

Friction and Wear Characteristics of Bio-Lubricants Containing Clove Oil as Antioxidant

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ABSTRACT

Vegetable oils as bio-lubricants have poor oxidation stability due to the unsaturated fatty acids in their composition. The oxidation in bio-lubricants can occur because they are exposed to heat, light, and oxygen. In this research, clove oil was used to reduce oxidation in vegetable oils. The effects of blending clove oil (0, 5, and 10% wt) with virgin coconut oil (VCO), hydrogenated coconut oil (HCO), and palm oil that have been exposed to oxygen for 30 days have been investigated. Viscometer and pin-on-disk tests were used to determine the physical and tribological properties of the bio-lubricants. The results show that the addition of clove oil to these oils could reduce the oxidation process. It was indicated by the reduced percentage increase in the dynamic viscosity of 10% wt clove oil in VCO of around 5.41% for 30 days. Results of wear rate indicated that the effect of adding clove oil to VCO and HCO was better than that of palm oil, where the wear rate of VCO and HCO decreased with an increasing clove oil composition. Meanwhile, their coefficients of friction were only affected at low speeds (500 rpm).

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1. INTRODUCTION

Nowadays, most of the total production of lubricants in the world comes from petroleum-based or mineral oil. The lubricant is non-renewable and toxic, and it is harmful to humans and the environment due to its low degradation and high toxicity [1]. Lubricants discharged into the environment can cause contamination in the air, soil, and water. So, the scientist is trying to develop a lubricant that has greater biodegradability and less toxicity. Lubricant obtained from bio-based sources, such as vegetable oil, is an alternative to

traditional mineral oil. Vegetable oils as bio-lubricants have excellent physicochemical properties, such as high viscosity indexes and flash points, good resistance to shear, and high biodegradability [2]. Despite the great potential of vegetable oil as a bio-lubricant, it is not widely commercialized due to poor oxidation stability and poor low-temperature properties [3].

Mineral oil is still used dominantly in industry, and the consequences of the disposal of lubricants are increasing each year, causing environmental damage [4]. Governments in

Europe and America have made regulations to encourage the use of environmentally friendly lubricants to mitigate the effects of the disposal of the lubricant [5]. The evolution of regulation has increased environmental awareness to minimize inappropriate disposal of lubricants and has also increased demand for biodegradable lubricants from vegetable oils. They present high lubricity and good metal adherence due to their fatty acid content [6]. The fatty acids can form a monolayer film using their closely packed polar carboxyl groups and can drastically decrease friction and wear in the boundary lubrication regime [7].

Vegetable oils have large amounts of unsaturated fatty acids that lead to poor thermal stability and are easily oxidized and damaged. The effect of oxidation in sunflower oil has been found by Fox et al. [8], who have shown an increase in wear and friction. Vegetable oils can be oxidized because they are exposed to heat, light and oxygen. Oxidative stability of vegetable oils is measured by induction time based on resisting to react with oxygen and breaking down. A longer induction time indicates that an oil is more resistant to oxidation. Factors that determine an oil's stability are the number of antioxidants, the type and ratio of fats, and the extent of refining. Antioxidants can protect against oxidation and play a key role in an oil's oxidative stability. Some vegetable oils contain various characteristic antioxidants, such as virgin olive oil, which contains mixtures of secoiridoids [9]. Polyunsaturated fats have two or more double bonds, which makes them prone to oxidation. The Refinement process on vegetable oils lowers the oxidative stability of the oil because the process strips away natural antioxidants and also exposes the oil to heat.

To reduce oxidation in vegetable oils, numerous things have been done by using several chemical modifications, such as inhibitors of oxidation, water-oil interfaces and antioxidant effectivity, natural antioxidants, and modifications to improve oxidative stability [10]. In natural sources such as vegetables, aromatic plants, and cereals, there are many types of compounds with natural antioxidant properties, such as phenolic compounds. These compounds can function as singlet and triplet oxygen quenchers to inhibit photo-oxidation and auto-oxidation, respectively, as well as free radical scavengers and peroxide decomposers [10]. The blending technique has been tested by Li et al. [11] and Ali

et al. [12] to improve the oxidative stability of soybean oil with the addition of rice brain oil as a natural antioxidant.

Indonesia is a tropical country that has an abundant source of clove oil and is the primary producer of clove oil in the world. It supplies more than 80% of its output to its domestic market [13]. The source of clove essential oil is obtained by hydro-distillation of the tree's different components, including leaves, cloves, and stems. The chemical composition of clove essential oil has some forty components [13]. Eugenol is the main component (68.05–82.38%) of clove oil, which has strong antioxidant activity [14]. Antioxidants are compounds that can delay or inhibit the oxidation of lipids or other molecules by inhibiting the initiation or propagation of oxidizing chain reactions [15]. In this paper, the effect of blending vegetable oils and clove oil as natural antioxidants as bio-lubricants will be investigated in terms of physical and tribological properties.

2. MATERIALS AND METHODS

2.1 Oil samples

In this research, coconut oils and palm oil were used as vegetable base oils. The used coconut oil types were without heat processing (VCO) and with heat processing (HCO) [16]. All three oils were obtained from commercial cooking oils available in supermarkets. The chemical properties of these oils in terms of saturated and unsaturated fatty acids are shown in Table 1. Clove oil was used as an additive with variations of 0, 5, and 10% wt and was blended into VCO, HCO, and palm oil. Base oils added with clove oil were stirred using a digital magnetic stirrer (MS 200) to ensure that the oils were perfectly blended. Stirring was conducted for 15 minutes at 90 °C with a speed of 200 rpm. Afterwards, the oil samples were measured for viscosity, wear, coefficient of friction, scar diameter of the pin, and scar width of the disk. The schematic diagram of sample oil preparation is shown in Figure 1.

Table 1. Composition of unsaturated and saturated fatty acids (UFA and SFA) in VCO, HCO, and palm oil [16,17].

No.	Bio-lubricant	UFA [%]	SFA [%]	Ratio UFA and SFA
1.	VCO	1.21	98.44	0.123
2.	HCO	7.89	91.64	0.086
3.	Palm oil	7.47	84.42	0.088

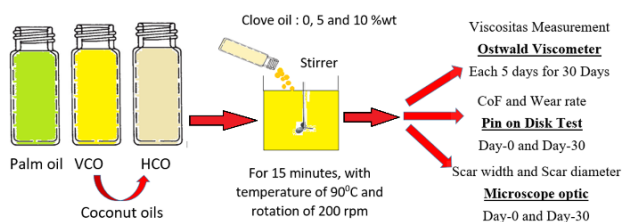


Fig 1. Schematic diagram of sample oil preparation.

2.2 Measurement of viscosity

The measurement of viscosity was performed using an Ostwald viscometer. The viscosity of the blended oils was measured every 5 days for 30 days. These oils had been exposed to oxygen to ensure that natural oxidation occurred. Oil samples of about 10-15 ml were inserted into the viscometer. The oil sample would rise to the upper bulb, and its surface would pass through the upper reservoir line. The bottom bulb was filled with about half the sample, then allowed to drain under gravity. The viscosity of the oil sample was determined by comparing the flow time and density with those of distilled water.

2.3 Wear and coefficient of friction (CoF) measurements

Wear and CoF measurements were performed using a pin-on-disk tribometer (Figure 2). The test was done by means of a rotating disk and a pin that pressed the sample oil. Wear volume was measured by determining weight loss of the disk after rotating for 50 minutes with different loads and speeds. To measure the weight loss of the disk, an electronic balance (SF-400C) with a pre-cession weight of 0.01 g. The weight loss of the disk was used to determine the volume of the worn disk and the wear rate of the worn disk. The mechanical properties and dimensions of the pin and disk are shown in Table 2.

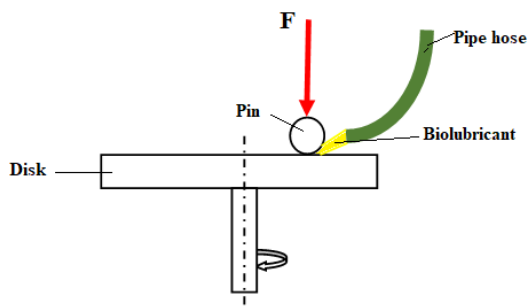


Fig. 2. Schematic diagram of testing wear and CoF using pin-on-disk apparatus.

During pin-on-disk testing, the arm of the pin holder would undergo deflection due to the frictional force between the pin and the rotating

disc. The large deflection that occurred on the pressing arm would be passed to the load cell in the form of voltage changes, which were representations of the friction force that occurred. Then, the voltage data was changed to force data calibration.

Table 2. Mechanical properties of pins and disks.

Element	Pin	Disk
Material	Stainless Stell 4450	AISI 1015
Hardness (BHN)	610	135
Ultimate Stress (GPa)	2.104	0.465
Shear Stress (GPa)	1.58	0.35
Dimension (mm)	7.938 dia	160 x10

The surface roughness of the disk was 0.972 Ra, and the surface roughness of the pin was mirror polished. The contact between the rough surface (disk) and the smooth surface (pin) resulted in the worn surface of the disk due to abrasive wear. To determine abrasive wear on the disk surface, the Archad wear equation was used based on the theory of asperity contact, and the equation to calculate the total volume of wear is shown in Equation 1.

$$Q = kW \tag{1}$$

Where k is known as a wear coefficient or wear rate. W is the normal load applied to the surface by its counterbody.

2.4 Surface marfology of the pin and disk

Observation of the surface marfology of pins and disks was conducted after the disk rotated for 50 minutes with loads of 50 and 100 N and speeds of 500 and 1400 rpm, respectively. The observation of the surface marfology of the pin and disk was carried out by using a stereo microscope (Olympus SZX 10) to analyze the forms of wear and measure the size of the scar diameter of the pin and the scar width of the disk.

3. RESULTS AND DISCUSSION

3.1 Results

Lubricant has an important role in reducing wear and friction on the two contact surfaces. Wear that occurs on two contact surfaces is caused by damage to a solid surface due to the relative motion between the surface and a contacting substance.

The quality of bio-lubricants in the contact area depends not only on their physical properties but also on their chemical properties. The addition of antioxidants to bio-lubricants will affect their physical and tribological properties. The most important physical properties of lubricant in the elastohydrodynamic lubrication regime are viscosity, whereas in mixed lubrication, not only physical properties (i.e., viscosity) but also chemical properties (i.e., fatty acids) of lubricant are most important. Both their properties as lubricants will influence tribological properties (i.e., wear and CoF) on the contacting surfaces.

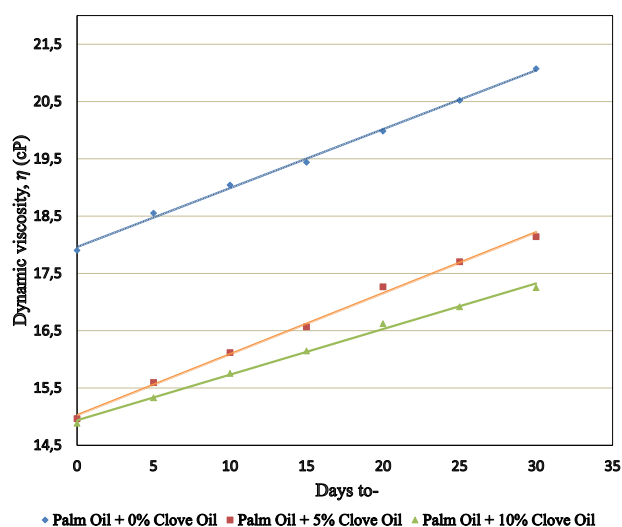


Fig. 3. Changes in the dynamic viscosities of palm oil after adding percentages of clove oils for 30 days exposure to oxygen.

The results of the measured dynamic viscosity of bio-lubricant can be seen in Figures 3 and 4. From the figures, when the vegetable oils and clove oil were blended, the dynamic viscosity of the resultant oils decreased due to the dynamic viscosity of clove oil being lower than that of bio-lubricants. After blending and exposure to oxygen, the dynamic viscosity of the oils increased linearly with the increasing days. The highest increase in dynamic viscosity occurred in the oils without the addition of clove oil; this is indicated by the gradient of a large increase in dynamic viscosity. Except for palm oil, where the increase in dynamic viscosity was larger for palm oil with the addition of 5% wt of clove oil.

The summary of changes in the dynamic viscosity of bio-lubricants after the addition of clove oil (i.e., 0, 5, and 10% wt) from day 0 to day 30 is shown in Table 3. From the table, it can be seen that the addition of clove oil to VCO, HCO, and palm oil increased the dynamic viscosity of the

sample oils at 27 °C. The oil with the highest percentage increase in the dynamic viscosity of the sample oils was palm oil.

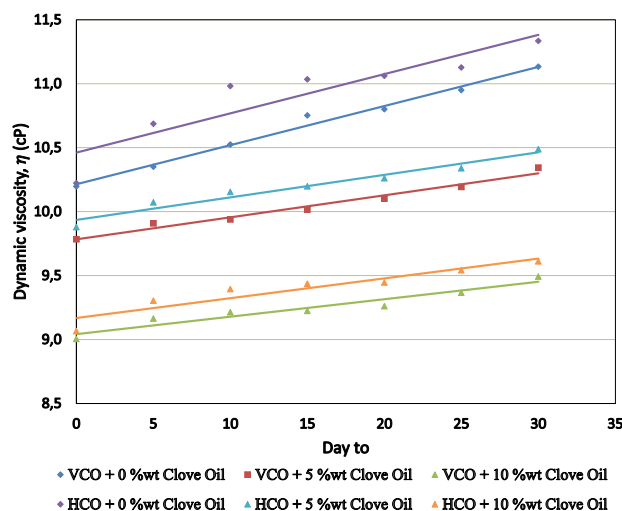


Fig. 4. Changes in the dynamic viscosities of VCO and HCO after adding percentages of clove oil for 30 days exposure to oxygen.

Table 3. Dynamic viscosity of bio-lubricants before and after adding clove oil with a percentage of 0, 5, and 10% wt after being exposed to oxygen for 30 days.

Bio-lubricant	Clove oil (%wt)	Dynamic viscosity at 27°C [cP]	
		Day-0	Day-30
VCO	0	10.20 ± 0.036	11.13 ± 0.013
	5	9.78 ± 0.006	10.34 ± 0.010
	10	9.00 ± 0.005	9.49 ± 0.010
HCO	0	10.22 ± 0.041	11.33 ± 0,006
	5	9.88 ± 0.006	10.49 ± 0.004
	10	9.07 ± 0.025	9.61 ± 0.011
Palm oil	0	17.90 ± 0.021	21.07 ± 0.065
	5	14.96 ± 0.039	18.14 ± 0.019
	10	14.88 ± 0.043	17.25 ± 0.027

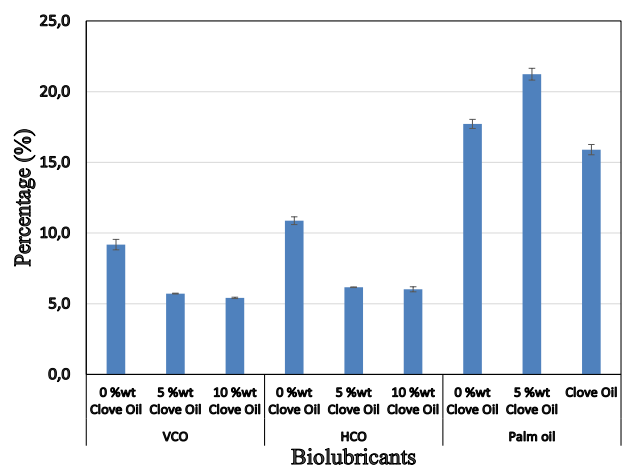


Fig 5. Percentage of increased dynamic viscosities of VCO, HCO, and palm oil after adding percentages of clove oil for 30 days exposure to oxygen.

The percentage of increasing dynamic viscosity of sample oils after adding clove oil can be seen in Figure 5. From the figure, it can be concluded that the addition of 10% wt clove oil to VCO and HCO could significantly suppress the increase in the dynamic viscosity of the sample oils, around 5.41% and 6.01%, respectively, after exposure to oxygen for 30 days. In contrast, palm oil with the addition of 5% wt clove oil could only reduce the increase in the dynamic viscosity to around 15.9%.

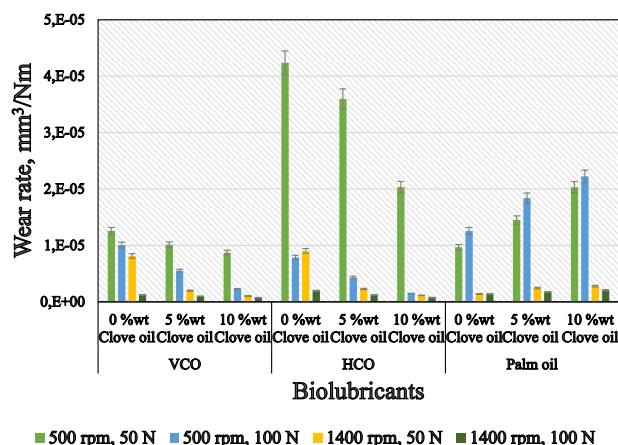
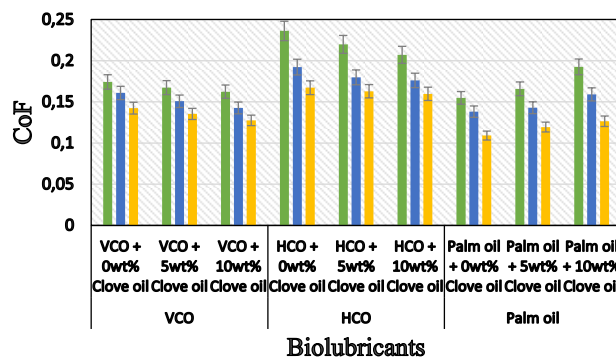


Fig. 6. Wear rate of disks with different loads and speeds lubricated by bio-lubricants with different compositions of clove oil.

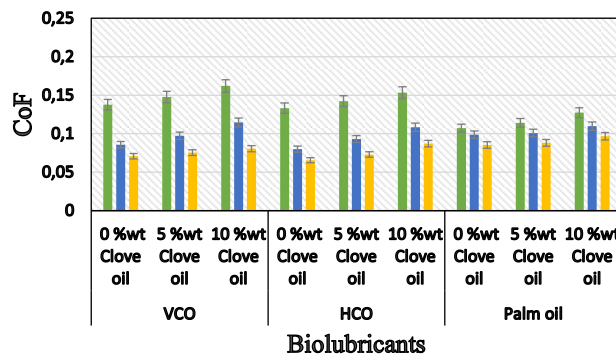
Pin-on-disk testing was conducted at low speed (500 rpm) and high speed (1400 rpm). The two levels of speed represent regimes of lubrication, which are boundary and mixed lubrication regimes, respectively, based on a theoretical prediction of the Stribeck curve [16]. Two loads used were 50 and 100 N, which were equivalent to 1.2 and 1.5 GPa, respectively, based on Hertzian pressure.

For each test, the wear coefficient or wear rate, k , was calculated from Equation 1, where volume, Q , was measured from the weight of removing the surface of the disk times density. The results of a series of wear tests are plotted as a function of bio-lubricants in Figure 6. The present experiments give wear rates varying over more than two orders of magnitude (7.147×10^{-7} to 4.236×10^{-5}). Wear rates were high for all bio-lubricants at low speeds with loads of 50 and 100 N. The highest wear rate occurred on disks lubricated by HCO with 0 and 5% wt of clove oil, where severe wear occurred. However, at high speed (1400 rpm), the wear rate of the disk decreased with increasing load. The lowest wear

rate occurred on VCO and HCO after adding 10% wt of clove oil. For both rotations (500 and 1400 rpm), the effect of adding clove oil to HCO and VCO is that the higher the load, the lower the wear rate; conversely, for palm oil, the higher the load, the higher the wear rate.



(a)



(b)

Fig 7. CoF of different bio-lubricants and loads at speeds (a) 500 rpm and (b) 1400 rpm by using pin on disk testing.

Figure 7 illustrates the effect of applied load and rotational speed on the friction coefficient of bio-lubricants with various contents of clove oil. Their CoF of bio-lubricants almost decreased linearly with the applied load. Additionally, it should be noted that the CoF of VCO and HCO with any composition of clove oil was always lower than that of pure base oil under the applied loads and speeds. At low speed (500 rpm) and the increased clove oil concentration, the CoF decreased slightly on VCO and HCO. A one-way ANOVA revealed that there was no statistically significant difference in mean CoF for HCO between clove oil concentration and load ($p > 0.05$). However, under increasing speed, clove oil in palm oil did not have an effect on decreasing CoF.

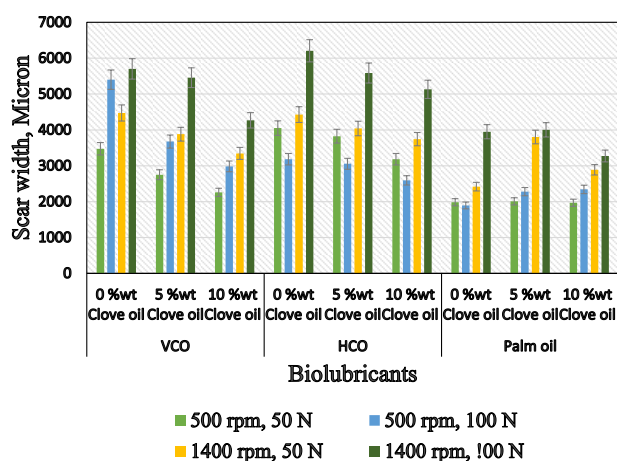


Fig. 8. The scar width of disks with different biolubricants, loads, and speeds.

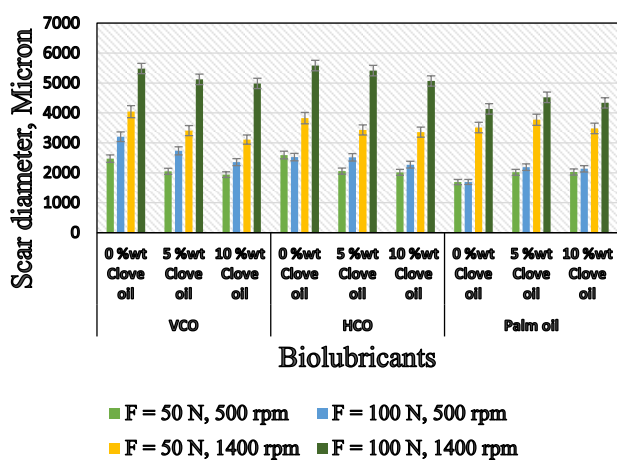


Fig. 9. The scar diameter of pins with different biolubricants, loads, and speeds.

Figures 8 and 9 show the scar width of the disks and the scar diameter of the pins, respectively, with different lubricant oils, loads, and speeds. Under low speed (500 rpm) with contact pressures of 1.2 and 1.5 GPa (i.e., 50 and 10 N, respectively), the scar width of the disk and the scar diameter of the pin were lower than those at high speed (1400 rpm). The effect of clove oil in VCO and HCO for both speeds decreased the scar diameter and width of the pin and disk; otherwise, clove oil in palm oil increased.

The morphologies of the worn surfaces of disks lubricated by VCO and HCO are shown in Figures 10 and 11 with various compositions of clove oil under the applied loads of 50 and 100 N at speeds of 500 and 1400 rpm. Under the lubrication of 0% wt clove oil in HCO with loads of 50 and 100 N and speeds of 500 rpm, the worn surfaces were not only very rough but also characterized by severe plastic deformation (Figures 10a and 10b), which

suggests that the disks suffer serious wear. The force applied to the surfaces in solid-state contact produces elastic deformation initially, and if the load is sufficiently high, plastic deformation or strain is produced in the material.

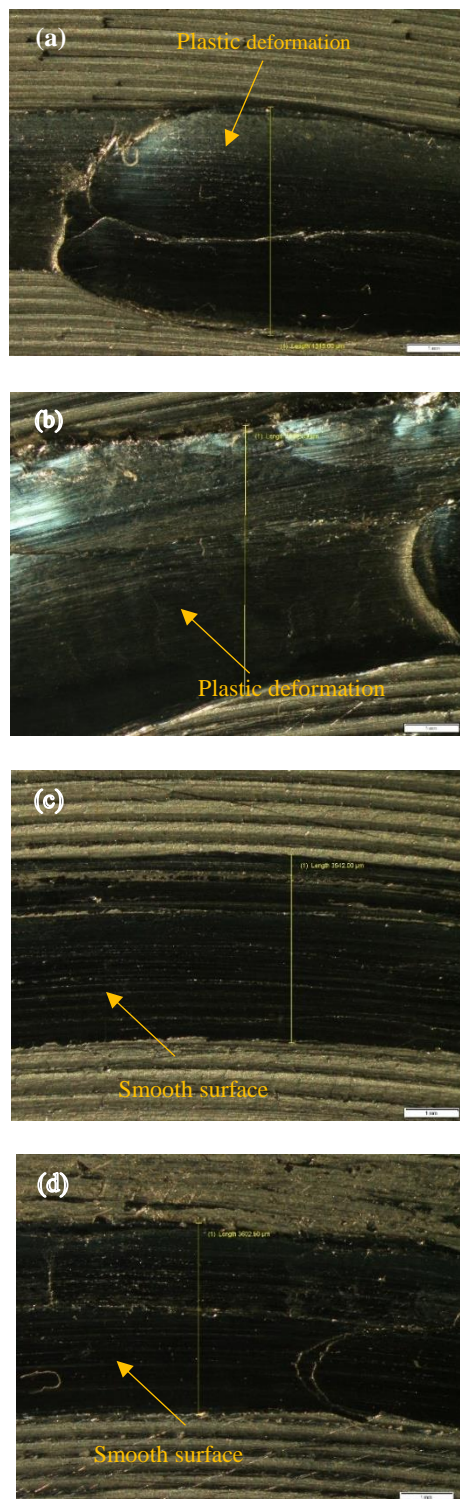


Fig. 10. Surface morphologies of disks lubricated by HCO with 0% wt clove oil at (a) 50 and (b) 100 N and VCO with 10% wt clove oil at (c) 50 and (d) 100 N with a rotational speed of 500 rpm.

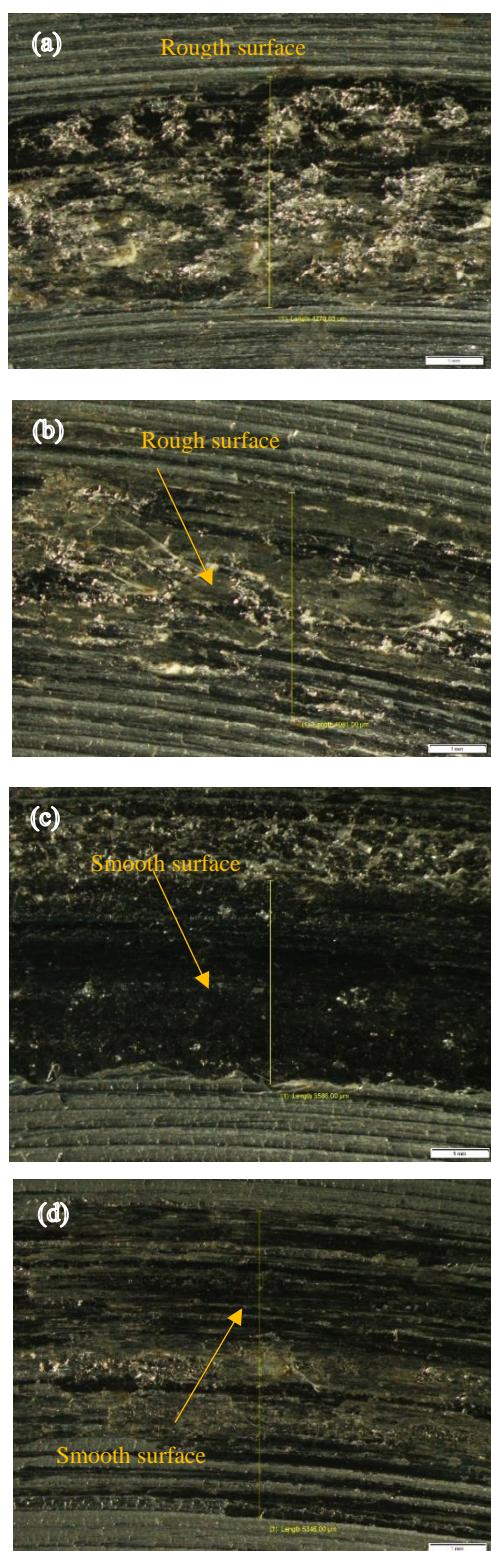


Fig. 11. Surface morphologies of disks lubricated by HCO with 0% wt clove oil at (a) 50 N and (b) 100 N and VCO with 10% wt olive oil at (c) 50 and (d) 100 N with a rotational speed of 1400 rpm.

As the concentration of clove oil in HCO is 0% wt with 1400 rpm and loads of 50 and 100 N (Figure 11), there were only some slight furrows on the worn disk surfaces (Figures 11a and 11b), which

could lead to moderate abrasive wear, consistent with Figure 4, where the wear rate of HCO with 0% wt clove oil is $4.2 \times 10^{-5} \text{ mm}^3/\text{Nm}$ (50 N) and $7.8 \times 10^{-6} \text{ mm}^3/\text{Nm}$ (100 N). Moreover, there was no obvious plowing damage with the exception of some fine scratches on the worn disk surfaces of steel lubricated by VCO with 10% wt clove oil (Figures 11c and 11d), where the wear rate was $1.0 \times 10^{-6} \text{ mm}^3/\text{Nm}$ (50 N) and $7.1 \times 10^{-7} \text{ mm}^3/\text{Nm}$ (100 N). Therefore, the bio-lubricants with clove oil have much better friction-reducing and antioxidant properties than those without clove oil.

3.2 Discussion

The effect of clove oil with different percentages in bio-lubricants could delay the oxidation process in VCO, HCO, and palm oil. This was indicated by a decrease in the percentage of dynamic viscosity of these oils after exposure to oxygen for 30 days. An antioxidant in clove oil in the form of eugenol can inhibit the oxidation process in these oils. According to Ilhami et al. [18], eugenol in clove oil can be used to minimize or prevent lipid oxidation in food. VCO has the lowest unsaturated fatty acid composition among these oils (Table 1) because the manufacturing process of VCO does not use a heating process [16]. The most common molecules that are attacked by oxidation are unsaturated fatty acids [10]. The use of antioxidants as one major means of ensuring oxidative stability [9,10].

The antioxidants in clove oil could not only influence a decrease in dynamic viscosity in these oils but also reduce the wear rate of the disk. The ratio of wear rate between bio-lubricants with 5% wt clove oil and the highest wear rate of the bio-lubricants is shown in Figure 12. From the figure, severe wear occurred on the surface of disks when they were lubricated by HCO and palm oil at a rotation speed of 500 rpm and a load of 50 N because they have a high unsaturated fatty acid content. The high unsaturated fatty acids in bio-lubricants can promote oxidation and increase the viscosity and breakdown of the oil [19]. There was no significant effect of clove oil in palm oil on reducing oxidation. It was also shown in increasing CoF with increasing clove oil concentrations, loads, and speeds, as shown in Figure 5. From Tukey's HSD test for multiple comparisons, it was found that the mean value of CoF was significantly different between clove oil concentration and load ($p < 0.05$). The effect of the breakdown of oils due to oxidation could influence the chemical and physical

properties of lubricants and increase CoF and wear on palm oil. Chemical and physical properties of lubricants play a role in protecting contact surfaces from wear, especially in the boundary lubrication regime [16]. The effect of clove oil in VCO and HCO in terms of CoF was reduced slightly with increasing clove oil concentrations at low speed (500 rpm). The reason might be that, under high load and low speed, clove oil could prevent the formation of oxidation in these oils, which not only had a low viscosity increase but also had a good lubricating effect. In contrast with the speed of 1400 rpm, CoF was increasing with increasing clove oil concentrations because the transition of regime lubrication occurred on the contact from the boundary to the mixed lubrication regime. In this regime, viscosity has an effect on CoF, and this is consistent with figures 3 and 4, where the addition of clove oil will reduce the viscosity.

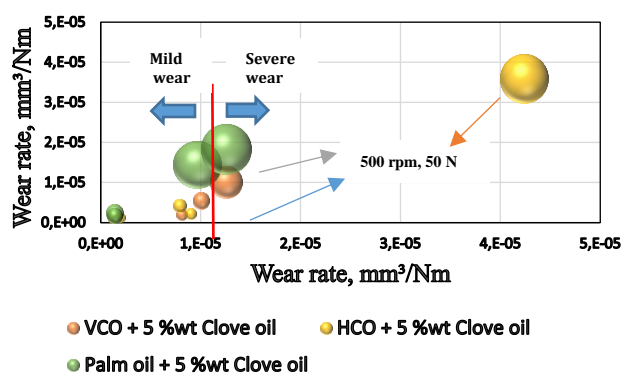


Fig. 12. Ratio between the wear rate of bio-lubricants with 5% wt of clove oil and the highest of wear rate of the bio-lubricants

The scar diameter of the pin (Figure 9) was smaller than the scar width of the disk (Figure 8). This is attributed to boundary lubrication, where contact between asperities occurs. With these high pressures, it would result in plastic deformation of the disk. This will result in deep wear on the disk, and it is consistent with Figure 6, where the volume of the worn disk was higher. Whereas under the high speed (1400 rpm) with these high pressures, the lubrication regime was mixed, where the contact was separated by asperity and lubricant. The chemical and physical properties of lubricant in this regime have the role of protecting the surface of the pin and disk from wear. The effect of clove oil in bio-lubricants under low speed could reduce the deep wear of the disk, whereas under high speed it could reduce wear on both the pin and the disk.

The morphologies of worn disk surfaces lubricated by the addition of clove oil in VCO protected the surface of the disk from plastic deformation and severe wear at low and high speeds, respectively. This agrees well with the results mentioned in Figure 6 when the wear rate is high, around $4.2 \times 10^{-5} \text{ mm}^3/\text{Nm}$ (50 N) and $7.8 \times 10^{-6} \text{ mm}^3/\text{Nm}$ (100 N). With the addition of clove oil to these oils (Figures 10c and 10d), the worn surfaces became smooth and the plastic deformation was greatly abated, indicating that clove oil had good antioxidant properties and reduced friction where the wear rate was low around $8.7 \times 10^{-6} \text{ mm}^3/\text{Nm}$ (50N) and $2.2 \times 10^{-6} \text{ mm}^3/\text{Nm}$ (100 N), as shown in Figure 6. Oxidation of unsaturated fatty acids in VCO could be prevented by clove oil, so unsaturated fatty acids formed a layer that could protect the disk surface from plastic deformation at low speed and severe wear at high speed. The ability of fatty acids to lubricate metal surfaces forms a monolayer film using their closely packed polar carboxyl groups, which can reduce friction and wear in boundary lubrication [7].

The increasing composition of clove oil in palm oil did not increase its tribological properties. It is caused by a high percentage increase in viscosity compared to VCO and HCO (Figure 5), where eugenol in palm oil couldn't suppress the oxidation rate. This will have an effect on its fatty acid content and viscosity, where these substances will affect the lubrication regime, especially in boundary and mixed lubrication regimes.

4. CONCLUSION

In this paper, the effect of adding clove oil to VCO, HCO, and palm oil was investigated under exposure to oxygen for 30 days in terms of physical and tribological properties. Through the viscometer and the pin on disk tests, the following conclusions were obtained:

- The dynamic viscosity of VCO, HCO, and palm oils by adding clove oil was reduced with an increase in the percentage of clove oil in biolubricants. The oil with the lowest decrease in dynamic viscosity by 5.41% was VCO due to the low content of its unsaturated fatty acids.

- b. The effect of adding clove oil to coconut oils (VCO and HCO) was better in terms of wear rate, scar diameter, and width than that of palm oil. An antioxidant in clove oil in the form of eugenol could delay the oxidation process in VCO and HCO and not destroy their chemical properties, which were indicated by lower wear and scars. The use of antioxidants in clove oil could ensure oxidative stability in VCO and HCO.
- c. The addition of clove oil to VCO and HCO only reduced the coefficient of friction at low speeds, while at high speeds it would reduce the coefficient of friction.

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