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Tribological study of vegetable oil and its TMP esters as biolubricants

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KEYWORDS

Biolubricant
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Wear preventive
Extreme pressure

ABSTRACT

Vegetable oil based biolubricants are presented as an environment friendly alternative to conventional lubricants. In this paper comparative study of tribological performance of palm olein oil, sunflower oil and chemically modified palm olein TMP ester and sunflower TMP ester as biolubricants has been done. This will assist researchers and industries to choose biolubricant best suited for their applications. TMP esters of sunflower oil and palm olein oil were synthesized and a four ball tribometer was used to study the wear preventive characteristics as well as extreme pressure characteristics of palm olein oil, sunflower oil, palm olein TMP ester and sunflower TMP ester as per ASTM D4172 and ASTM D2783 standards. Lower coefficient of friction for TMP esters of palm olein oil and sunflower oil was observed in comparison with natural palm olein oil and natural sunflower oil respectively. Improved Wear preventive characteristics for sunflower TMP ester in comparison with sunflower oil was observed whereas for palm olein TMP ester and palm olein oil wear preventive characteristics were comparable. Extreme pressure characteristics for natural oils as well as TMP esters found to be comparably the same. Overall, sunflower TMP ester exhibited better tribological performance among all the biolubricants and also found to have similar tribological performance with petroleum-based lubricants.

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ACRONYMS

CoF	Coefficient of friction
FTIR	Fourier-transform infrared spectroscopy
FTP	Flash temperature parameter
KOH	Potassium hydroxide
TMP	Trimethylolpropane
WSD	Wear scar diameter

1.0 INTRODUCTION

Vegetable oil based biolubricant has gained interest in recent years because of its biodegradability, non-toxicity and renewability. These properties of vegetable oils make them a good alternative to the conventional lubricant which ends up in environment leading to the pollution (Abdollah et al., 2020; Ahmad et al., 2020). But one of the major drawbacks with vegetable oil based biolubricant is its poor thermo-oxidative property which limits its application areas. Over the years researchers have suggested various chemical modification techniques to overcome this problem. One such technique is the transesterification of vegetable oils to form TMP (tri methylol propane) esters. TMP esters improve thermo-oxidative stability of vegetable oil through reduction of hydrogen molecule from beta carbon position as these are very reactive towards oxygen (Fadzel et al., 2018; Owuna et al., 2020). Other polyol esters are also used for transesterification reaction, but TMP has the lowest melting point among others (Yunus et al., 2004). Since the primary purpose of lubricant is to decrease the friction and wear between two mating parts, biolubricants has to be in par with the conventional lubricants in terms of reducing wear and friction (Hussain et al., in press). Syahrullail et al. (2013) found lower wear for refined, bleached and deodorized palm olein oil than hydraulic oil and also reduced coefficient of friction for studied sliding speed. Jabal et al. (2019) found improved antiwear performance of sunflower oil in comparison with mineral oil. The reason for better antiwear and antifriction performance of vegetable oil is its unsaturated fatty acid content which forms adsorption layer on the metal surface preventing direct metal to metal contact. The tribological performance of chemically modified TMP esters of vegetable oils with improved thermo-oxidative stability is also important to realize their suitability as lubricant. Tribological performances of some of the chemically modified TMP esters of vegetable oils are shown in Table 1.

Though there are significant numbers of tribological study of palm oil based TMP ester (Yunus et al., 2004; Zulkifli et al., 2014a; Yunus et al., 2020). But study for the tribological performance of sunflower TMP ester is limited. Also, there's limited study regarding the comparison of tribological performance of TMP esters of different vegetable oils. This comparison will help researchers and industries to choose biolubricant best suited for their application. In this paper, wear preventive characteristics and extreme pressure performance of palm olein oil, sunflower oil, palm olein TMP ester and sunflower TMP ester has been studied. Palm olein oil and sunflower oil has been selected for the study on the basis of cost, availability and stable market price (Rudnick and Bartz, 2020).

Table 1: Tribological performances of vegetable oil TMP esters.

Sr. No.	Biolubricant	Ref. Lubricant	Tribo system	Lubrication Condition	Findings	References
1	Palm oil TMP ester	Paraffin oil; Fully Formulated lubricant	Four ball wear test	Load: 392 N; Speed: 1200 rpm; Operation time: 60 min	-TMP ester showed better wear preventive property and lower coefficient of friction than paraffin oil.	Zulkifli et al., 2016
2	Palm oil TMP ester	Ordinary lubricant	Four ball wear test	Load: 40 kg; Speed: 1200 rpm; Operation time: 60 min; Operating temperature: 75°C	- Blends of TMP ester and ordinary lubricant had better wear preventive characteristics and coefficient of friction than ordinary lubricant.	Zulkifli et al., 2013
3	Methyl oleate TMP ester; Canola TMP ester	Diesel	High Frequency Reciprocating Rig test	Frequency: 50Hz; Operation time: 75 min	- Blend of canola TMP ester with diesel found to have better lubricity performance than methyl oleate-diesel blend	Sripada et al., 2013
4	Lunaria TMP ester	Conventional Group 1 base oil	Four ball wear test; High Frequency Reciprocating Rig test	IP 239 standard method; Frequency: 50Hz; Operation time: 60 min; Operating temperature: 100°C	-Better wear preventive characteristics and friction characteristics for Lunaria TMP ester	Dodos et al., 2015
5	Jatropha TMP ester	SAE20W40 mineral oil	Pin on disc wear tester	Normal load: 80 N; Sliding speeds: 1500 rpm; Sliding distance: 30000 m; Operation time: 60 min	-Mineral oil blend with Jatropha TMP ester showed improved coefficient of friction, lower specific wear rate and better wear preventive characteristics	Singh et al., 2019

2.0 EXPERIMENTAL PROCEDURE

2.1 Materials

Refined sunflower oil and refined palm olein oil were procured from local market. Fatty acid profiles of these oils analyzed through gas chromatography are shown in Table 2.

Methanol (99.8%), potassium hydroxide pellets, sodium methoxide (95%) powder, tri methylol propane (98%), sodium sulphate anhydrous (97%), ethyl acetate (99.5%) and filter paper, were obtained from M/s S. D. Fine Chemicals Ltd., Mumbai, India and were used in the synthesis process.

Table 2: Fatty acid composition.

Refined Sunflower oil		**Refined Palm olein oil**	
Fatty Acids	Percentage (%)	Fatty Acids	Percentage (%)
Linoleic acid	64.993	Oleic acid	43.297
Oleic acid	24.549	Palmitic acid	39.237
Palmitic acid	6.695	Linoleic acid	12.132
Stearic acid	3.116	Stearic acid	3.810
Behenic acid	0.647	Myristic acid	0.829
		Linolenic acid	0.364
		Lauric acid	0.175
		Arachidic acid	0.105

2.2 Synthesis of TMP Esters

The synthesis process was adopted from (Heikal et al., 2017). 300 g of vegetable oil was measured in an Erlenmeyer flask and heated upto 60^0C temperature. A known amount of methanol with molar ratio of 6:1 to oil was taken in a small beaker and 1% by wt KOH catalyst was added in the beaker and swirled to dissolve in methanol solution. This mixture was then added into the vegetable oil flask maintained at 60^0C and stirring was given to the mixture for about an hour. Then the entire mixture was poured in a separating funnel. Once settled, two layers were obtained with lower layer of glycerol and upper layer of vegetable oil methyl esters. The obtained vegetable oil methyl ester was then water washed with distilled water heated at 70^0C. After water washing vegetable oil methyl ester was passed through sodium sulphate bed and filter paper. After filtration, vacuum drying of vegetable oil methyl ester was done to remove any residual methanol.

The obtained vegetable oil methyl ester was taken in a 500ml three necked round bottom flask and a known amount of TMP with molar ratio 4:1 is added in the flask. Then the mixture was heated upto 120^0C before adding sodium methoxide catalyst (1% by wt). After the addition of catalyst, reaction temperature was maintained at 125-130 ^{0}C with magnetic stirring under vacuum. The vacuum was set to 50mbar gradually to avoid spillover reaction. The entire reaction is carried out for 4 hours after the addition of catalyst. Figure 1 shows the reaction setup. The product was then washed with ethyl acetate and filtered to remove the catalyst. Then vacuum distillation was done to remove any unreacted vegetable oil methyl ester, methanol and ethyl acetate.

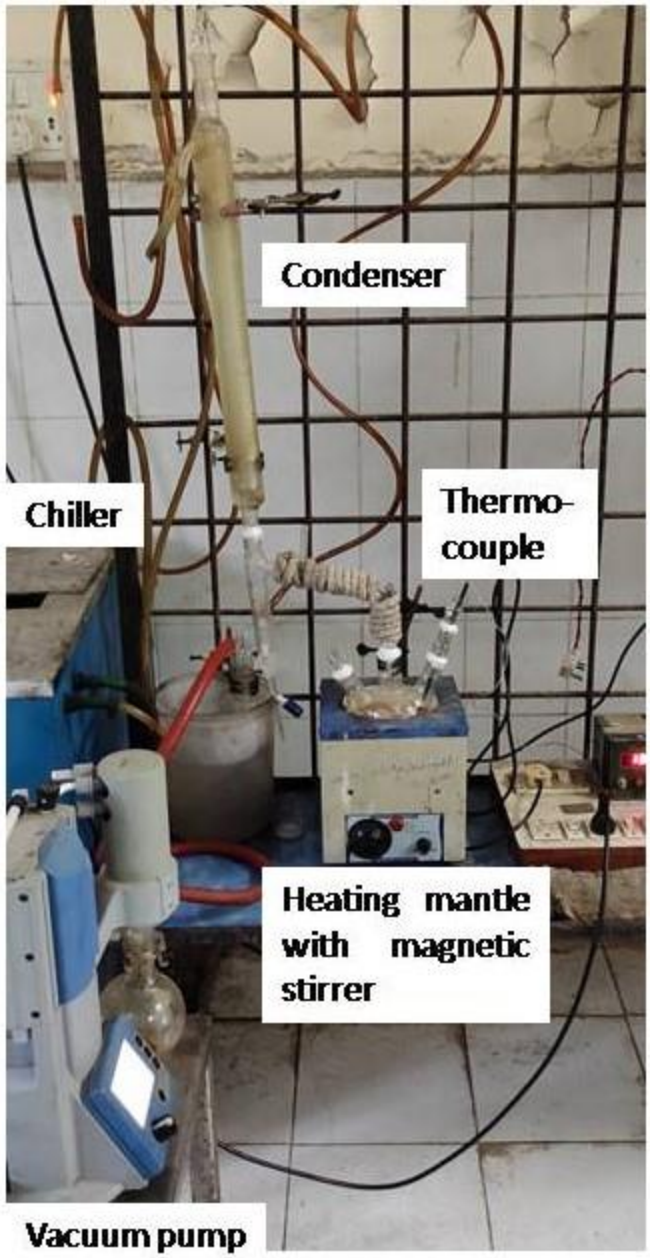


Figure 1: Experimental setup for TMP ester synthesis.

Fourier-transform infrared spectroscopy (FTIR) was done to confirm the esterification of product and physicochemical properties of the product was analyzed using ASTM standards to evaluate product's suitability as biolubricant.

2.3 Tribological Study Using Four Ball Tribometer

Ducom TR-30L tribometer shown in Figure 2 (a) was used for the tribological study of biolubricants. Chrome alloy steel ball of 1/2” diameter manufactured from AISI 52100 material with Rockwell C hardness 64-65was used. The chemical composition of the steel ball used for tribological study is given in Table 3.

Table 3: Steel Ball Composition.

Iron	**Carbon**	**Manganese**	**Silicon**	**Chromium**	**Sulphur**	**Phosphorous**
96.906%	0.990%	0.350%	0.240%	1.500%	0.003%	0.011%

Wear preventive characteristics of vegetable oils and their respective TMP esters were studied using ASTM D4172 standards. For the study, four steel balls were used with three balls being clamped together inside a test lubricant cup and the cup was then filled with the oil to be tested as shown in Figure 2 (b). The fourth ball was pressed against these three balls in the cavity between the clamped three balls with a load of 40±0.3 kgf forming three-point contacts. The oil temperature inside test lubricant cup was maintained at 75^0C using a thermocouple. The fourth ball was then given a rotational speed of 1200±10 rpm for about an hour. After that the ball-cup assembly was cooled to room temperature and the lower three balls were examined under optical microscope to measure the wear scar diameter. A total of six readings have been taken for the three balls to establish the average wear scar diameter. Wear scar diameter generated for the tested oil was then used to compare different oils for wear preventive characteristics. Also, the mean coefficient of friction for each test run is measured using the Equation 1.

$$\text{Coefficient of friction, } \mu = \frac{T\sqrt{6}}{3Pr} \tag{1}$$

Where *T* is the mean frictional torque in kg-mm measured once steady state condition is reached. *P* represents applied load in kg and *r* is the distance between centre of surface contact on lower balls and axis of rotation (3.67mm).

Flash temperature parameter (FTP) was calculated using equation 2. Flash temperature parameter is a number which is related to a critical flash temperature above which the lubricant fails to perform under the given conditions (Shahabuddin et al., 2013). FTP for oils used in present study were then compared with the existing studies on petroleum-based oils to check for the suitability as lubricant.

$$FTP = \frac{P}{WSD^{1.4}} \tag{2}$$

The extreme pressure property of test oils was measured using ASTM D2783 standard. For the test, upper fourth ball is rotated at 1760±10 rpm pressed under load against the lower three stationary balls which were covered in the oil to be tested. The temperature for the test was maintained at 34 ±1 ^{0}C. The load was increased gradually in steps as stated in the standard for each run of 10s duration until welding occurs. For each test run, oil was changed. Weld point was achieved at the lowest load at which the rotating ball welds to the lower three balls marking the extreme pressure limit for the test oil.

(a) (b)

Figure 2: (a) Four ball tribometer; (b) Lubricant cup holder.

3.0 RESULTS AND DISCUSSION

3.1 Characterization of Biolubricants

Vegetable oils and their respective TMP esters are shown in Figure 3. Their physicochemical properties were measured using ASTM standards as shown in Table 5. The FTIR spectrum for TMP esters of sunflower oil and palm olein oil are shown in Figure 4 a) and b) respectively.

(a) (b) (c) (d)

Figure 3: (a) Sunflower oil; (b) Palm olein oil; (c) Sunflower TMP ester; (d) Palm olein TMP ester.

In the figures, C=O stretching can be seen by the sharp peaks at 1740.83 cm^{-1} and 1741.48 cm^{-1} respectively, confirming the formation of ester after esterification with TMP. Also, the absence of -OH stretching peaks in the spectrum confirmed the conversion of carboxyl group of fatty acids indicating biolubricant esterification reaction completion. Table 4 summarizes the important FTIR peaks recorded.

Table 4: Summary of FTIR peak for TMP esters.

Functional Group	***Reference Peak (cm^{-1})**	**[b]TMP ester Peak (cm^{-1})**	
		Sunflower TMP ester	**Palm olein TMP ester**
-OH stretch	3300-2500[a]	-	-
	3600-3200[c]	-	-
CH-sp^3 stretch	3000-2850	2853.21,2922.41	2853.00,2922.08
C=O stretch	1750-1730[b]	1740.83	1741.48
CH_2 bend	1465	1461.58	1461.97
CH_2 long chain	720	723.15	721.97

[a] : acid, [b] : ester, [c] : alcohol, * (Fadzel et al., 2018)

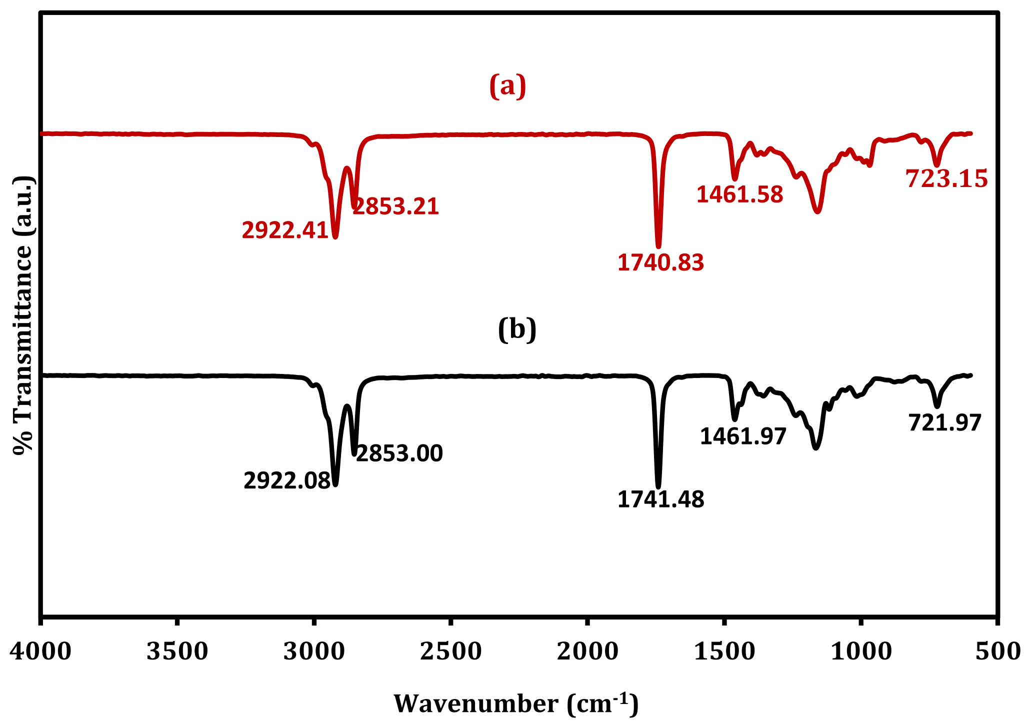


Figure 4: FTIR spectrum of (a) Sunflower TMP ester (b) Palm olein TMP ester.

Table 5: Physicochemical properties of biolubricants.

	Sunflower oil	Sunflower TMP ester	Palm olein oil	Palm olein TMP ester	Test method
Kinematic Viscosity (cSt) @40^{0}C	29.98±0.05	137.16±0.07	35.90±0.04	13.98±0.04	ASTM D445
Kinematic Viscosity (cSt)@100^{0}C	7.63±0.05	21.11±0.13	7.51±0.13	3.89±0.05	ASTM D445
Viscosity Index	240.72±2.56	179.57±1.33	183.47±5.77	188.86±7.13	ASTM D2270
Acid Value(mg KOH/gm)	0.59±0.20	2.14±0.41	2.15±0.49	1.43±0.55	ASTM D974
Iodine Value (gm I2/100gms)	118.17±2.78	93.29±2.72	63.48±2.89	62.28±3.04	ASTM D5554
Pour Point (^{0}C)	-13±1	-5±1	12±1	17±1	ASTM D97
Flash Point(^{0}C)	326±2	258±2	328±2	216±2	ASTM D92

3.2 Friction and Wear Characteristics

The mean coefficient of friction measured for the defined test conditions in the presence of different oils is shown in Figure 5. The lower the CoF value, better the friction characteristics of oil. Sunflower oil has more CoF than palm olein oil. This could be associated with the fatty acid structure of the natural oil. Palm olein oil has high oleic acid content whereas sunflower oil has high linoleic acid content. In general, saturated fatty acid and monounsaturated fatty acid forms effective monolayer over metal surfaces thus minimizing metal to metal contact whereas polyunsaturated fatty acid due to the existence of additional double bonds tends to decrease the density of fatty acid protective monolayer. This is in agreement with the research of (Reeves et al., 2015) for natural oils. TMP esters of sunflower oil as well as palm olein oil has better CoF than the natural oils themselves as shown in Figure 5. This could be due to the increased number of ester groups leading to the better binding of molecules offering greater resistance to shear forces (Havet et al., 2001). Also among TMP esters, sunflower TMP ester was having better CoF than palm olein TMP ester. This could be due to the difference in length of fatty acid which can be indicated by the viscosities of both the oils shown in Table 5. Viscosity increases with the fatty acid chain length (Wahyudi et al., 2018) and thus sunflower TMP ester being more viscous had increased fatty acid chain length in comparison to palm olein TMP ester. Also, adsorbed film thickness increases with length of fatty acid, which leads to increased protected surface (Zulkifli et al., 2014b).

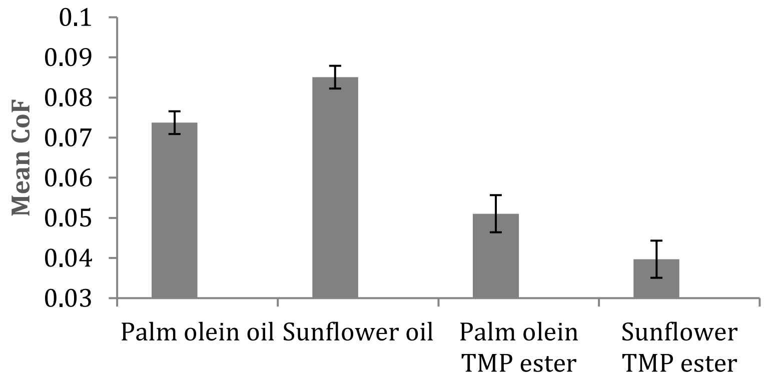


Figure 5: Mean coefficient of friction for biolubricants.

The mean wear scar diameter measured on three steel balls lubricated with different oils is shown in Figure 6. Lower the mean wear scar diameter better was the performance of oil as lubricant. As can be seen from Figure 6, mean wear scar diameter for different oils trailed the same trend as mean CoF. Higher the mean CoF, higher the wear scar diameter except for palm olein TMP ester. Palm olein TMP ester has comparable mean WSD with palm olein oil. This could be due to the lower viscosity of palm olein TMP ester caused by the lower length of fatty acid of TMP ester synthesized.

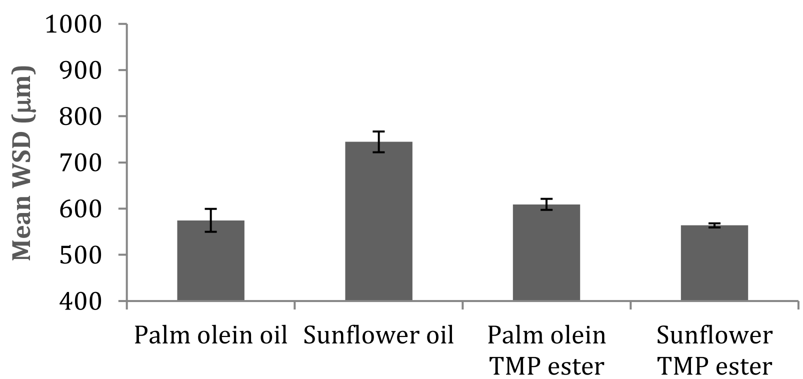


Figure 6: Mean wear scar diameter for biolubricants.

Figure 7 showed optical micrographs of wear scar diameter for different oils. Abrasive wear can be seen in all the four micrographs. The deeper the grooves are, darker they appear whereas shallow grooves are lighter in appearance (Suresha et al., 2020). Figure 7(c) shows smooth surface with lighter groove for palm olein TMP ester whereas deeper grooves can be seen for palm olein oil in Figure 7 (a). Similarly in Figure 7(b) darker grooves can be seen than in Figure 7(d) representing lesser wear for sunflower TMP ester than sunflower oil. Thus, optical micrographs suggest better wear performance for vegetable oil TMP esters than natural vegetable oil.

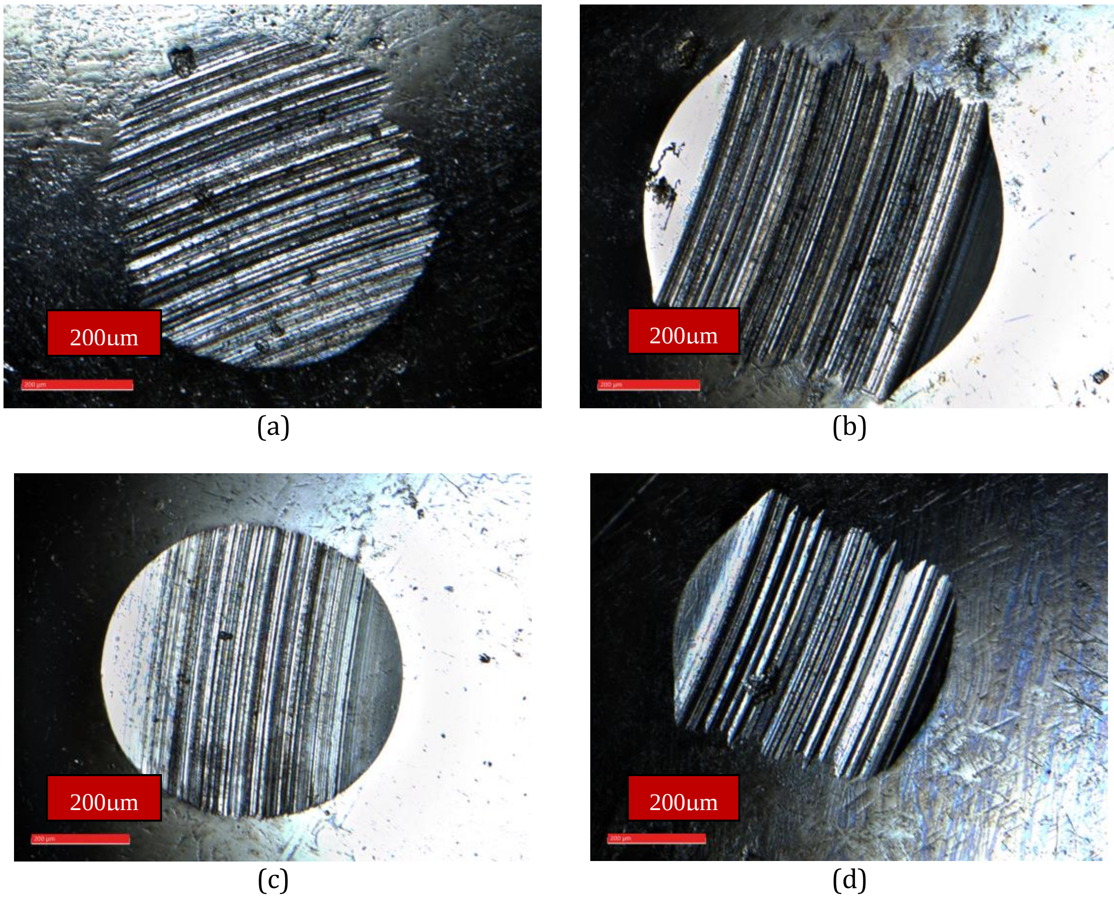


Figure 7: Optical micrographs for (a) Palm olein oil (b) Sunflower oil (c) Palm olein TMP ester (d) Sunflower TMP ester.

3.3 Flash Temperature Parameter

A comparison on flash temperature parameter of different oils used in the present study as well as with the petroleum-based lubricants from existing studies (Aravind et al., 2015; Charoo and Wani, 2017; Gupta and Harsha, 2018; Suresha et al., 2019) is shown in Figure 8. Existing studies selected for comparison based on similar testing conditions based on ASTM D4172. Higher the FTP value better is the lubricant as it indicated stable fluid film whereas a lower FTP value indicated poor lubricity due to the lubricating film damage. As FTP was inversely proportional to WSD, its trend is accordingly for biolubricants with sunflower TMP ester having the best FTP value. Palm olein TMP ester has better FTP value than paraffin oil which is in line with the study of Zulkifli et al. (2016) as shown in Table 1. From Figure 8, it is clear that sunflower TMP ester have comparable or better FTP values than the petroleum based lubricating oils. Thus sunflower TMP ester can be a suitable replacement for petroleum based lubricants.

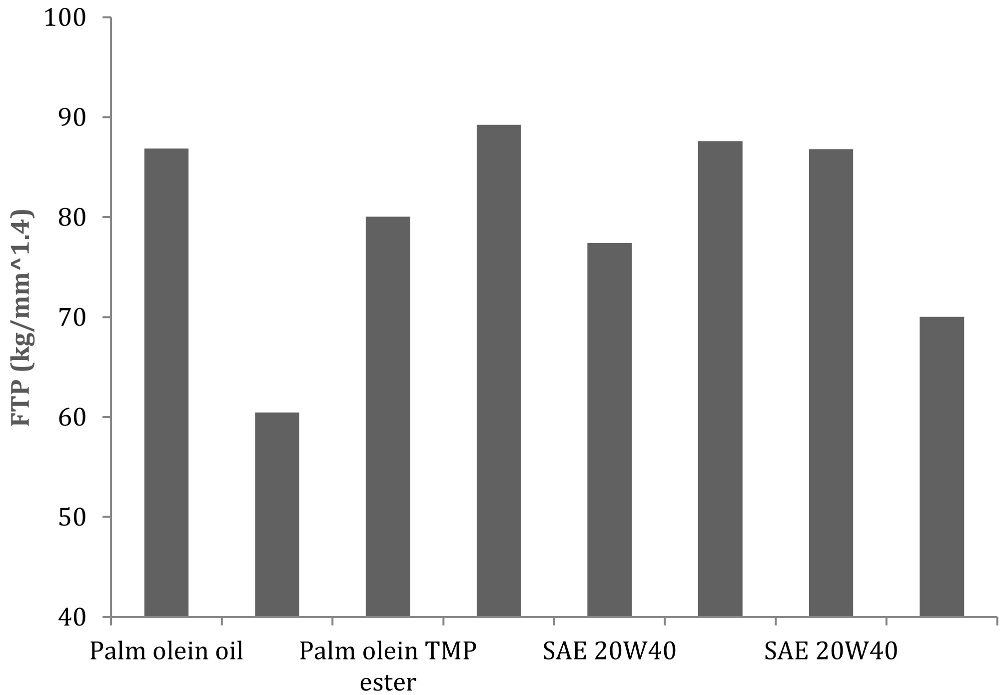
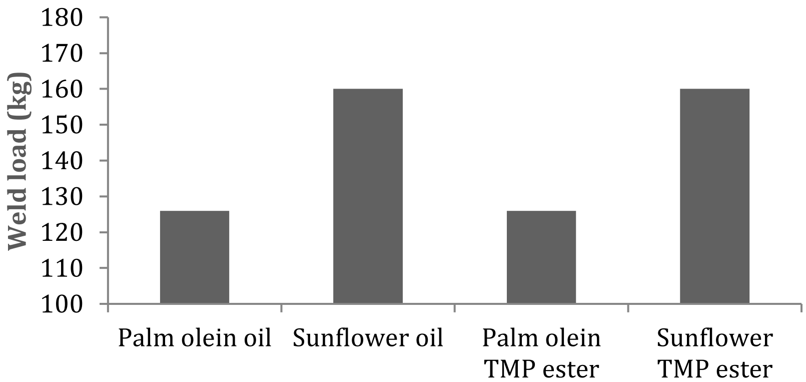


Figure 8: FTP of biolubricants from present study and petroleum-based lubricants from existing studies.

3.4 Weld Point

The welding loads for the steel balls under lubrication condition for different oils are shown in Figure 9(a). Figure 9 (b) showed steel balls at weld load after the extreme pressure test. Weld point determines the load carrying capacity of the lubricants. This extreme pressure property of vegetable oils and their TMP esters have shown a trend quite different from what was seen in case of CoF and WSD. It can be seen from the figure that the weld load for palm olein oil is same as palm olein TMP ester whereas weld load for sunflower oil was as same as sunflower oil TMP ester. Thus, TMP esterification of natural oils doesn't seem to change the extreme pressure property of vegetable oils. At higher loads, film thickness formed by fatty acids was reduced causing more metal-metal interaction which increased the temperature at which, oil film thickness was less stable leading to the breakdown of the lubricant film. Also, sunflower based lubricant had better extreme pressure property than the palm olein based. Sunflower oil had highest viscosity index among all oils indicating lesser change in viscosity with temperature whereas sunflower TMP ester has highest viscosity with good viscosity index. Thus, both the oils can have better viscosity at higher temperatures than palm olein oil and its TMP esters. Also, viscosity improved load carrying capacity. This explains better extreme pressure property of sunflower oil and sunflower TMP ester.



(a)

(b)

Figure 9: (a) Weld load for biolubricants (b) Steel balls at weld load.

CONCLUSION

In this paper, tribological performance comparison among palm olein oil, sunflower oil, palm olein TMP ester and sunflower TMP ester were done. As discussed, fatty acid composition has significant role in determining tribological performance of biolubricants. A significant reduction in coefficient of friction for TMP esters of palm olein oil and sunflower oil than the natural palm olein oil and sunflower oil was observed. Among all the studied biolubricants, sunflower TMP ester has shown least coefficient of friction. Wear preventive characteristics found to have improved significantly for sunflower TMP ester than the natural sunflower oil. But in case of palm olein oil and palm olein TMP ester, wear preventive characteristics were comparable based on mean wear scar diameter although optical micrography suggests better wear performance for palm olein TMP ester than palm olein oil. Extreme pressure characteristics of natural oil and their respective TMP esters were found similar with sunflower TMP ester and sunflower oil having higher weld load than palm olein oil and palm olein TMP ester. Out of all the biolubricants studied, sunflower TMP ester has shown best tribological properties which are comparable with the petroleum-based lubricants.

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APPENDIX

Tribological properties of petroleum-based oil.

Oil	Testing Conditions	Wear Scar diameter (mm)	FTP	References
SAE 20W40	40 kg, 1200 rpm, 3600 s, 75 C	0.624	77.410802	(Suresha et al., 2019)
SAE 20W50	40 kg, 1200 rpm, 3600 s, 75 C	0.57129	87.591249	(Charoo and Wani, 2017)
SAE 20W40	40 kg, 1200 rpm, 3600 s, 75 C	0.575	86.801056	(Aravind et al., 2015)
Paraffin oil	40 kg, 1200 rpm, 3600 s, 75 C	0.6703	70.029832	(Gupta and Harsha, 2018)