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Original article

Tribological properties of polyol ester – commercial motorbike engine oil blends



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ABSTRACT

This current study investigates the effect of blending bio-lubricant with commercial lubricant on physicochemical and tribological properties. The synthesis of TMP and PE ester are optimized to obtain 100% long chain tetra-esters and extreme care was taken during preparation of bio-lubricant to lessen deterioration of physio-chemical properties by blending with commercial oil. SM grade commercial petrol engine oil was blended with Tri-methylolpropane (TMP) ester, Penta-erythritol (PE) ester derived from Calophyllum-inophyllum in 10, 15, 20 and 25% v/v. The tests for anti-wear, anti-friction and extreme pressure properties are conducted on the sample oils on a four-ball wear tester. The friction coefficient and wear of bio-lubricant – commercial oil blends have decreased significantly up to 20% blending percentage. In the extreme pressure test, there is a substantial improvement in the weld load and marked improvement in the load wear index signifying the better load-bearing capacity of commercial oil – bio-lubricant blends. A synergy between bio-lubricant and additives in the commercial oil was found from a metallographic examination of worn balls after the EP test. The optimum volume percent of blend was found to be 15% PE blend and 10% TMP blend for optimum overall performance.

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1. Introduction

Fast utilization of petroleum resources significantly increased during the past decades owing to the fast growth in technology. Use of engine oil is essential to deter the formation of heat and to prevent corrosion. Several researchers (Adhvaryu and Erhan, 2004; Aziz et al., 2016; Erhan and Asadauskas, 2000; Fox and Stachowiak, 2007; Habibullah et al., 2014; Hamid et al., 2016; Havet et al., 2011; Heikal et al., 2017; Masjuki et al., 2013; Jayadas and Nair, 2006; Kamalakar et al., 2013; Kodali, 2002; Lathi and Mattiasson, 2007; Maleque et al., 2003; Mobarak et al., 2014; Panchal et al., 2017; Ponnekanti and Kaul, 2012; Salih et al., 2011; Laad and Jatti, 2018) investigated the use of biobased oils for commercial applications. Ting and Chen, 2011 estab-

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lished that use of esters extracted from oil bearing trees is helpful in the development of novel lubricants, thus decreasing the amount of toxicity and an eventual improvement of the biodegradability of the lubricant. Lathi and Mattiasson, 2007 established that biolubricants can be a substitute to petro-based oils. They are also found possess excellent tribological properties with high viscosity index and lubricity. Due to chemical structural similarities between mineral oils and plant based oils, the bio lubricants and/or their blends have the capability to be a substitute to mineral and synthetic oils. Though numerous efforts were made to find the relevant substitute to mineral based oils in IC engines, only a few researchers (Zulkifli et al., 2016; Jayadas and Nair, 2006) have highlighted the prospects of ester based biolubricants as a auxiliary to engine oil. Mobarak et al. (2014)) mentioned that apart from applications in automotive & aviation industry, petro based oils are also widely used in manufacturing industries. Hamid et al., 2016 analyzed that the bio-lubricants when blended with mineral oil based conventional lubricants can compete with commercial oils and outperform them in toxicity, biodegradability and low-cost reliability. Ponnekanti and Kaul, 2012 postulated that vegetable oils owing to intense interactions with the lubricated surfaces can act as anti-wear additives and friction modifiers. The polar groups in the long chain fatty acid structure of vegetable oil

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make them very effective in boundary as well as hydrodynamic lubrication conditions.

Notwithstanding the benefits like high stearic and oleic acid content, and biodegradability, the incidence of hydrogen atoms on the β -carbon atom in the esters makes them prone to oxidation, thus limiting their use to low temperatures. Furthermore, in the studies carried out by Ting and Chen, 2011; Zulkifli et al., 2016 and Havet et al., 2011, it was established that the load-carrying capacity of biolubricants is less failing them at higher loads thereby rendering them unsuitable for automotive applications. Nevertheless, the thermal stability of the bio-based lubricant can be enhanced by converting them to per-esters of sugar instead of glycerol esters. Several studies Zulkifli et al., 2016 and Havet et al., 2011 have found that by changing the glycerol group in the structure with an alcohol group, oxidation resistance and thermal stability could be enhanced. Polyol esters such as trimethylolpropane (TMP) ester penta-erythritol (PE) ester were studied as alternative bio-lubricants Jayadas and Nair, 2006, Salih et al. (2011)), Malegue et al., 2003 and Panchal et al., 2017. These

Table 1

Studies carried out by previous researchers on the percentage ester composition.

Author	Source of bio lubricant	Major findings
Zulkifli, 2013	TMP ester derived from Palm oil base	The triester content in palm oil based TMP ester is 95%. The composition of mono ester and diester are 5% respectively.
Zulkifli, 2016	Polylol esters of TMP and PE palm oil methyl esters.	The percentage fatty acid content in triester, diester and monoester are 82%, 9% and 1% respectively. Whereas, for PE ester the composition of tetraester, triester
Sripada et al., 2013	Methyl oleate and canola bio diesel based TMP ester	The highest yield of TMP tri-ester of 91.2% was observed at 140 °C, at a molar ratio of 5. The composition of diester was found to be 8.8% .

studies indicate that two important parameters namely; the structure of the polyol ester and the length of the fatty acid chain influence wear and friction under boundary lubrication regime. It was

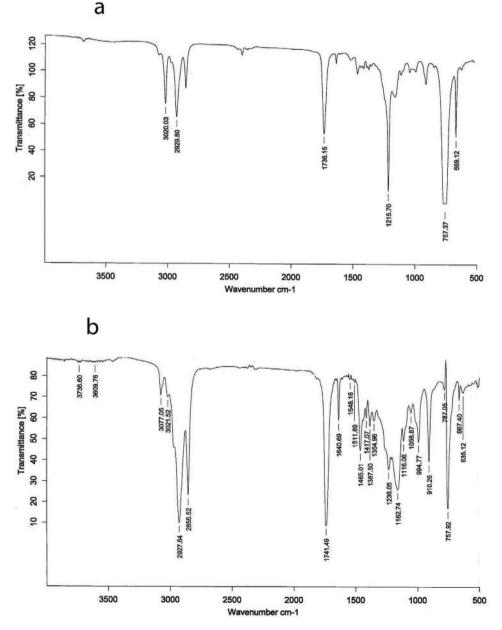


Fig. 1. FTIR spectrum of a) PE ester and b) TMP ester.

Table 2

Elemental analysis of formulated commercial lubricant.

Element	Test method	Quantity
Calcium, mg/Kg	ASTM D5185	1862
Zinc, mg/Kg	ASTM D5185	1037
Phosphorous, mg/Kg	ASTM D5185	946
Sulfur, wt%	ASTM D4951	0.286

Table 3

Test conditions during evaluation of lubricating properties on four ball tribometer.

Wear test as per ASTM D4172					
Temperature of the cabin Test duration	75 ± 2 ℃ 1 h				
Speed of the top ball	1200 ± 6 RPM				
Load applied Friction test as per ASTM I	40 kgf 05183				
Temperature of the cabin	75 ± 2 °C				
Duration of test	1 h (for wear in) Followed by friction test up to seizure load				
Speed of the top ball Load applied	600 RPM 40 kgf (During wear in) (In Friction test, starting from initial load of 10 kgf and incremented by 10 kgf every ten minutes till seizure load)				
Extreme pressure test as per Temperature of the cabin Duration Speed of the top ball Load applied					

Table 4

Load conditions during EP test.

S.No	Load, kgf	Compensation/actual scar diameter, mm
1	16	0.22
2	20	0.237
3	24	0.252
4	32	Actual scar diameters taken for calculations
5	40	
6	50	
7	60	
8	80	
9	120	
10	140	
11	160	
12	200	

Table 5

Physico-chemical properties of TMP ester bio-lubricants and commercial oil blends.

also found that lubricating performance of bio-based oils is affected by the number of ester groups, the length of fatty acid chains and linearity of the polyol.

1.1. Present studies

The current investigations dwell upon investigating the tribological properties of a blend of bio-lubricant (esters) derived from Calophyllum-inophyllum oil with commercial lubricant. Trimethylolpropane (TMP ester) and Penta-erythritol (PE) ester based bio-lubricant are selected in the current study owing to their typical four ester groups and longer chain. Previous studies described the preparation and tribological performance of Penta-erythritol (PE) ester containing 50–60% tetra-ester with tri-ester, di-ester and mono-ester as balance components. In the present study, in the synthesis, the reaction is done till 100% long chain tetra ester is obtained.

The commercial lubricant selected is formulated 4-stroke motorbike oil containing ZDDP additives. The Calophylluminophyllum oil or Tamanu seed oil was extracted from their seeds. The occurrence of sufficient amounts of oleic acid (>40%), stearic acid (>18%) and good physio-chemical properties makes it a worthy choice as bio-lubricant. TMP and PE esters were synthesized from the seeds of Tamanu tree and the synergic effect of additives in the commercial oil and polyol esters in bio-lubricant on the lubricating properties is investigated. Mixing is carried out in 10, 15, 20 and 25% v/v and due to the additives in the commercial lubricating oil, higher blending ratios are taken. Importance was given to find an best blending ratio for greatest performance.

In this study, all the lubricating properties are assessed using a four-ball tester. In a four-ball tester, the three point contact formed by the four balls under lubricated conditions reflects the boundary lubrication conditions. The relative performance of test oils in terms of friction, wear and extreme pressure properties are assessed using the method.

2. Materials and methods

2.1. Preparation of bio lubricant samples

The Calophyllum-inophyllum seeds were finely ground after drying and the oil was extracted in soxhlet apparatus using a solvent. For separation of TMP ester- Calophyllum fatty acids (300 g, 1.234 mol), trimethylolpropane (48.62 g, 0.362 mol),

S.No	Properties	Method	TMPester	SM grade motor Oil	SM oil + 10% TMP ester	SM oil + 15% TMP ester	SM oil + 20% TMPester	SM oil + 25% TMP ester
1	Acid number (mgKOH/g)	ASTM D664	0.22	1.05	0.99	0.95	0.97	0.93
2	Viscosity at 40 °C (cSt)	ASTM D2245	17.52	137.22	124.33	118.85	112.98	107.11
3	Viscosity at 100 °C (cSt)	ASTM D2245	4.71	15.72	14.42	13.68	12.91	12.24
4	Viscosity index	ASTM D2270	207.6	120	116	112	108	104
5	Coppers strip corrosion	ASTM D130	1b	1a	1a	1a	1a	1b

Table 6

Physico-chemical properties of PE ester bio-lubricants and commercial oil blends.

S.No	Properties	Methods	PE ester	SM grade motor Oil	SM oil + 10% PE ester	SM oil + 15% PE ester	SM oil + 20% PE ester	SM oil + 25% PE ester
1	Acid number (mgKOH/g)	ASTM D664	0.28	1.05	0.99	0.95	0.97	0.93
2	Viscosity at 40 °C (cSt)	ASTM D2245	23.85	137.22	124.33	120.21	114.5	108.87
3	Viscosity at 100 °C (cSt)	ASTM D2245	6.68	15.72	14.81	14.36	13.91	13.46
4	Viscosity index	ASTM D2270	263	120	119	117	116	111
5	Coppers strip corrosion	ASTM D130	1b	1a	1a	1a	1a	1b

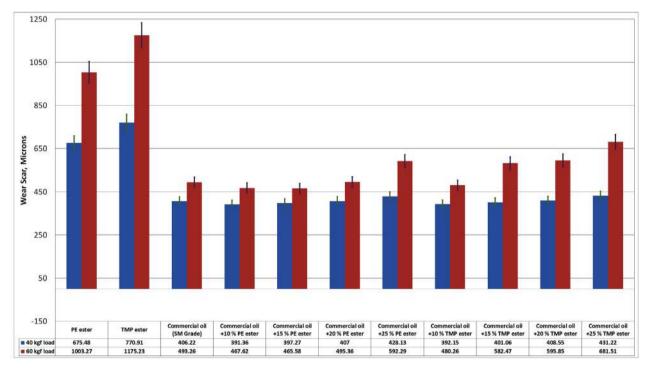


Fig. 2. Wear scars of test oils at 40 and 60 kgf load.

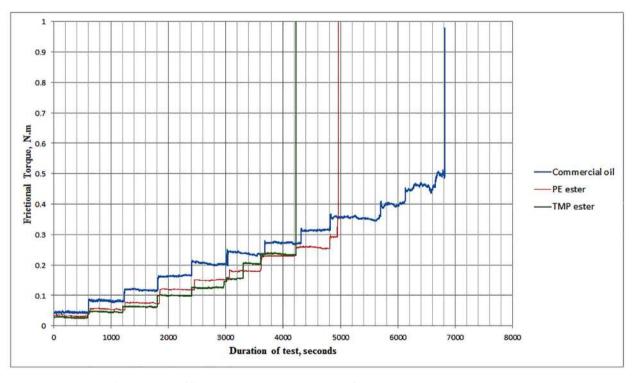


Fig. 3. Variation of friction torque during the test duration for commercial oil, TMP ester and PE ester.

p-TSA (3 g based on 1% weight of fatty acid) and 300 ml of toluene were stirred at reflux temperature. To prepare PE ester, Calophyllum fatty acid (250 g, 0.885 mol), pentaerythritol (30.43 g, 0.2234 mol), p-TSA (2.5 g based on 1% weight of fatty acid) and 250 ml of toluene were stirred at reflux temperature.

After reaction, the mix is taken to room temperature and toluene is removed under vacuum. The reaction mix is added with aqueous sodium bicarbonate solution, ethyl acetate and agitated for half an hour. The organic layer formed in the reaction was separated, washed two times with water, dried over anhydrous sodium sulphate and concentrated under vacuum. The crude esters were purified by basic alumina column chromatography using 95% hexane and ethyl acetate as eluent to obtain TMP and PE esters. These products are characterized by IR spectral studies on an FT-IR (Perkin-Elmer) spectrometer and investigated for physiochemical and basic lubricating properties. As perceived from Fig. 1, in IR spectrum C = O widening was witnessed at 1738 cm⁻¹ in PE ester and 1741 cm⁻¹ in TMP ester. Based on physico-chemical properties, the synthesis method is corrected and optimized for best properties.

Similar works were carried out by other authors (Fox and Stachowiak, 2007; Heikal et al., 2017; Mobarak et al., 2014) nonetheless the present work involves synthesis of pure 100% TMP and PE ester and mixed with commercial oil to estimate the tribological performance. The source of bio lubricant, the percentages of different ester of the previous works has been provided in Table 1.

2.2. Preparation of blends of bio lubricant and commercial lubricant

The commercial base lubricant in the present study is Racer 4 (SM grade four stroke motor bike engine oil) containing Zinc and phosphorous based additives. The elements present in the lubricant found from ICP analysis is as given in Table 2.

The Trimethylol-propane (TMP) and Penta-erythritol(PE) ester based biolubricants are mixed with commercial base lubricant in 10, 15, 20 and 25% volume percentage. After mixing the mixture is stirred for 5 min in a ultra-sonic bath sonicator to obtain a uniform dispersion.

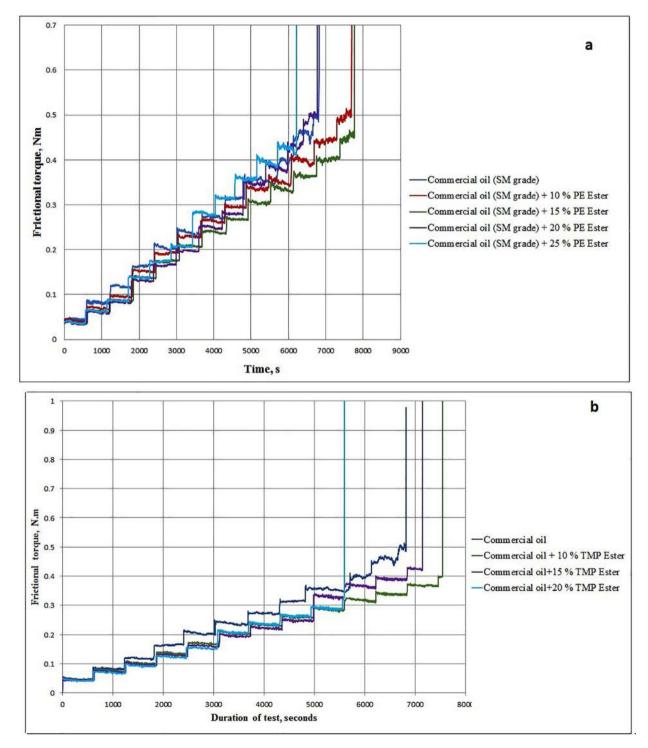


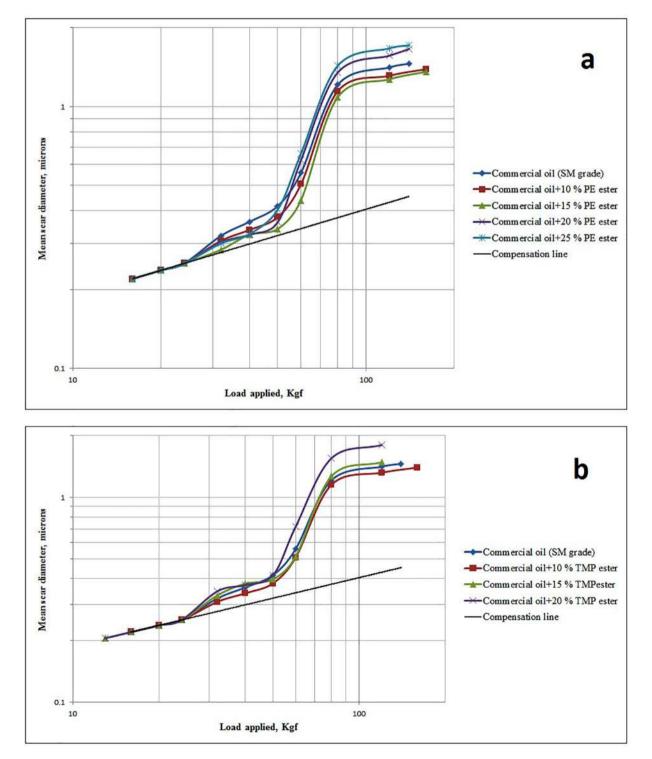
Fig. 4. Variation of friction torque during the test duration with a) PE ester based oils b) TMP based oils.

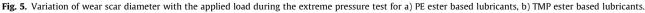
2.3. Assessment of tribological properties

In 4 S motorbikes, the same lubricating oil acts as engine oil acts as gear oil and hence the lubricant is required to lessen friction &wear and sustain extreme pressures in gears. In the study, four ball tribometer was used for the assessment of tribological properties. The wear, friction and extreme pressure test were performed as per ASTM standards. In all the tests, three half inch diameter steel balls are held together and dipped in the lubricating fluid. A similar fourth ball called "top ball" is forced against the three clamped balls by a load, thereby creating a 3 point contact. All the test conditions during wear, friction and EP tests are given in the Table 3.

For wear test as per ASTM D4172, the top ball is rotated against the three bottom balls, at a certain load and a wear scar is formed on the 3 balls due to rupture. The mean value of the scar diameters on the 3 balls is taken as wear scar diameter.

Friction test is carried out as per ASTM D 5183 standard, under changing load conditions simulating an internal combustion engine. Initially wear-in is performed under the aforesaid (Table 3)





test conditions. After the end of wear-in test for one hour, the used lubricating oil was removed and without removing the worn steel balls, the ball pot was cleaned and 10 ml of pristine lubricating sample is filled again for friction test. During the friction test, initial load was set at 98.1 N and the load is increased in increments of 98.1 N every 10 min of time interval until sharp increase in the friction torque is detected which represent the seizure.

The lubricant's extreme pressure properties are evaluated as per ASTM D 2783 for weld load and load wear index of the oil. A series of ten tests of 10 s duration was carried out with load increased during each tests until a weldment of all four balls is formed under extreme pressure which is called the weld load. The series of tests are conducted starting at an initial load of 32 kgf and the successive tests were carried out with increased loads until the four balls weld under extreme pressure. As per the standard, for loads below 32 kgf, the corrected load is calculated using compensation scar diameter and the corrected load is calculated for all ten reading. After each test, the oil is flushed out and the scar diameter on the balls is noted down. Likewise all the tests in the ten test sequence are conducted with fresh oil and fresh ball set but with

Table 7

Results of EP test of test oils.

Oil	Weld load	Load wear index
Commercial oil (SM Grade)	160	30.07
Commercial oil + 10% PE ester	200	32.76
Commercial oil + 15% PE ester	200	34.62
Commercial oil + 20% PE ester	160	29.7
Commercial oil + 25% PE ester	140	28.6
Commercial oil + 10% TMP ester	200	32.61
Commercial oil + 15% TMP ester	160	30.5
Commercial oil + 20% TMP ester	140	26.61

varying load. The load-wear index (LWI) indicate the overall performance of the engine oil in a range loads between well below seizure and welding and is calculated from the formulae given below.

Corrected load =
$$\frac{LD_h}{X}$$

where L = load applied, kgf, X = mean scar diameter on the worn balls, Hertzscardiameter, $D_{\rm H}=8.73\times10^{-2}~(L)^{1/3}$ and The load wear index, LWI = (A/10), (kgf) where, A = sum of the corrected loads in all ten tests.

The details of the loads applied and compensation scar diameter taken are shown in Table 4.

2.4. Metallographic investigation

The worn balls prior to weld load during EP test was analyzed using metallurgical microscope to check the pattern of wear. The worn surfaces were examined by EDX spectra for deposits on the surface to assess the synergic effect of esters in bio-lubricant and additives in the commercial oil.

3. Results and discussion

3.1. Physico-chemical properties of bio-lubricant and blends

The physio-chemical properties of bio lubricants have a significant role in sustaining tribological properties. Any decline of properties owing to faulty synthesis of biolubricant would lead to low performance. The bio-lubricant synthesis is optimized after assessing the properties using standard test methods after ascertain they are in the tolerable range.

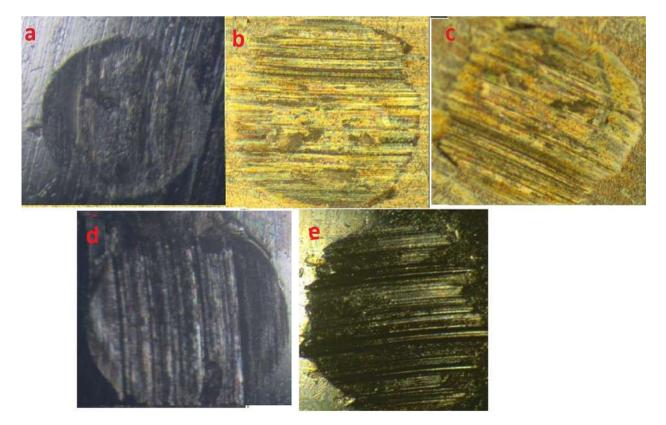


Fig. 6. Metallographic images of worn balls in extreme pressure test a) Commercial oil b) Commercial oil + 10% PE ester c) Commercial oil + 15% PE ester d) Commercial oil + 20% PE ester e) Commercial oil + 25% PE ester.

It is observed that the acid number of all test oils is well under 1.5 mg/KOH/g as approved by SAE standard (see Tables 5 and 6).

A minor decline in the viscosity and viscosity index above 20% blending percentage is observed with the viscosity index remaining above 90 as required for engine oils. Copper strip corrosion test was performed at 50 °C for 3 h shown that up to 15% mixing percentage the corrosion deterrence is found be brilliant and in line with commercial lubricant. For the blends with 20 and 25% bio lubricant, the corrosion resistance is found to be optimal marked by 1b on corrosion standard. The TMP ester based biolubricant mixtures possess lesser viscosity when matched with PE ester based biolubricant blends. This is owing to longer chain length of PE ester. The decrease in viscosity and viscosity index of TMP ester based lubricants could have a bearing on the lubricating properties and engine performance.

3.2. Tribological properties

The tribological tests were performed in ten reproducible experiments and the average values were taken. Seizure load during friction test plays a vital role in augmenting the efficacy of the lubricant. From the results, it is established that the PE ester in interaction with the additives of the commercial oil demonstrating a good enhancement in the tribological properties. The results of wear test for various test oils at 40 kgf load and 60 kgf load are as given in Fig. 2. From Fig. 2, the anti-wear performance of test oils at 40 kgf and 60 kgf load can be gauged. PE ester based lubricants performed better than TMP ester based lubricants. The performance of commercial base lubricant mixed with PE ester up to 20% blending ratio is found to be optimum with deterioration observed past 20%. In case of TMP ester mixed with commercial lubricant base the optimum blending ratio is found to be 85:15.

From the above findings, it can be predicted that although PE ester offered higher wear scar, blending PE ester in commercial oil up to 15% blending volume percentage, due to lower coefficient of friction of ester there is a significant improvement in the anti-wear properties and anti-friction properties. When the volume percent is increased beyond 20%, there is slight decline in the wear resistance with an increase in the wear scar diameter. Fig. 3 compares the performance of commercial oil and ester of TMP and PE. It can be seen that although esters could offer low friction coefficient, their performance is limited by low seizure loads. As can be observed the PE ester gave very low coefficient of friction but possess a low seizure load of 90 kgf which is the main disadvantage of bio-lubricants. TMP ester gave the lowest coefficient of friction among all but failed at 80 kgf. By mixing PE ester in 10% and 15%

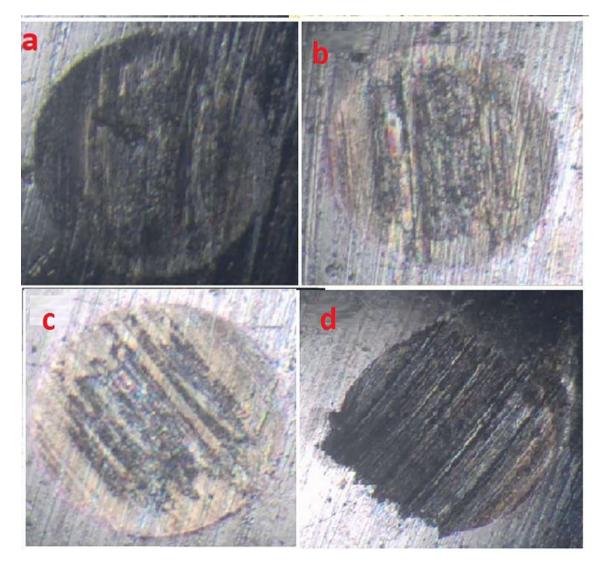


Fig. 7. Metallographic images of worn balls in extreme pressure test a) commercial oil b) Commercial oil + 10% TMP ester c) Commercial oil + 15% TMP ester d) Commercial oil + 20% TMP ester.

in commercial base lubricant, there is a good improvement in both seizure load as well as the friction coefficient.

Fig. 4, it can be observed that at lower loads the frictional torque of all lubricants is in the same range. However, a significant parity can be seen between commercial lubricant and its blends in terms of frictional toque as the load increases.

From Fig. 4a and b for blending percentage of 20%, it can be observed that the friction torque reduction took place with an eventual decrease in the seizure load which resulted in early failure of the ester compared to commercial lubricant. For commercial lubricant blended with 25%, there is a reduction in friction torque up to 60 kgf load and beyond that point there is a jump in friction torque indicating beyond 20% the action of esters is more prevalent compared to the additives in the lubricant.

In EP test results seen from Fig. 5a and b, with blending of PE ester up to 15% volume and TMP ester up to 10%, there is an

enhancement in the weld load and load wear index with 15% PE ester blend and 10% TMP ester blend giving best results.

As seen from Fig. 5a and b, up to last non seizure load point (C) all blends performed very well indicating effectiveness at lower loads. In the incipient seizure region (C-D) and immediate seizure region (D-E), the blends with 20 and 25% PE ester experienced a sharp increase in the wears scar diameters affecting the load wear index and the weld load. Due to this, although the weld load weld load remained the same as that of commercial oil, there is a reduction in the load wear index due to poor anti-wear performance of the blends after seizure load. Blends with percentage of PE ester below 20% and TMP ester below 15% could fare very well in these regions. This can be ascribed to the synergy between esters and additives in the commercial oil leading to an enhanced surface lubricity. Poor performance at higher percentages of ester can be attributed to the thinning of oil which could lead to premature failure.

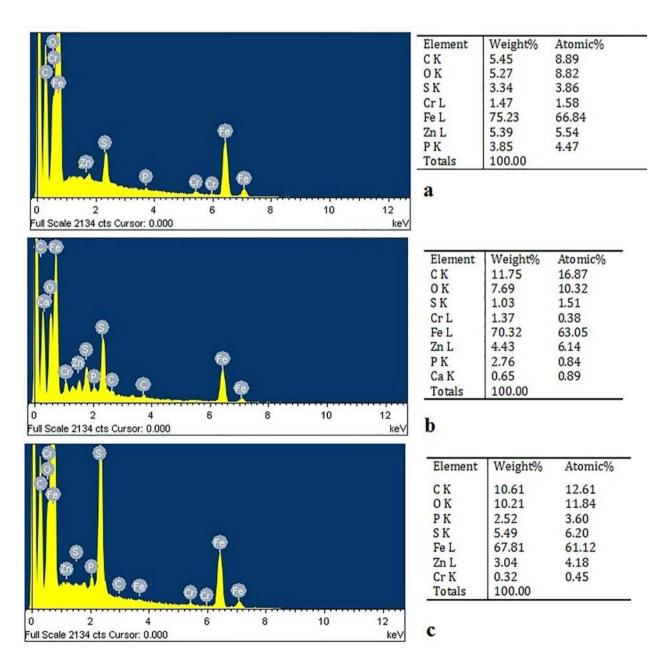


Fig. 8. EDX spectrum of worn balls during EP test a) with commercial oil b) with commercial oil + 15% PE ester c) with commercial oil + 10% TMP ester.

The following Table 7 summarizes the extreme pressure properties of all test oils

3.3. Results of metallographic test

Lubricating oils apart from ZDDP based additives for anti-wear and anti-friction performance, also comprise of Sulphur as EP additive. Under extreme temperatures produced by extraordinary operating pressures, the EP additives in the commercial oil react with the metal surfaces creating new compounds like iron phosphides and iron sulfides on the contact surface. These metal compounds form a chemical film on the surface which acts as a barrier thereby decreasing friction, wear and lessen the chance of welding. The worn surfaces of the balls prior to weld load during EP test were characterized in a metallurgical microscope for the structure. Figs. 6 and 7 depict the images taken from metallographic microscopes with worn balls of EP test prior to weld load. The images are taken at 20x resolution on a metallurgical microscope. It can be seen that base oil and base oil mixed with 10% and 15% PE ester as well as TMP have performed very well.

The wear scar in all cases is less and the esters could prevent abrasive wear during extreme pressure conditions. In case of pure ester and oils mixed with 20% and 25% PE and TMP esters, a severe abrasive wear could be noted indicating ineffectiveness at higher concentrations. The balls were also tested on scanning electron microscope equipped with Energy-dispersive X-ray spectroscopy (EDS) for metallographic depositions on the worn surfaces.

Fig. 8a–c show the EDS analyses quantifying the elements deposited on the worn surfaces of the balls tested with commercial oil, commercial oil blended with 15% PE ester and commercial oil blended with 10% TMP ester respectively.

From Fig. 8a for worn balls tested with commercial oil have their surfaces covered with some amount of zinc and phosphorous along Sulphur due to formation of tribo-film consisting of ZDDP (Zinc dialkyldithiophosphate) which reduce the chances of seizure. Elemental carbon can also be seen in the EDX spectrum which can be probably due to the combustion products. Fig. 8b and c, display the EDX spectrum of the worn ball tested with commercial oil blended with 15% PE ester and 10% TMP ester. From the elements in the spectrum, it can be concluded that the tribo-film on the surface comprises of additives in the lubricant (Zinc and phosphorous) along with Sulphur, calcium and greater amounts of carbon deposit compared to EDX spectrum of ball tested with commercial oil. This may be due to combustion products and also deposition of esters on the mating surface which decreased friction thereby improving seizure load and weld load.

4. Conclusions

- 1. 100% long chain PE ester and TMP ester when blended with commercial oil in 10–15 vol% improved the tribological properties without affecting the physio-chemical properties.
- A notable decrease in wear and friction coefficient of oils due to mixing with PE and TMP ester.
- 3. The extreme pressure characteristics have also enhanced with improvement in weld load and load wear index.
- 4. For top results, the mixing percentage should be below 20% for PE ester based lubricant mixtures and 15% for TMP ester base lubricant blends.
- An increase in the blending percentage would result in reduction of viscosity and viscosity index resulting in untimely failure of the engine oil.
- 6. The metallographic studies suggest synergy between esters and additives in the commercial oil resulting in superior performance of commercial biolubricant blends.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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