



## Tribological behaviour of palm oil mixed with fly ash microparticles as a bio-based lubricant for manufacturing processes

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KEYWORDS	ABSTRACT
Fly ash Palm oil Four ball tribometer Tribological behaviour	In this study, the effect of different concentrations of fly ash microparticles in palm oil bio-based lubricant was investigated on the coefficient of friction, wear scar diameter, and worn surfaces of steel ball. The bio-based lubricants ranging from 0 to 0.14 wt% were prepared by dispersing fly ash microparticles and surfactant Span 85 in the palm oil by using ultrasonication technique. The tribological test was conducted using Four Ball Tribometer based on ASTM D4172-94 standard. The surface morphology was analysed using Scanning Electron Microscopy (JEOL6010PLUS). The results show that the inclusion of fly ash into palm oil could significantly improve the tribological behavior compared to that of pure palm oil. At the optimum fly ash concentration of 0.12 wt%, lowest coefficient of friction, smallest wear scar diameter and smoothest worn surface can be obtained.

### 1.0 INTRODUCTION

Lubricant is a substance that used to reduce friction, heat and wear between two moving surfaces. According to Sulaiman et al. (2021), during the manufacturing processes, lubricant is normally flushed to the coated tool surface before the workpiece material enters the deformation region between the tool/workpiece interface to cool down the temperature. Lubricants are classified into four groups which are solids, semi-solids, gas and liquids (Bart et al., 2013). Currently, the most commonly used lubricants in the market are mineral oils due to its low cost and good tribological performance. However, these mineral oils demonstrated a destructive

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environmental effect because they are not biodegradable and harmful. This will further lead to a higher cost in waste management (Abdollah et al., 2020).

Nowadays, bio-based lubricants are gaining popularity because of their biodegradable properties. Examples of bio-based lubricants that currently being researched on are sunflower oil, neem oil, and palm oil (Suresha et al., 2020 and Cortes et al., 2020).

According to Abdollah et al. (2020), palm oils produced a higher wear rate than mineral oils due to its incomplete oxidation and thermal stability, solidified at low temperature and low viscosity. However, these can be fixed by mixing up with additive materials through ultrasonication process. Not only that, the mixing of additive materials into lubricants also produced a longer tool life span, less coefficient of friction and better performance on machining process (Chan et al., 2018). Dmitri (2012) stated that the most widely used additive materials in the lubricants are graphite, molybdenum disulfide, tungsten disulfide ( $WS_2$ ), ester and zinc dithiophosphate (ZDP).

Fly ash is a by-product of coal thermal power plants. Hower et al. (2017) predicted that every year, about 750 million tons of coal ash are generated all over the world. Fly ash can cause environmental pollution if it does not dispose properly. Recently, many efforts have been made to recycle fly ash in concrete and ceramic industry. For example, Guo and Zhang (2020) used the fly ash as raw materials to produce autoclaved and modified wall blocks. Fly ash was also used to produce green concrete which is an environmentally friendly concrete that has higher strength and durability than the common concrete (Jiang et al., 2020). Although extensive research has been carried out using fly ash in various applications, however, the used of fly ash as additive materials in lubricant is still scarce.

Fly ash contains few major oxides like silicon dioxide ( $SiO_2$ ), ferric oxide ( $Fe_2O_3$ ) and aluminum oxide ( $Al_2O_3$ ) (Salah et al., 2017). Based on Cortes et al. (2020), addition of  $SiO_2$  nanoparticles as lubricant additives in sunflower oil could reduce the coefficient of friction and volumetric wear by 77.7% and 74.4%, respectively when compared to sunflower base oil. The polishing effect that produced by the  $SiO_2$  nanoparticles also could be confirmed by the surface enhancement of the worn surfaces.

Therefore, in this study, recycled fly ash which is high in  $SiO_2$  can be a great economical alternative to replace the expensive conventional ceramic particles as lubricant additive. The main aim of this study was to investigate the tribological behaviours of palm oil mixed with fly ash in different concentrations (0.02 wt.% - 0.14 wt.%). The experiments were carried out on the four ball tribometer in accordance to ASTM D4172-94 standard, and the coefficient of friction, wear scar diameter, and worn surfaces of steel ball were evaluated.

## 2.0 EXPERIMENT METHOD

### 2.1 Characterization of Materials

The fly ash microparticles used in this study were produced in Manjung Power plant, Perak and collected from YTL Sdn. Bhd (M). The fly ash chemical composition was characterized by using PANalytical X'Pert Pro X-Ray Diffractometer. Table 1 shows the chemical composition of the fly ash microparticles. Based on the Table 1, fly ash microparticles contains of 44.1% of  $SiO_2$ , followed by 19.1% of  $Al_2O_3$ , 13.5% of  $CaO$ , 12.4% of  $Fe_2O_3$ , 4.7% of  $MgO$ , 2.9% of  $Na_2O$ , 1.4% of  $K_2O$ , 1.0% of  $SO_3$ , 0.7% of  $TiO_2$ , 0.1% of  $Mn_2O_3$ , and 0.1% of  $P_2O_5$ . Figure 1 shows the Scanning Electron Microscope (SEM) micrograph of the fly ash microparticles. It is clearly seen that the fly

ash microparticles have a spherical shape and various sizes.

Table 1: Chemical composition of fly ash microparticles.

Chemical Composition	Concentration (%)
SiO <sub>2</sub>	44.1
Al <sub>2</sub> O <sub>3</sub>	19.1
CaO	13.5
Fe <sub>2</sub> O <sub>3</sub>	12.4
MgO	4.7
Na <sub>2</sub> O	2.9
K <sub>2</sub> O	1.4
SO <sub>3</sub>	1.0
TiO <sub>2</sub>	0.7
Mn <sub>2</sub> O <sub>3</sub>	0.1
P <sub>2</sub> O <sub>5</sub>	0.1

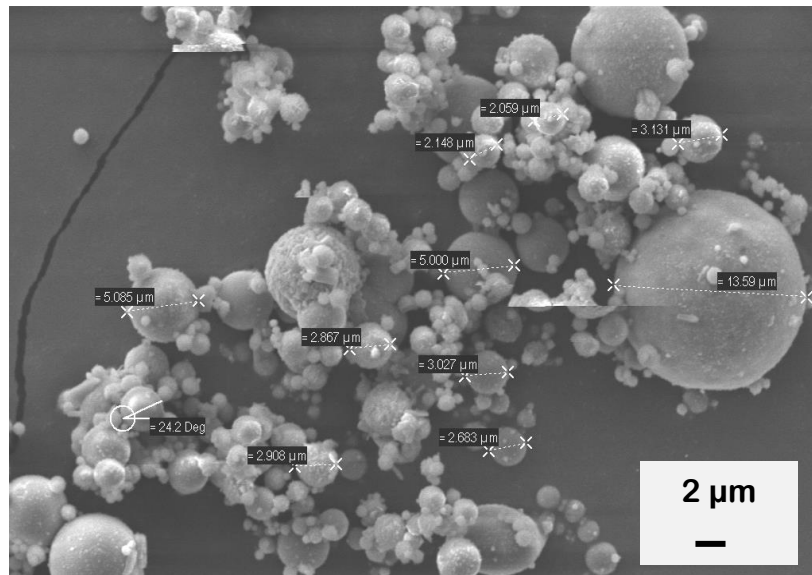


Figure 1: SEM micrograph of fly ash microparticles.

## 2.2 Preparation of Bio-Based Lubricant

In this study, palm oil (Seri Murni brand) which manufactured by FFM Marketing Sdn. Bhd was used as the base oil. The physico-chemical properties of the palm oil are shown in Table 2. The bio-based lubricant was prepared by using ultrasonication process. Desired amount of fly ash (0.02 wt.% - 0.14 wt.%) was dispersed together with same ratio of Span 85 as surfactant into palm oil, then sonicated for 1 hour under condition of 50% amplitude and 0.5s interval. Equation 1 presents the formula used for the lubricant preparation.

$$\text{Concentration of fly ash microparticles (wt.\%)} = \frac{\text{Weight of solute (g)}}{\text{Weight of solution (g)}} \times 100\% \quad (1)$$

Table 2: Physico-chemical properties of the palm oil (Darfizzi & Jumat, 2014).

Physico-Chemical test	Value
Iodine value	61.67
Acidity (%)	3.01
Saponification value (mgKOH/g)	197.18
Average molecular weight (gmol <sup>-1</sup> )	854.3
Unsaponifiable matter	0.17
Moisture content (Karl Fischer) (%)	0.22
Cloud point (°C)	7(±1)
Pour point (°C)	5(±2)
Flash point (°C)	>320(±5)
Fire point (°C)	>320(±5)
Oxidative stability (DSC) (°C)	179
Kinematic viscosity, 40°C (cSt)	45.9
Kinematic viscosity, 100°C (cSt)	9.4
Viscosity index	195

### 2.3 Tribological Test

Four ball tribometer was used to run the test for all the lubricants. Figure 2 illustrates the schematic diagram of four ball tribometer. The ASTM D4172-94 standard test method for wear preventive characteristics of lubricating fluid (four ball method) was used as a guideline to conduct the current tribological process and research parameters selection in order to develop the precision data and the parameters were presented in Table 3. Moreover, 3 repetitions for each of the test was conducted to detect the abnormality or outlier in the research data. Four steel balls were used in every test. Before the test, all the steel balls were cleaned using acetone in an ultrasonic bath for 2 minutes and then dried. Three steel balls were placed into the steel cup and then tightened using a torque wrench. One steel ball was then tightened into the machine spindle. Next, the lubricant was introduced slowly into the steel cup to prevent air bubbles from forming. After that, the steel cup was installed into the disc securely. 392N of load was applied slowly onto the steel cup to prevent shock from happening. Then, the specimen was heated to a temperature of 75°C. After that, spindle speed of 1200rpm was set to drive the spindle that holds the single steel ball. The test was conducted for a period of 1 hour. After 1 hour, the heater was turned off and steel cup was removed from the four ball tribometer. The lubricant was drained from the steel cup and the scar area produced on the three steel balls were cleaned using acetone and observed by using SEM. The test was performed thrice on the same concentration to ensure the accuracy of the results. The steps were repeated for different concentration of fly ash in palm oil. After the test, the coefficient of friction for each concentration was calculated using Equation 2. Then, the coefficient of friction for each concentration was averaged to achieve the result.

$$\text{Coefficient of friction, } \mu = \frac{T\sqrt{6}}{3Wr} \quad (2)$$

Where:

$T$  = Frictional force (Nm)

$W$  = Load

$r$  = distance from center of contact surface on lower balls to the axis of rotation = 3.67mm

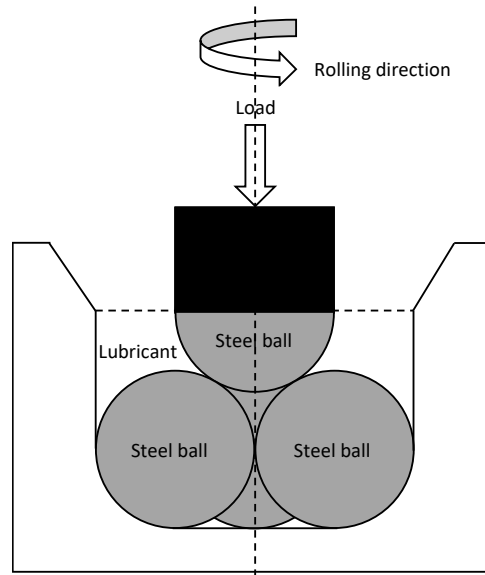


Figure 2: Schematic diagram of four ball tribometer.

Table 3: Parameter of four ball tribometer.

Parameters	Value
Load, N	392
Temperature, °C	75
Spindle Speed, rpm	1200
Duration, min	60
Repetition, run	3

#### 2.4 Surface Morphology Observation

Surface morphology observation was conducted on the steel balls after the tribology test. 3D Non-Contact Optical Profilometer was used to determine the wear scar diameter. The average wear scar diameter on the steel ball was calculated using the Equation 3. Then, the surface morphology of the worn surface was observed using the SEM.

$$\text{Average wear scar diameter, in } \mu\text{m} = \frac{HR_1 + VR_1 + HR_2 + VR_2 + HR_3 + VR_3}{6} \quad (3)$$

Where:

$HR$  = Horizontal reading of wear surface

$VR$  = Vertical reading of wear surface

## 2.5 Z-Score Test

Z-score test was conducted to determine the acceptable range of the result that obtained from the experiment. The confidence level of this experiment was set to 95%, therefore based on the normal distribution, the data with standard Z-scores between the ranges of -1.96 to 1.96 will be considered acceptable. The Z-Score of the result was calculated based on Equation 4 (Molugaram & Rao, 2017).

$$Z = \frac{X - \mu}{\sigma} \quad (4)$$

Where:

$X$  = Result obtained

$\mu$  = Mean

$\sigma$  = Standard deviation

## 3.0 RESULTS AND DISCUSSION

### 3.1 Coefficient of Friction (COF)

Figure 3 shows the coefficient of friction by using different concentrations of fly ash microparticles. The coefficient of friction values for all the samples ranged from 0.062 to 0.140, and the Z-scores of all values ranged from -1.40 to 1.41, which is within the acceptable range of the 95% confidence level (standard Z-scores is between -1.96 to 1.96). Based on the Figure 3, it was observed that pure palm oil (without fly ash) has the highest coefficient of friction value, which is 0.14. Evidently, when the fly ash microparticles dispersed into the palm oil, there were reductions in the coefficient of friction of the bio-based lubricant samples. These results are in agreement with those obtained by Mushtaq and Hanief (2021). As shown in Figure 3, 0.12 wt.% of fly ash microparticles possesses the lowest coefficient of friction, which is 55.71% lower than that of pure palm oil. The reductions in coefficient of friction value after the addition of fly ash microparticles into palm oil was probably because of the ball-bearing effect which caused by the fly ash microparticles. The spherical shape fly ash microparticles flowed in between the two contact surfaces and caused the contacted surface to roll instead of sliding.

However, when the fly ash concentration increased to 0.14 wt.%, the coefficient of friction increased instead of decreased. The increment of coefficient of friction may be caused by the agglomeration of fly ash microparticles in the bio-based lubricants. According to Salah et al. (2017) and Abdollah et al. (2020), agglomeration could form at higher concentration of additives, and increased the shear strength and led to a high coefficient of friction. Van der Waals forces could cause particles to clump together which will lead to agglomeration of particles during dispersion process (Werth et al., 2003). It is possible that at high fly ash microparticles concentration, the Van der Waals forces are higher than dispersion forces, thus causing the microparticles to agglomerate and leading to the increment of coefficient of friction. But it is noteworthy that the coefficient of friction at 0.14 wt.% was still lower compared to the value at pure palm oil. This result is consistent with those obtained by Mushtaq and Hanief (2021), where the lubricating capabilities were considerably enriched after adding the lubricant additive in the pure oil.

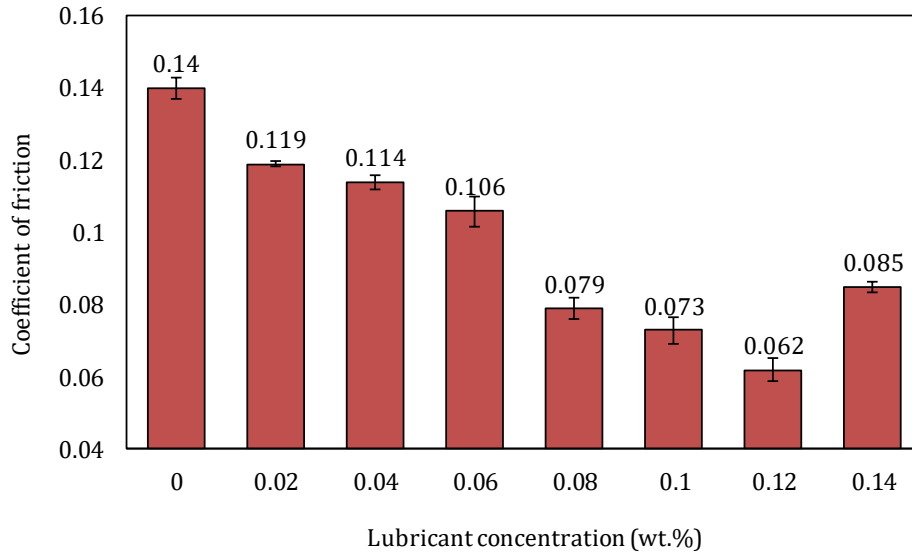


Figure 3: Effect of different lubricant concentrations on the coefficient of friction.

### 3.2 Wear Scar Diameter of Steel Ball

Figure 4 shows the wear scar diameter of steel balls after the wear test. All of the Z-scores values ranged from -1.41 to 1.41, which is within the acceptable range of the 95% confidence level (standard Z-scores is between -1.96 to 1.96). As can be seen in the Figure 4, the average wear scar diameter for pure palm oil was the highest in comparison to palm oil mixed with fly ash microparticles. However, as the fly ash concentration in the palm oil increases, the average wear scar diameter decreases gradually until it reaches the optimum value at 0.12 wt.% of fly ash microparticles. Similar results were obtained by Abdollah et al. (2020) and Suresha et al. (2020). According to them, inclusion of particles in the oil can significantly reduce the wear scar diameter and this reduction was due to the improvement in the oil viscosity which helped in the formation of thin-film. The presence of particles may reduce the shear force that acting on the steel ball interface, and causing in the reduction of contact area.

Nevertheless, when the concentration of fly ash increased further (i.e. 0.14 wt.%), the wear scar diameter tends to increase. As known from Singh et al. (2019), at a high concentration of particles (behind the optimum concentration limit), abrasion of the surfaces will be occurred and resulted more wear rate and higher wear scar diameter. However, it is worth noting that the wear scar diameter that produced by 0.14 wt.% fly ash is still lower compared to that of pure palm oil. This result indicates that dispersion of fly ash could decrease the wear scar diameter (wear rate) significantly.

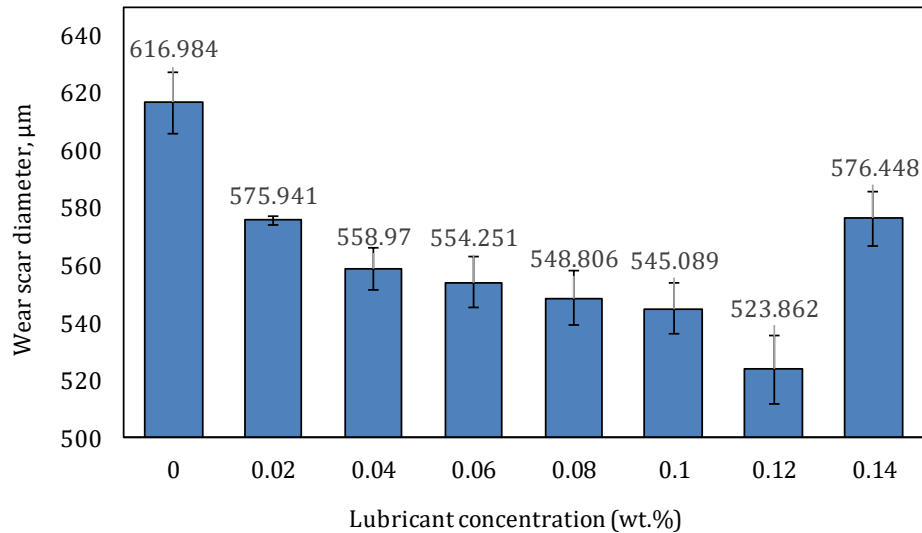


Figure 4: Effect of different lubricant concentrations on the wear scar diameter.

### 3.3 Surface Morphology

Figure 5 shows the surface morphology of the worn surface for all the concentrations of fly ash microparticles. The study of worn scar surfaces is critical for identifying metallic failures that caused by friction and wear between the contact surfaces (Awang et al., 2020). Based on Figure 5(a), it was observed that by using pure palm oil, the steel ball shows harsh surface with various rough grooves, dense scratches and deep furrows, which depicted the abrasive wear. However, as the fly ash concentration in the palm oil increases, the worn surface shows brighter grooves and smoother wear track. In the present study, the optimum fly ash concentration was at 0.12 wt.% (Figure 5g), where the steel ball shows the smoothest and brightest wear track with fewest grooves, scratches and shallow furrows. These results are consistent with findings obtained by Singh et al. (2019). The addition of fly ash to the palm oil reduced the severity of wear, and this improvement can be attributed to fly ash's ability to react with the palm oil to form a defensive tribo-layer on the steel ball surface. This layer may help to prevent surface contact between the steel balls, resulting in less friction and wear, and a smoother surface (Mushtaq and Hanief, 2021). Besides that, with the addition of nanoparticles in the oil, the grooves that formed on the steel ball may also help in holding the nanoparticles back on the wear surface, and causing reduction in deep groove formation (Suresha et al., 2020).



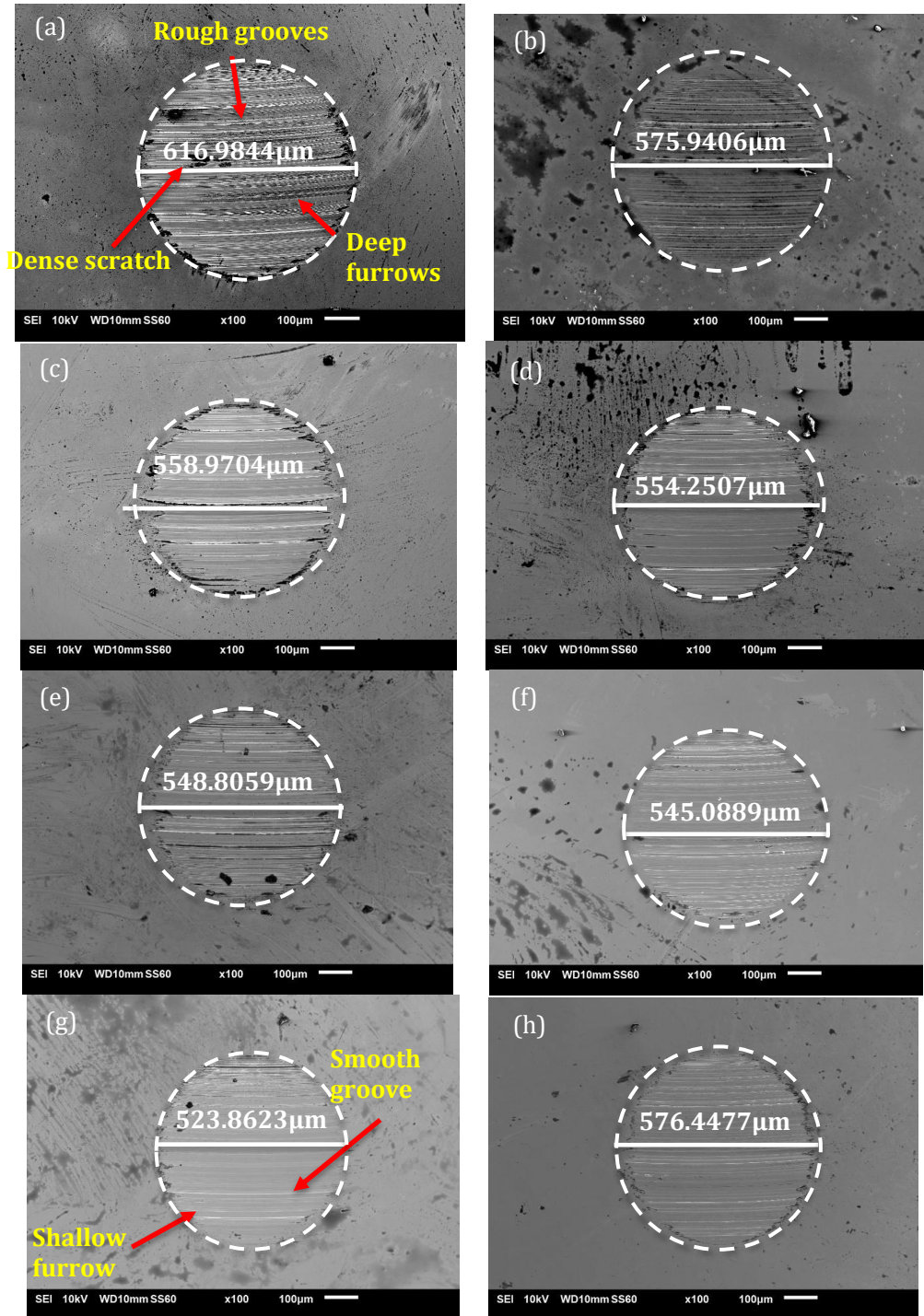


Figure 5: SEM image of worn surface on steel balls (a) Pure palm oil; (b) 0.02 wt.% fly ash; (c) 0.04 wt.% fly ash; (d) 0.06 wt.% fly ash; (e) 0.08 wt.% fly ash; (f) 0.10 wt.% fly ash; (g) 0.12 wt.% fly ash; (h) 0.14 wt.% fly ash.

## CONCLUSIONS

In this study, the tribological behaviours of palm oil mixed with fly ash microparticles were investigated, in terms of coefficient of friction, wear scar diameter and worn surface of the steel ball. Eight different concentrations of fly ash microparticles were used in the research, ranging from 0 – 0.14 wt.%. The bio-based lubricants were prepared using palm oil as base oil, fly ash microparticles as lubricant additives and Span 85 as surfactant. The significant findings of the study are summarized as follows:

- (a) Addition of fly ash microparticles in the palm oil gives a better tribological behaviour than pure palm oil.
- (b) The coefficient of friction, wear scar diameter and worn surface can be significantly reduced with lower concentration of fly ash microparticles.
- (c) The optimum concentration is at 0.12 wt.%, where it reduces the coefficient of friction and wear scar diameter by 55.71% and 15.09% respectively compared to that of pure palm oil.
- (d) With the addition of 0.12 wt.% fly ash, the worn surface has the smoothest grooves and shallowest furrow.

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