Study on the Friction and Wear Characteristics of Bio-lubricant Synthesized from Second Generation Jatropha Methyl Ester

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Keywords:
Bio-lubricant
Jatropha Methyl Ester
Anti-wear
Extreme-pressure
Friction

A B S T R A C T

The demands for eco-friendly bio-lubricants are growing due to the environmental concern and the rapid depletion of petroleum oil. This paper outlines the tribological evaluation of jatropha methyl ester (JME) based bio-lubricant by analyzing its anti-wear (AW) and extreme pressure (EP) characteristics. The AW and EP tests were conducted using a four-ball tribotester with standard test methods of ASTM D 4172 and ASTM D 2783, respectively. After each test, the wear scar diameter, flash temperature parameter, viscosity and viscosity index (VI) were measured. The SEM analysis characterized the surface structure of the worn surface. The properties of formulated bio-lubricants were compared with the commercial lubricant SAE 15W-40. Experimental results showed that under boundary lubrication, the bio-lubricants showed excellent tribological properties up to the initial seizure load (ISL). Over the ISL, the friction and wear were increased slightly as compared to the commercial lubricant. The final seizure load (FSL) found for the bio-lubricant (BL 10), and commercial lubricant was 220 kg. The bio-lubricant with 10% JME (BL 10) was found to be the most favorable, which met standard ISO requirements except for pour point.

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1. INTRODUCTION

Lubricants are used as anti-friction or anti-wear agents to minimize the wear and friction from the interacting working surfaces, which are under mechanical stresses. Most of the lubricants which are available in the market are based on mineral oils formulated from petroleum oil. The mineral oil contains many classes of chemical components, including paraffin, naphthenes, aromatics, and heteroatom species [1]. These mineral oils are toxic and non-biodegradable; thus, they are not environmentally friendly [2]. Besides, the uncertainty regarding the availability of the crude oil and its higher prices has drawn attention of the researchers to formulate bio-lubricants which are non-toxic and
biodegradable [3]. Bio-lubricants derived from vegetable oils and animal fats have the potential to substitute petroleum-based lubricants partially [4,5].

Bio-lubricants derived from vegetable oils are composed of fatty acids and possess a double-bonded triacylglycerol structure, which makes them an excellent candidate as a lubricant in automotive engine application [6,7]. The monomolecular and multimolecular surface films caused by the triacylglycerol structure of the molecules prevent contact between the metal surfaces. Hence, the progression of pits and asperities on the metal surfaces are restricted [8,9]. The coefficient of friction and wear rate on the metal surface depend on the degree of adsorption from the lubricants and the strength of fluid film [10]. The oxygenated compounds such as fatty acids play a significant role in reducing adhesion between the contacting metals by reacting or adsorbing on the surfaces. Previous investigations reported that the addition of a small amount of fatty acid to non-polar mineral oil or a pure hydrocarbon leads to a significant reduction in friction and wear [11]. Both boundary and hydrodynamic lubrication can be achieved from the bio-lubricant due to its long-chain free fatty acid [12,13].

Despite numerous advantages, the use of bio-lubricants is limited because of their instability under severe operating conditions [1-14]. Lubricating oil undergoes various unfavorable conditions such as extreme pressure, temperature, and shear-stress. During several tribological processes, lubricating oils should have the ability to retain their properties. Different feedstocks have been used by different research groups to formulate bio-lubricants. Bio-lubricants formulated from rapeseed, palm, karanja, and jojoba oil have shown excellent tribological properties and compatibility with automotive applications [15-17].

The current investigation uses Jatropha methyl ester (JME) as a source of bio-lubricant. The parent oil of JME contains 44.7 % oleic acid, 32.8% linoleic acid, 14.2 % palmitic acid, 7 % stearic acid, and other minor fatty acids. The technical properties of JME are found to be favorable compared to jatropha oil, especially the thermo-oxidative stability and cold flow property [18].

In literature, some studies on palm methyl ester-based bio-lubricants are found. However, the palm is an edible source, thus contend with the food. Unlike palm oil, jatropha oil is a non-edible renewable source; thus, can be an important feedstock for its more extensive application as bio-lubricants. However, there are limited studies on the tribological characteristics of JME based bio-lubricant. Therefore, this investigation aims to determine the tribological properties of JME based bio-lubricant aiming at its wider application.

2. MATERIALS AND METHOD

2.1 Lubricant sample preparation

Bio-lubricants were prepared by blending 2.5 to 12.5 % of JME with the commercial SAE 15W-40. The details of the lubricant samples are presented in Table 1. The commercial SAE 15W-40 oil was chosen as a base lubricant. The properties of JME are presented in Table 2.

Table 1. Composition of tested bio-lubricant samples.

| Sample | Lubricant composition |
|--------|-----------------------|
| BL 0   | Commercial lubricant SAE 15W-40 |
| BL 2.5 | 2.5% JME with 97.5% SAE 15W-40 |
| BL 5   | 5% JME with 95% SAE 15W-40 |
| BL 7.5 | 7.5% JME with 92.5% SAE 15W-40 |
| BL 10  | 10% JME with 90% SAE 15W-40 |
| BL 12.5| 12.5% JME with 87.5% SAE 15W-40 |

Table 2. Properties of Jatropha methyl ester (JME).

| Property                              | Unit     | Value  |
|---------------------------------------|----------|--------|
| Density                               | kg/m³    | 875    |
| FFA content                           | %        | 0.20   |
| Acid value                            | mg KOH/g | 0.50   |
| Water content                         | mg/kg    | 500    |
| Kinematic viscosity @40°C             | mm²/s    | 4.71   |
| Kinematic viscosity @100°C            | mm²/s    | 1.83   |
| Induction period                      | h        | 3.02   |

2.2 Four-ball configuration

In this study, a four-ball tribotester was used to study anti-wear (AW) and extreme pressure (EP) characteristics of bio-lubricants. The ASTM D 4172 and ASTM D 2783 were used for AW and EP testing, respectively. The schematic diagram of the four-ball machine is shown in Fig. 1. In this machine, three stationary balls are placed in a cup below a fourth ball, which is connected to a rotating shaft via a chuck. The frictional torque exerted on the three lower balls can be measured...
by a calibrated arm, which is connected to the spring of a friction recording device.

\[ T = \mu \times \frac{3W \times r}{\sqrt{6}} \Rightarrow \mu = \frac{T \sqrt{6}}{3W \times r} \]  

(1)

where \( \mu \) - coefficient of friction, \( T \) - frictional torque in kg-mm, \( W \) - applied load in kg, \( r \) - distance from the center of the contact surfaces of the lower balls to the axis of rotation, which is 3.67 mm.

Following the test, the wear scar diameter (WSD) was measured and analyzed by Ducom software. The Flash Temperature Parameter (FTP), which is used to express the critical temperature above which the lubricant fails to withstand its properties, was calculated using the following formula [20]:

\[ FTP = \frac{W}{d^3} \]  

(2)

where \( W \) - load in kg, and \( d \) - mean wear scar diameter in mm.

Before and after the test, the viscosity of the lubricant samples was measured using an Automatic Anton Paar SVM 3000 Viscometer using ASTM Method D 455 with a controlled bath temperature of both 40 °C and 100 °C. Before the test, the viscometer was calibrated using a standard sample. The pour point of the lubricant samples was measured using ASTM D 97 method by placing the samples in a refrigerator. The pour point was identified as the temperature when the lubricant stopped to pour.

3. RESULTS AND DISCUSSION

3.1 Friction properties analysis

Figure 2 shows the friction force (Nm) of carbon chromium steel balls under different percentages of JME based bio-lubricants. The BL 12.5 (12.5 % JME) showed the lowest friction force, which is almost similar to BL 10 (10 % JME). The base lubricant (BL 0) showed maximum friction force throughout the operation time. The fluctuation rate of different bio-lubricants was almost identical, while for base lubricant the fluctuation rate was slightly higher, which was increased with time. The bio-lubricants especially, BL 10 and BL 12.5 had a strong affinity to act as a friction-reducing additive. Furthermore, they showed the ability to retain their property by maintaining a constant rate of friction force without much fluctuating.
Fig. 2. The fluctuation of friction force for different percentages of bio-lubricants, recorded for 60 sec.

Figure 3 shows the variation in the coefficient of friction (μ) for different bio-lubricant samples at a constant load of 40 kg. The coefficient of friction (CoF) from BL 10 was found to be lowest with a value of 0.069, while the CoF was highest for base lubricant with a value of 0.12. All bio-lubricants showed lower CoF as compared to the base lubricant. Due to the low CoF, bio-lubricants had the excellent boundary lubrication properties as well as better lubricating film stability to protect metal to metal contact. The bio-lubricants formed a multi or monolayer between metal surfaces, which prevented metal to metal contact and thereby reduced the CoF. The reduction in CoF from bio-lubricants can be explained as the increases in nominal contact surfaces and decreases of pressure in interacting friction zones [21].

Fig. 3. Coefficient of friction (μ) vs. percentage of bio-lubricants at a constant load of 40 kg.

During the tribochemical process, the chemical reaction between the functional groups of the bio-lubricants and the metal ion formed a protective boundary layer. This layer helped to reduce the coefficient of friction and wear [4]. It can be seen that, up to 10 % addition of JME, the decreasing trend in CoF was existed, while further addition of JME, for example, 12.5 %, affected the friction property adversely. Masjuki and Maleque (1997) [22] investigated the anti-wear characteristics of palm methyl ester (PME) based bio-lubricants and showed that the bio-lubricant with 5 % PME had the highest ability to reduce the friction. However, up to 10 % JME added bio-lubricant is found to be favorable based on the current finding.

3.2 Wear characteristics analysis

Figure 4 shows the effect of the percentage of bio-lubricant on the wear scar diameter (WSD). The lowest WSD was found from BL 10, which is about 21 % lower than base lubricant. The WSD was increased when 12.5 % of JME was added to the base lubricant. With a higher addition of JME, the multi or monolayer of the bio-lubricant could not withstand because of the unsaturated palmitic acid. The increased corrosive effect of the higher percentage of bio-lubricants also affected the wear characteristics of the metal surface. From PME based bio-lubricant, a similar phenomenon was also observed by Maleque et al. (2000) [23] and Adhvaryu et al. (2004) [24].

Fig. 4. Effect of different percentages of bio-lubricant on wear scar diameter (WSD) at a constant load of 40 kg.

3.3 Flash temperature parameter analysis

The flash temperature parameter (FTP) indicates the critical temperature below which the lubricant film will withstand without breakdown. According to Fig. 5, it can be seen that the maximum FTP was found from BL 10, while the minimum FTP was obtained from the base lubricant. The results implied that BL 10 had the maximum lubricating performance and the highest possibility to withstand its lubricating film without breakdown. This
phenomenon was also observed by Bhattacharya et al. (1990) [25]. The FTP depends on the applied load and WSD. Since the applied load is constant, thus, higher the WSD lower the FTP. The results also depict that with the addition of above 10 % JME in the base lubricant, the FTP was decreased, which can be explained as the degradation of bio-lubricants’ molecular properties. Hence, the rubbing surfaces came closer to each other and increased the surface contact between the surfaces. The addition of 2.5 % and 5 % JME in base lubricant did not play any significant role in increasing the FTP, while a significant change was observed with 7.5 and 10 % addition of JME.

3.4 Viscosity analysis

Figure 6 illustrates the viscosity test result before and after the four-ball testing at 40 °C and 100°C. The purpose of this test was to check the degradation of lubricants’ quality after the test being conducted. According to Fig. 6a, after the test at 40 °C, the biggest change in viscosity was observed for base lubricant (4.91 cSt), while the lowest change was observed for 10 % bio-lubricant (0.201 cSt). Bio-lubricants retained their property with a slight change in viscosity.

Figure 6b shows the change in viscosity at 100 °C after the one hour operation of the four-ball tribometer. Again, the maximum and minimum changes were observed for the base lubricant and 10 % JEM added bio-lubricant, respectively. However, the changes were minimal as compared to the change that occurred at 40 °C.

3.5 Compatibility of JME based bio-lubricants in automotive and machinery application

The quality and compatibility of the lubricants are generally expressed by its viscosity, viscosity index, and pour point. Table 3 presents the ISO viscosity grade requirement and comparison with that of JME based bio-lubricant BL 10.
Table 3. Comparison of bio-lubricant BL 10 with ISO viscosity grade [12].

| Parameter                        | ISO VG32 | ISO VG46 | ISO VG68 | ISO VG100 | JMEBL 10 |
|----------------------------------|----------|----------|----------|-----------|-----------|
| Kinematic viscosity (cSt) at 40°C| >28.8    | >41.4    | >61.4    | >90       | 63.35     |
| Kinematic viscosity (cSt) at 100°C| >4.1     | >4.1     | >4.1     | >4.1      | 10.63     |
| Viscosity index (VI)             | >90      | >90      | >198     | >216      | 157.7     |
| Pour point (°C)                  | < -10    | < -10    | < -10    | < -10     | -6        |

The JME based bio-lubricant satisfied the ISO standard except for pour point. In this investigation, the highest pour point for the BL 10 was found as -6 °C, which did not meet the standard requirement of -10 °C. This shortcoming does not affect much in tropical countries.

3.6 Worn surface analysis

Figure 8 shows the scanning electron microscopy (SEM) surface structure of the worn surface of steel balls under different percentages of JME based bio-lubricant.

Fig. 8. SEM images of the worn surface of steel balls under different percentages of bio-lubricant.

It can be seen that the size of the WSD using BL 2.5 and BL 5 were larger as compared to the base lubricant. The WSD was decreased for 7.5 % to 10 % of JME added bio-lubricant. Above 10 % JME added bio-lubricant, the WSD was increased again. The maximum WSD was found for BL 2.5, while the minimum WSD was for BL 10. A relatively smoother wear track was observed for the BL 10. A comparatively large amount of metal was fused from the surface of the ball specimen under base lubricant and lower percentages of bio-lubricants. The surface phenomenon observed for the BL 10 can be explained as the better stability of the lubricant film thickness after one-hour operation of the tribometer.

A greater worn surface under 12.5 % added bio-lubricant with compared to that of 10 % added bio-lubricant indicates that some corrosive wear was initiated due to the increases of the JME.

3.7 Extreme pressure characteristics

In extreme pressure experiment, the test was conducted using a load range between 40 kg to the final seizure load (FSL) that is the load required to weld balls specimen.

Figure 9 shows the variation in CoF for different bio-lubricants under different loading conditions. The magnitude of the CoF indicates that the lubrication regime that occurred in the rubbing zone was boundary lubrication. Most of the bio-lubricants showed less CoF in comparison to the base lubricant. The initial seizure load (ISL) for base lubricant was higher than bio-lubricants. The base lubricant and BL 10 were found to be more stable in terms of FSL. At higher load, the lubricating film thickness becomes thinner than some of the asperities present in the boundary lubrication regime. However, these asperities were covered by the long chain fatty acids and the esters of the bio-lubricants. The BL 10 showed the best performance in terms of reducing CoF. The fatty acid present in the JME acted as an active surface material, which was adequate in 10% JME added bio-lubricant and above that the fatty acid did not play any role.

Fig. 9. Variation of the coefficient of friction with load for different bio-lubricants.

Figure 10 illustrates the variation of WSD under different loads using different percentages of
bio-lubricant. The WSD was increased with increasing load. Up to the load of 80 kg, the WSD for base lubricant and other bio-lubricants were almost the same. However, after ISL, the WSD of bio-lubricants were higher than that of base lubricant. It can also be seen that BL 10 had the WSD close to that of the base lubricant and retained its property up to 220 kg, which is the same as the base lubricant.

![Graph showing variation of wear scar diameter with load for different percentages of bio-lubricants.]

**Fig. 10.** Variation of wear scar diameter with load for different percentages of bio-lubricants.

![Graph showing variation of initial and final seizure load and corresponding wear scar diameter for different bio-lubricants.]

**Fig. 11.** Variation of initial and final seizure load and corresponding wear scar diameter for different bio-lubricants.

Figure 11 shows the variation in ISL, FSL, and WSD for different bio-lubricants. The highest ISL was found for base lubricant (160 kg), while the minimum ISL was found for JMEBL 12.5 (100 kg). Among the bio-lubricants, BL 10 had the highest ISL of 140 kg. At ISL load, the minimum WSD was found for BL 7.5 (1.78 mm); this was followed by BL 10 (2.04 mm) compared to the WSD of 2.19 mm using base lubricant. When the load reached the ISL, adhesive wear occurred between the rubbing surfaces, and thereby the CoF was increased sharply. The FSL is the maximum load at which the lubricant film fails, and the interacting surfaces become welded. The results of Fig. 11 indicate that the tribological properties of JME based bio-lubricants are better compared to the base lubricant up to the loading condition of ≤100 kg. However, at higher pressure, except BL 10, other bio-lubricants did not work well compared to the base lubricant as the bio-lubricants failed to retain their properties at a lower load.

4. CONCLUSIONS

In this investigation, anti-wear and extreme pressure characteristics of different bio-lubricants were investigated using a four-ball tribotester. Based on the experimental results, key conclusions are as followed:

**Anti-wear (AW) investigation:**

1. The CoF of steel balls was reduced with the addition of JME up to 10 % in the base lubricant. Besides, the fluctuation of friction force was the highest for base lubricant, while the lowest and the almost same trends were observed for 10 % and 12.5 % JME added bio-lubricants.

2. The viscosity and VI of tested bio-lubricants and base lubricant were reduced after one hour operation of the four-ball machine. However, the reduction was lowest for BL 10.

3. The maximum flash temperature parameter was found for BL 10, while it was minimum for base lubricant. The minimum WSD was found for BL 10, while it was maximum for base lubricant. Moreover, above 10 % JME, the WSD was further increased.

4. Based on the viscosity and the pour point, JME based bio-lubricants met the standard requirement for viscosity, while the pour point was not within the standard specified limit.

**Extreme pressure investigation:**

1. The addition of JME in the base lubricant acted as a potential additive, which enhanced the lubrication properties of the base lubricant. Bio-lubricants formed a saturation layer between contacting surfaces and the addition of 10 % JME was the most suitable.

2. Before ISL, the bio-lubricants showed better tribological properties compared to the base lubricant. However, after ISL, at a particular load, the CoF of bio-lubricants was slightly higher than base lubricant.

3. The FSL of 220 kg was found for base lubricant and BL 10, which indicates that 10
% of JME added bio-lubricant is capable of maintaining its properties at higher load and extreme pressure.

Finally, it can be concluded that JME based bio-lubricants have the potential to reduce the dependency on petroleum-based lubricants partially.

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