performed at a temperature of 55°C using n-hexane. At a temperature of 55°C and particle size of 0.5 mm, the extraction was carried out for 150 min, to obtain optimum oil yield. The oil yield obtained at the end of the 150 min extraction time for the extraction conditions was calculated and recorded. The extraction temperature was measured using an electronic thermometer (Hanna HI-9063), while the time was measured using a stop watch. The oil yield was calculated (see Equation 1) using AOAC method no. 920.85 at the end of extraction period of 150 min. After each extraction batch/cycle, the solvent was removed at 60°C using rotary evaporator (model N-1000S-W, EYELA, Tokyo, Japan). The solute to solvent ratio used for the extraction process was 1:5 (15 g: 150 ml). The entire extraction process carried out under the set of conditions was performed three times and the average value reported, while the total extraction yield was obtained using AOAC 920.85 standard method.

The oil yield of sample was calculated using Equation (1).

\[
\%\text{Oil yield} = \frac{\text{Weight of oil extracted (g)}}{\text{Weight of sample (g)}} \times 100. \tag{1}
\]

The extracted oil sample was PKO.

2.3 | Physicochemical properties of PKO and PKO biolubricant samples

The oil yield (AOAC 920.85) was determined according to AOAC approved techniques. In this method, the palm kernels were milled with a manual grinder and passed through a 0.5 mm-mesh sieve size in order to obtain a particle size of 0.5 mm. Thereafter, 15 g of the milled kernel powder was packed in a thimble. Oil was then extracted with N-Hexane of boiling point of 68.7°C for 150 min at a temperature of 55°C, using Soxhlet apparatus (Soxtect 2050, FOSS, Denmark). After each extraction batch/cycle, the solvent was removed at 60°C using rotary evaporator (model N-1000S-W, EYELA, Tokyo, Japan), while the yield was calculated using Equation (1), as earlier presented. At the end of the oil extraction, the oil was dried at temperature of 105°C for 5 h so that residual water and hexane could evaporate.

Viscosity index (ASTM D2270), viscosity (ASTM D445) and specific gravity (ASTM D1217 – 15), were determined using ASTM standard methods. In addition, the pour and flash points were determined using ASTM D97 and ASTM D93 standard methods, respectively. Each physicochemical property was measured three times and the average values of the properties were determined and noted.

2.4 | Fatty acid composition of PKO and PKO biolubricants samples

Fatty acid profile was evaluated in line with the AOAC 996.06 (1990). In this procedure, a GC (Shimadzu GC–14B, Model 910), was used to determine quantitatively, the prevalent fatty acids in the PKO and PKBL samples. According to the
equipment, a HP 88 capillary column (0.25 mm i.d. × 100 m, film thickness 0.25 μm—Shimadzu Corporation, Tokyo, Japan), was used to equip the GC’s flame ionization detector and integrator. This was achieved by using 250°C temperatures for both the injector and detector. However, the oven temperature was retained at 190°C for a period of 15 min. Thereafter, this temperature was then increased intermittently, up to 230°C, at the rate of 5°C per minutes. Afterward, it was maintained at this temperature for the same time interval as the initial step. The carrier gas used was nitrogen which was maintained at a pressure of 500 kPa. Finally, the prevalent fatty acids were identified and compared with standard compounds while the quantity of each fatty acid was calculated from the percentage area of the individual fatty acid. The analysis was carried out three times.

2.5 | Transesterification experiment

2.5.1 | Synthesis of palm kernel methyl ester

In other to synthesize the palm kernel methyl ester (PKME), 25 ml of the PKO (triglyceride) sample was poured into 250 ml conical flasks and heated to 60°C using a water bath. Solution batches of potassium methoxide were prepared by dissolving 5.1 g (30 wt%) of KOH pellets in an agitated 250 ml beaker containing 150 ml anhydrous methanol. The potassium methoxide solution was then transferred into warm 25 ml PKO (triglyceride) sample at a methanol to oil ratio of 6:1. The solution was then stirred vigorously, using a magnetic stirrer at 500 revolutions per minute for 120 min. In other to ensure proper settling, the mixture was left undisturbed in a separating funnel for 24 h. At the end of the settling process, the upper layer (methyl ester sample) was poured into a beaker and afterward properly washed with distilled water. This was aimed at removing unreacted methanol, catalyst, glycerin, soap, and other impurities. The demoisturization of the fatty acid methyl ester (FAME) sample was then carried out by heating slowly to constant temperature of 100°C. Finally, the lower layer that consists of glycerol and soap was collected via the bottom of the funnel. The processes performed under standardized conditions occurred three times and the average value reported.

The percentage methyl ester yield of the PKO sample in each case was calculated using the relationship in Equation (2):

\[
\% \text{Methyl ester yield} = \frac{\text{Mass of methyl ester produced (g)}}{\text{Mass of oil sample used (g)}} \times 100. \tag{2}
\]

PKME, generally referred to as FAME is the product of the transesterification of the extracted PKO; see Figure 1. On the other hand, while using the same method, FAME of PKO was also obtained with the procedure. Here, a mixture comprising of 300 g of PKO (triglyceride), 100 g of methanol, as well as 1% wt/wt orthophosphoric acid catalyst was decanted into a continuously stirred reactor. This reactor was equipped with a water-cooled reflux condenser to ensure complete reaction. Thereafter, the mixture was then heated to 65°C and maintained at this temperature for 1 h 30 min. After the reaction, the mixture was dosed with 0.2 molar solution of sodium trioxocarbonate IV, to ensure complete neutralization of the acid, and eventually stop the reaction. The mixture was then poured into a separating funnel and subsequently allowed to stand for 24 h to ensure complete separation of methyl esters and glycerol phases. Glycerol phase at the bottom was emptied into a clean container and allowed to stand. The PKME (FAME) was then heated to 65°C to ensure the removal of the residual methanol. Finally, the remaining catalyst in the PKME was removed by successively rinsing with hot distilled water at 80°C. After which the remaining water in the PKME was eliminated by oven-heating at 100°C. This standardized process took place three times with the average value reported.

[Figure 1: Reaction scheme for transesterification reaction of triglyceride (PKO) with methanol to obtain methyl esters (FAME). FAME, fatty acid methyl ester; PKO, palm kernel oil]
2.5.2 Synthesis of biolubricant from PKME using TMP

The synthesis of biolubricant adapted was as described by Surapojet al.\textsuperscript{26} with slight modifications. The obtained PKME or FAME was synthesized with TMP to obtain TMP ester or palm kernel biolubricant (PKBL\textsubscript{T}) as shown in Figure 2.

Here, TMP was initially heated using a transesterification experimental set-up. This set-up comprises a 50 ml three-necked round-bottom flask that was fitted to a water-cooled reflux condenser, a thermometer, Kipp's apparatus and a stirrer operated at 1000 rpm. At 1000 rpm, and under the flow of CO\textsubscript{2}, the TMP in the flask was heated to 110°C and maintained at this temperature for 15 min, then subsequently cooled. To ensure the evolution of moisture from the TMP, the temperature was maintained at 110°C. Thereafter, a Ca(OH)\textsubscript{2} catalyzed batch transesterification reactions between PKME (FAME) and already cooled TMP were conducted at PKME–TMP ratios: 3:1, 4:1, 5:1, 6:1, and 7:1, using the same experimental set-up. At 80, 100, 120, 140, and 160°C, each of the stated PKME–TMP ratios was subjected to transesterification. At intervals of 1, 2, 3, 4, and 5 h, samples from the respective individual runs of the experiment (at a particular molar ratio and temperature) were monitored, collected and analyzed. In this study, the catalyst was recycled by transferring it back to the reaction vessel and adding more catalyst and the TMP and repeating the process for additional period of time. After every reaction, the mixture was allowed to cool to room temperature, prior to carrying out filtration process. This was to allow for separation of the residual solid catalyst from the liquid mixture, which is the palm kernel biolubricant (PKBL\textsubscript{T}). The filtered palm kernel bio-based stock was analyzed using the GC to determine the product composition. Furthermore, prior to the characterization of the bio-based TMP ester, unreacted methyl ester was not expunged. This was aimed at the improvement of the wear resistance of the bio-based TMP ester, as well as to prevent conjugation reaction that occurs at elevated temperatures (180–200°C), which involves polyunsaturated fatty acid (PUFA).\textsuperscript{17} The standardized process was performed three times and the average value reported.

2.5.3 Synthesis of biolubricant from PKME using epoxidation-esterification reaction

In the synthesis of biolubricant from PKME using epoxidation-esterification reaction, 50 g of the PKME sample was placed in a three-necked flask. Seven gram of acetic acid was placed in the round bottom flask, followed by the addition of 0.02 g (1.5 wt%) of H\textsubscript{2}SO\textsubscript{4} to the flasks. The mixtures were stirred continuously, followed by the addition of 15 g (1.5 molar) of hydrogen peroxide (15 wt%). The mixture was added to the flask containing 7 g acetic acid to produce peracetic acid (PAA). The methyl ester sample in the three-necked flask was then heated to 70°C, followed by the addition of PAA mixtures. These were allowed to react at 70°C for 7 h with continuous stirring at 1200 rpm. Samples were taken out every 1 h from each of the reaction set up for FTIR analysis to determine the effect of reaction time on the yields (i.e., changes in functional groups as the reaction proceeds) at 70°C. At the end of the reaction, 3 ml of the epoxy methyl ester sample was then removed twice using diethyl ether (2 x 20 ml) in separating funnels. The organic phases (oily layer) of samples were purified (washed) three times with 5% saturated sodium bicarbonate NaHCO\textsubscript{3} (3 x 15 ml) to neutralize the unreacted acid present. Thereafter, the organic phase of the sample was also washed three times with saturated solutions of sodium chloride NaCl (3 x 15 ml) to obtain epoxy methyl ester of the sample. The sample was dried over anhydrous magnesium sulfate. The solvent (diethyl ether) was then removed using a rotary evaporator. The epoxy oxygen (oxirane) content and iodine value (IV) of the sample was measured according to the procedure described by Arumugam et al.\textsuperscript{27} and Arumugam and Sriram.\textsuperscript{28} The process carried out under the set of conditions was performed three times and the average values epoxy oxygen (oxirane) content and IV of the sample reported.

The obtained epoxy methyl ester sample was epoxy methyl ester (oxirane) of palm kernel oil (EMPKO\textsuperscript{6}). Figure 3 shows the reaction scheme for the epoxidation reaction of PKME to obtain EMPKO\textsuperscript{6}.
2.5.4 | Esterification ring opening reaction

In the esterification ring opening reaction, 20 g of epoxy methyl esters (oxirane) sample (EMPKO<sup>0</sup>) was placed in a round bottom flask and 10 ml of ethyl acetate was added to it. The mixture was stirred continuously, followed by the addition of 4 g of acid anhydride. The flask was purged with nitrogen (to exclude air), followed by the addition of 1 ml boron trifluoro diethyl etherate. The epoxy methyl esters (oxirane) sample in the flask was heated to 70° C, after which the mixtures were left to react at this temperature for 7 h. in fume cupboard with continuous stirring. The organic phase (oily layer) of the sample was purified three times with 5% NaHCO<sub>3</sub> (3 × 15 ml) to neutralize the unreacted acid present. Thereafter, this sample was also washed three times with saturated solutions of sodium chloride NaCl (3 × 15 ml) to obtain the branched methyl ester sample. The sample was dried over anhydrous magnesium sulfate and the solvent (ethyl acetate) removed using a rotary evaporator. The unreacted anhydrides were removed at atmospheric pressure using distillation at 80° C. The distillation unit can only process about 20 ml of sample each time, limiting the amount of sample that can be processed.<sup>2</sup>

The obtained branched methyl ester sample was then used as a precursor for the synthesis of modified triester-derivatives by acetylation with octanoyl chloride to obtain the palm kernel biolubricant (PKBL<sub>EE</sub>). The standardized process was performed three times and the average value reported. Figure 4 shows the reaction scheme for the esterification of EMPKO<sup>0</sup> (epoxide).

3 | RESULTS AND DISCUSSION

3.1 | Transesterification of PKME with TMP for palm kernel biolubricant production

Figures 5 and 6 show the progress of the transesterification reactions at different times for 3.0 and 4.0 molar-ratios, respectively. From the plots, it can be seen that transesterification advanced in a stepwise manner. At first, monoester (ME)
formation reached a maximum value, and this was immediately followed by diester (DE) formation. At maximum DE formation stage, triester (TE) formation increased at a fast rate. This was attributed to the fact that during the transesterification of FAME with TMP which occurs stepwise, intermediate products are produced, prior to the final preferred product formation, which is the triester (TE).\textsuperscript{29} First, the ME that is a single branch polyol ester was formed during the reaction. However, increasing the ME quantity, resulted to the immediate conversion to DE with another FAME molecule. Finally, the TE was formed by the reaction of DE and FAME (PKME). It is worth noting from the plots that the TE concentration increased with decrease in concentrations of DE and ME, as evident in Figures 5 and 6. This reaction mechanism has been reported by a number of other researchers.\textsuperscript{4,30,31}

### 3.1.1 Temperature effects

To determination the effect of temperature on the synthesis of biolubricant from PKME, the experiment was conducted at FAME to TMP molar ratio of 4:1, with catalyst concentration of 1.0% wt/wt of reaction mixture. At these conditions,

![Figure 5](https://example.com/figure5.png)

**Figure 5** Transesterification between TMP and PKME of 140°C and 3:1 molar-ratio, using 1 wt% Ca(OH)\textsubscript{2} catalyst. PKME, palm kernel methyl ester; TMP, trimethylolpropane

![Figure 6](https://example.com/figure6.png)

**Figure 6** Transesterification between TMP and PKME of 140°C and 4:1 molar-ratio, using 1 wt% Ca(OH)\textsubscript{2} catalyst. PKME, palm kernel methyl ester; TMP, trimethylolpropane
the reaction was carried out at 80, 100, 120, 140, and 160°C, in order to study the effect of temperature on biolubricant synthesis. Figures 7 and 8 show the temperature effect results for the synthesis PKO biolubricant. It can be seen from Figure 7 that an increase in temperature gave rise to the increase in TE composition, until at about a temperature of 140°C when the increase in TE composition became negligible. This was attributed to the fact that at higher temperature (see Figure 8); the quantity of FAME in the reactor was low due to vaporization, favoring the reverse reaction. In other words, the recondensation of the FAME vapor back into the reactor. Hence, the reverse reaction would be contained; thereby leading to more esterification of the DE to TE. This is the reason for the significant difference in TE composition, compared with those of DE and ME. Therefore, it is crucial for the condenser water to be cold enough, in other to ensure condensation of the vaporized FAME back into the reactor.4,16

3.1.2 Mole ratio effects

In other to ensure improved yield of the triester during the transesterification of FAME with TMP, excess quantity of FAME or TMP is used. This is because transesterification reaction is a reversible reaction. However, in this study, excess
FAME was chosen over TMP due to economic reasons of its lower cost. The molar ratio of FAME to TMP was varied at 3:1, 4:1, 5:1, 6:1, and 7:1, at temperature of 100°C, time of 5 h and Ca(OH)\textsubscript{2}/wt catalyst weight of 1.0 wt%. Figure 9 shows the effect of PKME: TMP mole ratio on % composition of TMP ester at 100°C and catalyst loading of 1% each. In addition, Figure 10 shows the temporal yield of palm kernel triester at various mole ratios for temperature of 100°C and 1% catalyst loading. It can be seen from Figure 9 that as the molar ratio of FAME: TMP was increased, the yield of TE increased as well. In order to obtain better and more product yield, the reactants molar ratios were kept above the stoichiometric values. This is because the reaction was driven more toward completion. However, in Figure 10, it is evident that increasing the molar ratio beyond 4:1 gave negligible improvement in TE yield for PKO biolubricant. This phenomenon can be attributed to the low rate of conversion of DE to TE, as well as occurrence of reverse reaction that caused the breaking of DE to TE.\textsuperscript{4} Encinar et al.,\textsuperscript{11} Bahadi et al.,\textsuperscript{12} and Menkiti et al.,\textsuperscript{10} reported that for transesterification of rapeseed, palm kernel, and fluted pumpkin methyl esters using TMP, maximum conversions were attained at molar ratios of 3:1, 3.05:1, and 6:1, respectively. In this work, it can be seen that the conversion to TE increased from 71.34% to 77.10% as the molar ratio increased from 3:1 to 4:1 (see Figure 9). However, a small amount of DE was an added value to the properties of the lubricant. Meanwhile, the excess of FAME remaining in the final product would affect the physical properties of lube and additional energy would be required to remove it.\textsuperscript{10,30}
3.2 Epoxidation and subsequent esterification of PKME for biolubricant production

The result of the effect of temperature and time at different times and temperatures, on the relative fractional conversion of the PKME sample to oxirane is presented in Figure 11. The conditions used for this study were H$_2$O$_2$-ethylenic unsaturation molar ratio of 1.5:1, 2.5 wt% H$_2$SO$_4$ catalyst concentration and 1200 rpm stirring speed.

The temperature and time effects on the rate of epoxidation of the PKME sample were determined at 35, 45, 55, 65, and 75°C. The result indicated that the relative fractional conversion to oxirane, increased directly with reaction time during the early stages of the reaction as can be seen in Figure 11. However, it started decreasing afterward with additional increase in time of the reaction. This abnormality associated with additional increase in time was due to the opening of oxirane ring. It was also discovered that temperature increase favored peracetic acid formation. Hence, the resulting implication was not limited to accelerated epoxidation, but also enhanced hydrolysis rate; that is oxirane ring opening of the product.

Furthermore, Figure 11 shows the times necessary to achieve maximum relative conversion at different temperatures. As can be seen in the figure, the times necessary to attain maximum relative conversion were at the 8, 7.9, 6, 6, and 4 h, at temperatures of 35, 45, 55, 65, and 75°C, respectively. From Figure 11, it is observed that smaller rates were noticed for the reactions at lower temperatures of 35 and 45°C. Though more stable oxirane rings were obtained at these temperatures, compared with those of higher temperatures 55, 65, and 75°C, which showed higher rate but more unstable oxirane ring which led to higher degradation of the epoxide. It is important to state that at the higher temperatures of 55, 65, and 75°C; the relative conversion of the PKME sample to oxirane attained maximum values in 6, 6, and 4 h, respectively. Nevertheless, it was noticed that there was a decrease in the relative conversion to oxirane with additional increase in time. For instance, at temperature of 75°C, the observed decrease in the relative conversion to oxirane was very clear, since it began after 4 h. In the same way, at temperatures of 55 and 65°C, their relative conversion started after 6 h. Nevertheless, at 35 and 45°C, more stable rings were obtained for the sample. This is because there was no decrease in the relative conversion to oxirane within the studied time intervals (see Figure 11).

Similar results were also obtained by Agu et al., Wei et al., Borugadda and Goud, Jalil et al., and Hong et al. for the epoxidations of Terminalia catappa L. methyl ester, methyl oleate, castor oil FAME, PKO, and linoleic acid of J. curcas oil, respectively. The highest fractional conversion to oxirane obtained was 0.87, at 75°C in 4 h as shown in Figure 11.

3.3 Physicochemical properties of PKO and PKO biolubricants

The physicochemical properties of the PKO and the biolubricants produced by transesterification with trimethylolpropane (TMP) and epoxidation-esterification methods are presented in Table 1. The obtained oil yield of palm kernel was 49.82%. This value was slightly higher than 49.2% yield reported for palm kernel by Hossain et al., but significantly
TABLE 1 Physicochemical properties of PKO and modified PKO biolubricants

| Property | units | PKO | PKBL-T | PKBL_E | Petrolubricant | Methods |
|----------|-------|-----|--------|--------|---------------|---------|
| Oilyield | %     | 49.82 | 0.990 ± 0.0005 | 4.10 ± 0.005 | 0.848 | AOAC 920.85 |
| Specific gravity | g/ml | 0.910 ± 0.0005 | 0.990 ± 0.0005 | 1.10 ± 0.005 | 0.848 | ASTM D1217-15 |
| Viscosity @ 40°C | cSt | 24.08 ± 0.008 | 42.53 ± 0.005 | 46.69 ± 0.005 | 46.476 | ASTM D445 |
| Viscosity @ 100°C | cSt | 5.02 ± 0.008 | 10.65 ± 0.005 | 11.42 ± 0.005 | 6.940 | ASTM D445 |
| Viscosity index | 180 ± 0.5 | 139 ± 0.5 | 132 ± 0.5 | 105 | ASTM D2270 |
| Pour point | °C | 18 ± 0.5 | −11 ± 0.5 | −12 ± 0.5 | −20 | ASTM D97 |
| Flash point | °C | 210 ± 0.5 | 235 ± 0.5 | 240 ± 0.5 | 220 | ASTM D93 |

Abbreviations: PKBL_E, palm kernel biolubricant obtained by epoxidation-esterification; PKBL_T, palm kernel biolubricant obtained by transesterification of TMPPKO; PKO, palm kernel oil.

higher than the 47% yield reported by Zaidul et al. However, the 49.82% yield obtained for PKO in the present study is slightly lower than 51.35%, reported by Yerima et al. As such, the oil yield obtained in this study is similar to that reported by Hossain et al. and Yerima et al. This slight difference in the PKO yields could be attributed to the extraction method and operation condition used.

A number of studies have shown that vegetable oils have shortcomings with respect to its use as biolubricant without modification of its structure. These limitations include thermal, oxidative, and hydrolytic instability, as well as inadequate low temperature fluidity as a result of their high pour points. These limitations are caused by the glycerol moiety which is a major constituent in vegetable oil. Most of these problems are significantly reduced by chemical modification of the vegetable oil, which in this case is PKO. Hence, the reason for the modification of the PKO using the methods of transesterification with TMP and epoxidation-esterification.

The synthesis of PKO biolubricant using two stage transesterification methods causes the removal of the β-hydrogen atom from the oil structure. Hence, this provides an ester with high oxidative and thermal stability. In this two stage transesterification process, the unstable hydrogen that is bared by the glycerol molecule is substituted by a more stable TMP. In the first stage of the transesterification reaction, the glycerol molecule is removed, while in the second stage, a more effective TMP molecule, replaces the glycerol. Thus, trimethylolpropane triester with more superior properties and performance is produced.

Similarly, the synthesis of PKO biolubricant was also achieved using the three step synthesis of the methyl ester that involves epoxidation, ring opening, and esterification steps. The epoxidation process helps in the removal of unsaturation in the methyl ester by converting them into epoxy-groups which helps to improve the oxidative stability. It is a known fact that the existence of double bonds in vegetable oil chains accelerates oxidative degradation. On the other hand, there are poor low temperature properties in the oil, leading to solidification at lower temperatures like 0°C. These shortcomings, limit their application at low operating temperature, especially as biolubricants for automotive and industrial purposes. Hence, the suitable method to improve the low temperature flow properties is to attach branching sites at the epoxy carbons. This leads to the need for the ring opening step of this synthesis approach. This was achieved by carefully carrying out esterification ring opening reaction using acid anhydride and boron trifluoro diethyl etherate catalyst. The obtained branched methyl ester was used as precursors for the synthesis of modified triester-derivatives by acetylation with octanoyl chloride.

Conventional lubricant, which is the petroleum lubricant sample, was analyzed using similar parameters as those determined for the biolubricant; in order to evaluate the close relation of the properties of the biolubricant samples to the conventional lubricant. Table 1 shows the physicochemical properties of the PKO, palm kernel biolubricant (PKBL_T) synthesized by transesterification of TMP, palm kernel biolubricant (PKBL_E) synthesized by epoxidation-esterification of the methyl ester and the conventional petroleum lubricant.

It could be observed from the table that the biolubricants samples were more viscous and have higher weight than their petroleum lubricant counterpart. Hence, these properties have the advantages of better mechanical load and thermal resistance of the biolubricants over mineral lubricant. Furthermore, the viscosity indices and pour points values of the studied samples were similar to the petroleum lubricant sample. The flash point values of the synthesized biolubricant samples were higher than the PKO sample. This is an indication that the synthesized samples had enhanced thermal resistance, as a result of the transesterification with TMP and epoxidation-esterification reactions. The flash point values of the synthesized biolubricant samples were close to that of petroleum lubricant; thus, an indication of their greater thermal stability.
3.3.1 Specific gravities of synthesized PKO biolubricants

As seen in Table 1, the specific gravity value of PKO biolubricant PKBL_T synthesized by transesterification with TMP (0.990 g/ml), is similar to that of PKBL_E synthesized by epoxidation-esterification (1.10 g/ml). However, it can be seen from the table that there was an increase in the specific gravity of these two samples when compared with the specific gravities (SG) of the raw PKO sample (0.910 g/ml). However, the petroleum lubricant had the least SG value of 0.848 g/ml. The higher SG values of the PKBL_T and PKBL_E samples, compared with the PKO value, was attributed to increase in molecular complexity, resulting from the TMP backbone and the elongated chain of the triester, which is caused by the epoxy ring opening.1,4,7 It can be seen also that the specific gravity value for the biolubricant samples were higher than the petrolubricant. This could be attributed to chemical structural change in the constituent molecules. It is noteworthy that the specific gravity change leads to a corresponding change in the mass of the products. In other words, the higher the specific gravity, the heavier and more viscous the lubricant oil would be. Hence, the biolubricant has the advantages of better sustenance at elevated temperature, as well as the ability to withstand greater loads.1,49 The compatibility of biolubricant products with either the heavy or light duty engines is determined by its SG. This compatibility is the ability of the sample to mix with other liquids.49 As such, materials with lower SG (<1) floats in water, whereas those with higher SG (>1), sink in water. Therefore, biolubricants with higher SG and viscosity, last longer on the applied surfaces and joints.1,16,49

3.3.2 Viscosities of the synthesized PKO biolubricants samples at 40°C and at 100°C

The viscosities of PKO biolubricants synthesized by transesterification with TMP PKBL_T were 42.53 cSt at 40°C and 10.65 cSt at 100°C, respectively. Similarly, the viscosities of PKO biolubricants synthesized by epoxidation-esterification PKBL_E were 44.69 cSt at 40°C and 11.42 cSt at 100°C, respectively. From the values in Table 1, it can be seen that viscosities values of the PKBL_E sample were comparatively higher than those of the PKBL_T sample. The viscosity of the PKO sample was lower than the values reported for PKO, by Alan et al.1 The viscosity was also lower than the value for Jatropha and palm oils by Menkiti et al.,4 and Reddy et al.,50 respectively. These values for PKO biolubricant samples were less than those of biolubricant from PKO reported as 480.63 cSt at 40°C and 20.54 cSt at 100°C, by Alan et al.1 Similarly, the values of viscosities at 40 and 100°C for PKO biolubricant samples in this study were higher than the 32.67 and 6.10 cSt, respectively, reported for palm kernel biolubricant by Shaba et al.16 Furthermore, Bahadi et al.12 and Dandan et al.,51 reported that the viscosities of palm kernel biolubricant samples at 40 and 100°C as, 41.76, 8.73 cSt and 35.36, 11.24 cSt, respectively. These cited values were relatively close to the values obtained in this work. The differences in the values of the palm kernel biolubricant samples obtained in this work, and those reported in the literature could be attributed to factors such as, process parameters, geographical location, and methods of production.48 In addition, the values of the viscosities of PKO biolubricants in this work were lower, when compared with Jatropha biolubricants with values of 55.17 cSt at 40°C and 10.96 cSt at 100°C, as reported by Bilal et al.52 These values are however comparable to those of petroleum lubricants which were evaluated to be 46.476 cSt and 6.940 cSt, at 40 and 100°C, respectively. However, it is vital to state that the viscosities at 100°C of the PKO biolubricants samples obtained in this work were greater than that of the petroleum lubricants. As such, the synthesized PKO biolubricants exhibits greater thermal stability and can endure greater mechanical stress than petroleum lubricants.

3.3.3 Viscosity index

The viscosity indexes values for the PKO biolubricant synthesized by transesterification with TMP PKBL_T, synthesized by epoxidation-esterification PKBL_E, and petroleum lubricant are 139, 132, and 105, respectively. These values are higher, though close to that of the petroleum lubricant value. Viscosity index is defined as the property of a liquid that helps to resist changes in viscosity with increases or decreases in temperature. In lubricants, it is important to have higher viscosity index values. This is because lubricants with higher viscosity index significantly resist viscosity changes with changes in temperature. The viscosity index of PKO in this work is 180. This value is lower than 185 and 232, reported by Alan et al.1 for PKO and Shaba et al.,16 for palm kernel biolubricant, respectively. In addition, the values of palm kernel biolubricant samples in this study, were both lower than 154, reported by Bahadi et al.12 Furthermore, this value was lower than the 233 viscosity index value for Jatropha oil, reported by Menkiti et al.,4
3.3.4 | Pour points

The temperature, at which oil solidifies enough to resist flow, is the pour point of the oil sample.49 The pour point reduced from 18°C for PKO to −11 and −12°C for biolubricants after double transesterification and epoxidation-esterification processes, respectively. However, the pour point of the petroleum lubricant is −20°C. Hence, these results show that the pour point of the biolubricants and petrolubricant are good enough to permit their use at low temperatures. This was possible because, in the biolubricants, the products thermal resistance was greatly enhanced, due to the fact that the thermally fragile glycerol in the PKO triglycerides were replaced by the TMP backbone and the elongated chain obtained during epoxy ring opening, which are thermally stable. Hence, the significant thermal stability and cold temperature properties of the PKO biolubricants, compared with the PKO. Similar results were obtained by Alan et al.,1 Bahadi et al.,12 Shaba et al.,16 Dandan et al.,51 and Musa49 for the synthesis of biolubricant using PKO; as well as by Ishola et al.14 and Bello et al.,53 for the synthesis of biodiesel from palm olein and PKO, respectively.

3.3.5 | Flash points

Flash point of a fuel can be defined as the temperature at which the fuel can ignite when exposed to a heat source. This is of importance in safe handling, storage and transportation.2 The flash points of PKO and palm kernel biolubricants samples PKBL_T and PKBL_E were 210, 235, and 240°C, respectively. Thus, the products are categorized as nonhazardous products due to their high flash point values. With respect to lubricants, flash point is the temperature at which some vapor is emitted from the lubricant to temporarily ignite a flame.1 Flash point is an important property that must be considered in evaluating the overall flammability hazard of a biolubricant and other similar materials. On the other hand, the flash point of the petroleum lubricant was 220°C. The obtained results were similar to those reported by Alan et al.1 (>210°C), Shaba et al.16 (216°C) and Bahadi et al.12 (320°C), for PKO biolubricants. These flash point values of the obtained PKO biolubricants samples were also in the range of those reported by Aji et al.44 for Neem biolubricant (262°C) and by Bilal et al.52 for Jatropha biolubricant (274°C).

3.4 | FTIR analyses of PKO and PKO biolubricants

The surface chemistry of the extracted PKO sample and the synthesized biolubricants samples were analyzed using FTIR spectroscopy in order to determine the functional groups present in them. The results in Figures 12–14 were analyzed and likened with known signature of identified materials in the FTIR library.54 For all the samples, the main peaks of importance would be highlighted and briefly discussed.
For the PKO sample (Figure 12), the peak at 1047.5 cm\(^{-1}\) is a characteristic of C–O stretching, indicating the presence of alcohol and phenol which are oxygen-containing compounds. Similarly, the peak at 2020.284 cm\(^{-1}\) is characteristic of combination N–H stretching, combination O–H stretching, indicating the presence of organic compounds. Furthermore, the peaks centered at 3002.457 and 3289.143 cm\(^{-1}\) are characteristic of O–H stretching, indicating the presence of carboxylic acids, which are oxygen-containing compounds and water; which results in the easy biodegradability of the oil sample.

Figure 13 shows the FTIR spectrum of synthesized biolubricant confirming that the transesterification reaction between methyl esters and TMP actually occurred. From the spectrum, the peak at 1773.514 cm\(^{-1}\), which falls in the range of carbonyl (C=O) group, indicates the absorption for esters.\(^5\) Similarly, the absorption peaks at 2931 and 2886 cm\(^{-1}\), are within the absorption range for C–H stretching in the hydrocarbon component of the biolubricant. Finally, the broad peak at 3340 cm\(^{-1}\), indicates the presence of O–H groups, indicating the presence of oxygen-containing compounds and water molecules which appear as impurities in the TMP.
Figure 14 shows the FTIR spectrum ranges of the PKO biolubricant obtained by the epoxidation-esterification of the methyl ester sample. This spectrum indicates that epoxidation had taken place. For instance, the appearance of the band peaks of 1100 and 1110–1290 cm\(^{-1}\), which were not present in the methyl ester sample, is characteristics of the epoxide. This is because the functional groups of C–O–C stretching of ethers oxirane ring and Aliphatic C–O stretching of esters, indicate the presence of oxygen-containing compounds.\(^5\) The band corresponding to the carboxylic acid functional group is located at 3509–3533 cm\(^{-1}\). The presence of this group is associated with the ester hydrolysis during epoxidation reaction.\(^2\) Furthermore, the band corresponding to the alkane group is located at 2850–2873 cm\(^{-1}\). The presence of this functional group which was absent in the raw PKO sample indicates the stability of the biolubricant sample obtained by epoxidation-esterification. Finally, predominance of epoxy groups 1100–1290 cm\(^{-1}\), indicates that epoxidation reaction actually occurred.

3.5 | Fatty Acid composition of PKO, and synthesized biolubricant

The fatty acid compositions of PKO, PKO biolubricant synthesized by transesterification with TMP (PKBL\(_{T}\)) and PKO biolubricant synthesized by epoxidation-esterification (PKBL\(_{E}\)) methods are presented in Table 2. From the results in the table, it can be seen that the saturated and unsaturated fatty acid (UFA) compositions of PKO, PKBL\(_{T}\), and PKBL\(_{E}\), were (72.69% and 27.31%), (81.81% and 18.19%), and (83.45% and 16.55%), respectively; indicating that the PKO, PKBL\(_{T}\), and PKBL\(_{E}\) samples were all highly saturated. For the PKO sample, the predominant SFA was lauric acid, with 41.85%, while oleic acid was the predominant UFA, with 17.45%. Similar fatty acid composition was obtained for PKO as reported by Tambun et al.\(^5\) However, there is the need to further improve on the saturation level of the PKO by chemical modification, prior to their possible application as biolubricant.\(^4\)

From the results presented in Table 2, it can be observed that the saturated and UFA compositions of PKBL\(_{T}\), were 81.81% and 18.19%, respectively. These results show that the percentage compositions of the SFA for the PKBL\(_{T}\) sample increased while that of the unsaturated decreased after transesterification of the PKO sample using TMP. This could be due to the heat assimilation. This is because as the heating temperature rises, it results in the fatty acids modification. Hence, the saturation level increased due to decreased prevalence of two or three double bonds.\(^2\) Hence, this results in the PUFAs decrease, with corresponding increase in the SFA.\(^5\) Therefore, transesterification of PKO using TMP increases its saturation level, making the modified oil more usable for use as biolubricant. The predominant SFA in PKBL\(_{T}\) sample was lauric acid, with composition of 47.61%. On the other hand, oleic acid was the predominant UFA, with composition of 16.11%. Similar fatty acid composition was obtained for transesterification with TMP of methyl ester of \(J.\ curcas\) oil.\(^4\)

From the results presented in Table 2, it can be seen that the saturated and UFA compositions of PKBL\(_{E}\) sample, were 83.45% and 16.55%, respectively. From these results (Table 2), it is can be seen that the percentage compositions of the

| Fatty acid | Fatty acid type | PKO (%) | PKBL\(_{T}\) (%) | PKBL\(_{E}\) (%) |
|------------|----------------|---------|-----------------|----------------|
| C8:0 (Caprylic acid) | Saturated | 0.11 | 0.58 | 1.89 |
| C10:0 (Capric acid) | Saturated | 5.08 | 2.13 | 6.71 |
| C12:0 (Lauric acid) | Saturated | 41.85 | 47.61 | 45.36 |
| C14:0 (Myristic acid) | Saturated | 18.34 | 20.42 | 19.08 |
| C16:0 (Palmitic acid) | Saturated | 6.13 | 8.52 | 7.56 |
| C18:0 (Stearic acid) | Saturated | 1.18 | 2.55 | 2.85 |
| C18:1 (Oleic acid) | Unsaturated | 17.45 | 16.11 | 15.44 |
| C18:2 (Linoleic acid) | Unsaturated | 9.86 | 2.08 | 1.11 |
| Saturated fatty acids (%) | 72.69 | 81.81 | 83.45 |
| Unsaturated fatty acid (%) | 27.31 | 18.19 | 16.55 |
| Total (%) | 100.00 | 100.00 | 100.00 |

Abbreviations: PKBL\(_{E}\), palm kernel biolubricant obtained by epoxidation-esterification; PKBL\(_{T}\), palm kernel biolubricant obtained by transesterification of TMPPKO; PKO, palm kernel oil.
SFA increased, leading to decrease in percentage of UFA (oleic acid and linoleic acid). It can be observed that there was a greater increment in the percentage of SFA present in the PKBL_E sample when compared with that of PKBL_T samples. However, like in the case of the transesterification of methyl ester with TMP, there was an increase in the percentage of SFA. This was due to the heating, which caused an increase in temperature and is associated with epoxidation reaction. This is because of the fact that heat treatment of oils or methyl esters, induces fatty acids modifications with two or three double bond. These explanations, substantiates the reason for the decrease and increase of the UFA and SFA, respectively. The improvements in the stability of the PKBL_E and PKBL_T samples were due to increase in SFA samples as evident in Table 2. Just like in the cases of PKO and PKBL_T, the predominant saturated and UFA in PKBL_E sample, were lauric acid and oleic acid, respectively.

4 | CONCLUSION

The production of biolubricant from PKO by the transesterification PKME using TMP and epoxidation-esterification processes was achieved successfully, since no article in the literature has compared the processes for biolubricant production from PKO, which justifies the importance of this study. However, the PKBL_E sample, produced by transesterification of PKBL_T using TMP, conformed better to the stipulated standard, when compared with the PKBL_E sample obtained by the epoxidation-esterification method. That notwithstanding, both samples have shown potential for utilization as biolubricant. The presence of the C–H, OH functional groups identified by the FTIR results, is an indication that the biodegradability of the produced biolubricant samples would be enhanced. The effects of time, temperature and mole ratio significantly affected the transesterification reaction with TMP; while time, temperature and molar ratio of H₂O₂ significantly influenced the epoxidation reaction. Therefore, chemical modifications of PKO for biolubricant production using the aforementioned methods were useful in successful development of PKO biolubricant samples, for possible use in the lubrication of equipment, especially in the food industry, since PKO is an edible oil.

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NOMENCLATURE
AOAC Association of Official Analytical Chemists’
DE diester
EMPKO° epoxy methyl ester (oxirane) of palm kernel oil
FAME fatty acid methyl ester
FTIR Fourier transform infrared
GC gas chromatography
ME monoester
PAA peracetic Acid
PKBL<sub>E</sub> palm kernel biolubricant obtained by epoxidation-esterification
PKBL<sub>T</sub> palm kernel biolubricant obtained by transesterification of TMP
PKME palm kernel methyl ester
PKO palm kernel oil
PKTE palm kernel triester
PUFA polyunsaturated fatty acids
RPM revolutions per minute
SFA saturated fatty acids
TE triester
TMP trimethylolpropane
TMPTE trimethylolpropanetriester
UFA unsaturated fatty acids

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