A Pin-on-Disk Experimental Study on a Green Particulate-Fluid Lubricant

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The present investigation analyzes a green, petroleum-free lubricant that is produced by mixing two environmentally benign components—canola oil and boric acid powder. To study the influence of boric acid particle size and solid volume fraction on the proposed lubricant performance, pin-on-disk experiments were conducted with spherical copper pins (radius 6.5 mm) and aluminum disks (Ra = 1.35 μm). Friction coefficient measurements were taken at more than 20 distinct operating conditions while varying the lubrication condition (unlubricated, boric acid, canola oil, boric acid/canola oil mixture), boric acid volume fraction, and boric acid particle size. Based on the experiments, it was determined that a solid volume fraction of 7% with 350–700 μm particles was the optimum green particulate lubricant candidate for minimizing the friction at the conditions tested. This work also uncovered an inverse relationship between the friction coefficient and boric acid particle size (in canola oil at 7% solid fraction). Micrographs of the pin and disk wear track were analyzed to study this frictional behavior of the interface materials. Additionally, rheological tests were conducted to measure the viscosity of the canola oil and boric acid powder mixture as a function of particle size, and it was found that the viscosity increased with particle size over the size range tested. Finally, the results indicated that the boric acid-canola oil lubricant mixture demonstrated excellent potential for use as lubricants in industrial applications such as sheet metal forming. [DOI: 10.1115/1.2908913]

Keywords: sheet metal forming, tribometer, pin-on-disk test, environmentally friendly lubricants, powder lubrication

1 Introduction

The U.S. consumed more than 2.5×10^9 gallons of lubricants in 1997. Fifty-four percent of these were automotive lubricants—engine oil and transmission fluid—whereas 44% were industrial lubricants such as hydraulic fluid and gear oil. Over the past decade, the landscape of the lubrication marketplace has significantly changed because of a combination of environmental, health, economic, and performance challenges. To address these challenges, it is essential to develop and implement lubricants that come from natural resources. Environmentally friendly (EFL) or "green" lubricants are renewable and usually made from vegetable oils (e.g., rapeseed/canola, corn, or soybean oil), synthetic esters, polyalkylene glycols (PAGs), or severely hydrotreated petroleum based oils. When compared to mineral and synthetic oils, vegetable oils have a number of distinct advantages including significantly higher lubricity and viscosity, lower volatility, and higher shear stability, detergency, and dispersancy. With better biodegradable and toxicity properties than conventional petroleum based products [1], vegetable oils have tremendous potential for use in the industrial sector. The ultimate motivation for this work is to research and develop a novel green multifunctional lubricant that can be used in room-temperature manufacturing processes such as sheet metal forming.

In recent work, Lovell et al. [2] investigated the performance of a mixture of canola oil and boric acid powder (5 wt % and 100 μm average particle size) in specialized sheet metal forming experiments. The experiments indicated that a green canola oil and boric acid particle mixture could significantly outperform pure canola oil, pure boric acid, and conventional petroleum based lubricants. Numerous other related studies have been carried out over the past several decades on the lubrication properties of boric acid powder alone.

Some of these investigations [3,4] primarily focused on the performance of boric acid in high temperature applications. Others, including Wornyoh et al. [5], evaluated boric acid and numerous layered lattice powders or "powder lubricants." Comprehensive studies aimed at determining the feasibility of using boric acid in general engineering systems were carried out by Erdimir et al. [6–8]. Their research indicated that boric acid's unique layered lattice structure made it a very promising solid lubricant material because of its high load carrying capacity and low steady-state friction coefficient.

In research directly related to metalworking, others have studied the use of boric acid as a lubricant in manufacturing operations. In forming processes, it was observed that boric acid provided very low friction (0.04) between an aluminum work piece and a steel forming tool and that the postfabrication cleaning of the lubricant was environmentally safe, nontoxic, and water soluble [9,10]. In drilling experiments conducted with sapphire tools [11], the addition of boric acid to distilled water was found to increase the rate of drilling of polycrystalline alumina by a factor of 2. Additionally, boric acid was found to help reduce friction and corrosion when mixed with cutting and grinding fluids during machining [12,13]. Recently, the friction and wear performance of boric acid lubricant combinations was studied in extended duration operations [14]. That study showed that the boric acid either required replenishment or a carrier fluid to be effective for extended duration operations. This work focuses on determining an optimal boric acid lubricant combination by examining its tribological performance in pin-on-disk tests as a function of composition, solid fraction, and particle size.

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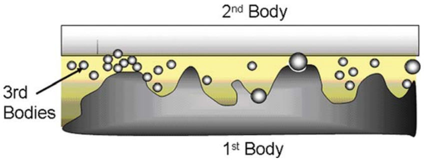

Fig. 1 Boric acid-canola oil lubricant PAML interface

2 Combined Boric Acid and Canola Oil Lubricant

The boric acid powder and canola oil lubricant mixture demonstrates multifunctional lubrication performance, where surfaces are separated by a liquid lubricant film and protected by solid powder. Boric acid powder (H_3BO_3) is a lamellar solid that has low frictional behavior and shear strength. It exists in large supplies and the United States Environmental Protection Agency has classified it as benign to the environment—a "green lubricant." Technical details of the physical, chemical, and tribological characteristics of boric acid are discussed in other works [14,15].

2.1 Lubrication Properties of Canola Oil. The development of natural lubricants to displace petroleum based products is an emerging area of research in the manufacturing community. This development has been driven by dramatic increases in the price of crude oil and the movement toward using biodegradable oils from renewable resources. Vegetable oils such as canola oil are becoming more attractive in a wide range of engineering applications [1,14,16]. As outlined in the Introduction, vegetable oils have a number of distinct advantages over mineral and synthetic oils [17]. In fact, vegetable oils offer a large range of kinematic viscosity, from $2.37\ \mathrm{mm^2/s}$ to $8.53\ \mathrm{mm^2/s}$ at $100^\circ\mathrm{C}$ and from $7.85\ \mathrm{mm^2/s}$ to $35.01\ \mathrm{mm^2/s}$ at $40^\circ\mathrm{C}$ [18]. Canola oil—which is extracted from rapeseed—was specifically chosen as the natural lubricant in the present investigation because it is inexpensive, readily available, and has a viscosity and surface tension similar to fluids used in sheet metal stamping processes [19]. In addition, reports in the literature have indicated that canola oils with an ultrahigh oleic ($>80\%$) content surpass Group 1 petroleum oils at room temperatures [16].

2.2 Properties of the Combined Lubricant Mixture. One of the drawbacks for using dry boric acid powder in sheet metal stamping processes is the fact that it must be continuously "sprayed" into the tool-workpiece contact interface. Such a spray system is sophisticated to develop and expensive to implement, and therefore does not represent a viable lubrication alternative for industry. For this reason, a natural canola oil-boric acid mixture is proposed as an alternative to petroleum based lubricants in room-temperature forming processes. As conceptually depicted in Fig. 1, the canola oil would carry the third-body boric acid particles, providing a particle-augmented mixed lubrication (PAML) slurry. In preliminary research by the authors [19], such a concept was found to be highly effective at reducing wear when boric acid was suspended in canola oil. The lubricant mixture reduced the wear between sliding metal components by nearly a factor of 10 when compared to unlubricated and conventional oil lubricated conditions [14]. While the boric acid-canola oil mixture in that work showed very low wear, its frictional performance showed moderate improvements. This was likely due to the fact that an optimal boric acid solid fraction and particle size was not determined. Therefore, this work aims to conduct pin-on-disk experiments with varying particle properties to determine the optimal boric acid and canola oil lubricant composition.

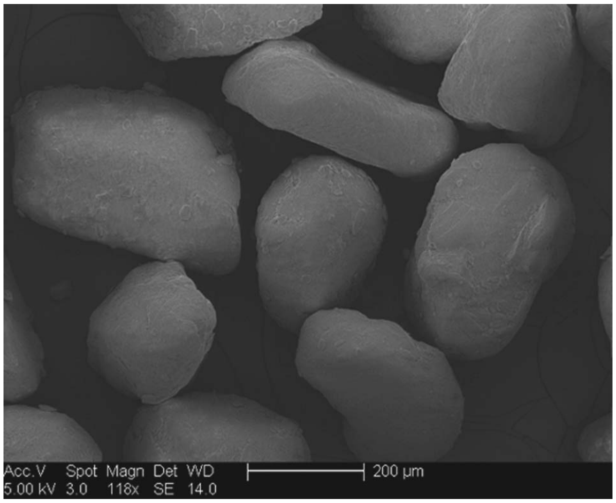

Fig. 2 SEM image of boric acid powder/crystals

3 Experimental Conditions

To study the influence of boric acid particle size and solid volume fraction on the combined lubricant's performance, pin-on-disk experiments were conducted using a commercially available tribometer. More than 20 distinct operating conditions were investigated using spherical (6.5 mm diameter) copper pins with and aluminum disks that had an average roughness (Ra) of $1.35\ \mu\mathrm{m}$. Before each experiment, the disk and pin surfaces were cleaned with acetone. A uniform film of lubricant was then carefully placed on the disk so that the surface of the disk was in a fully flooded condition. For the experiments, a 100 g normal load was applied with a constant sliding velocity of 100 mm/s. All experiments were carried out at room temperature ($23^\circ\mathrm{C}$).

Using the tribometer, friction coefficient measurements were taken while varying the lubrication conditions (unlubricated, boric acid, canola oil, boric acid/canola oil mixture), boric acid solid fractions (3.5%, 7%, 10.5%, and 21%), and boric acid particle sizes ($0-100\ \mu\mathrm{m}$, $100-150\ \mu\mathrm{m}$, $150-180\ \mu\mathrm{m}$, $180-350\ \mu\mathrm{m}$, and $350-700\ \mu\mathrm{m}$). Table 1 presented the particle sizes and solid volume fraction used in this experimental investigation. To obtain the different solid fractions, the particles were mixed with canola oil using a vortex generator that homogenized the mixture. The boric acid particles were crushed and screened into different sizes, and a solid volume fraction of 7% was used for investigating the effect of particle size on the coefficient of friction. A scanning electron microscope (SEM) image of the boric acid powder is shown in Fig. 2. As listed in Table 1, the particle size range of

Table 1 Particle sizes and solid volume fraction used in this experiment

Solid fraction boric acid (%)	Particle size (μm)				
	0–106	106–147	147–180	180–350	350–710
3.5				※	
7	※	※	※	※	※
10.5				※	
21				※	

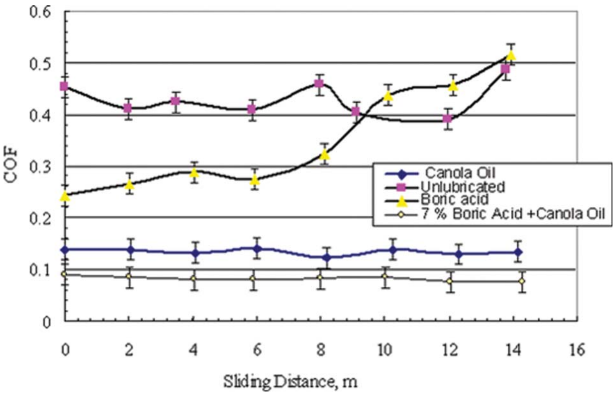

Fig. 3 COF with sliding distance

180–350 μm was used to study the influence of solid fraction on the lubricant's performance.

4 Results and Discussion

In order to develop a base line for performance comparison, the measured coefficient of friction (COF) values were plotted as a function of the sliding distance in Fig. 3 for all of the lubrication conditions investigated. As expected, Fig. 3 shows that the unlubricated and boric acid (only) cases yielded the highest friction coefficients over the sliding distances tested. In the unlubricated case, the friction is large because the surface interaction is dominated by metal to metal asperity contact. In the boric acid only condition, the powder lubricant's performance is poor because the solid lubricant particles are forced out of the contact interface during each sliding cycle; hence, the boric acid only provides separation of asperities at the early stages of sliding but ultimately generates the same frictional value as the unlubricated case. In fact, Fig. 3 shows that the friction coefficient steadily increases for the boric acid only case from an initial value of 0.24 to more than 0.50 at the end of the experiments. This increase in friction was identically found by Deshmukh et al. [14], where the boric acid required replenishment to effectively lubricate over time. Higgs and Wornyoh [20] recently demonstrated that other thin film lamellar powders, namely, MoS_2, must be replenished in order to lubricate in dry powder filled interfaces. Without replenishment, the powder was ejected from the sliding interface and the asperity interaction of the contacting surfaces became more pronounced. Future work will also seek to investigate whether or not some of the resulting wear particles oxidized.

The most effective lubricants in the experiments were the canola oil and canola oil-boric acid mixture. For these cases, the surface tension properties of the canola oil allowed the lubricant to remain in the contact interface and partially separate the contacting asperities for the duration of the experiments. The decreased asperity interaction is signified by the fact that the friction coefficient is relatively low for these cases and remains constant or slightly decreases over the sliding distances tested. Furthermore, as illustrated in Fig. 3, the lubricant mixture of 7% boric acid and canola oil clearly had the lowest COF. This follows prior experimental results by the authors [2,14] where it was found that the canola oil and boric acid mixture exhibited multifunctional lubrication behavior. Essentially, the viscosity and surface tension properties of the canola oil minimized the friction coefficient and surface interaction, while the powder particles formed a protective boundary layer that coated the tribosurfaces.

To determine the optimum composition of the boric acid-canola oil lubricant mixture, a series of experiments was conducted at different volume fractions of canola oil with a constant particle size distribution of 180–350 μm. Based on these experiments, the influence of the boric acid solid volume fraction ν on the frictional interaction was plotted in Fig. 4. As illustrated in the figure, the mixture of $\nu=7\%$ outperformed the other lubricant mixtures. Somewhat surprisingly, the next best lubrication conditions were, respectively, found to be $\nu=3\%$, 10.5%, and 21%. Such a finding suggests that there is an optimum or critical volume fraction (ν_{cr}) of 7% that maximizes the tribological performance of the lubricant mixture. For volume fractions below the critical level ($\nu < \nu_{cr}$) the friction coefficient decreases with increasing solid fraction, indicating that more boric acid enhances the lubrication effects. For $\nu > \nu_{cr}$, the friction coefficient increases with solid fraction, which suggests that the lubricant has entered a "particulate frictional fluid" regime akin to "granular flow" [21]. Although more detailed studies are needed to obtain a complete understanding of this phenomenon, the friction can also be studied as a function of the lubricant's mixture viscosity. The viscosity η can be determined as a function of the particle-free fluid viscosity η_0 and the solid (volume) fraction ν using the Einstein [22] viscosity equation for dilute (low) solid fractions as

$$\eta = \eta_0(1 + 2.5\nu) \tag{1}$$

Since the viscosity varies with solid fraction (see Eq. (1)), a plot of the friction coefficient versus viscosity would qualitatively resemble Fig. 4. Similar to the volume fraction results, a critical viscosity value (~0.103 Pa s) was found for the lubricant mixture. Surprisingly, the optimal friction coefficient that occurred at a solid fraction $\nu=7\%$ (Fig. 4) would violate a linear rule of mixtures for the particulate-oil lubricant, which would have predicted that the friction for the mixture should fall somewhere between the friction coefficients of the dry (boric acid powder) and wet (canola oil) components of the mixture.

4.1 Determination of the Pin-on-Disk Lubrication Regime. In an effort to determine which lubrication regime our lubrication

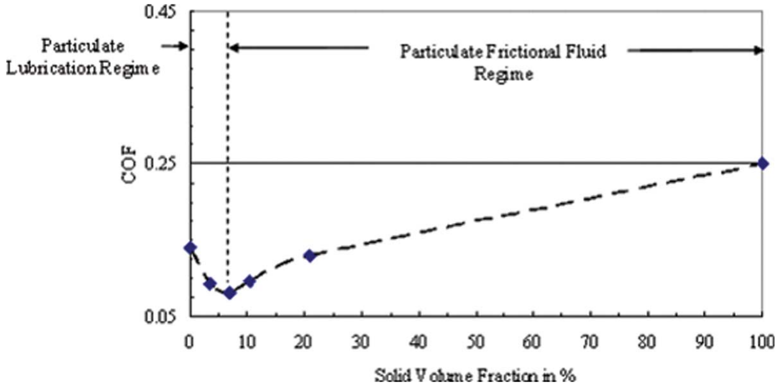

Fig. 4 COF versus solid volume fraction

Table 2 Geometry and lubricant constants

Ball radius, R_x	0.0065 m
Effective elastic moduli of pin and disk, E'	9.63×10^{10} Pa
Normal load, F	0.98 N
Viscosity, η	0.1–0.1105 Pa s
Elliptical parameter, $k=a/b$	1.03
Asymptotic isoviscous pressure, $P_{\mathrm{iv\ as}}$	48.24×10^{6} Pa
Dimensionless speed parameter, U	8.11×10^{-12}
Dimensionless material parameter, G	1996
Dimensionless load parameter, W	2.41×10^{-7}

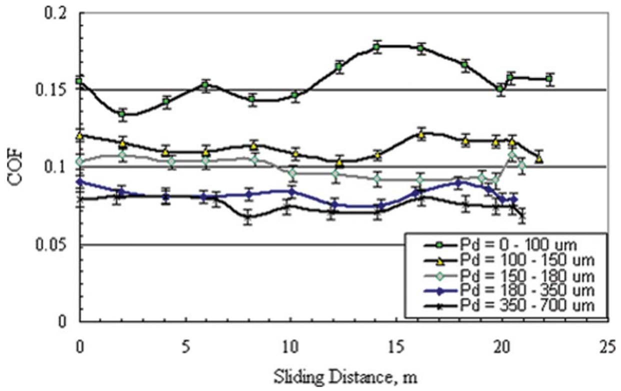

Fig. 5 COF with sliding distance

system was operating, Hamrock and Dowson's [23] elastohydrodynamic film thickness calculations were utilized. As described by Lovell et al. [24], Hamrock and Dowson's theory can be used to predict the lubricant separation thickness for a given speed, load, lubricant, and pin and disk material and geometry. According to their work, the lubricant's central and minimum film thicknesses are given by

$$H_c = 2.69 U^{0.67} G^{0.53} W^{-0.067} (1 - 0.61 e^{-0.73\ \mathrm{K}}) \tag{2}$$

$$H_{\min} = 3.63 U^{0.68} G^{0.49} W^{-0.073} (1 - e^{-0.68\ \mathrm{K}}) \tag{3}$$

In Eqs. (2) and (3), the dimensionless speed, material, load, and ellipticity parameters are, respectively, defined by

$$H_c = h/R_x \tag{4}$$

$$U = \eta_0 V/(E' R_x) \tag{5}$$

$$G = E'/p_{\mathrm{ivas}} \tag{6}$$

$$W = F/(E' R_x^2) \tag{7}$$

$$k = a/b \tag{8}$$

where η_0 is the lubricant viscosity, V is the surface velocity of the disk, E' is the effective elastic moduli of pin and disk, h is the film thickness of lubricant, R_x is the curvature of the pin, $P_{\mathrm{iv\ as}}$ is the asymptotic isoviscous pressure, F is the normal load exerted on the pin, a is the semiminor axis of the Hertzian contact ellipse, and b is the semiminor axis of the Hertzian contact ellipse.

Utilizing the parameters listed in Table 2 for our system, Hamrock and Dowson's elastohydrodynamic film thickness equations, respectively, predicted central and minimum film thicknesses within the range of 0.071–0.076 μm and 0.042–0.045 μm. With an average surface roughness of 1.35 μm for our pin and disk components, these values clearly indicate that our system operates in the boundary and mixed lubrication regimes rather than the elastohydrodynamic regime. Consequently, one would expect decreased friction with increased lubricant viscosity. The fact that a critical viscosity value was found in the experiments, however, indicates that the increased presence of boric acid powder helped the lubricant provide partial separation, but then hindered favorable lubrication. It should be noted that since the disk has an average roughness Ra=1.35 μm, the asperities can be assumed to have a negligible effect on the particulate lubrication performance since virtually all of the particles tested have a particle diameter $P_d \gg \mathrm{Ra} = 1.35$ μm.

Graphically depicted in Fig. 5, the boric acid powder diameter P_d is the final parameter that was studied. Based on the experiments, it was found that the coarsest powder size range (P_d $=350-700$ μm) showed the COF with increasing sliding distance. This can more clearly be depicted by Fig. 6, where the steady-state coefficients of friction from Fig. 5 were plotted against their respective mean particle diameters and shown to be inversely related. Such a trend is counterintuitive because one might presume that the smaller boric acid particles would more readily enter the contact interface between the pin and the disk and provide enhanced particle-augmented mixed lubrication.

4.2 Analysis of the Particle Size Effects on Friction. To examine the inverse relationship between the friction coefficient and the particle size, abbreviated pin-on-disk tests (5 min) were conducted. These tests were at the same experimental conditions described in Sec. 3 for a canola oil mixture of 7% boric acid volume fraction and varying boric acid particle sizes. Micrographs of the wear tracks on the disk surfaces were taken using an optical microscope at 50× magnification both (a) *before* and (b) *after* each pin-on-disk test (see Figs. 7–9). Examining the figures, it is clearly shown that a greater number of the smaller boric acid particles (P_d=0–100 μm) remain within the contact area of the disk as compared to the larger particle sizes (P_d=350–700 μm). In Fig. 7(*b*), for example, one can see that most of the disk surface is covered with boric acid particles as opposed to canola oil for the tests run with the particle size range of P_d=0–100 μm. Most of the surface in Fig. 8(*b*) with the particle size range P_d=150 –180 μm also appears to be covered with powder as opposed to oil, but it is inconclusive on whether it has more powder coverage than Fig. 7(*b*). Figure 9(*b*) shows that the disk wear track has the least powder coverage for the largest particle sizes P_d =350–700 μm, which implies that most of the lubricant within the sliding contact interface was composed of canola oil. As shown in Fig. 3, canola oil was found to have a lower friction coefficient than boric acid in our pin-on-disk tests. Therefore, it appears that the smaller particles can more easily enter the contact interface with the oil, which causes more of the friction properties

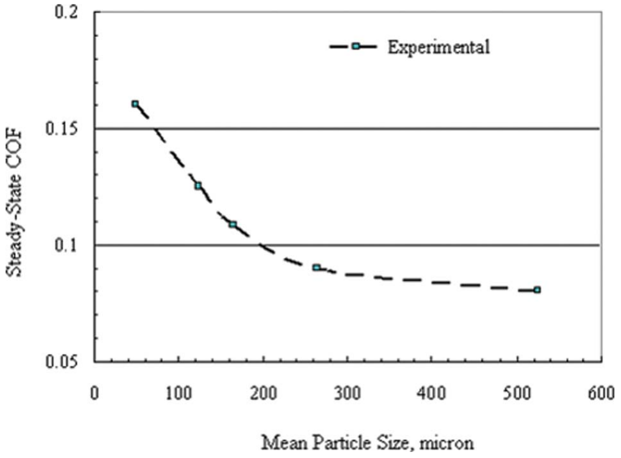

Fig. 6 Steady-state COF with mean particle size

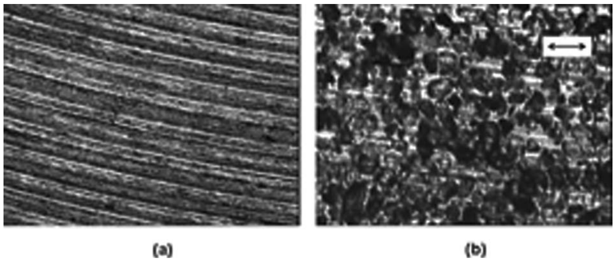

Fig. 7 Particles on disk wear track before and after test: 0–100 μm

within the pin/disk interface to be controlled by the higher coefficient boric acid powder. The micrograph results in Figs. 7–9 shed some light on the trend in Fig. 6; however, it does not quantitatively explain how the combination of 7% boric acid powder and canola oil brings the COF to a value that is lower than the canola oil COF. This suggests that there are likely chemical and microstructural changes in the mixture components that enhance lubrication performance. Analysis of the pin micrographs was not able to shed more light into the phenomena at play.

Finally, viscometric tests were conducted using a Rheometrics© rheometer to measure the canola oil/powder mixture viscosity as a function of particle sizes $P_d = 0-150$ μm (see Fig. 10). These tests revealed that the viscosity did indeed increase with particle size for the range of particles tested. However, the rheometer could not accept the particle sizes over 150 μm. Yet, since the surface separation performance in the pin/disk interface would be enhanced by this increase in viscosity, one could postulate that this would also cause the inverse relationship between particle size and COF, as shown in Fig. 6.

5 Conclusion

In the present investigation, pin-on-disk experiments were carried out to determine the lubrication performance of a novel green particulate-fluid lubricant that could be used in industrial applications. As previously reported [2,14], the proposed boric acid and canola oil mixture outperformed other lubricants to demonstrate excellent potential for use as a completely natural lubricant alternative. Based on the experiments, it was determined that the boric acid and canola oil mixture with a solid fraction $\nu = 7\%$ and $P_d = 350-700$ μm particles would be the optimum green lubricant for minimizing the friction in the conditions tested. This work also uncovered a critical solid fraction ν_{cr} for the pin-on-disk conditions studied. Below ν_{cr}, the friction coefficient was found to decrease with increasing solid fraction; whereas above ν_{cr}, the friction coefficient increased with increasing solid fraction. Thus, ν_{cr} may be useful as a parameter for exploring the various tribological regimes of a particulate fluid as a function of solid fraction.

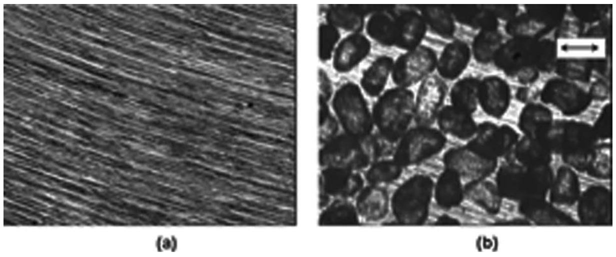

Fig. 8 Particles on disk wear track before and after test: 150–180 μm

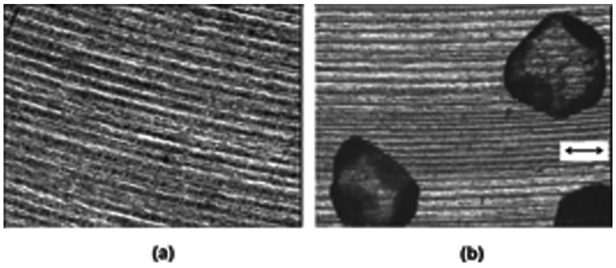

Fig. 9 Particles on disk wear track before and after test: 350–700 μm

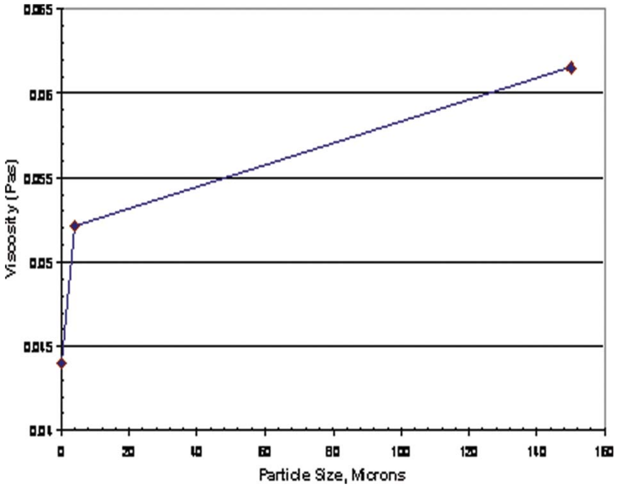

Fig. 10 Viscosity versus particle size P_d

An inverse relationship between the friction coefficient and boric acid particle size (in canola oil at 7% solid fraction) was also uncovered. Based on optical microscope images taken of the disk wear tracks after the pin-on-disk tests, it was found that more of the smaller boric acid particles studied remained in the wear track than the larger particles. Since boric acid powder had a higher dry friction coefficient than the canola oil lubricant, it was deduced that this greater percentage of boric acid in the sliding contact led to the inverse relationship between particle size and friction coefficient. Additionally, preliminary rheological test results showed that the oil viscosity increased with particle size, which would also suggest an inverse relationship between COF and particle size. Finally, the optimum lubricant performance was found to occur for canola oil and 7% boric acid powder for the largest particle size range ($P_d = 350-700$ μm).

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