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Investigation of tribological properties of CuO/palm oil nanolubricant using pin-on-disc tribotester

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It has been proposed that nanoparticles have the ability to enhance the tribological properties of lubricants. In the present research, the tribological properties of a palm-oil-based nanolubricant with copper (II) oxide (CuO) nanoparticles have been investigated experimentally using a pin-on-disc tribotester. The experiments were conducted at a sliding speed of 0·2 m/s and an applied load of 9·8 N, which corresponds to a contact pressure of 0·93 GPa for 60 min duration. Commercial mineral oil (SAE40) was used for comparison purposes. The results show that the tribological properties of the palm-oil-based lubricant were improved with the presence of copper (II) oxide nanoparticles, showing huge reductions in coefficient of friction and wear scar diameter of about 56 and 48%, respectively. The findings revealed that the presence of copper (II) oxide in palm kernel oil (PKO) reduced the roughness of the pin surface by 43·7%. The results also show that the tribological properties of the copper (II) oxide/palm-oil-based nanolubricant were comparable to those of SAE40.

Notation

E'	effective modulus of elasticity
F	normal load exerted on the pin
g	gravity
h_{min}	film thickness
k	elliptical parameter
L	distance along the scar diameter
R_a	arithmetic mean roughness
R_x	curvature of the pin
r	radius of nanoparticles
V	surface velocity of the disk
v_z	settling velocity
η_0	dynamic viscosity
μ	viscosity of the lubricant
ρ_F	density of lubricant
ρ_{NP}	density of nanoparticles

1. Introduction

The formulation of lubricants to be used in transportation sectors must fulfil the latest standards and guideline regulations in terms of minimum fuel consumption and environmental protection.[1,2] An increase in fuel efficiency and service lifetime plays a crucial role in reducing fuel consumption for transportation sectors globally.[3] In this regard, the improvement of the tribological properties is important to reduce friction and wear. The use of antiwear (AW) and extreme pressure (EP) additives is important under severe frictional conditions.[4] However, conventional additives such as zinc dialkyl-dithiophosphate and molybdenum dialkyl-dithiocarbamate contain harmful elements such as sulfur, chlorine and phosphorus that can negatively affect the environment.[5] Therefore, there is an urgent need to develop new AW and EP additives in lubricants with significantly improved tribological properties to be used in transportation sectors.

The advent of nanotechnology has allowed the synthesis of nanomaterials in various fields, including optic and optoelectronics,[6] photocatalysis,[7,8] electrical and sensor devices,[9] biomedical applications[10] and so on due to their excellent chemical, thermal and mechanical properties. Recently, the application of nanomaterials in lubricants has brought about a significant amount of experimental research into the use of nanoparticles as lubricant additives (also known as nanolubricants). This is because nanolubricants are relatively insensitive to temperature and show limited tribochemical reactions, compared to conventional additives. It has been reported that nanoparticles dispersed in lubricant can enhance the tribological performance of the lubricant.[11–21] Several mechanisms are involved in the enhancement of the tribological properties, including reduction of metal-to-metal contact by material filling, mending and polishing effects,[20,22] tribosintering of nanoparticles on the surface,[15,23] tribofilm formation[16,24] and rolling of nanospheres.[22]

Many researchers focus on the evaluation of nanoparticles as additives in mineral-oil- and synthetic-oil-based lubricants; only a few experiments have tested the addition of nanoparticles to vegetable-oil-based lubricants. The formulation of a vegetable-oil-based lubricant with nanoparticles could be advantageous in terms of environmental benefits. In Malaysia, the potential use of palm oil as an alternative lubricant has been studied extensively for various applications since Malaysia is the world's second largest producer of palm oil.[25–32] Nowadays, the trend changes to evaluate the tribological properties of palm-oil-based nanolubricant.[12,33,34] However, there are still limited studies on the evaluation of copper (II) oxide (CuO) nanoparticles as an additive in palm kernel oil (PKO). Basically, palm fruit produces two different types of oils: (*a*) palm oil, which is used in food applications, and (*b*) PKO, which is used in non-food applications. In the present research, PKO was selected as it would not disturb the food proportion and also to optimise the utilisation of the excess oils. Thus, the present research evaluated the tribological properties of the refined, bleached and deodorised (RBD) PKO with and without copper (II) oxide nanoparticles using a pin-on-disc tribotester in accordance with ASTM G 99.[35] The results will be compared with commercial mineral oil SAE40, since a formulated vegetable-oil-based lubricant should be able to provide at least the same tribological properties as commercial mineral oil in order to be used as an alternative lubricant. Viscosity tests were conducted using a rotary viscometer. In addition, the wear and surfaces analysis was evaluated using a high-resolution microscope. The worn surface was further characterised by using energy dispersive X-ray spectroscopy (EDX) in order to detect the elements present on the pin's surface.

2. Experimental detail

2.1 Preparation of nanolubricant

RBD PKO (from Keck Seng (Malaysia) Berhad, Johor, Malaysia) was used as the base lubricant for the nanolubricant. Copper (II) oxide nanoparticles with composition of 79·87% copper (Cu) and 20·10% oxygen (O_2) were commercially purchased from MK Impex Corporation Canada and diluted in the PKO at a concentration of 0·34 wt%. To synthesise a nanolubricant of a particular concentration, the weight of the required amount of nanoparticles in a given amount of lubricant was calculated. In the present research, the total volume of PKO is fixed at 100 ml and weighted using weighing balance. Then, the required mass of nanoparticles is calculated and weighed to make a 0·34 wt% nanolubricant, which means 0·314 g of the nanoparticles is dispersed in 92·023 g of the PKO. The mass percentage calculation is calculated by using the mass of a solute (nanoparticles) and the mass of a solution (lubricant + nanoparticles) as shown in Equation 1. The selection of the nanoparticle concentration was based on the optimum concentration from the experiment done by Thottackkad *et al.*[13] The properties (Table 1) and the component content (Table 2) of the nanoparticles were provided by the supplier. Commercial mineral oil (SAE40) from Malaysia was used for comparison purposes. The nanolubricant was prepared by diluting the copper (II) oxide nanoparticles in the PKO base lubricant and mixed using high-shear homogeniser (IKA T25 digital Ultra-Turrax, Germany) at a speed of 7000 revolutions per minute for 40 min. The use of the high-shear homogeniser may result in better dispersion since it can break the aggregates by the effect of extremely strong shear forces. Figure 1 shows photographs of copper (II) oxide/PKO nanolubricant in the test tube after a period of time with a sedimentation rate of 1·23 × 10^{-9} m/s where it was calculated using Stokes' law as shown in Equation 2,[34] where the copper (II) oxide/PKO nanolubricant is shown to be fully precipitated in 16 d.

$$\text{mass percentage: wt\%} = \frac{\text{mass of solute(nanoparticles)}}{\text{mass of solution(lubricant + nanoparticles)}} \times 100\%$$

1.

Table 1. Properties of copper (II) oxide nanoparticles

Nanoparticles	Morphology	Purity: %	Size: nm	Surface area: m^2/g
Copper (II) oxide	Spherical	99	40	80

Table 2. Component contents of copper (II) oxide nanoparticles

Component	Contents: ppm
Barium	0·8
Calcium	400
Cadmium	2·4
Cobalt	6·4
Iron (Fe)	87
Potassium (K)	300
Magnesium	72
Manganese (Mn)	3·2
Phosphorus	300
Lead	100
Strontium	2·4
Zinc	200

ppm, parts per million

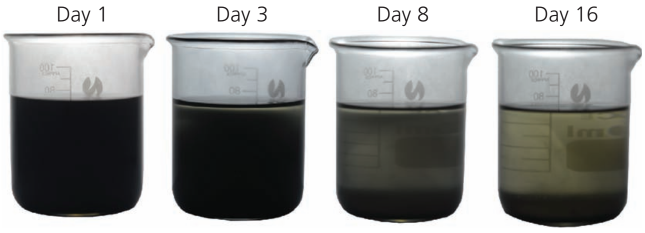


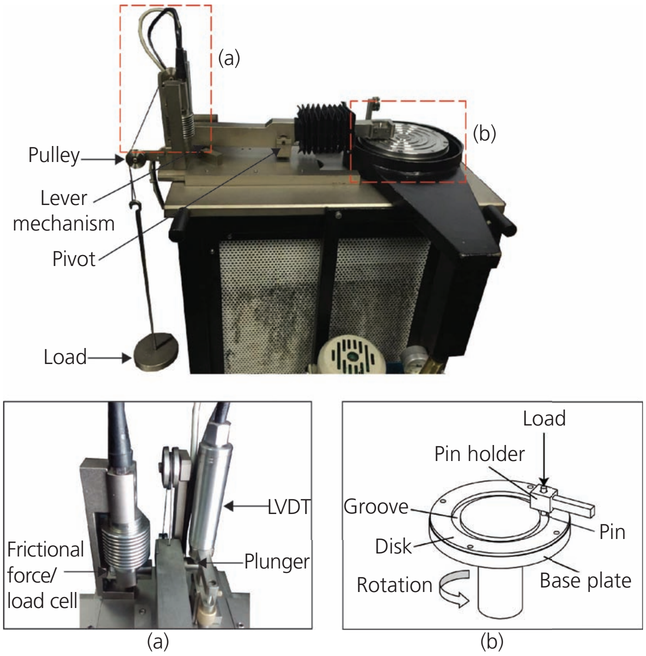
Figure 1. Sedimentation of copper (II) oxide/PKO nanolubricant over a period of time

$$v_z = \frac{2(\rho_{NP} - \rho_F)\boldsymbol{g}r^2}{9\mu} \quad 2.$$

where v_z is the settling velocity, ρ_{NP} is the density of nanoparticles (6·49 g/cm^3), ρ_F is the density of lubricant (0·89 g/cm^3), $\boldsymbol{g}$ is gravity (9·81 m/s^2), r is the radius of nanoparticle (2×10^{-6} cm) and μ is the viscosity of the lubricant at room temperature (0·0397 Pa s).

2.2 Evaluation of tribological properties

The tribological properties of the tested lubricants (SAE40, PKO and copper (II) oxide/PKO nanolubricant) were evaluated using a pin-on-disc tribotester (model TR-20LE, Ducom Intsruments Pvt., Ltd, Bangalore, India) (see Figure 2) in accordance with ASTM G99. The normal force was applied by placing the required weight on a loading pan. The frictional force sensor or load cell measures the friction between the stationary pin and rotating disc. The signal output of friction was obtained directly from the data acquisition system. The frictional force was then divided with normal load to obtain the coefficient of friction (COF). The COF was taken as a measure of antifriction performance of the tested lubricants. In the present research, the stationary pin made of pure aluminium A1100 formed a point contact with the rotating SKD11 tool steel disc with densities of 2·71 and 7·85 g/cm^3, respectively. The dimensions of the hemispherical pin were 30 mm long and 6 mm dia. with 3 mm radius of curvature on one end; while the geometry of the disc was 165 mm × 8 mm. The initial arithmetic mean roughnesses (R_a) of the pin and disc surfaces were 0·01 and 0·3 μm, respectively. Only a small amount of lubricant (0·2 g) was placed on the disc surface, and the disc was designed to have a groove to prevent the lubricant from flowing out during the rotation of the disc.



(a) (b)

Figure 2. Pin-on-disc tribotester (a) linear variable differential transformer sensor and load cell (b) schematic diagram of pin-on-disc. LVDT, linear variable differential transformer

The experiment was carried out at constant applied load of 9·81 N, which corresponds to a contact pressure of 0·93 GPa for 60 min of time duration at the sliding speed of 0·2 m/s. The antiwear performance of the tested lubricants was evaluated by measuring the wear scar diameter (WSD) of the pin specimen using an optical microscope after each experiment. The pins were cleaned with acetone after each test to ensure that there was no excess oil on the pin surface. The worn surface of the pins was also analysed, and it was measured using a high-resolution optical microscope. The slider profile of the pin surfaces was also taken for further confirmation of the wear behaviour of the pin surfaces. Further analysis was done by characterising the worn surfaces of the pin using scanning electron microscopy (SEM) and EDX in order to detect the elements present on the pin's worn surface.

3. Results and discussion

3.1 Viscosity and viscosity index

Figure 3 presents the kinematic viscosity and viscosity index of the RBD PKO with and without copper (II) oxide nanoparticles in comparison with commercial mineral oil SAE40 under 40 and 100°C temperatures. The kinematic viscosity of the tested lubricants was measured using a rotary viscometer. From this figure, the kinematic viscosity of the tested lubricants generally decreased as the temperature increased. This is because temperature increment results in weak intermolecular forces.[36] The presence of copper (II) oxide nanoparticles in PKO shows a slight reduction in the kinematic viscosity at the temperatures of 40 and 100°C. Similar findings were obtained by Ali *et al.*[20] for aluminium oxide and titanium dioxide nanolubricants, which reduced the viscosity of the mineral oil. This is because layers in the nanoparticles resulted in ease of relative movement due to weak intermolecular forces.[20,37] As can be seen in Figure 3(b), the viscosity index of PKO was higher than that of SAE40. This is due to the presence of the triglyceride structure in PKO that can maintain stronger intermolecular interactions with increasing temperature than the branched hydrocarbons or esters in SAE40.[38] Moreover, the results reveal that the viscosity index of the copper (II) oxide/PKO nanolubricant was increased slightly by 0·9% compared to the PKO without copper (II) oxide nanoparticles. The minimum changes in viscosity with temperature of the copper (II) oxide/PKO nanolubricant proved that the presence of copper (II) oxide nanoparticles in PKO was able to maintain the thickness of the oil film and thus keep the friction to a minimum.

3.2 Tribological properties

In order to evaluate the tribological properties of the tested lubricants (SAE40, PKO and copper (II) oxide/PKO), a pin-on-disc tribotester

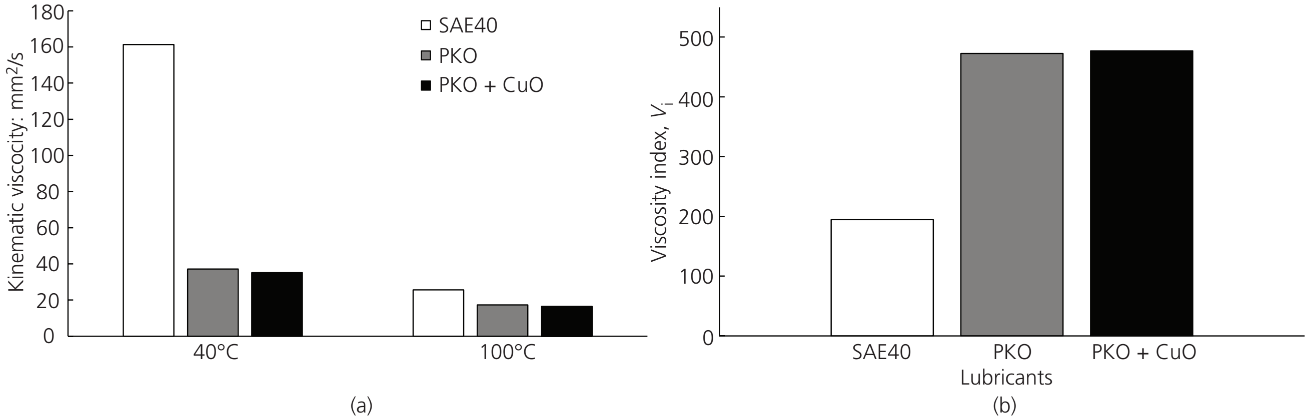


Figure 3. (a) Kinematic viscosity and (b) viscosity index of the tested lubricants

was employed. Figure 4 shows the COF and WSD of SAE40 mineral oil and PKO with and without copper (II) oxide nanoparticles at a sliding speed of 0·2 m/s. From this figure, it can be seen that both the COF and WSD of PKO were higher than SAE40. This is because fatty acids have long been used as ‘friction modifiers’ in mineral oils under boundary lubrication conditions. Free fatty acids in mineral oils tend to interact with the steel surface and therefore produce a more effective lubricant layer than vegetable oils alone.[39] Both the COF and WSD of PKO appeared to have decreased dramatically when copper (II) oxide nanoparticles were added – by 56 and 48%, respectively. The results also show that copper (II) oxide/PKO nanolubricant has better antifriction and antiwear performance than SAE40 mineral oil.

Previous research has reported that there are several mechanisms involved in making nanoparticles act as powerful additives to improve the tribological properties of a lubricant. The rolling friction of sphere-like copper (II) oxide nanoparticles at the contact surface could improve the tribological properties of the base lubricant.[4,24,40] Another plausible mechanism involved is the sliding mode of the nanoparticles that served as spacers between sliding surfaces, thus preventing surface contact.[41] The concentration of nanoparticles in the lubricant is one of the contributing factors that make the nanoparticles serve as spacers. Higher nanoparticle concentration increased the tendency of particle agglomeration; at high applied load, this particle agglomeration could provide supporting effort over a larger contact area that could play a role as a spacer in reducing surface contact.[42] It is also found that nanoparticles will act as a third body for material transfer: layers of copper (II) oxide are gradually transferred from the nanoparticles onto the contact surfaces and act to reduce friction between the two contact surfaces.[43] Moreover, a previous observation by Alves *et al.*[14] confirms that the enhancement of the tribological properties was attributed to the deposition of copper (II) oxide nanoparticles on the contact surface, which can reduce the shear stress. They confirmed the deposition of copper (II) oxide nanoparticles by analysing the element content using an EDX analyser. Gulzar *et al.*[34] reported that the presence of the element copper on the worn surface of the specimen shows the mechanism of tribosintering at the contact surface forming a film that enables the protection against friction and wear.

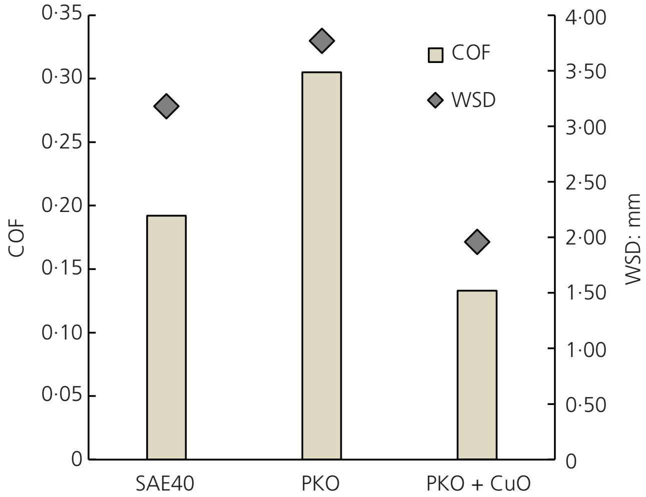


Figure 4. COF and WSD of the tested lubricants

According to Castillo and Spikes,[44] the rolling effect of spherical nanoparticles depends on the base oil film thickness. When the particle diameter of nanoparticles is greater than the film thickness, the nanoparticles will be easily trapped at the interfaces. The rolling surfaces of the trapped nanoparticles were then deformed plastically and formed a transfer film. Rapoport *et al.*[45] stated that when the film thickness is close to the size of the nanoparticles, the shape of the nanoparticles is preserved and leads to rolling friction of nanoparticles at the interfaces. In the present research, minimum film thickness can be obtained approximately by using elastohydrodynamic film thickness calculations as shown in Equation 3.[46]

$$h_{\min} = 3{\cdot}63R_x\left(\frac{\eta_0 V}{E'R_x}\right)^{0{\cdot}68}\left(\alpha E'\right)^{0{\cdot}49}\left(\frac{F}{E'R_x^2}\right)^{-0{\cdot}073}\left(1-e^{-0{\cdot}68k}\right) \tag{3.}$$

where η_0 is the dynamic viscosity at 27°C, V is the surface velocity of the disc, E' is the effective modulus of elasticity, R_x is the curvature of the pin, α is the viscosity–pressure coefficient, F is the normal load exerted on the pin and k is the elliptical parameter. From Equation 3, the calculated value of the lubricant film thickness is 36·28 nm, which is close to the diameter of copper (II) oxide nanoparticles (40 nm). This calculated value proved that spherical copper (II) oxide nanoparticles in the present research effectively roll at the interfaces, and the particles were not deformed or trapped at the interfaces, thus reducing friction and wear. The identification of element content on the worn surfaces of the pin was performed using EDX spectroscopy, as shown in Table 3. The EDX analysis shows that there is no copper element content found when lubricated with PKO + copper (II) oxide. It was clearly proven that there are no particles trapped at the interfaces. Similarly, a previous observation by Thottackkad *et al.*[42] confirms that there is no copper element on the pin surface, indicating that the nanoparticles did not penetrate into the pin surface and the nanoparticles act as a rolling medium. The rolling effect of copper (II) oxide nanoparticles was also reported by Wu *et al.*[16] This mechanism was clearly illustrated in Figure 5. This result is also consistent with the finding from Battez *et al.*[43] that could not detect copper element by EDX analysis on the wear surfaces. They stated that the nanoparticles serve as a third body to reduce the contact surfaces, thus decreasing the contact surfaces.

Figure 6 presents the wear worn surfaces and the slider profile of the friction surface on pin specimens lubricated with SAE40, PKO and copper (II) oxide/PKO nanolubricant. In the wear worn surface images, the horizontal scratches result from the friction test, and the arrow shows the sliding direction. The slider profile was taken perpendicular to the sliding direction, where L represents the distance along the scar diameter. Comparing the worn surfaces, it seems that deeper parallel grooves were found on the surfaces lubricated with SAE40 and PKO, while the worn surface lubricated with copper (II) oxide/PKO nanolubricant shows a smooth surface. It can also be observed that the worn surface of SAE40 was rougher than the worn surface of PKO, where the slider profile shows that the pin surface lubricated with SAE40 formed a steeper peak with a relatively high surface roughness of R_a = 0·316 μm than that of PKO (R_a = 0·244 μm). The steeper peak means more material removal of the friction surface from the pin specimen and represents a deeper abrasive groove on the pin surfaces. Although PKO has a lower surface roughness value and a smoother worn surface than that SAE40, it has a high COF. The presence of copper (II) oxide nanoparticles made the PKO able to minimise direct metal-to-metal contact, thus resulting in less material removal and a smoother worn surface. The slider profile of the pin surface lubricated with copper (II) oxide/PKO nanolubricant shows a moderate peak and has a lower surface roughness value (R_a = 0·123 μm) than that with PKO. The results revealed that the surface roughness of PKO with the presence of copper (II) oxide was reduced by 43·7%. The present research suggests that copper (II) oxide nanoparticles smoothen the surface through a rolling mechanism.

4. Conclusion

In the present research, the tribological properties of PKO with and without copper (II) oxide nanoparticles have been evaluated using a pin-on-disc tribotester. The results showed that the presence of copper (II) oxide nanoparticles in PKO has improved the tribological properties by reducing the COF and WSD by 56 and 48%, respectively. Surface analysis by way of SEM and EDX suggested that copper (II) oxide nanoparticles act as a rolling

Table 3. Chemical elements found on the pin worn surfaces with EDX analysis

Sample	Element	Weight: %	Atomic: %
SAE40	Carbon (C)	9·76	36·31
	Oxygen (O)	0·70	1·95
	Aluminium (Al)	72·02	47·58
	Manganese	10·15	8·26
	Iron	7·37	5·90
PKO	Carbon	2·20	11·21
	Oxygen	0·61	2·34
	Aluminium	84·44	72·35
	Manganese	6·43	7·16
	Iron	6·32	6·93
PKO + copper (II) oxide	Carbon	2·90	13·82
	Oxygen	1·09	3·89
	Aluminium	82·26	68·06
	Manganese	7·47	7·79
	Iron	6·28	6·44

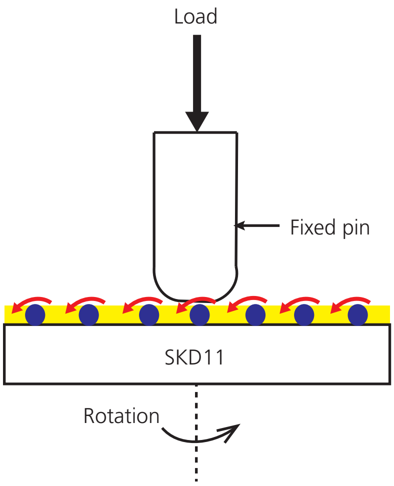


Figure 5. Rolling effect mechanism of nanolubricant

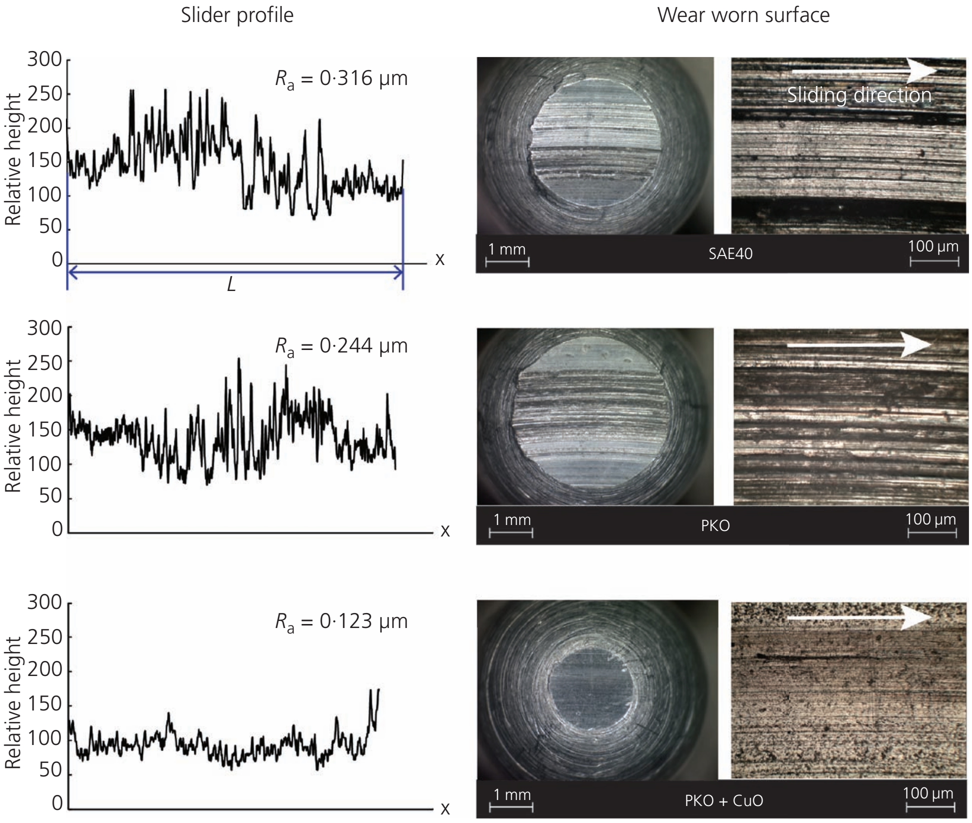


Figure 6. Wear worn surfaces and surface profile of the tested lubricants. *L* is the distance along the scar diameter

medium to improve the tribological properties of the lubricant. For future work, research to investigate the potential and feasibility of nanolubricant in real systems should be sustained.

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