



Friction and wear characteristics of palm mid olein base stock with Tertiary Butyl-Hydroquinone and Ethylene Vinyl Acetate as bio-based lubricant

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KEYWORDS

Tribology
Bio-based lubricant
Anti-oxidant
Oxidative stability
Viscosity improver

ABSTRACT

In this present study, the physicochemical properties of Palm Mid Olein (PMO) were enhanced by using an anti-oxidant agent, Tertiary Butyl Hydroquinone (TBHQ), and viscosity improver, copolymer Ethylene Vinyl Acetate (EVA) at a certain ratio. The oxidative stability of PMO was evaluated in terms of oil induction time (OIT) by using Differential Scanning Calorimetric (DSC). Meanwhile, the tribological performances of PMO were evaluated by using a pin on disc tribotester. The results were compared to the pure PMO and mineral-based oil VG68 mutually. It was observed that the presence of anti-oxidant TBHQ in PMO base stock was able to increase the oxidative stability about 4.5 times. PMO + TBHQ + EVA also demonstrated a significant reduction in friction coefficient and wear scar diameter compared to that of pure PMO. It showed that the presence of an anti-oxidant was able to prolong the existence of a thin layer of soap film in protecting the contact surfaces.

Received 15 February 2024; received in revised form 7 March 2024; accepted 18 May 2024.

To cite this article: Paiman et al., (2024). Friction and wear characteristics of palm mid olein base stock with Tertiary Butyl-Hydroquinone and Ethylene Vinyl Acetate as bio-based lubricant. Jurnal Tribologi 41, pp.10-23.

1.0 INTRODUCTION

The global market size for lubricants is expected to reach USD 182.6 billion in 2025, approximately a 15% increase compared to the year 2020. This is contributed by the rapid growth of industrialization in the Asia Pacific, the Middle East, and Africa, increasing demand in automotive industries, and process automation in most industries (Ho et al., 2019; Lubricant Market, 2021). It shows that the demand for lubricant oil is grown exponentially with the technological revolution. Lubricant oil is largely consumed as engine oil, followed by hydraulic oil, metalworking fluid, gear oil, turbine oil, and many more. Meanwhile, mineral-based resources remain the largest base stock that produces most of the lubricant oil in the world, followed by synthetic-based and bio-based oil.

The potential of bio-based lubricants to be widely used in engineering applications has been highlighted in the last few decades. This is one of the most studied resources instead of synthetic-based oil which required very high production costs and is not economically funded by most researchers. Masripan et al. (2020) summarized in their study that inedible plant oil promises high viscosity index and high lubricity that prolongs the service life of the equipment and has an ability to carry a higher load, therefore minimizing the trace metal carryover. Singh et al. (2020) reviewed and discovered that vegetable-based oil provided better physical properties, high lubricity, and high bio-degradability in comparison to mineral-based oil. They emphasized that by shifting the resource, industries may save a lot of money in maintaining the equipment and simultaneously protecting the environment. Barbera et al. (2022) concluded that lubricants are made up of 70 – 100% base oil and 0 – 30% of additives or surfactants. This opens the opportunity for the bio-based lubricant to be formulated along with the mineral-based oil and reduced the reliance on mineral resources. In a review conducted by Owuna (2020), they summarized that the utilization of bio-based lubricants in the industry faces many issues and challenges such as low thermal-oxidative stability and poor low-temperature behavior. Therefore, more research is needed to be done comprehensively to overcome the issues. Yahaya et al. (2018) simplified in their review that even the potential of bio-based lubricants appears to be promising, but their commercial deployment is still limited. Therefore, more significant works need to be carried out from the perspective of design optimization up to industrial-scale processing.

1.1 Oxidative Stability of The Bio-based Lubricant

Oxidative stability is defined as the sustainability of the oil and fat from being oxidized with the presence of oxidation catalysts including oxygen molecules, light, heat, pressure, presence of water, contaminants, and many more (Mobarak et al., 2014). The higher oxidative stability indicated that the oil resisted having any reaction that can produce corrosive and unstable compounds thereby affecting the viscosity and lubricity of the oil (Santos et al., 2015; Sutar et al., 2018). Generally, bio-based oil has balanced saturated and unsaturated fatty acid molecules. These unsaturated fatty acids, particularly polyunsaturated compounds such as linoleic and linoleic have a double bond on their ninth and tenth chains and are susceptible to the oxidation process (Rudnick, 2020). This double bond was actively reacted to oxygen molecules and formed free radicals which are then degraded into the form of hydroperoxides, aldehydes, ketones, and carboxylic acids (Maglimao et al., 2020). The rate of oxidation is influenced by the degree of unsaturation whereby the higher the degree of unsaturation, the higher the oxidation rate (Syahir et al., 2017). Oxidation has to be avoided as it resulted in polymerization and degradation of the lubricant oil. Polymerization leads to an increase in viscosity (oil becomes thick and plastic-like structure) while degradation enhanced the production of corrosive products thereby leading to

weakening the oil properties (Zainal et al., 2018; Barbera et al., 2022). Usually, the oxidative stability of lubricant is measured by using Gas-Liquid Chromatography (GLC) machine. It is a powerful and informative tool (Jim, 2016). Commonly, the oxidative level of the oil is determined in terms of oil induction time (OIT) or onset temperature (OT) (Saldana et al., 2013).

1.2 Viscosity Behavior of The Bio-based Lubricant

Some of the bio-based lubricant especially those derived from palm oil has low viscosity properties at ambient temperature. The purpose of adding a viscosity improver is to increase the oil viscosity so that the bio-based oil can be used as a lubricant under specific operating conditions. An application like journal bearing required optimum viscosity in order to create such hydrodynamic pressure to support the entire given loads. The polymer-based additive is widely used as it was able to stabilize the viscosity behavior towards the increase in temperature (Rudnick, 2017). For example, Ethylene Vinyl Acetate (EVA) copolymer is one of the viscosity improvers that has been most paired with bio-based lubricants in the previous study. It has a high molecular weight polymer that can increase the viscosity of the oil at high temperatures by steric effect (Loehle, 2017). Quinchia et al. (2010) studied the effect of adding viscosity improver to the tribological performances. They blended ethylene vinyl-acetate copolymer (EVA) and ethyl cellulose (EC) with sunflower, soybean, and castor oil at a concentration of 4 % and 1 % respectively. They observed that EC responded efficiently in castor oil in reducing wear and friction, especially in mixed lubrication regimes. Meanwhile, EVA is much more effective in both boundary and mixed lubrication regimes. The same team further investigated their research into the real engineering application. They found that the addition of EVA was able to increase the oil viscosity from around 330 % to 420 % at a temperature of 40°C (Quinchia et al., 2014). Moreover, the combination of castor oil, sunflower oil, and EVA at a weight ratio >1 is capable to increase the blended oil viscosity up to 320 cSt which is suitable for gearbox and engine applications. Rani et al. (2015) studied the rheological properties of rice bran oil (RBO) enhanced with low-density polyethylene (LDPE) and EVA. They discovered that the addition of EVA into RBO was significant in improving the oil viscosity only at a low temperature of 40°C. Meanwhile, LDPE was much more effective at higher temperatures. These gave the options to the users depending on their applications.

2.0 EXPERIMENTAL PROCEDURE

In this study, the research methodology is divided into three parts which are oxidative stability test, viscosity analysis, and tribological test by using pin on disc tribotester. The bio-based lubricant that has been used in this study is Palm mid Olein (PMO) and the performance will be compared to the mineral-based oil VG68.

2.1 Oxidative Stability

In this study, the oxidative stability was improved by adding the organic anti-oxidant agent Tertiary-butyl hydroquinone (TBHQ) at concentrations of 450 ppm. The sample of calculation on how to determine the concentration in ppm is shown in Equation (1). TBHQ is outsourced from GC Chemical Sdn Bhd and its technical specifications are listed in Table 1. TBHQ was in powder form, therefore, direct blending by using a motorized stirrer and a little bit of heating around 45°C is needed to ensure that the TBHQ powder is well blended. Visual observation was also executed to confirm that the TBHQ was totally dissolved into the based oil. The oxidative stability was

evaluated in terms of their oxidative induction time (OIT) by using Differential Scanning Calorimetric (DSC) machine following ASTM E1858.

$$TBHQ (ppm) = \frac{TBHQ (gram)}{TBHQ (gram) + Solution (gram)} \times 1000000 \quad (1)$$

Table 1: Technical specifications of TBHQ.

| Details | Specification |
|-------------------------|---|
| Product name | Tertiary – Butyl Hydroquinone (TBHQ) |
| Chemical name | 2-(1,1-Dimethylethyl)-1,4-benzenediol |
| Appearance/ Description | White to a crystalline powder |
| Molecular formula | $C_{10}H_{14}O_2$ |
| Functional class | Antioxidant |
| Solubility | Soluble in alcohol and insoluble in water |
| Purity (Assay) | 99% of $C_{10}H_{14}O_2$ |

2.2 Viscosity Improver (VI)

Additive containing polymeric compound ethylene vinyl-acetate (EVA) was used as viscosity improver (VI) in this study. It was outsourced from Sigma Aldrich, United States of America (USA). The technical specifications of EVA are listed in Table 2. EVA copolymer was blended with palm oil-based lubricant by using a motorized stirrer similar to TBHQ powder at a concentration of 1.3 %, 1.5 %, and 2 %. The sample calculation of EVA concentration is shown in Equation (2). Since EVA copolymer has a high melting point, the blending process took 2 hours of continuous stirring at a temperature of 120°C. After that, the blended oil is left at ambient temperature for natural cooling before being ready to be used.

$$EVA (\%) = \frac{EVA (gram)}{EVA (gram) + Solution (gram)} \times 1,000,000 \quad (2)$$

Table 2: Technical specifications of copolymer EVA.

| Details | Specification |
|------------------------|--|
| Product name | Poly (Ethylene-co-vinyl-acetate) |
| Appearance (color) | Colorless |
| Appearance (form) | Beads (0.6 – 0.8 mm in diameter) |
| Melt index (8g/10 min) | 190°C/2.16 kg |
| Melting point | 75°C |
| Density at 25°C | 0.9480 g/ml |
| Content | 25 wt% vinyl acetate and enriched with 200 – 900 ppm BHT |

2.2.1 Viscosity Analysis

In this study, the viscosity of the lubricant was measured by using a rotational viscometer. This machine is equipped with stainless steel spindle and has high accuracy. The density of the lubricant was first determined by using a hydrometer. Then, the lubricant was poured into a 250 ml beaker and placed on the heater. The heater, which is also equipped with a magnetic stirrer was placed just below the spindle. During the testing, the lubricant was heated and stirred slowly until reached the temperature of 100°C. The test was conducted by following ASTM D2983. The viscosity readings were recorded automatically by the machine. Once the lubricant temperature reached 100°C, then it was cooled down naturally to the ambient temperature. Similarly, the viscosity values were also recorded during the cooling process. Then, the average viscosity values for the particular temperature were averaged to get the actual reading.

2.3 Pin on Disc

Pin-on disc tribotester was used in this study as shown in Figure 1. This was to replicate the actual material in contact with a sliding motion. In this testing, the friction coefficient and wear scar diameter were evaluated. The friction coefficient value was calculated by using Equation (3). Meanwhile, the wear scar diameter was measured by using a high-definition microscope.

$$\text{Friction coefficient, } \mu = \frac{F}{N} \quad (3)$$

Where F is friction force, and N is applied load.

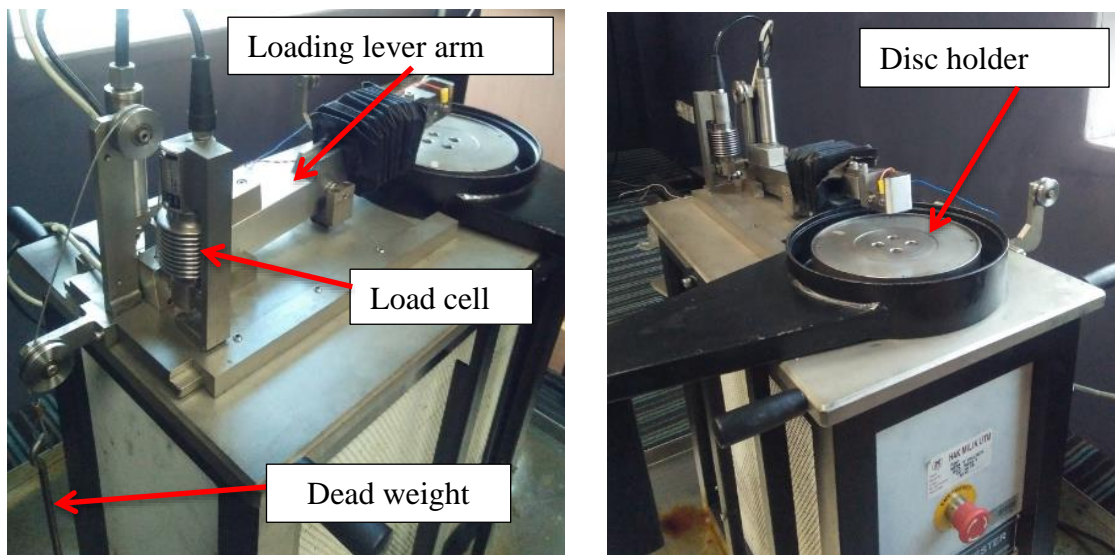


Figure 1: Pin on disc tribometer.

2.3.1 Experimental Conditions

The experimental condition for the test using a pin on the disc tribotester is tabulated in Table 3.

Table 3: Experimental condition for the pin on disc test.

| Details | Description |
|---------------------|------------------------------------|
| Applied load | 0.5 kg; 1.0 kg; 1.5 kg |
| Rotational speed | 0.5 m/s; 1.0 m/s; 1.5 m/s; 2.0 m/s |
| Duration | 1 hour |
| Temperature | Ambient temperature |
| Standard | ASTM G99 |
| Amount of lubricant | 50 ml |

3.0 RESULTS AND DISCUSSION

3.1 Oxidative Stability of Palm mid Olein (PMO)

The oxidative stability of all tested lubricants was evaluated by using Differential Scanning Calorimetric (DSC) analysis. The DSC analysis can be carried out on various key parameters including oxidative induction time (OIT), onset, and peak maximum temperature (Saldana et al. 2013). In this present study, the effect of adding TBHQ into PMO was measured by OIT as illustrated in Figures 2. The oxidation experiment was conducted in isothermal mode. OIT is best described as the period of time with consistent heat flow and the time length is considered the measurement of lipid stability. A liquid sample of about 12 mg was first loaded into the DSC pan by using a Pasteur pipette. The hermetically sealed DSC pan must have a pinhole so that the oxygen can be purged and reacted to the liquid sample. The sample was then heated rapidly at a rate of 10°C/min under Nitrogen (N₂) purging until it reaches the corresponding temperature of 180°C (T1). After that, the sample is continuously heated with constant heat flow but under oxygen atmospheric conditions. The OIT is calculated from the time of Oxygen being induced (T1) until the heat flow signal deviates from the baseline (T2). This point was also known as the end of induction time and indicates the starting point of the oxidation process or initiation stage (Saldana et al., 2013). The length of induction time (T2-T1) presented the oil stability measurement and there is no chemical reaction taking place. This can be simplified that the longer the induction time, the more stability of oil towards oxidation.

In Figures 2, it is clearly shown that PMO+TBHQ demonstrated a longer oxidative induction time (OIT) compared to PMO at a constant temperature of 180°C. PMO+TBHQ recorded an OIT of 53.21 minutes while PMO alone can only sustain until 12.3 minutes. Therefore, it can be stated that adding 0.045 % of TBHQ improved the oxidative stability of the PMO by approximately 4.5 times. As expected for VG68, the end of induction time (T2) was not detected after running the analysis for 60 minutes, and this indicates that the OIT for VG68 is longer than PMO and PMO+TBHQ.

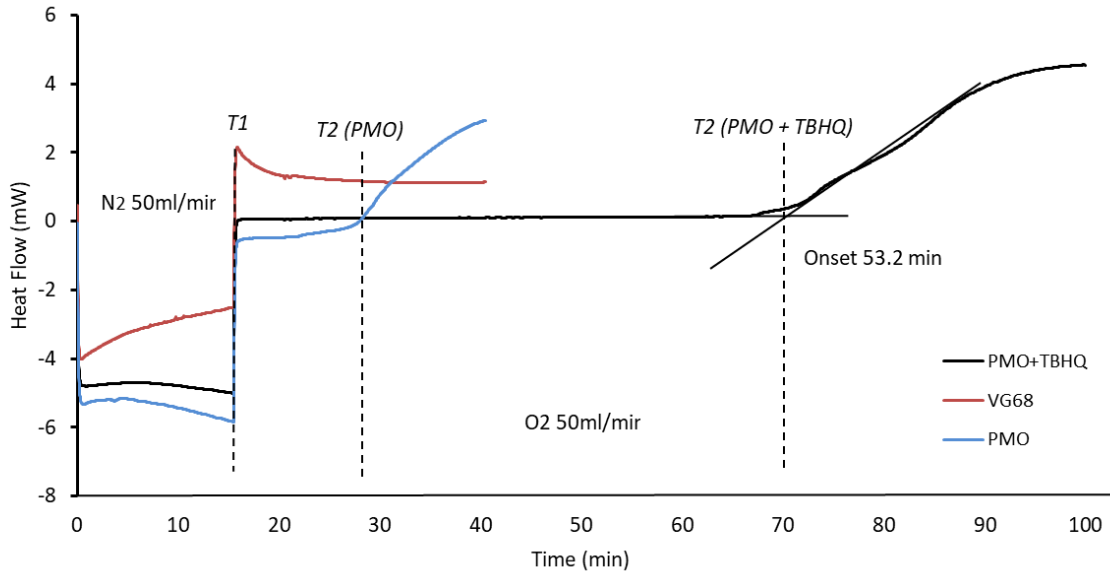


Figure 2: Oxidative induction time (OIT) for PMO, PMO+TBHQ, and VG68.

3.2 Viscosity Profile of PMO Added with Viscosity Improver EVA Co-Polymer

The effect of adding viscosity improver (VI), ethylene vinyl acetate (EVA) copolymer to the PMO was shown in Figure 3. It was observed that the higher the concentration of EVA being added, the higher the viscosity of the oil at a particular temperature. PMO has low viscosity at 40°C because of its physical properties itself. VG 68 has higher viscosity at 30°C and 40°C compared to PMO, however, it showed an identical viscosity profile with PMO at temperatures of 50°C onwards. Since this study is targeting the application that is operating at temperatures of 35°C to 40°C, therefore the addition of a viscosity improver is needed to serve the purpose. The additional 1.3 % concentration of EVA co-polymer has increased the viscosity index (VI) of PMO to about 19.16 %. Even higher viscosity oil is good in providing thickening effects, but the fluidity and pour point capability must also be considered. In this study, the pour point of PMO added with 450 ppm of TBHQ and a few concentrations of EVA was evaluated by using ASTM D97-93. It was noticed that 1.3 %, 1.5 %, and 2 % of EVA resulted in pour points of 6°C, 12°C, and 15°C respectively.

The viscosity of the lubricant oil is critical, especially in journal-bearing applications as the viscosity is one of the main mechanisms to create the hydrodynamic condition. It also determined the layer of thickness film that separated the mating surfaces. The lubricant must be able to experience less variant of viscosity following the increase in rotational speed, temperature as well as applied load. Al-Araji and Sarhan (2011) stated that the reduction in viscosity caused the incapability to provide sufficient protection against asperities contact.

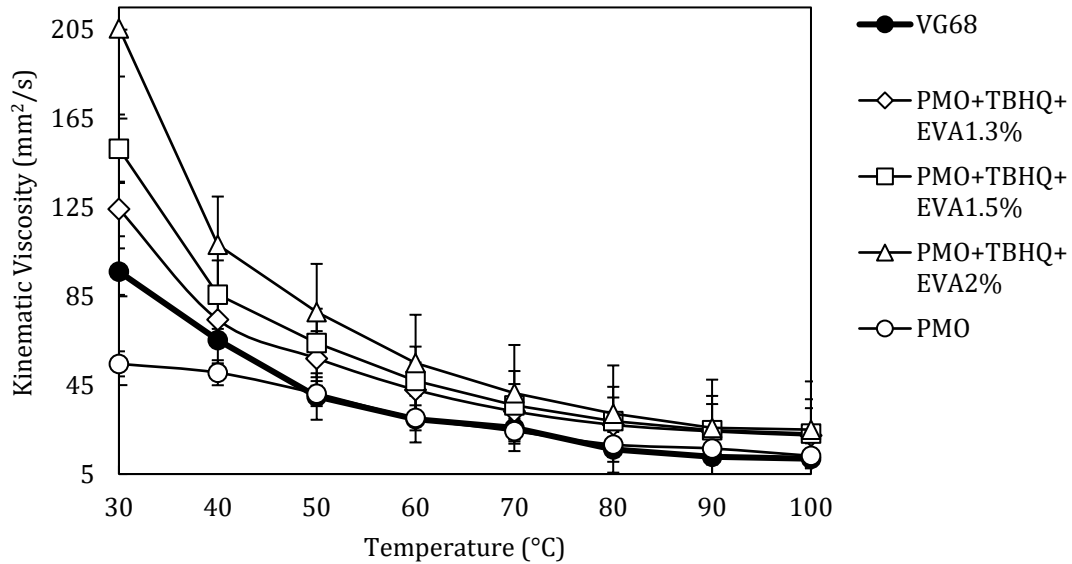


Figure 3: Viscosity profile of PMO, PMO+TBHQ+EVA, and VG68.

3.3 Friction Behaviors of PMO+TBHQ+EVA Using Pin on disc Tribotester

The friction behaviors of PMO added with TBHQ and EVA were plotted in Figure 4(a) for the load of 0.5 kg, (b) 1.0 kg, and (c) 1.5 kg, respectively. For a load of 0.5 kg, PMO+TBHQ+EVA showed a lower friction coefficient compared to PMO and VG68. However, it increased as speed increased before decreasing at the highest speed of 2 m/s. PMO shows more consistent trends at all sliding speeds. There are no significant differences observed for PMO and PMO+TBHQ at a medium load of 1 kg. The only differences were seen at higher speeds 2 m/s where PMO+TBHQ+EVA exhibited a lower friction coefficient compared to PMO. At a higher load of 1.5 kg, huge differences were noticed at a lower speed whereby PMO+TBHQ+EVA shows a lower friction coefficient compared to others. Only small differences were found at higher speeds and PMO+TBHQ+EVA still demonstrates a lower friction coefficient. VG68 experienced an increase in friction as sliding speed increased at a load of 1 kg and 1.5 kg. In response to the increase in applied load, PMO recorded a friction coefficient increment of about 0.062 or 54.42 % on average while PMO+TBHQ+EVA showed a lower friction increment at 0.033 or 41.03 % respectively. On top of that, VG68 shows the least increment at about 34.64 % even demonstrating higher friction coefficient value at an average of 0.155.

In the pin-on-disc machine, the experiment was conducted in the open system. The tendency of the oil to move to the peripheral area was higher at a higher rotational speed. In comparison to palm oil-based lubricant, VG68 possibly has a lower natural affinity to the contact surface. It was easily slipped away to the peripheral areas at a higher speed resulting in less protection to the contact surface. This is the possible reason why VG68 exhibited quite high friction at the higher speed of 2 m/s for each load, compared to PMO and PMO+TBHQ+EVA.

The main reason polymeric-based additives are being added to PMO is to improve the viscosity behavior and increase the viscosity index (VI) of the base oil. The polymeric additive is able to minimize the variations of viscosity toward temperature rise and provided high sustainability to

the existence of a thin layer of soap film to protect the mating surfaces (Rudnick, 2017). Polymer compounds have high thickening efficiency and good shear stability. The increase in temperature increased the solvency power thus, resulting in an increase in coil expansion and thickening efficiency (Covitch and Trickett, 2015). Therefore, the polymer counteracted the intrinsic thinning of the base oil. Similar findings were found by Smeeth et al. (1996) where the addition of EVA was able to maintain low friction, especially at low speed. They suggested that this behavior is observed when the contact inlet is filled with adsorbed polymer layers that cause them to become more viscous and thicker than the bulk solution. This situation leads to greater lubricant entrainment and provides thicker film protection, thus reducing friction and wear. As the sliding speed and load are increased, the presence of anti-oxidant TBHQ surely helped in prolonging the existence of the thin layer of soap film formed by fatty acid molecules. It was proven that the combination of these TBHQ and EVA in PMO base oil has shown a significant impact in reducing friction.

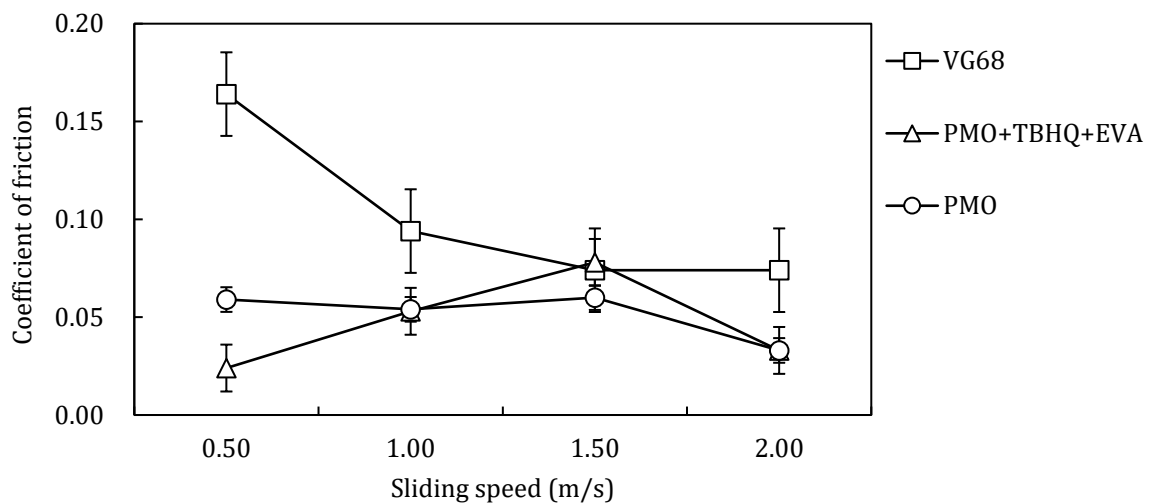


Figure 4(a): Friction coefficient trends at a load of 0.5 kg.

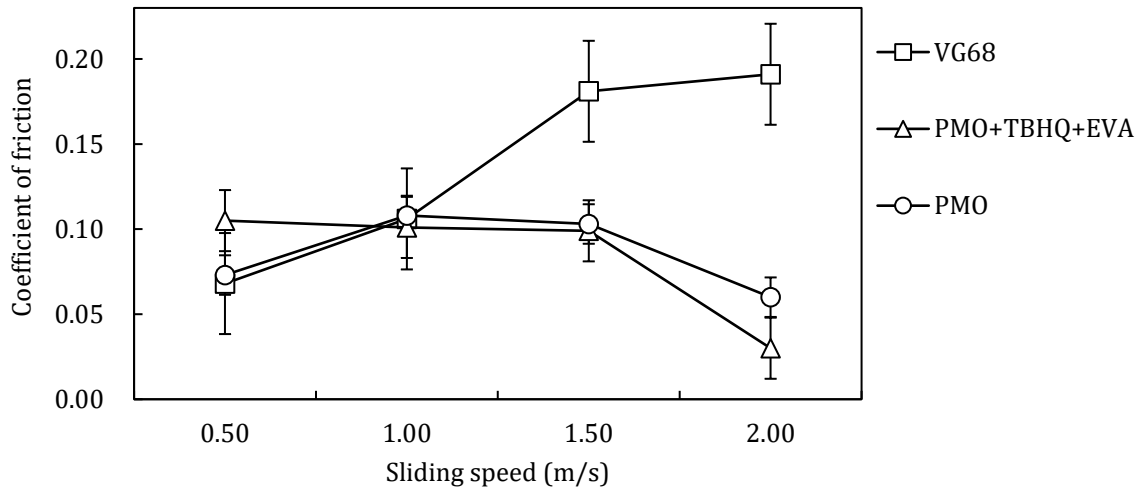


Figure 4(b): Friction coefficient trends at load of 1 kg.

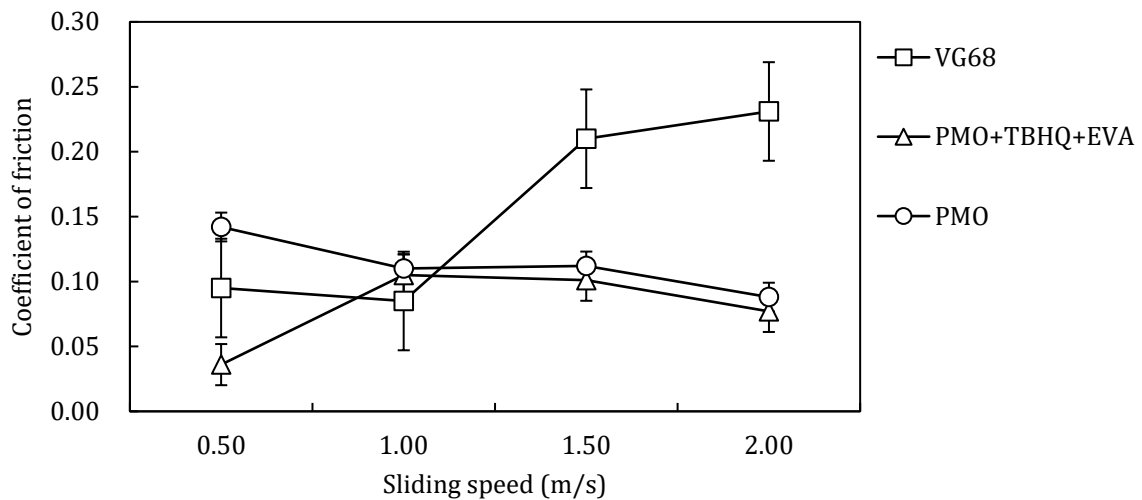


Figure 4(c): Friction coefficient trends at a load of 1.5 kg.

3.4 Wear Scar Diameter Analysis of PMO+TBHQ+EVA

The wear scar diameter (WSD) analysis of all tested lubricants was plotted as shown in Figure 5 (a) for 0.5 kg, (b) 1 kg, and (c) 1.5 kg. It was found that VG68 shows no significant impact on the increase in sliding speed and applied load as it was able to maintain the low WSD at all testing conditions and even experienced a higher friction coefficient in the previous analysis. The results were contradicted for both PMO and PMO+TBHQ+EVA in which they exhibited lower friction but experienced higher WSD in comparison with VG68. A similar finding was found by other researchers (Jayadas et al., 2007), who suggested that friction behaviors do not proportionally

affect the -wear performances. As discussed in the earlier analysis, the reaction of fatty acids with metal surfaces has degraded the thin layer of soap film and leads to severe damage on the contact surface. When sliding speed and load are increased, more surface area is contacted, which leads to an increase in temperature (Zulkifli et al., 2016). Mobarak et al. (2014) believed that the increase in speed definitely embarked on the initiation stage of oxidation. By comparing PMO and PMO+TBHQ+EVA, it was observed that PMO+TBHQ+EVA shows lower WSD in all cases. It proved that the addition of both additives TBHQ and EVA was able to reduce the oxidation effects and sustain the formation of a thin layer of soap film to protect the contact surface.

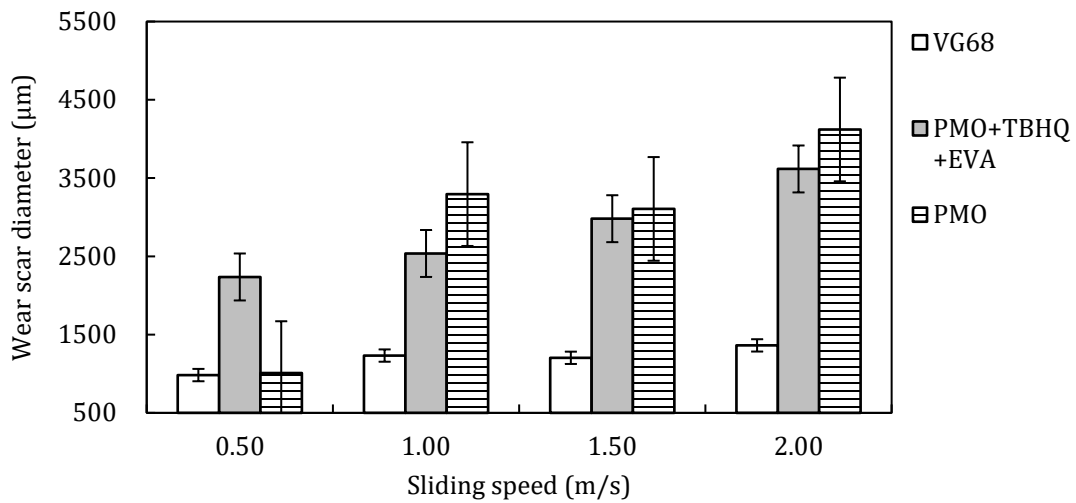


Figure 5(a): The trend of WSD at a load of 0.5 kg.

Figure 5(b): The trend of WSD at a load of 1 kg

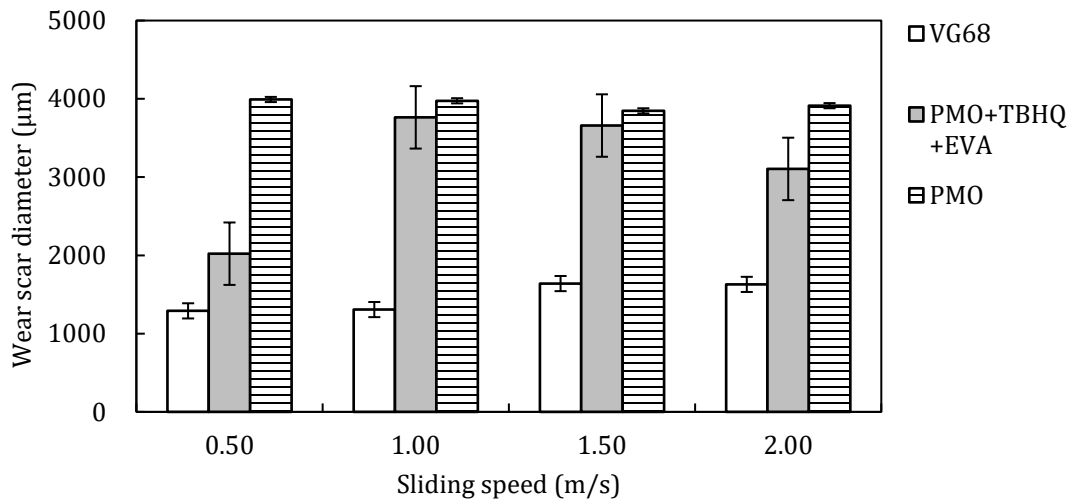
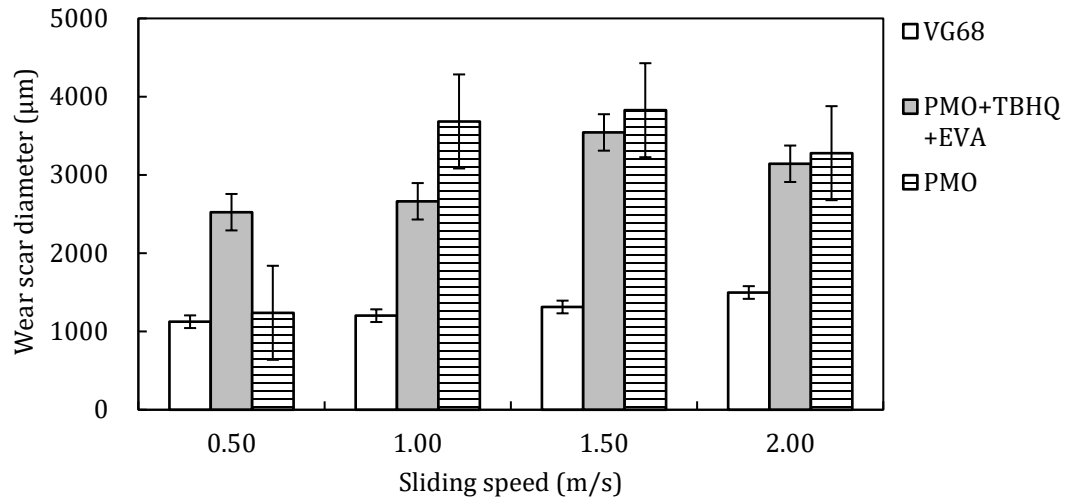


Figure 5(c): The trend of WSD at a load of 1.5 kg.

CONCLUSIONS

In this study, the antioxidant and viscosity improver have been added to the bio-based lubricant to enhance their properties. The tribological performances of this oil were evaluated and compared to the mineral-based oil mutually. It can be concluded that:

1. The addition of TBHQ into PMO was able to increase the oil induction time (OIT) by 4.5 times. TBHQ acts as a radical scavenger that breaks the auto-oxidation process, thus improving the oxidative stability of the PMO.

2. The additional TBHQ and EVA was able to reduce the friction coefficient as compared to pure PMO and mineral-based oil VG68
3. The addition of TBHQ and EVA tends to reduce the wear behavior when compared to pure PMO but is still higher than mineral-based oil VG68

ACKNOWLEDGEMENT

This research was supported by the Ministry of Higher Education of Malaysia and Universiti Teknologi Malaysia (UTM) through the Encouragement Research Grant (31J31), and Fundamental Research Grant (23H32).

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