

Esterification and Evaluation of Palm Oil as Biolubricant Base Stock[†]

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Refined, bleached, and deodorized palm oil become one of the potential renewable resources in biolubricant application. However, palm oil cannot be used directly as biolubricant because of its low oxidative stability. It is due to the presence of oxidation active site α -hydrogen in a glycerol backbone structure. This drawback can be overcome by molecule structural redesign through chemical modification process such as esterification with polyhydric alcohol. The synthesis of biolubricant was carried out via esterification of palm oil fatty acids with trimethylolpropane in a mole ratio of 3.5:1, 1.34% sulphuric acid as the catalyst at 154°C for 5.97 h. Gas chromatography equipped with a flame ionization detector was used to determine the percentage of ester composition in palm oil trimethylolpropane (POTMP) ester. The structure of POTMP ester was confirmed by Fourier transformation infra-red (FTIR), proton and carbon Nuclear magnetic resonance ($^1\text{H-NMR}$ and $^{13}\text{C-NMR}$) spectroscopy. The result showed that POTMP ester had successfully synthesized with 92% yield, 100% composition of triesters and POTMP existed in liquid form at room temperature. The existence of the ester functional group evidenced by FTIR at 1740 cm^{-1} , the chemical shift of $^1\text{H NMR}$ at 2.29-2.36 ppm and $^{13}\text{C NMR}$ at 173.53-173.57 ppm. Physicochemical properties analysis showed that POTMP ester had oxidative stability at 183°C, pour point at 8°C, the flash point at 290°C and 158 of viscosity index which made POTMP plausible to be used in many industrial lubrication applications.

Key words: Esterification; oxidative stability; palm oil fatty acids; trimethylolpropane

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The development of products from renewable resources has emerged in the last decade due to the increasing concerns over the use of petroleum-based products which cause progressive reduction of fossil fuels and concerns over the impact on the environment [1, 2]. Use of renewable resources in the industry is vital to ensure sustainable development. In the field of lubricant, plant oils are found to be the best alternative source not only because they are renewable raw materials, but also because they are biodegradable and non-toxic compared to conventional mineral-based oils [2, 3, 4]. Plant oils exhibit excellent lubricity with high viscosity index and biodegradability properties which they are being more closely examined as base stock for lubricants and functional fluids [5, 6, 7].

However, most plant oils have several disadvantages that will cause instability and limit their application in the lubricant industries. Plant oil formulations provide many challenges, such as low oxidative stability [2, 8]. It is due to the presence of oxidation active sites α -hydrogen in glycerol backbone structure and a double bond in the

unsaturated fatty acid structure which will cause plant oil to become unstable at higher temperature [9]. The α -hydrogen is easily removed from triacylglycerol molecules and it will form acid and olefin (unsaturated compounds). The resulting unsaturated compounds will undergo polymerization and will cause the formation of the precipitate that will increase the viscosity of plant oil [10].

Scientific approaches need to be taken to overcome the oxidative stability drawback that incurs in the plant oil. Molecule structural redesign through chemical modification of plant oils becomes one of the attractive ways to solve this problem. One modification that can be used is esterification with polyhydric alcohol such as neopentyl glycol, trimethylolpropane (TMP) and pentaerythritol (PE) which will replace glycerol backbone in plant oil structure. The advantage of using polyhydric alcohol is the absence of α -hydrogen which will enhance the thermal and oxidative stability of the lubricant at high temperature [11, 12, 13]. Among these polyhydric alcohols, TMP is well known polyhydric alcohol used in lubrication fields. Moreover,

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TMP has a relatively low melting point and branched which are an important characteristic of biolubricant base oil production [14]. TMP is polyhydric alcohol that has three hydroxyl groups.

Recently, refined, bleached and deodorized (RBD) palm oil which abundantly available all over Malaysia become one of the potential plant oils that can be used as biolubricant base stock [15, 16]. There are many studies reported on the production of biolubricant from palm oil and TMP. The production of biolubricant *via* transesterification of palm oil methyl ester (POME) with trimethylolpropane (TMP) was reported by Yunus *et al.* [17]. The synthesis of palm oil TMP ester was carried out in a mole ratio of 3.9:1 (POME:TMP), 120°C, 20 mbar pressure, and 0.8% of sodium methoxide catalyst in 1 hour and the reaction has successfully produced 98% palm oil TMP triesters. The optimization of transesterification of POME and TMP was also reported by Sulaiman *et al.* [18]. The optimum conditions obtained were 3.8:1 (POME: TMP) mole ratio, 120°C, 20 mbar pressure and 0.9% of sodium methoxide catalyst in 2 hours. The result showed 86% of palm oil TMP triesters were successfully synthesized.

Most of the studies used palm oil methyl ester (POME) as a starting material in transesterification with TMP. There is less study of the uses of palm oil fatty acids (POFAs) as a starting material in the esterification process. Hence, in this paper, the esterification of POFAs with TMP to produce palm oil TMP (POTMP) ester as shown in Figure 1, is reported. Fatty acid composition of POFAs and POTMP ester, as well as the percentage of ester composition in POTMP ester would be analyzed by GC-FID, while FTIR and NMR spectroscopy confirmed the structure of POTMP ester. Physicochemical properties of POTMP ester were examined by using several tests such as oxidative stability, pour point, flash point, and viscosity index.

MATERIALS AND METHODS

Materials

RBD palm oil was obtained from Jomalina Refinery, Teluk Panglima Garang, Selangor, Malaysia. Trimethylolpropane (97%) was purchased from Sigma Aldrich. Sulphuric acid, toluene, ethyl acetate, sodium bicarbonate, sodium chloride, sodium sulphate, hydrochloric acid, *n*-hexane, potassium hydroxide, and ethanol were purchased from System.

Hydrolysis of RBD Palm Oil

Hydrolysis process involves two stages which are saponification and acidification. In saponification, 50

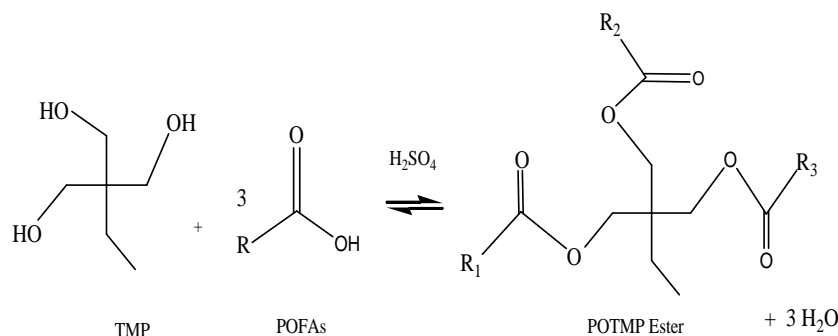
gram of RBD palm oil was mixed with alkaline ethanol and heated at 60°C for 2 hours. Then, the mixture underwent acidification process in which hydrochloric acid 6N was added to neutralize the alkaline solution. The washing was continued using distilled water and hexane as solvent. The product which is palm oil fatty acids (POFAs) was kept for overnight by adding anhydrous sodium sulphate. The product was filtered by Whatman No 1 filter paper and the solvent used was isolated by rotary evaporator at 70°C. The product (POFAs) was analyzed using GC-FID.

Esterification of POFA and TMP

The esterification process was conducted by mixing palm oil fatty acids (POFAs), and TMP in a mole ratio of 3.5:1. 1.34% of sulphuric acid and 100 ml toluene were added to the mixture. The mixture was heated at 154°C for 5.97 hours. The product (POTMP ester) was neutralized with sodium hydrogen carbonate solution, sodium chloride solution, and ethyl acetate. The product was kept for overnight by adding anhydrous sodium sulphate. The product was filtered by Whatman No 1 filter paper and the solvent used was isolated by rotary evaporator at 70°C. The POTMP ester was analyzed using GC-FID, FTIR, and NMR.

Fatty Acid Composition Analysis

Fatty acid composition of RBD palm oil, POFAs, and POTMP ester were analyzed using GC-FID (column BPX-70). The fatty acid methyl esters (FAMES) were prepared using two methods: base catalyzed for RBD palm oil and POTMP ester and acid catalyzed for POFAs. For base catalyzed, FAME was prepared by blending 0.1 ml of the sample with 1 ml of hexane. 1 ml of sodium methoxide solution (1.55 gram NaOH and 50 ml of methanol) was added to the oil solution and stirred the solution vigorously using Vortex stirrer for 10 seconds. The solution was allowed to stand for 10 minutes to separate the clear solution of FAME from the cloudy aqueous layer. The upper FAME layer was slowly collected and injected into GC for analysis. For acid catalyzed, 1 gram of POFAs was mixed with 3.75 ml methanol. 0.75 ml reagent mixture (5 ml methanol and 1.25 ml concentrated hydrochloric acid (36.5%)) was added, followed by 0.75 ml of toluene. The mixture was heated at 65°C for 1.5 hours. The mixture then transferred into separation funnel. 7.5 ml of hexane and 5 ml distilled water were added to the mixture. The mixture was allowed to stand to separate out two layers. The upper layer was slowly collected and dried using anhydrous sodium sulphate for overnight. The sample was filtered and injected into GC for analysis.



R= Mixed palm oil fatty acids (myristic, stearic, palmitic, oleic and linoleic acid)

Figure 1. Esterification of palm oil fatty acids (POFAs) with trimethylolpropane (TMP).

POTMP Ester Analysis

The percentage of ester composition in POTMP ester was determined using GC-FID (column DB-5HT). The sample was prepared by mixing 0.3 μl of POTMP ester with 1 ml of ethyl acetate. The structure of POTMP ester was confirmed using FTIR and NMR. FTIR spectra was recorded on a Perkin Elmer Infrared Spectrophotometer in the range 500-4000 cm^{-1} . ^1H and ^{13}C NMR were recorded on JEOL-ECP 400 spectrometer (400MHz ^1H /100.61MHz ^{13}C) using CDCl_3 as a solvent.

RESULTS AND DISCUSSION

RBD palm oil is golden yellow and exists in semi-solid form at room temperature. hydrolysis process

was carried out to separate fatty acid from the glycerol backbone. The percentage yield of hydrolysis was 95%. Figure 2 shows the GC-FID (column BPX-70) chromatogram of fatty acid compositions in RBD palm oil (before hydrolysis) and POFAs (after hydrolysis). The fatty acid composition has been simplified in Table 1. The major fatty acid composition in POFAs is oleic acid with 43.5%, followed by palmitic acid (42.5%), linoleic acid (9.5%), stearic acid (3.7%) and myristic acid (0.8%). The percentage of unsaturated fatty acids in POFAs is 53% and saturated fatty acids are 47%. The fatty acid composition of both samples are approximates close to the value of fatty acid composition of palm oil reported by Chowdhury *et al.* [19] in 2007.

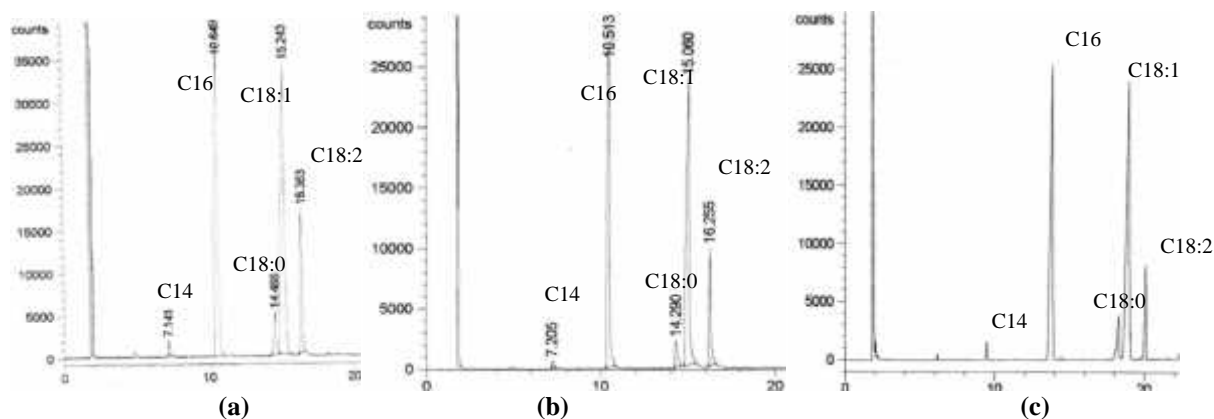


Figure 2. GC-FID (column BPX-70) chromatogram (a) RBD palm oil; (b) POFAs; (c) POTMP ester.

Table 1. Fatty acid composition (percentage) of RBD palm oil, POFAs and POTMP ester.

Fatty acid compositions	RBDPO	POFAs	POTMP Ester	Reference ^a
Myristic acid (C14)	0.6	0.8	0.9	1.2
Palmitic acid (C16)	41.9	42.5	41.5	41.8
Stearic acid (C18)	3.6	3.7	5.1	3.4
Oleic acid (C18:1)	44.8	43.5	45.6	41.9
Linoleic acid (C18:2)	9.1	9.5	6.9	11

^aChowdhury *et al.* [19]

The esterification process was carried out between POFA and TMP in the presence of sulphuric acid as a catalyst to produce POTMP ester. The percentage yield of POTMP ester was 92%. Figure 2(c) shows fatty acid compositions in POTMP ester, and the percentage composition of fatty acids also has been simplified in Table 1. From the GC result, it showed that POTMP ester contained 47.5% saturated acid and 52.5% unsaturated fatty acid. The higher percentage of unsaturated fatty acids made POTMP ester exists in liquid form at room temperature.

Figure 3 shows the GC-FID (column DB-5HT) chromatogram of POTMP ester. There are three types of esters that will be produced from the esterification process which are monoesters, diesters and triesters. In the esterification process, monoesters and diesters were formed as intermediate products towards the completion of the reaction producing POTMP triesters [20]. Type of esters formed were identified by making comparison with the TMP ester formation in the previous study reported in 2002 by Robiah *et al.* [21].

In this study, the esterification of POFA and TMP had successfully produced 100% of triesters. It showed all hydroxyl groups in TMP were successfully esterified with POFA. This result was

supported by low hydroxyl value of POTMP ester which was 26.12 mg KOH/g compared to hydroxyl value of TMP which was 1254 mg KOH/g. The hydroxyl value of POTMP ester approximates close to the hydroxyl value of RBD palm oil which is 19 mg KOH/g.

FTIR Analysis

The formation of POTMP ester was confirmed by FTIR analysis. The comparison between FTIR spectra of POFA (before esterification) and POTMP ester (after esterification) is shown in Figure 4. FTIR spectrum of POTMP ester shows the appearance of C=O ester stretching peak at 1740 cm^{-1} . On the other hand, FTIR spectrum of POFA shows the stretching of C=O group of carboxylic acid appeared at 1696 cm^{-1} . The stretching of C-O ester group also exist at 1155 cm^{-1} and 1235 cm^{-1} in POTMP ester spectrum. This is due to the combination of sp^2 and sp^3 C-O stretching vibrations of ester. While the sp^2 C-O stretching of carboxylic acid exist at 1291 cm^{-1} and 1247 cm^{-1} in POFA spectrum. The shifted wavenumbers for both spectra showed that POFA was successfully esterified with TMP to form POTMP ester. In addition, the -OH stretching peak which initially existed in POFA spectrum ($3000\text{--}3400\text{ cm}^{-1}$) was no longer visible in POTMP ester spectrum.

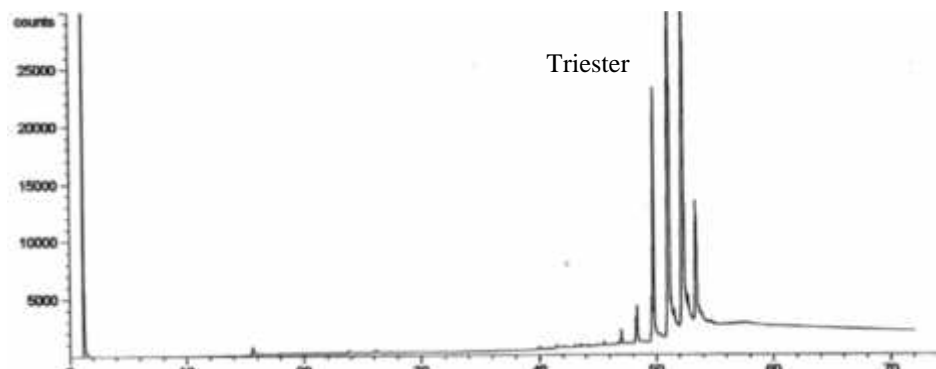


Figure 3. GC-FID (column DB-5HT) chromatogram of POTMP ester.

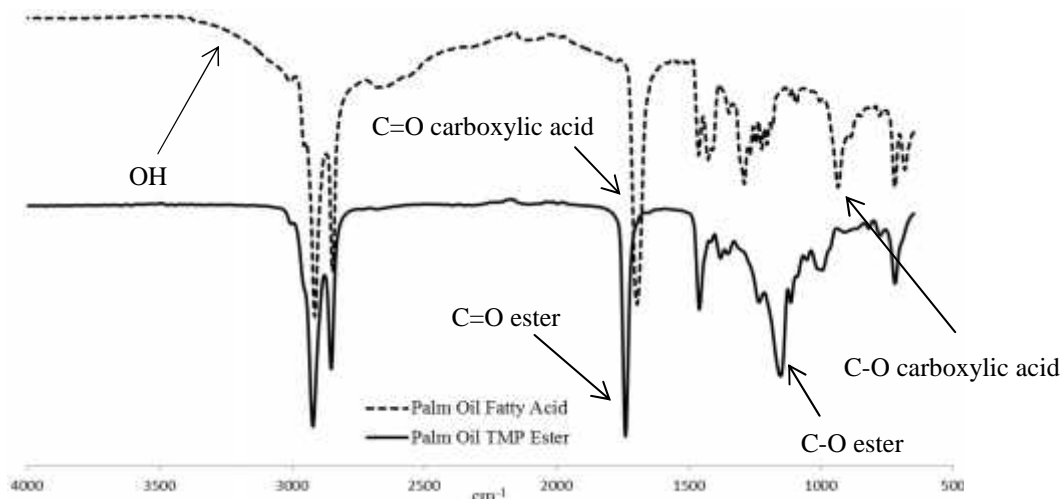


Figure 4. FTIR spectra of POFA and POTMP ester.

NMR Spectroscopy

The structure of POTMP ester was analyzed by NMR spectroscopy. Figure 5 shows ^1H NMR spectrum of POTMP ester. In ester structure, there are two type of protons which are proton of $\text{CH}_2\text{-O}$ and proton of $\text{CH}_2\text{-C=O}$. In this study, NMR spectrum shows proton of $\text{CH}_2\text{-O}$ for POTMP ester was detected at 4.02-4.03 ppm, and proton of $\text{CH}_2\text{C=O}$ was detected at 2.29-2.36 ppm. These values were in agreement with the reference which is the chemical shift for proton $\text{CH}_2\text{-O}$ is at 3.5- 4.8 ppm while the chemical shift for proton $\text{CH}_2\text{C=O}$ is at 2.1- 2.5 [22].

POTMP ester consists of the mixture of acyl groups such as myristate, palmitate, stearate, oleate, and linoleate. The presence of unsaturated fatty acids was determined by identification of the alkene group. In alkene group, there were two types of protons identified, which are proton of the double bond (-C=C-H) at chemical shift 4.5-6.5 ppm and proton of methylene group which bound to double bond (-C=C-C-H) at chemical shift 1.6-2.6 ppm [22]. From Figure 5, the existence of both protons can be detected at chemical shift 5.33 -5.39 ppm (-C=C-H) and 1.99-2.06 ppm (-C=C-C-H). The presence of

both peaks proved the existence of unsaturated fatty acids in POTMP ester.

Spectrum ^{13}C NMR for POTMP ester is shown in Figure 6. From Figure 6, it shows that there is the existence of carbon carbonyl ester (C=O) in POTMP ester at a chemical shift of 173.53-173.57 ppm. This value was in agreement with Pavia *et al.* [22] which showed that the chemical shift for carbon carbonyl ester (C=O) was in the range of 155-185 ppm. For carbon C-O which bound the fatty acid to TMP, its signal peak was detected at the chemical shift of 63.65 ppm. The presence of both carbons indicates the presence of ester bond between POFAs and TMP forming POTMP ester.

TMP carbon that has triester structure ($\text{C}_2\text{H}_5\text{-C-(CH}_2\text{OCOR)}_3$) was detected at chemical shift of 40.54 ppm and this value was in agreement with the reference which is chemical shift for quaternary carbon is 20-60 ppm [22]. The presence of unsaturated acyl groups was determined by identification of the alkene group (C=C). According to Pavia *et al.* [22], the chemical shift for alkene carbon (C=C) was in the range of 100-150 ppm. The alkene group (C=C) in POTMP ester was detected at the chemical shift of 127.88-130.18 ppm.

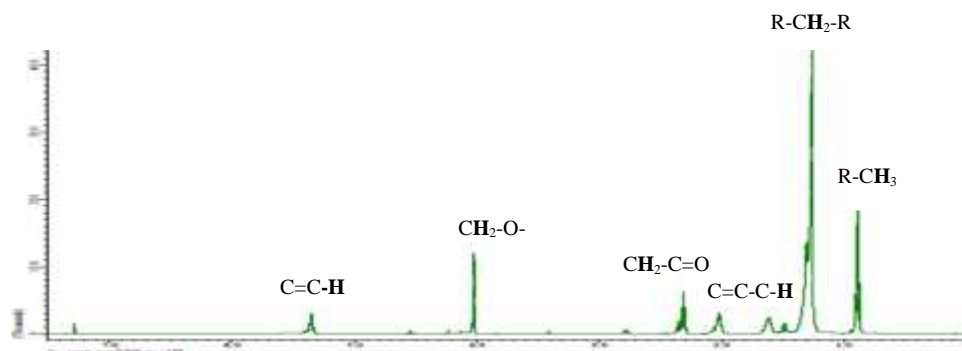


Figure 5. ^1H NMR spectrum of POTMP ester.

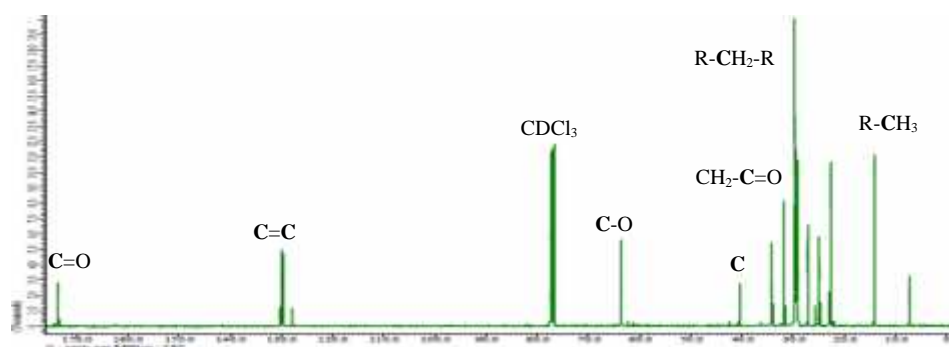


Figure 6. ^{13}C NMR spectrum of POTMP ester.

Table 2. Physicochemical properties of RBD palm oil and POTMP ester.

Physicochemical properties	RBD palm oil	POTMP ester
Oxidative stability (°C)	181	183
Pour point (°C)	7	8
Flash point (°C)	240	290
Kinematic viscosity at 40°C	56.97	61.79
Kinematic viscosity at 100°C	9.24	14
Viscosity index (VI)	130	158

Physicochemical Properties of POTMP Ester

There were tests conducted to determine the physicochemical properties of POTMP ester. The results of the physicochemical properties obtained are shown in Table 2.

The ability of a substance to resist oxidative degradation is an important property for biolubricants. Oxidative stability is determined by the onset temperature (OT). OT is the temperature at which a rapid increase in the rate of oxidation is observed at a constant, high pressure (200 psi). A high OT would suggest high oxidation stability of the material [2]. In this study, POTMP ester possessed good oxidative stability with high onset temperature which was 183°C. It was due to the decreasing percentage of unsaturated fatty acid (52.5%). Reduction of the double bond in unsaturated fatty acid had reduced the active oxidation site which gave higher oxidative stability.

Pour point is one of the important quality parameter to determine cold flow properties of the lubricant. The ability of a substance to remain liquid at low temperatures is an important attribute for a number of industrial materials, such as biolubricants, surfactants and fuels [23]. POTMP ester had high pour point (8°C) due to the increasing saturated fatty acids content. High saturated fatty acids content have caused the lubricant to form macro crystalline structures through uniform precipitation at low temperatures.

Another important factor in evaluating the performance of biolubricant is the flash point. Flash point is often used as a descriptive characteristic of fuel oil and it is also used to describe oils that are not used as fuels such as lubricant. Flash point refers to both flammable oils and combustible oils. The oils with a flash point value less than 43°C are flammable, while those having a flash point above this temperature are combustible oils [2]. POTMP ester showed higher flash point which is 290°C compared to RBD palm oil (240°C). This is due to the increasing molecular weight of POTMP ester with the presence of the ethyl group, which shows that the lubricant needs more energy to burn. High flash point value is important to ensure that the biolubricant is not burned in the engine during its operation [2].

Viscosity is also an important factor that influences the efficiency of the biolubricant in reducing friction and wear. The presence of ethyl group on the TMP structure has contributed to the increasing molecular weight of POTMP ester, thus increasing its viscosity. The viscosity of POTMP ester is in medium range which is 61.79 cSt at 40°C and decreasing to 14 cSt at 100°C. It shows the viscosity of oils decreases as temperature increases. The least viscous biolubricant which still forces the two moving surfaces apart is desired. If the biolubricant is too viscous, it will require a large amount of energy to move and if is too thin, the surfaces will rub and friction will be increased [2]. Viscosity index indicates the change of viscosity over the variation of temperatures [2]. POTMP ester has high viscosity index which is 158 compared to RBD palm oil (130). It shows viscosity of POTMP ester is less affected by temperature changes and does not drastically change when the temperature varying. This is a good indicator, and it is suitable for use at a large temperature range. The best oil for lubricant is the oil that has high viscosity index and will not vary much in viscosity over temperature range and therefore it will perform well [2].

CONCLUSION

POTMP ester was successfully synthesized from palm oil fatty acids and TMP with 92% of yield and 100% of triesters composition. The liquid POTMP ester could be potentially used as biolubricant basestock. POTMP ester showed good lubrication properties which had high oxidative stability, high flash point and high viscosity index compared to RBDPO. The removal of -hydrogen in RBDPO and replacing TMP polyhydric alcohol in POTMP ester successfully increased the lubricity properties. This could make POTMP ester a plausible biolubricant to be used.

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