Research Article

Bio-Based Lubricant Synthesis by Chemical Modification of Linoleic and Oleic Acid Mixture

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A B S T R A C T

The presence of increasing concerns and enforcement of growing regulations over environmental pollution are nowadays at play. The pollution arising from mineral oils is among the major concerns. With the gradual reducation of the world oil reserves, an increasing pressure comes into play for finding sustainable alternatives. Being appealing alternatives, vegetable oils consist of different fatty acids; however, they cannot be applied directly to internal combustion engines owing to their poor oxidation stability and high pour point value. Biolubricants are considered to be a new generation of lubricants, which are renewable and biodegradable and are produced from the chemical modification of vegetable oils. There are few studies investigating the feasibility of using the mixture of fatty acids as biolubricant feedstock. In this study, epoxidation, oxirane ring opening with palmitic acid and p-Toluenesulfonic acid, esterification reaction with octanol, and reaction of the remaining hydroxyl group with stearic acid were applied to modify the mixture of oleic and linoleic fatty acids and produce biolubricant. For this purpose, the IR spectrums of each epoxide, monoester, diester, and triester products were obtained and analyzed. At the end of the experiments, monoester, diester, and triester were obtained with 94 % yield, with 96 % yield, and with 98 % yield, respectively. Eventually, the final product was found with physicochemical properties comparable with the physicochemical properties of the lubricant standard ISO VG10.

1. INTRODUCTION

Nowadays, some phenomena like reduction of oil reserves and the increasing rate of energy price have made economic consumption of energy and its optimization in energy consumptive systems an indispensable task. Thus, renewable energy sources, including wind, solar energy, and biofuels (e.g., biodiesel and bioethanol) have attracted the attention of researchers and scientists. In this regard, the biggest quota of energy use is attributed to internal combustion engines, which are considered to be among the highest energy consumers. Accordingly, it is very important to devise various methods for reducing energy consumption by such engines. It is noteworthy that a third of the energy consumed by these engines is attributable to frictional losses; therefore, it seems reasonable to opt for the lubrication of different parts of such engines for the sake of reducing the degree of friction and

abrasion in their moving parts. This can exert an enormous effect on the reduction of energy and fuel consumption [1]. In fact, engine oil is an integral fluid for engines whose role can be likened to that of blood in the body system in which engine oil accomplishes this important task by lubricating the moving parts of engine. In addition, this fluid is petroleum-based, similar to other oils employed in engine and is also dependent upon costly and non-renewable sources. This has made it unreliable to be used in the long run. On the other hand, vast amounts of lubricants are used every year and their disposal as burned oils to nature brings about environmental pollution [2]. In addition to the harmful effects and air pollution caused by burning the lubricant of the engine fuel, this pollution also arises. Thus, lubricants are nowadays generated from renewable and eco-friendly resources such as vegetable oils, which are referred to as biolubricants and are dependent upon renewable vegetable resources. The desirable biodegradation of such oil ensures that no environmental pollution comes into play by these vegetable oils [3]. The materials existing in vegetable oils are both sustainable and biodegradable in a way

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that may lead to the decrease of global warming. Accordingly, such materials are becoming popular in the market from different perspectives, especially due to their availaibility, economic aspect, and environmentally friendliness [4]. Figure 1 shows different applications of biolubricants in different parts of a vehicle and values of their physicochemical properties including pour point, flash point, and Viscosity Index (VI) [5].

Biolubricants, which are made from chemical modification of vegetable oils, enjoy a good potential to act as an alternative to petroleum-based lubricants [6]. Vegetable oils are available in two types of edible and non-edible oils. The edible oils like canola, soybean, and sunflower oils are utilized for foods and the production of biodiesel and biolubricants. The non-edible oils like kitchen waste cooking oils and seed oils like Jatropha and castor are used for producing biofuels and biodiesels. It should be mentioned that the majority of vegetable oils are obtained from Triacylglycerol, which is a glycerol triester where ester linkage to three glycerol hydroxyl groups links three fatty acids together (Figure 2).
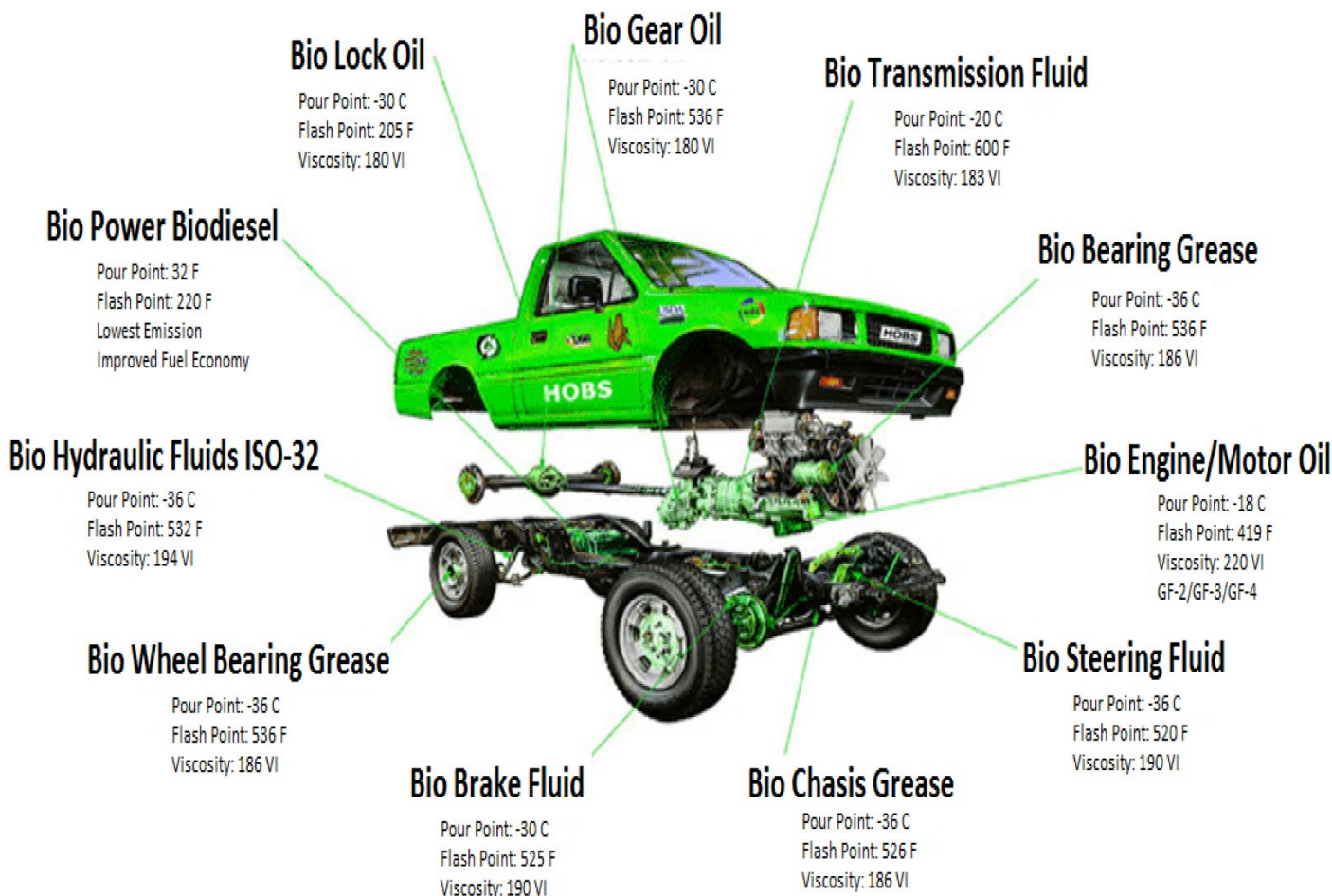

Figure 1. Different applications of biolubricants in various parts of a vehicle [5]
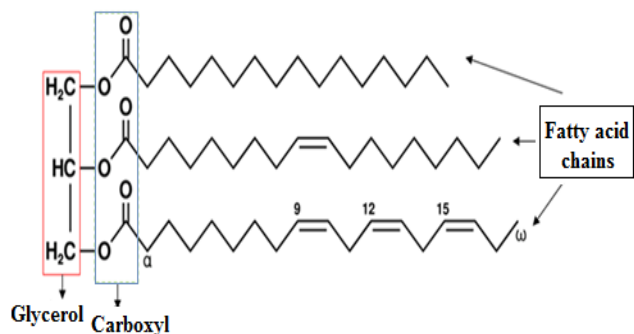

Figure 2. Triacylglycerol structure of vegetable oils

Two main factors, including the glycerol structure of Triacylglycerol and the composition and distribution of fatty acids are determinnets of the chemical and physical properties of vegetable oils [7]. Indeed, the Triacylglycerol structure is appropariate for lubrication because combination of carboxyl group and long-chain fatty acids with each other leads to highly strong lubrication. The presence of 14 to 22 carbon atoms with varying saturations in the majority of fatty acids of vegetable oils is evident. The delineation of higher interaction of triacylglycerol molecules with metal surfaces compared to that of hydrocarbons has made triacylglycerol molecules a highly strong lubricant to metal surfaces. Therefore, they contribute to the reduction of abrasion and friction [8].

However, it is not possible to use vegetable oils as lubricants without any intermediacy. As shown in Figure 3, it is evident that double bonds in the fatty acids of triacylglycerol are prone to reaction with oxygen in the air. Thus, some oxidation comes into play and as a result, it makes the insoluble materials deposited and the value of acid and oil viscosity raise. In the same way, it is possible to conveniently separate β hydrogen in glycerol section from the molecule structure and convert ester into acid and olefin. Low stability against oxidation and low pour point can be enumerated as the

negative effects of direct employment of vegetable oils as lubricants [9].

To date, different methods have been used by researchers to see whether or not vegetable oils can be used as lubricants. In this regard, some reserachers have examined polyols like Trimethylolpropane having a vegetable oil base and glycerol structure as an alternative to raise the stability of oxidation [10-13]. Some others have employed epoxidation as an alternative to stabilizing vegetable oils against oxidation [6, 14]. The hydrogenation, transesterification reaction, and genetic change of vegetable oils with long-chain alcohols, that is, 8-15 carbon atoms, were examined, as well [1, 15-17]. As a major additive reaction with double bonds, epoxidation results in forming oxirane rings and consequently, the oxidation stability of vegetable oils increases (Figure 3).
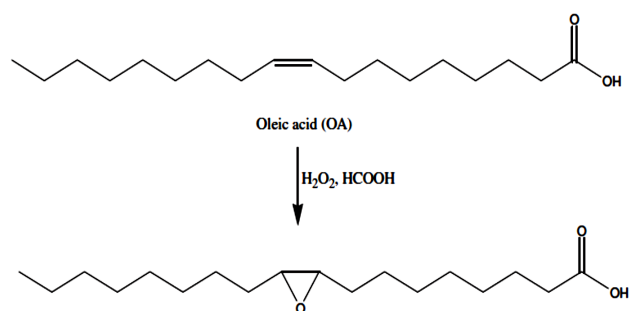

Figure 3. Epoxidation reaction

Considering the fact that natural compounds are available as fatty acid mixtures, conducting a survey on applied reactions in the mixture of these compounds supplies less expense for initial consumable ingredients than purified fatty acids and their following reactions. Therefore, a mixture of oleic and linoleic acid was used in this study. Through a chain of reactions, such as epoxidation, oxirane ring opening, and the esterification of epoxide, monoester, diester, and triester compounds were synthesized and analyzed by the functional FTIR spectroscopy. Indeed, most of previous research studies have focused on transesterification reaction as the most common route for biolubricant production. However, this paper addresses a multi-stage production process including the above-mentiond stages.

2. EXPERIMENTAL

2.1. Meterial

The mixture of oleic and linoleic acids was purchased from Alandshimi Company, Iran. Magnesium sulfate 97 %, sodium chloride 99 %, para toluene sulfonic acid 98 %, and octanol 99 % were purchased from Sigma Company, while formic acid 88 %, hydrogen peroxide 30 %, palmitic acid 99 %, and stearic acid 99 % were provided from Merck Company. Antonpaar viscometer SVM-3000, ignition point determiner (Model: P611A, Analysis Company, Belgium), rotary evaporator (Model: STRIKE 202, Italy), and overhead mixer (Wreke Eurostar Digital (S1), IKA Germany) in the speed range of 50-2000 rpm were also applied in this study. Similarly, the cloud and pour point refrigerator (Model: STEA assembled in England), spectroscope (FTIR) (Model: Tensor 27, Brucker Germany) were employed in the course of this study.

2.2. Procedure

2.2.1. Preparation of epoxide from mixed fatty acids (oleic acid and linoleic acid)

In order to prepare epoxide, 30 g of mixed fatty acids was poured into a beaker (100 ml), which had been placed in an ice bath until it reached 4 °C. Then, 28 ml of formic acid 88 % was gradually added. About 16 ml of hydrogen peroxide 30 % was slowly added using a burette at 0-4 °C. Next, the mixing process began at a speed of 800 rpm for 2-5 hours till a white precipitate was formed. To extract formic acid, the resulting precipitate was washed five times with the distilled water (50 ml) and was then filtered using a vacuum filter. After filtration, the precipitate was placed in a vacuum desiccator to dry and allow for the discharge of the extra formic acid.

2.2.2. Preparation of monoester from fatty acid epoxide

To perform this examination, 31 g of fatty acid epoxide, 6 g of palmitic acid, and 5 g of para toluene sulfonic acid were dissolved in 70 ml of toluene. This mixture was, then, poured into a two-necked flask (capacity: 250 ml). The reaction took place through stirring at 70-80 °C for 90 minutes. Thereafter, the temperature was raised to 90-100 °C and the admixture of the compounds was refluxed and mixed for 3 hours. When the reaction was over, the flask contents were replaced into a separatory funnel and remained there for 12 hours at ambient temperature. Next, the funnel's contents were washed 5 times using 50 ml of distilled water until the wash water had a neutral pH. The organic phase was, first, dehydrated using magnesium sulfate and was then filtered via vacuum filtration. Following the filtration, toluene was separated from the admixture using rotary evaporator (temperature: 90 °C; speed: 100 rpm).

2.2.3. Preparation of diester from fatty acid monoester

First, 10 g of fatty acid monoester and 27.8 g of octanol were poured into a two-necked flask (capacity: 100 ml), while 4 drops of sulfuric acid (10 %) were added as a catalyst. The reaction lasted for 10 hours (temperature: 60 °C). When the reaction was over, the flask contents were replaced into a separatory funnel (capacity: 250 ml). Then, 50 ml of normal hexane was added to separate and purify the product. Following the separation of the aqueous phase from the organic phase, 5 ml of saturated solution of sodium hydrogen carbonate was added to the organic layer to separate the lower phase (pH = 10). After that, 10 ml of saturated solution of sodium chloride (2 times) was added to the organic layer and the lower phase was separated once more (pH = 8). Finally, the organic phase was dehydrated using magnesium sulfate and was filtered by vacuum filtration. After being filtered, normal hexane was extracted from the recent admixture using rotary evaporator (temperature: 80 °C; speed: 100 rpm).

2.2.4. Preparation of triester from fatty acid diester

At the outset, 5 g of fatty acid diester and 5 g stearic acid were poured into a two-necked flask (capacity:100 ml), while 2 drops of sulfuric acid (10 %) were added as a catalyst. The reaction took place, while the mixture was being mixed and refluxed for 10 hours at 100-110 °C. At the end of the reaction, the flask contents were placed into a separatory funnel (250 ml) and 20 ml of normal hexane was added to it so as to separate and purify the product. After the separation

of the aqueous phase from the organic phase, 5 ml of the saturated solution of sodium hydrogen carbonate (2 times) was added to the organic layer so as to separate the lower phase (pH = 10). Afterwards, 10 ml of the saturated solution of sodium chloride (2 times) was added to the organic layer in order to separate the lower phase (pH = 7). At the end, the organic phase was dehydrated and filtered using magnesium sulfate and vacuum filtration, respectively. After filtration, normal hexane was extracted from the recent admixture using rotary evaporator (temperature: 80 °C; speed: 100 rpm).

The synthesis process of triester from oleic acid and linoleic acid is given in Figures 4 and 5.

2.3. Standard tests to identify lubricant physicochemical properties

2.3.1. Flash point

Flash point is the lowest temperature at which the sample is evaporated adequately and the combination of evaporated sample and air produces a flammable admixture. Thus, when the flame is drawn to this gas, it ignites for a moment and

then, dies out. The flash point should be high so as to lower the risk of volatility at high temperatures and increase the level of safety [18, 19]. During this examination, the flash point was measured by a flash point apparatus in accordance with ASTM D-93 standards.

2.3.2. Pour point

Pour point was measured using specific equipment in accordance with ASTM D-79. In order to measure the cloud point, the sample was poured into a test tube and placed in the measuring instrument. The contents were refrigerated in butyl glycol bath gradually. As soon as the sample got opaque or a milky cloud appeared, the temperature displayed on the monitor was written down. If the sample gets cooler, the process of freezing starts and the temperature in this state is defined as the freezing point. If the tube is taken out and held in a horizontal position, after some time, the sample gets warmer and begins to flow. The temperature in this state is defined as the pour point, which is a few grades above the freezing point.
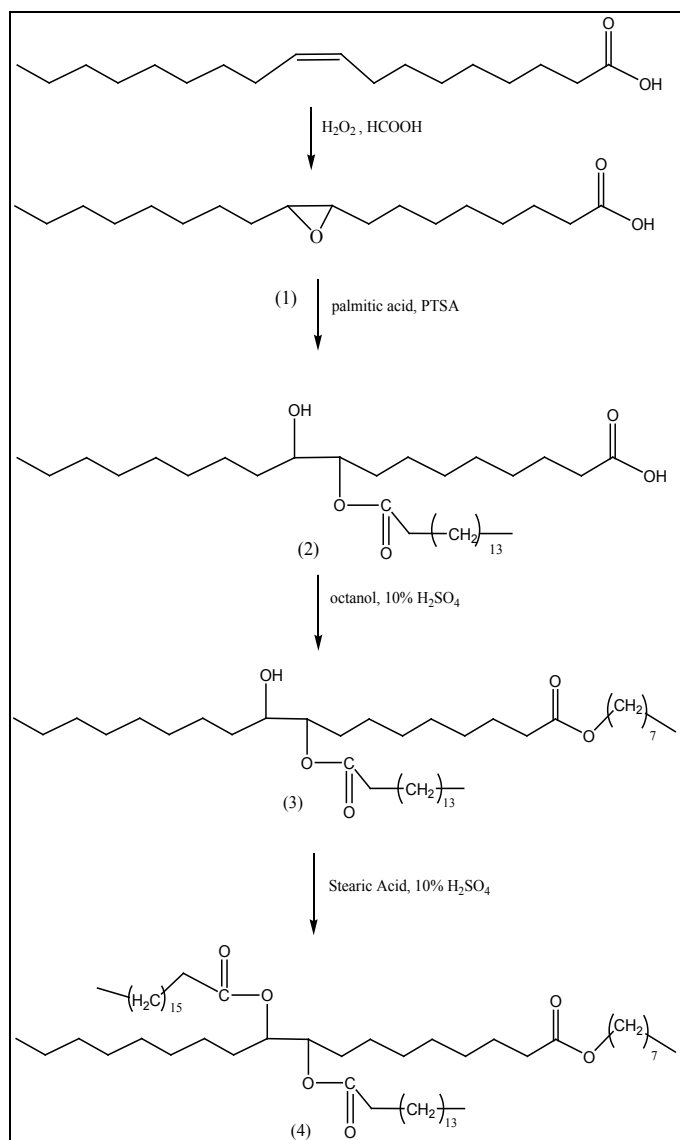
Figure 4. The process of preparing triesters from oleic acid: step (1): epoxidation, step (2): oxirane ring opening, step (3): esterification using octanol and step, (4): esterification using stearic acid

Figure 5. The process of preparing triester from linoleic acid, step (1): epoxidation, step (2): oxirane ring opening, step (3): esterification using octanol, and step (4): esterification using stearic acid
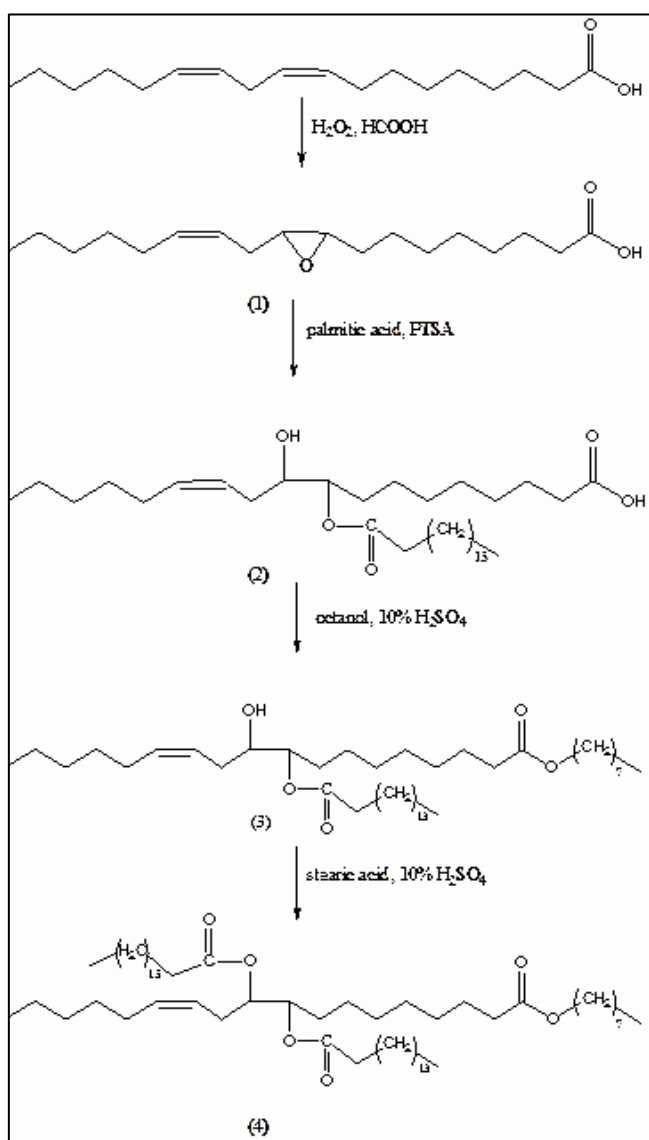
2.3.3. Viscosity index

Viscosity is defined as the resistance of fluid to moving and flowing. In order to produce a lubricant film with desired width, viscosity should be high; otherwise, it increases friction. Consequently, the dissipation power and abrasion grow.

Viscosity index is a dimensionless contract parameter that identifies the extent of kinematic viscosity which depends on the temperature. Low viscosity index represents wide fluctuations in viscosity in response to temperature changes, bringing about a steep slope in the viscosity-temperature curve. The more the viscosity index is, the less the viscosity fluctuates will be in response to temperature changes. Under these conditions, the slope of the viscosity-temperature curve remains constant and the lubricant provides a better function [18].

3. RESULTS AND DISCUSSION

3.1. IR spectrum analysis

In this study, 863 negligible cm^{-1} and 924 cm^{-1} peaks in mixed fatty acid (oleic acid and linoleic acid) epoxide correlated with oxirane ring carbons and 2927 cm^{-1} and 2852 cm^{-1} peaks correlated with the stretching vibration of CH_2 (aliphatic carbons). Analysis of epoxide spectrum also indicates that 1711 cm^{-1} and 1466 cm^{-1} peaks correlated with the stretching vibration of carbonyls and the flexion vibration of CH_2, respectively. Also, 721 cm^{-1}, 1412 cm^{-1}, and 3343 cm^{-1} peaks belonged to the oscillation vibration of CH_2, flexion, and stretching vibrations of O-H, respectively (Figure 6).

Regarding monoesters, 2924 cm^{-1} and 2854 cm^{-1} peaks were related to the stretching vibration of CH_2, while 1709 cm^{-1} and 1726 cm^{-1} peaks correlated with the stretching vibration of carbonyl ester group and carboxylic acid section, which represented the oxirane ring opening phenomenon. In monoester spectrums, 1098 cm^{-1} and 1175 cm^{-1} peaks correlated with the stretching vibration of C-O section of an ester; and 1290 cm^{-1}, 1376 cm^{-1}, and 1464 cm^{-1} peaks correlated with the stretching vibration of C-O section of acid, symmetric flexion vibration of CH_3, and flexion vibration of CH_2, respectively (Figure 7).
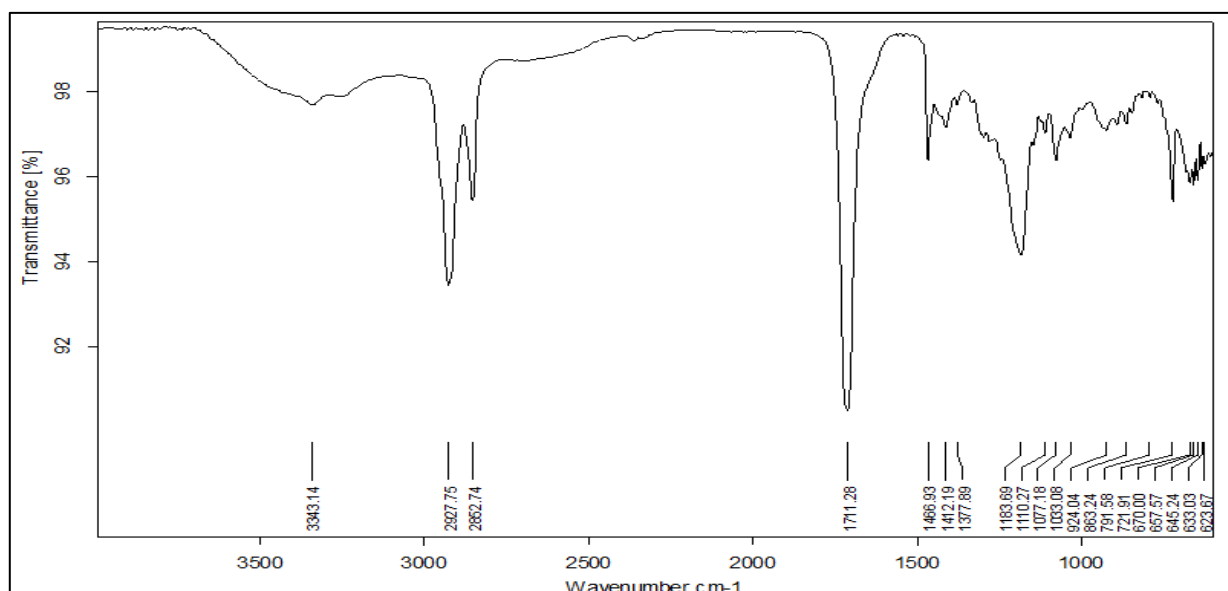

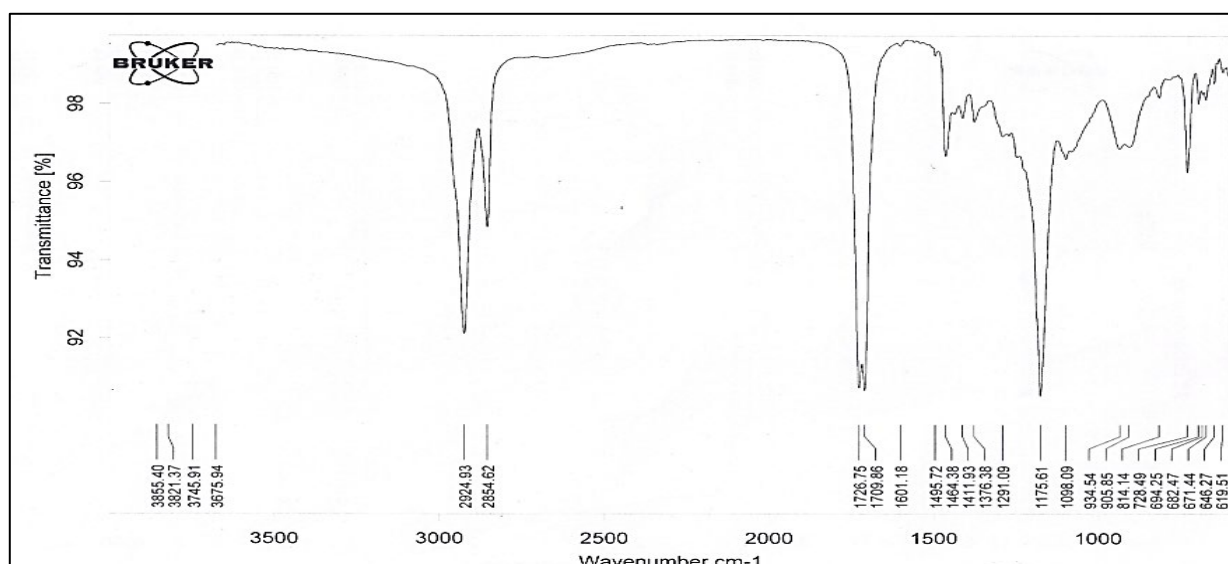**Figure 6.** IR spectrum for the mixed fatty acid epoxide

Figure 7. IR spectrum for fatty acid monoester

The analysis of the diester spectrums indicates that 3350 cm^{-1} peaks belonged to OH stretching vibration, 2925 cm^{-1} and 2855 cm^{-1} peaks correlated with CH$_2$ stretching vibration, and 1737 cm^{-1} peak correlated with carbonyl stretching vibration. Moreover, 1117 cm^{-1}, 1174 cm^{-1}, and 1246 cm^{-1} peaks correlated with the stretching vibrations of C-O part of an ester. Considering this spectrum, 1465 cm^{-1}, 1377 cm^{-1}, and 722 cm^{-1} peaks correlated with the flexion vibration of CH$_2$, symmetric flexion vibration of CH$_3$, and oscillation vibration of CH$_2$, respectively (Figure 8).

In triester spectrums, 1738 cm^{-1} peak belonged to carbonyl stretching vibration and the absence of OH peak in 3400 cm^{-1} area indicated the triester structure. On the other hand, 2923 cm^{-1} and 2853 cm^{-1} peaks correlated with the stretching vibrations of CH$_2$, and 1465 cm^{-1} and 1377 cm^{-1} peaks correlated with the flexion vibration of CH$_2$ and symmetric flexion vibration of CH$_3$, respectively. Peaks in 1116 cm^{-1}, 1170 cm^{-1}, and 1245 cm^{-1} areas correlated with the stretching vibration of C-O section of an ester (Figure 9) [6, 16].
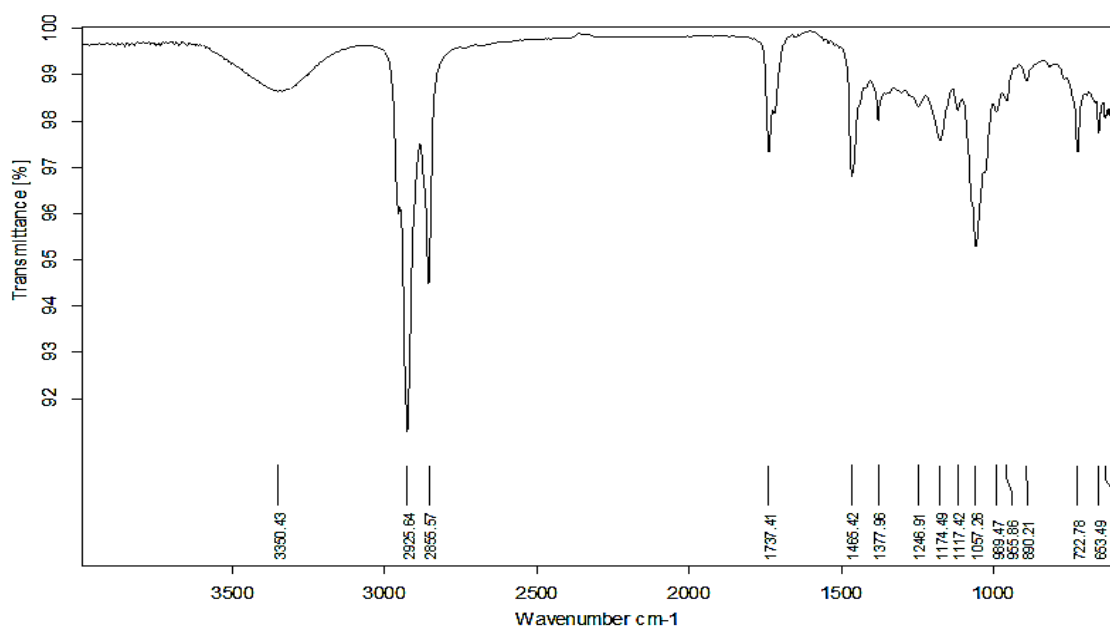

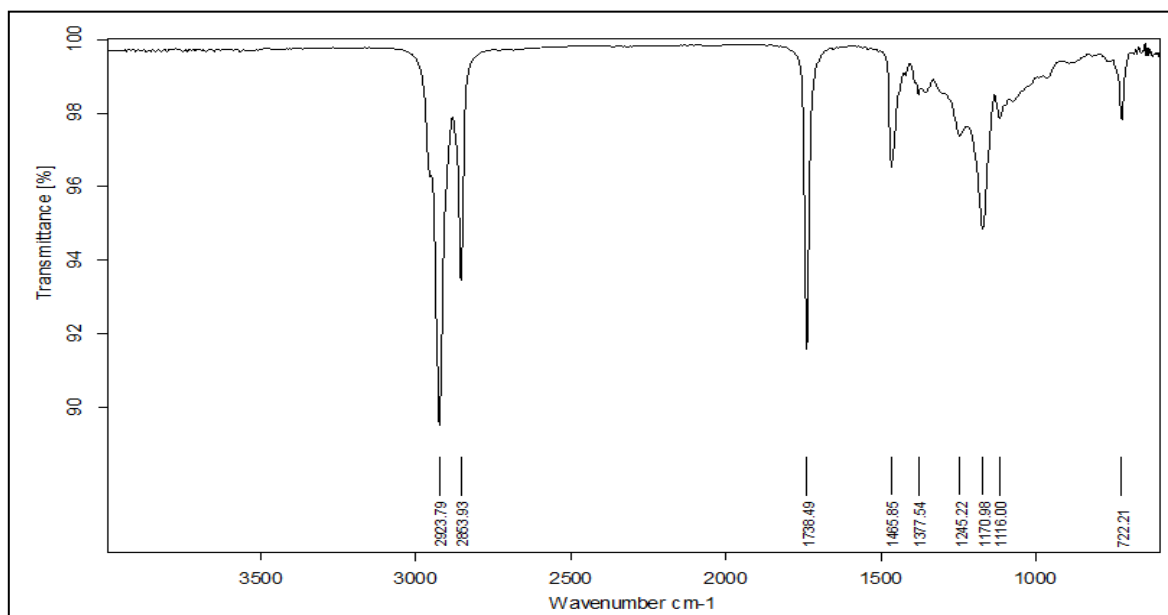
Figure 8. IR spectrum for fatty acid diester

Figure 9. IR spectrum for fatty acid triester

Based on these spectrums, it can be concluded that the elimination of 863 cm^{-1} peak in Figure 6 was related to oxirane ring opening and the formation of 1709 cm^{-1} and 1726 cm^{-1} peaks in Figure 7 was related to the production of ester groups. In Figure 8, the 3350 cm^{-1} peak was related to the formation of an OH group, which was eliminated during the production process of the triester (Figure 9). The results

obtained in this section are comparable with findings obtained by Bashiri et al. [6].

3.2. Physicochemical test results

The physical properties of the synthetized diester and triester and their comparison with lubricant ISO standard are presented in Table 1. The ISO VG standard is an international

standard for lubricants classification and indicates the main physicochemical qualifications of a standard lubricant. It can be observed that the physicochemical properties of the obtained biolubricant (triester) are compatible with the ISO VG 10 standard and the triester has better values in some features like VI and flash point, while there is a negligible difference between pour point values of the triester and the ISO standard.

Table 1. Physical properties of synthetized diester and triester

	Viscosity, 40 °C, cSt	Viscosity 100 °C, cSt	VI	Pour point °C	Flash point °C
Diester	9.89	2.63	90.27	-24	153
Triester	12.50	3.3413	138.77	-27	238
ISO VG10	> 9	> 2.68	104	-30	154

According to the results of lubricant standard tests, esters exhibited better lubricant behavior than diesters at low temperatures. There are two possible reasons for this phenomenon:

1- The presence of side chains attached to the main fatty acid skeleton prevents the individual molecules from accumulation. In the presence of stearic acid side chains in triester structure, spatial steric hindrance is greater compared to diesters. As a result, the pour point of fatty acid triester is lower than that of diester.

2- Diesters have one hydroxyl group less than monoesters and the absence of a hydroxyl group in triester's structure results in the reduction of hydrogen bond quantity and pour point value.

Lubricant performance, as a friction reducer, greatly depends on its viscosity. In fact, lubricant viscosity should be high; otherwise, it leads to an increased friction; as a result, greater abrasion and dissipation may occur.

Moreover, to ensure the safety and lower the possibility of evaporation at high temperatures, the flash point should be high. The flash points of diesters and triesters are 153 °C and 238 °C respectively. Triester compounds own a higher flash point than diesters since they consist of one more side chain.

4. CONCLUSIONS

In this paper, a multi-step synthesis process through a chain of reactions including epoxidation, oxirane ring opening, and the esterification of epoxide was applied to biolubricant (triester) production and the final product was analyzed by the functional FTIR spectroscopy. The calculated efficiencies of epoxidation reactions, oxirane ring opening, esterification by octanol, and esterification by fatty acids were 96.6, 94, 96, and 98 %, respectively. These values clarify the fact that applying mixed fatty acids can be more cost-effective. Moreover, considering the low cost of linoleic and oleic acid mixtures compared with mineral lubricants and the fact that these products are biodegradable and generate lower environmental pollution, their production was found to be justifiable.

According to kinematic viscosity values for diesters and triesters at 40 °C and 100 °C, the viscosity indexes for diesters and triesters were determined as 90.27 and 138.77, respectively. These values describe a negligible viscosity fluctuation in response to temperature changes. Finally, it can be concluded that triester derived from fatty acids, due to their greater viscosity index than diesters, can act as an ideal substitute for petroleum-based lubricants.

5. ACKNOWLEDGEMENT

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