



Tribological behavior investigation of Ficus Carica oil blended ethylene-vinyl acetate polymer on AISI 52100 steel using four-ball tribometer

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KEYWORDS

Fig oil
EVA copolymer
FT-IR
Bio-material

ABSTRACT

Lubrication has remained a source of concern in industries owing to its role in reducing excessive friction and wear during machine operation. In line with global sustainability development goals, bio-materials are regarded as substitutes to fossil lubricants due to their environmentally friendly nature. In this present research, the Ficus Carica oil (Fig oil) with Ethylene-vinyl acetate polymer additive were characterized in terms of functional properties and tribological strength. The base Fig oil was produced, characterized using chromatography, FT-IR along with the selected polymer additive for their compatibility, and also conducted tribological test using four ball tester. The result shows the presence of functional groups of OH/N-H, C-H, C=O, CH₃, C-O, CH₂ for Fig oil and inclusion of co-polymer, with a slight shift in band formation, describing excellent compatibility. Under 1.5 % EVA, the coefficient of friction was reduced with EVA polymer by 9.1% and 17% for 40 kg and 100 kg. However, for 75 °C under 40 kg and 80 kg, the wear scar diameter was reduced by 33% and 14% under 1200 rpm when compared to base Fig oil. With these features from Fig oil incorporated co-polymer, good tribological performance will be achieved, leading to a reduction in patronage of fossil oil products in lubricant usage.

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1.0 INTRODUCTION

Global population growth, combined with industrialization and modernization, has resulted in increased energy consumption. For the last decade, societies have relied heavily on fossil fuels, causing a gradual depletion of fuel reserves to the point where it is predicted that this non-renewable energy source will be depleted in the medium term, resulting in increased research and development efforts for alternative chemicals and energy sources that could replace traditional fossil products in lubrication (Opia et al., 2020; Beard et al., 2017).

As a result, many products like synthetic, petroleum, and vegetable oils have been investigated to ascertain their lubricity characteristics. Among them, vegetable oil seems to be the only alternative product to fossil products with renewable, degradable, and eco-friendly features (Opia et al., 2020). In this regard, bio-based lubricants (bio-lubricants) have emerged as potential replacements for traditional petroleum-derived mineral oils (Nagendramma & Kaul, 2012 ; McNutt & He, 2016). Bio lubricants have excellent physicochemical properties such as viscosity and flash points, as well as shear resistance and bio-degradability (Opia et al., 2022 ; Mobarak, 2013). However, due to several drawbacks, including poor thermo-oxidative stability and low-temperature behavior, vegetable oils are not considered suitable for direct use as a lubricant. Previous researchers conducted extensive research to minimize the weaknesses of vegetable oils using a chemical modification technique, which included the addition of chemical additives such as anti-oxidants, pour point depressants, and viscosity modifiers. In this regard, due to the working conditions of machines, vegetable lubricant necessitates the addition of some additives, such as polymers or viscosity index improvers (VII), to help withstand the temperature. As a result, it is necessary to analyze the performance of a novel bio-base lubricant, *Ficus carica* (Fig), as an example of a vegetable class, to see if it has good tribological properties, which might help reduce the use of edible vegetable products as lubricants.

Viscosity index improver (VII), also known as viscosity modifier (Müller et al., 2006), is an important type of additive. The viscosity index measures the change in viscosity as temperature changes. The higher the viscosity index (VI), the less an oil's viscosity changes with temperature (Shara et al., 2018). Viscosity index improvers are used to slow the rate at which viscosity changes with temperature. These enhancers have no effect on viscosity at low temperatures. However, as the oil heats up, the improvers allow the viscosity of the oil to rise within the limits set by the type and concentration of the additive. The *Eichhornia Crassipes* Carboxymethyle cellulose (EC-CMC) was used as polymer and the effect in lubrication under base rapeseed oil was investigated (Opia et al., 2021). When compared to pure rapeseed oil, a sample with 1wt. percent EC-CMC resulted in a 47.1 % reduction in wear scar diameter. The functional groups discovered by FT-IR analysis are credited with EC-good CMC's performance (Opia et al., 2021). Therefore, this research employed Fig oil together Ethylene–vinyl acetate (EVA) polymer under sliding contact, to examine its lubricant behavior under different sliding condition. The idea of using the materials chosen for this research is to create an environmentally friendly lubricant that can substitute popular petroleum products that are linked to environmental contamination (Opia et al., 2022; Shah et al., 2021). Additionally, if found to be competitive with petroleum products in terms of lubrication, *ficus carica* cultivation will be encouraged, and its cost on the market will be decreased.

2.0 MATERIALS AND METHODS

2.1 Vegetable Oils and Additives

The environmentally friendly base-stocks (vegetable oils) used in this work are Fig oil, while the copolymer additive used was EVA, and was supplied by Sigma Aldrich (Poland). A commercially fully formulated lubricant, SAE 20W-30, was utilized as a benchmark for comparison of the performance. The steel balls used had a diameter of 12.7 mm and were made of AISI 52100 steel. The extraction of Fig oil can be done by the hand press technique as presented in Figure 1. The physical properties of the vegetable oils are shown in Table 1. Ethylene-vinyl acetate copolymer (EVA) with 33% vinyl acetate content, was kindly supplied in the form of pellets.

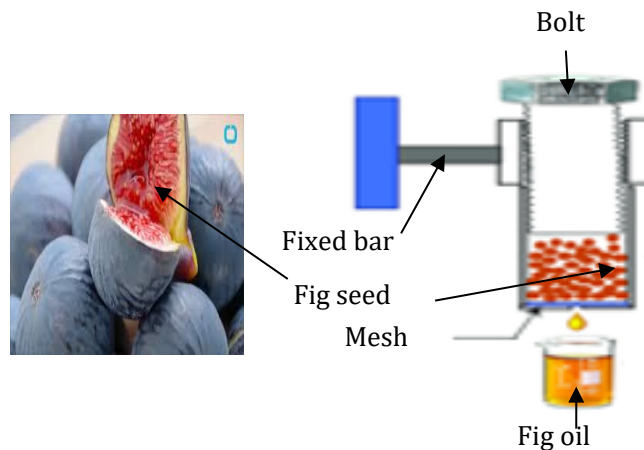


Figure 1: Extraction of Fig oil from *Ficus carica* fruits.

Table1: Physiochemical properties of Fig oil vegetable oil.

Veg. oil	Palmitic 16:0	Stearic 18:0	Oleic 18:1	Linoleic 18:2	α -Linolenic 18:3	Eichsenoic acid (n-9)
Fig oil	5.0-9.0%	2.0-4.0%	14.0-24.0%	20.0-35.0%	32.0-50.0%	0.5%

2.2 Characterization

The morphology of the EVA polymer was conducted using a SEM machine. In the area of molecular structure and bonding in the samples, Fourier-transform infrared spectroscopy (FT-IR) analysis was conducted to know the functional groups and also for the compatibility possibility between oil lubricant and formulated EVA. The study was conducted based on a previous study guide (Shara et al., 2018).

2.3 Preparation of the Lubricant Used

The new fig oil was blended with the EVA copolymer at different concentrations of 0.5%, 1%, 1.5% and 2%, respectively. Blends were prepared using a homogeneous mixer at constant velocity (1300 rpm). After that, when the polymers were completely dissolved, the mixtures were returned to room temperature by natural convective cooling. Dynamic viscosities were measured with a rotational controlled-strain rheometer (ARES, TA Instruments, USA), over a temperature

range of 25°C – 100°C. The viscosity indexes (VI) were obtained according to ASTM D-2270. EVA copolymer is considered an inert, nontoxic, and stable material. It is not expected to be biodegradable but is not hazardous, according to the Commission Directive 93/21/EEC.

2.4 Friction and Wear Measurements

The tribological performances of Fig oil and copolymer EVA as an additive were evaluated using a four-ball tribo-tester. The ASTM D4172 test was performed at a rotating speed of 1,200 rpm for 1 hr under 392 N and 980 at 75°C. The friction tests were repeated three times for each sample material to ensure that the experiments were reproducible. Figure 2 depicts the geometric configurations of a four-ball tribo-tester with one steel ball under load rotating against three stationary steel balls. The steel balls used had a diameter of 12.7 mm and were made of AISI 52100 steel (0.98-1.1% C, 0.15-0.30% Si, 0.25-0.45% Mn, 1.30-1.60 % Cr, 0.025% P, 0.025% S) with a hardness of 64–66 HRC. The WSD was determined using an optical microscope, and the COF was obtained directly from the electronic display. The worn surfaces of the balls were then examined using high-powered microscopes.

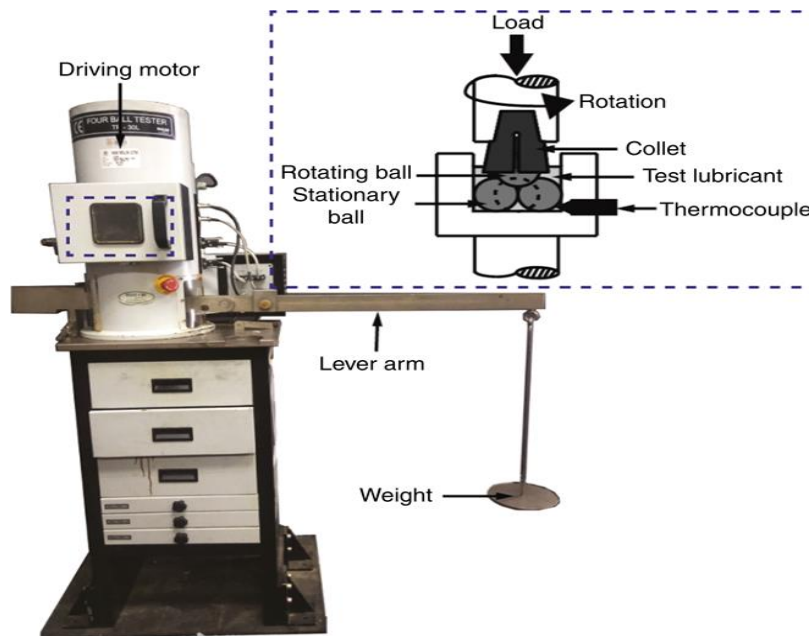


Figure 2: Image of the Four ball tester used, showing various parts.

3. RESULTS AND DISCUSSION

3.1 Characterization Result and Physical Properties of the Tested Lubricants

Figure 3 depicts SEM images of the EVA copolymer. The image shows the structure of no crystal or particles, indicating the possibility of dissolving in solution. The elements in the used copolymer were detected using EDX, with carbon and oxygen possessing high percentages, as shown in Fig.3. The FT-IR spectra were examined to determine the chemical structures of the Fig

oil and EVA blended as shown in Figure 4 (a). Compared to the Fig Oil spectra, a large and strong band at almost 3010 cm⁻¹ was detected in the EVA blended spectra due to the O–H/N–H group. The asymmetric and symmetric stretching of CH in oleic acid found in the samples resulted in absorption peaks at 2914 and 2903.77 cm⁻¹ on both samples. This is similar to the observations by Shara et.al, (Shara et al., 2018). But the EVA blended absorption peaks were observed to be more pronounced. The C=O group stretching vibration in the additives was due to the peaks at 1718 and 1688.3 cm⁻¹, which were very sharp in formation. However, a significant shift in the EVA blended sample was seen around 1367 cm⁻¹ due to the low concentration of the COO-group and a break in bond in the Fig oil sample. The two samples' similar behavior and wave indicated good compatibility, making them suitable for use in lubrication.

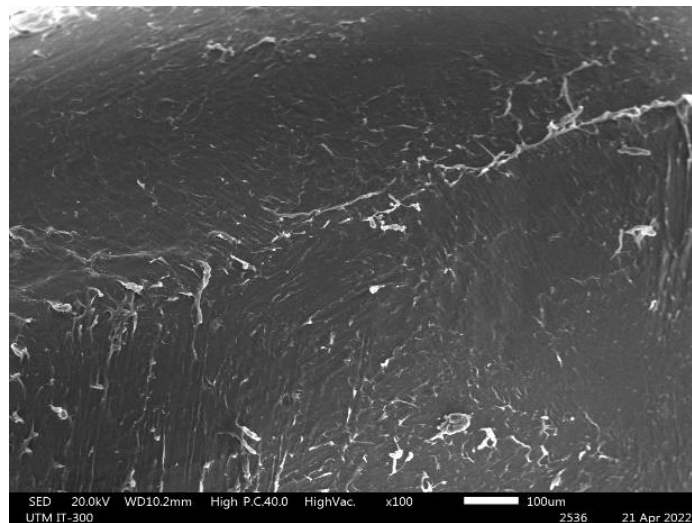


Figure 3: SEM image of EVA additive.

The density, kinematic viscosity, and VI of the tested lubricants are shown in Table 2. From Table 2, it can be seen that the kinematic viscosity of the tested lubricants generally reduced as the temperature increased due to the weakening of intermolecular attractive forces that cause rapid motion of molecules. The variation in the behavior of EVA nanoparticles in Fig oil under temperature/viscosity relationship is shown in the graph presented in Figure 4 (b). The decrease in the viscosity of the lubricants with an increase in temperature was due to degradation of the lubricant viscosity. The investigation found that there was very little difference in the viscosity values of the nano-lubricant between 100 °C and 150 °C, indicating quick degradation of the polymer at higher temperatures, bringing it to a close range with 0.5 wt% EVA. This has shown that larger EVA concentrations may not have as much resistance to high temperatures. As demonstrated in Table 2, the results showed that density changes as concentration rises at 15 °C. This is so because an increase in additive concentration makes the fluid thicker, which raises lubricant density. As a result, it required more time for the particles to dissolve fully as performed in the study under homogeneous mixing techniques.

Table 2: Lubricant (Fig. oil + EVA) physical properties as used in the study.

Sample	Viscosity 40°C		Viscosity 100°C		Viscosity Index	Density 15°C
	Dynamic ($\times 10^3$ Pa.s)	Kinematic (cSt)	Dynamic ($\times 10^3$ Pa.s)	Kinematic (cSt)		
Fig. oil	36.00	39.30	8.4	9.2	229.84	0.8312
Fig. oil + 0.5% EVA	63.1	70.4	9.4	9.7	119.01	0.8501
Fig. oil + 1% EVA	73.3	76.1	10.2	11.5	143.74	0.8611
Fig. oil + 1.5% EVA	71.9	73.3	9.7	10.8	135.84	0.8572
Fig. oil + 2% EVA	67.9	97.8	9.5	10.2	126.28	0.8394

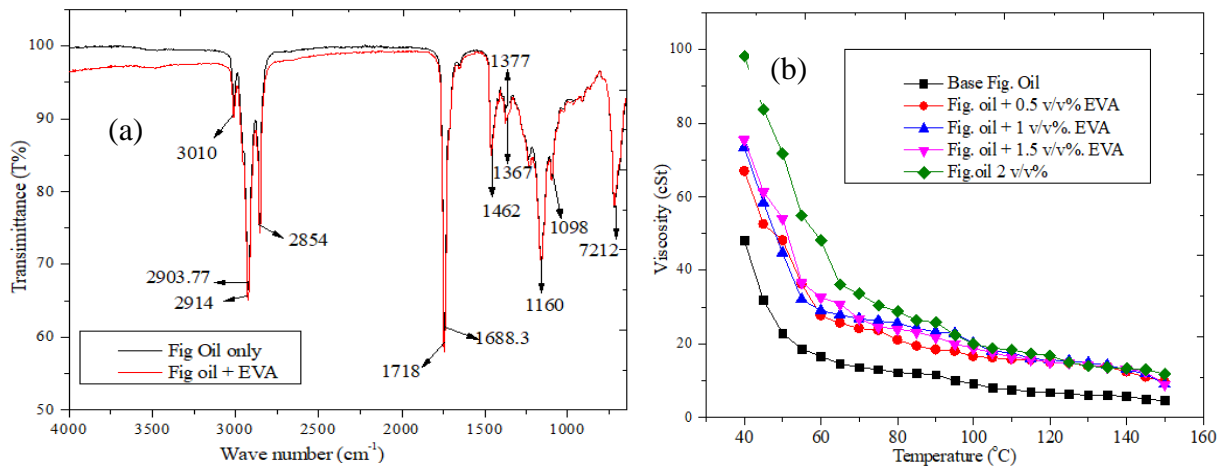


Figure 4: FT-IR analysis of Fig. oil and inclusion of EVA (a) and the effect of temperature on the viscosity behavior of the formulations (b).

3.2 Frictional Characteristics of Lubricating Fluid

To ascertain the best concentration of EVA polymer in Fig oil for optimal tribological performance. The friction reduction and anti-wear effects of the different concentrations of EVA additive (0.5, 1, 1.5, and 2 v/v%) compared to base oil under 80 kg were studied at different temperatures to gain more insight into the additive behavior during the testing as presented in Figure 5. The study discovered that as the temperature increased from 50 to 100 °C, the average friction coefficient and ball wear scar diameter decreased more strongly relative to the base lubricant. This is due to the growth of the formulated film into the contact region under additives application leading to surface protection (Guegan et al., 2019). This is similar to the observation made under the application of organic polymers in rapeseed oil (Opia et al., 2021). During the analysis, the COF and WSD of the base lubricants under 50, 75, and 100 °C were 0.063, 0.163, 0.177 and 0.471, 0.588, 0.707mm respectively. The commercial SAE 20W-30 demonstrated the lowest values both in COF (0.141) and WSD (0.21) compared to other samples. During the analysis, 0.5, 1, 1.5, and 2 v/v% EVA polymer as used under temperatures of 50, 75, and 100 °C,

the COF and WSD reductions were presented in Table 3. The investigation revealed that 1.5 v/v% of EVA blended gives the best tribological performance both in COF and WSD reduction and therefore should be used in the further analysis.

Table 3: The COF and WSD reduction during the various lubricant lubrication.

Parameter	COF					WSD				
	Fig oil only	0.5 v/v% EVA	1 v/v% EVA	1.5 v/v% EVA	2 v/v% EVA	Fig oil only	0.5 v/v% EVA	1 v/v% EVA	1.5 v/v% EVA	2 v/v% EVA
50 °C	0.063	25%	18%	17%	11%	0.471	33%	43%	57%	51%
75 °C	0.163	9%	15%	18%	12%	0.588	26%	39%	60%	44%
100 °C	0.177	10%	12%	8%	6%	0.707	31%	43%	57%	56%

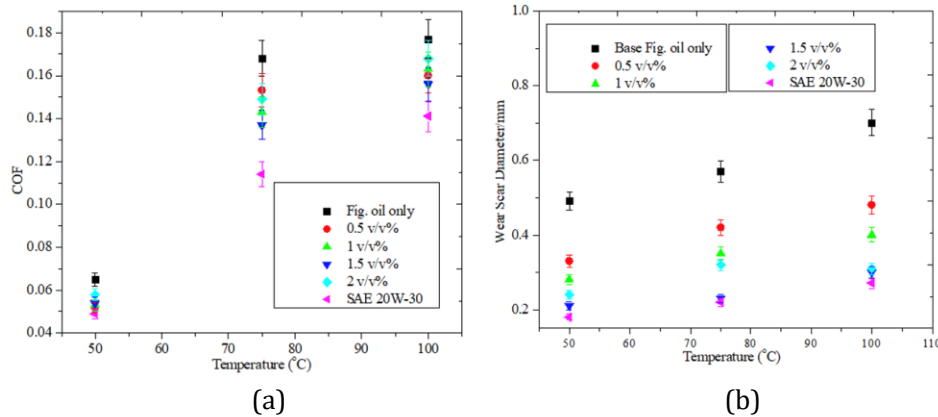


Figure 5: Avg. COF (a) and WSD (b) of the base oil (Fig. oil) and lubricant containing 0.5, 1, 1.5 and 2 w.% EVA polymers under different temperatures at constant load 80kg and speed 1200 rpm.

Figure 6 (a) displays the results of the COF of the Fig oil containing EVA additives under working conditions of 392 N (40 kg) 1,200 rpm and 980 N (80 kg) 1200 rpm for 1 hr at 75 °C. These results indicate that Fig oil without EVA under 40 kg and 80 kg has a higher COF of 0.0647 and 0.1684. It can be observed that the addition of EVA shows a good improvement in the COF reducing performance. The COF of Fig oil blended with 1.5 EVA under 40 kg and 80 kg was 0.0525 and 0.1374, respectively, as shown in Figure 6 (a). The COF was reduced by 18.4% and 18.9%, respectively, under 40kg and 80kg compared to base Fig oil. The effect of the EVA polymer on the COF during sliding operation was more pronounced under 80 kg load compared to a smaller load of 40 kg. This is attributed to the generation of more frictional energy for the formation of tribofilm. Also, the formation of a physical deposition film is responsible for the improvement in the tribological performance of the formulated lubricant. As shown in Figure 6(b), the performance of Fig oil and the inclusion of EVA was investigated at a speed of 1500 rpm. The COF of base Fig oil under 40 kg and 100 kg at 1500 rpm was 0.287 and 0.317, respectively. The addition of 1.5% EVA at 40 kg and 100 kg yielded 0.261 and 0.263, respectively, showing similar results. According to the findings, the COF was reduced by 9.1% and 17% for 40 kg and 100 kg, respectively,

compared to base Fig oil. This indicated that EVA with Fig oil yielded better protection at higher working temperatures, owing to the good formation of film at the sliding contact.

In analyzing wear scar diameter (WSD), inclusion of EVA in base Fig oil shows a reduction in the sliding wear of the bearing used compared to the raw Fig oil as presented in Figure 7 (a and b). Though, the application of EVA at the lower load of 40 kg under 1200 rpm demonstrated similar wear protection with base Fig oil. The resistance of fast degradation, due to the inclusion of EVA in Fig oil for 40N and 80N, respectively, under 1200 rpm, also reduction in WSD under application of 40N and 80N at a speed of 1500 rpm, respectively.

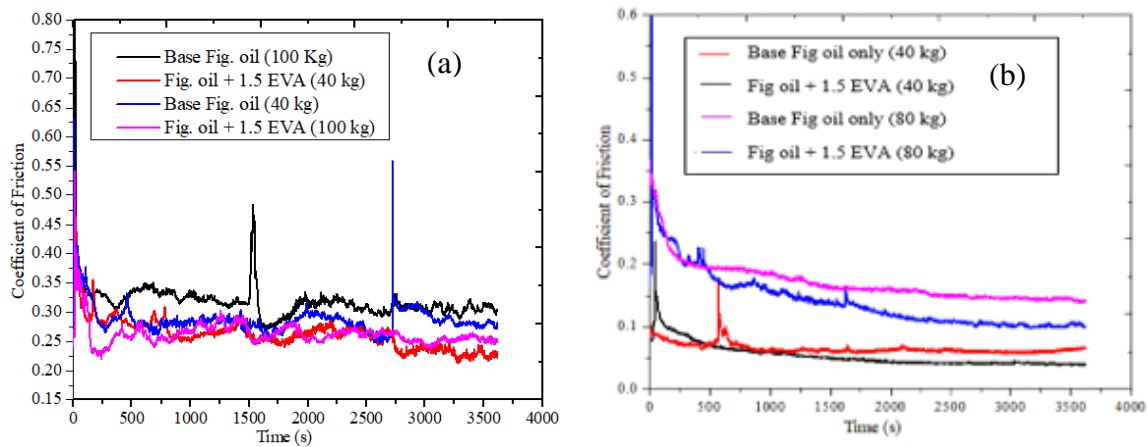


Figure: 6. Avg. COF (a) 1200 rpm and (b) 1500 rpm of the base oil (Fig. oil) and lubricant containing 1.5 v/v% EVA polymer under different load and constant temperature (75 °C).

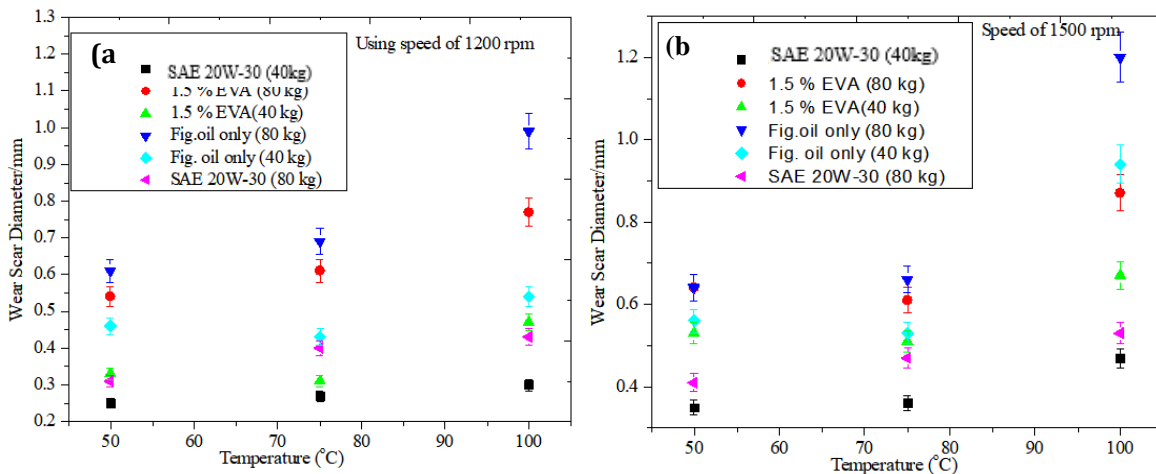


Figure 7: WSD (a) 1200 rpm and (b) 1500 rpm of the base oil (Figure oil), lubricant containing 1.5 % EVA polymer and SAE 20W-30 oil under different temperature (50, 75 and 100 °C) using constant load of 100kg.

The wear scar diameter for base Fig oil lubricated samples under 50, 75, and 100 °C were 0.51, 0.42, 0.47 mm and 1.01, 0.7, 0.601 mm, for 40 kg and 80 kg, respectively. For 1.5 wt.% EVA inclusion (50, 75 and 100 °C), yielded wear reduction of 11.7%, 33%, 31.9% and 30%, 14%, 8% for 40 kg and 80 kg under 1200 rpm respectively. Using commercial lubricant of SAE 20W-30 under 40k and 80kg gives 45%, 38%, 48% and 59%, 43%, 50%, respectively. Similar wear reduction was recorded under 1500 rpm. The study further observed that higher load gives better wear protection but shows more film cut, leading to a sharp peak on the graph as illustrated in Figure 6 (b). This is attributed to weak intermolecular and strong intramolecular bonding in between the layers of their formulated films (Zhao et al., 2017).

3.3 Worn Surfaces Analysis

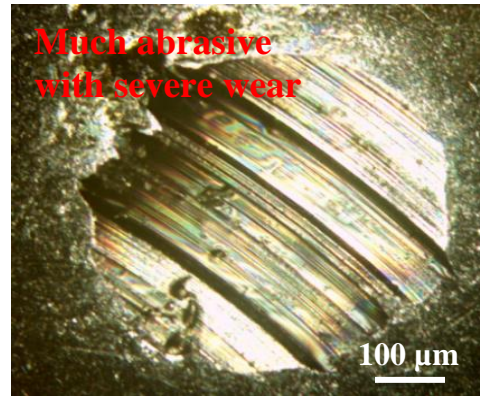
Figure 8 shows the low-resolution microscope SEM images of the worn scars on the ball bearing specimen. To ascertain the performance of each lubricant, the analysis is concentrating on the ball surface morphology. As previously indicated, base FO lubricant samples perform the worst, showing deep grooves on the sliding surfaces with little or no surface protection. The application of 1.5% EVA polymer with FO, on the other hand, resulted in wear reduction and performance that was comparable to that of a reference commercial lubricant. From the images, it could be seen that operations with a 40 kg weight resulted in less wear than those with an 80 kg load. Much abrasive wear, cracks with big grooves was seen with an 80 kg load, but much with base lubricant operation. By forming a tribo-film that acts as a protective barrier by preventing direct contact during sliding, the 1.5% EVA addition was able to minimize wear. However, higher wear reduction was observed when commercial SAE 20W-30 lubricant was used, as indicated in Figure 8, as a result of superior film formation. As observed from the lubricated surfaces (Figure 8), the introduction of an additive helped to prevent direct contact between the sliding element and the sliding element through particles that were diffused at the contact zone. The nanoparticles aid in film formation with low COF, and WSD, protecting components from rapid damage.

CONCLUSIONS

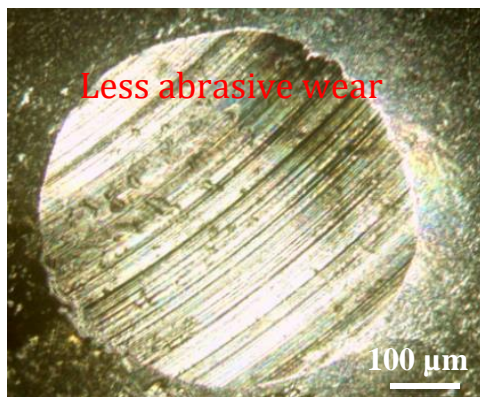
The experiment results showed that using EVA polymer as a lubricant additive significantly increased the tribological performance of the base Fig oil lubricant. All lubricant samples enriched with EVA polymers outperform Fig. oil samples in terms of COF reduction at both low and high loads, according to the tribological properties. Under 40 kg, 1.5 % EVA inclusion has the greatest effect on COF and WSD. However, the application of an 80 kg load yields some increased results in COF with 1.5 % EVA compared to 40 kg but lower than the COF of base Fig oil. The inclusion of 1.5 % EVA polymer in the surface analysis was proposed to help establish smoother contact surfaces to avoid the phenomenon of serious wear and large grooves. Furthermore, the improved performance of EVA necessitated sufficient frictional energy, facilitating tribo-chemistry operation between the sliding surfaces to generate an active tribo-film.



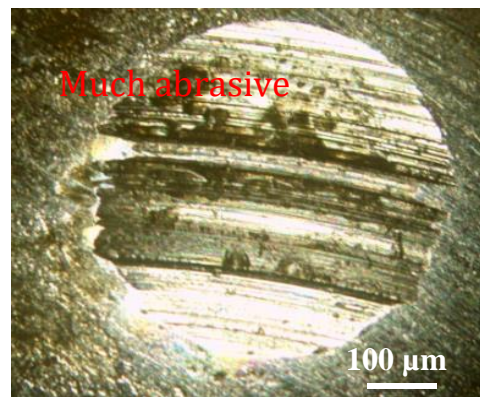
Base Fig. oil (40 kg)



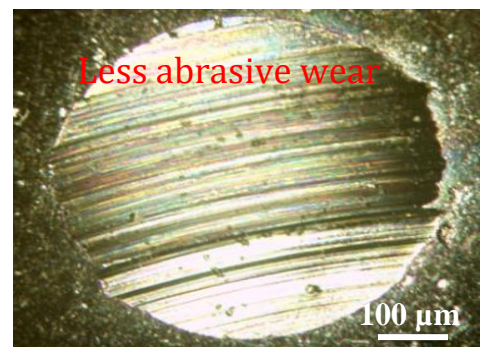
Base Fig. oil (80 kg)



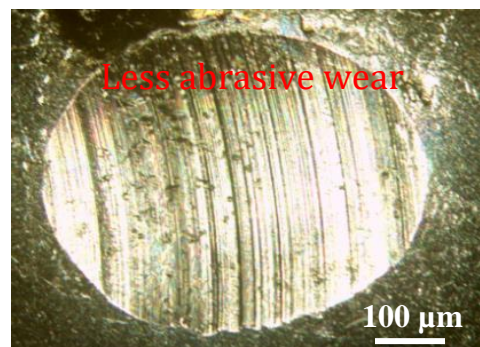
1.5 % EVA + Fig. oil (40 kg)



1.5 % EVA + Fig. oil (80 kg)



SAE 20W-30 (40 kg)



SAE 20W-30 (80 kg)

Figure 8: Wear images of various lubricants (base Fig. oil, 1.5% EVA and SAE 20W-30) under 1200 rpm and 75°C.

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