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Investigation of Modified RBD Palm Oil-Based Hybrid Nanofluids as Metalworking Fluid

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Abstract: The utilisation of hybrid nanofluid as a lubricant has a synergistic impact. This work investigated the tribological and physicochemical aspects of modified refined, bleached, and deodorized (RBD) palm oil (MRPO) comprising 0.025 weight % of hexagonal boron nitride (hBN) + titanium dioxide (TiO₂) (MRPOht) and hBN + tungsten disulfide (WS₂) (MRPOhw). The nanofluids' physicochemical parameters were assessed using kinematic viscosity, viscosity index and thermal conductivity. Furthermore, the tribological performance of the nanofluids were assessed using a four-ball test. The MRPO samples were evaluated against the marketable Synthetic Ester (SE). The outcomes presented that MRPOht has exceptional physical properties, with kinematic viscosity values of 22.82 mm²/s at 40 °C and 7.01 mm²/s at 100 °C. Moreover, MRPOht boasts the highest viscosity index, which stands at an impressive 303. MRPOhw had higher thermal conductivity of 0.1580 W.m.K and exhibits an outstanding coefficient of friction (COF) of 0.0662. The average wear scar diameters (MWSD) of MRPOs were smaller than those of SE. Overall, the combination of MRPO with hybrid additives exhibits exceptional physical, chemical, and friction-related properties, which establishes it as a feasible choice for an environmentally friendly metalworking fluid.

Keywords: nanofluids, hybrid nanofluids, nanoparticles, metalworking fluids

1. Introduction

As a heat-removal agent, a metalworking fluid is often employed in most industrial applications to minimize friction between surfaces and protect against wear. Mineral-based oils have been used in manufacturing for decades as lubricants. However, since mineral-based lubricants have limited biodegradability, high toxicity, and near-impossibility of disposal, they have caused severe environmental and health problems¹. A hazardous and unwelcoming working environment is created when personnel using machines are subjected to mineral oil-based MWF oil mist and smoke. Furthermore, nitrosamines, heavy metal compounds, and bactericides present in MWFs, as well as respiratory issues induced by inhaling hazardous fumes and contact with oil fogs and fumes, are all severe health risks associated with the use of mineral oil-based metal working fluids^{2,3}. In addition, greener products are becoming more popular due to global issues such as the high oil price, the environmental impact

of landfills, and the increased demand for environmentally friendly solutions⁴.

For the production of this plant oil or bio-lubricants, a diverse range of plants with oil-rich seeds, such as palms, jatropha, and soybeans, is required. Although bio-lubricants have several drawbacks, for example restricted thermal and oxidative steadiness, inadequate flow of fluids, and hardening at low temperatures, they nonetheless offer significant lubrication^{5,6}. Due to this flaw, vegetable oil is under chemical modification and additives reformulation to improve its properties^{7,8}. Chang et al.⁹ assessed the effectiveness of modified palm oil as a cutting fluid. The researchers discovered; modified palm oil exhibits a similar outcome to the commercially available trimethylolpropane trioleate brands. At a temperature of 40°C, it demonstrates a high viscosity of 44.3 mm²/s and an exceptional viscosity index of 219. Moreover, Salleh et al.¹⁰ conducted research on the tribological behavior of altered RBD PO as a lubricant.

There was a range of three to six hours for the reaction times. The results were compared with fully synthetic oil and RBD palm oil. From the result, they concluded that modified palm oil with a longer reaction time exhibits a lower coefficient of friction.

A nanoparticle's diameter can range from 1 to 100 nanometers, making it a novel material with unique properties. Due to its nanoscale size, it has the potential to occupy the empty space created by surface roughness (asperity). Nanomaterials characterization will enhance surface chemical reactions, resultant in the construction of a tribo-film. Nanomaterial's spherical shape of the nanomaterials promotes a rolling effect¹¹. Jamaluddin et al.¹² examined the physical and friction-related characteristics of MJO by including hBN as an additive at concentrations of 0.01wt.% to 0.05wt.% as MWFs. The tribological performance was assessed using a four-ball experiment. Incorporating 0.05wt.% hexagonal boron nitride (hBN) into MJO led to increased levels flash point and viscosity. However, MJO with 0.025wt.% shows the lowest COF. They also mentioned that a low concentration of additives was insufficient in improving the properties and the base oil. Moreover, they also mentioned that a higher number of additives is not preferable because it may cause higher COF. Ruliandini et al.⁵ inform that adding a small concentration of hBN into TMP Triester-based bio-lubricant shows great stability of the dispersion of hBN inside bio-lubricants. Previous research explored the tribological characteristics of WS₂ as lubricant additives¹³. It was found that the lubricant with additives had a lower coefficient of friction (COF) than the lubricant by itself. They also mention that WS₂ has great antiwear and antifriction properties. Singh et al.¹⁴ used TiO₂ in castor oil to act as additives and studied its friction and wear characteristics. The nanoparticles are mixed with the basic castor oil at a rate of 0.1 to 0.2%. The COF is lowest at 0.2% nanoparticle concentration and increases at 0.3% concentration. In terms of wear rate, the pin wears out at 0.2% concentration.

A considerable amount of study has been undertaken on the topic of machining using MWFs containing one kind of nanoparticles. However, to the best knowledge of the researcher, only a few studies involving hybrid nanofluids have been conducted. No standard model be able to foresee the viscosity values of hybrid nanofluids with any degree of accuracy given the several factors involved, including temperature, particle concentration, size, and shape¹⁵. There is study on the influence of various concentration of hybrid ceria-zirconia nanoparticles-coconut oil based on their tribological performance¹⁶. They found that the ideal concentration of hybrid additives was 0.62wt.% to achieve the maximum performance of the based oil. Singh et al.¹⁷ tested the alumina-graphene hybrid nano-cutting solution at 0.25, 0.75, and 1.25 vol.% volumetric concentrations. The tribological analysis shows that wear is reduced with rising nanoparticle concentration. Furthermore, the hybrid

nanofluid exhibits the slightest wear. Hybrid nanofluid has better wettability than base fluid and single nanofluid. The effects of hybrid alumina/graphene additives on the tribological characteristics of a cutting fluid used for AISI 304 steel turning were studied. There are three concentrations of the hybrid nano-lubricant that have been developed: 0.25, 0.75, and 1.25 vol.%. Hybrid nanofluids show a lesser coefficient of friction (COF) compared to alumina nanofluids¹⁸. Meanwhile, Du et al.¹⁸ examined the lubricating possessions of graphene oxide-TiO₂ nanofluid. They discovered that a nanofluid containing 0.5 wt.% GO-TiO₂ had superior friction-reducing and wear-resistant properties. Expectedly, they stated that GO-TiO₂ shows a synergistic effect as lubricant additives.

Hence, hybrid nanofluids derived from modified RBD palm oil (MRPO) were developed in this research. MRPO was combined with two hybrid nanoparticle additives at a concentration of 0.025 wt.% nanoparticles to create sustainable MWFs. MRPOs was mixed with hexagonal boron nitride (hBN) nano additives that were hybridized with titanium dioxide (TiO₂) and tungsten disulfide (WS₂). The kinematic viscosity and viscosity index and of hybrid nanofluids were determined, as well as the tribological characteristics of the hybrid nanofluids. The outcomes were then compared to the pure forms of MRPO and SE. In the lubricating sector, novel hybrid formulation nanofluids may open new possibilities as MWFs.

2. Methodology

2.1 Hybrid nanofluid sample preparation

A two-step transesterification process was conducted to refine, bleach, and deodorize (RBD) palm oil. In 1% sodium hydroxide (NaOH) presence, RBD palm oil reacts with methanol (CH₄O) (water bath condition at 60°C). This mixture was stirred continuously for 2 hours and produced fatty acid methyl ester (FAME)¹⁹. Following that, FAME undergo second transesterification process by reacting with trimethylolpropane (TMP) in the presence of 1% (wt/wt) sodium methoxide (NaOCH₃)⁷. The FAME and TMP molar ratio (FAME: TMP) was 3.5:1. This process took place bath at a constant temperature of 120°C in an oil bath condition. This solution was stirred continuously for 24 hours in vacuum condition at a constant pressure of 20kpa. This process produces the base oil for metalworking fluid, modified RBD palm oil (MRPO).

As indicated in Table 1, hybrid nanofluids were created by combining MRPO with solid hybrid nanoparticles, hBN with WS₂ and TiO₂, respectively with fixed weight concentration of 0.025 wt.% based on the based oil weight as shown in Equation 1. Table 2 indicates the properties of each nanoparticle. The based oil was heated until 60°C, mixed with hybrid nanoparticles for 30 minutes, and stirred at 700rpm. The mixture was homogenized for 30 minutes using a Bandelin HD3200 type ultrasonic homogenizer with frequency of 20 kHz 200 W of power

to increase the mixture stability²⁰). All combinations of precipitation and layer separation were examined in the last phase. By using this method, hybrid nanoparticles were uniformly dispersed throughout MRPO. In addition, the sedimentation was observed to identify the stability of the dispersion²¹). In comparison to MRPO, the density of hybrid nanoparticles is higher. Therefore they tend to sink over time. Because there is no visible sedimentation after a short time of observation, it has been determined that the hybrid nanoparticle dispersion in MRPO has excellent dispersion stability. All hybrid nanofluid samples were then compared with SE.

$$wt. \%_{np} = \frac{wt. np}{wt. bo} \times 100 \quad (1)$$

Table 1. Hybrid nanofluid samples description.

Symbol	Description
MRPOht	MRPO + (hBN + TiO ₂)
MRPOhw	MRPO + (hBN + WS ₂)

Table 2. Nanoparticles properties.

Properties	hBN	WS ₂	TiO ₂
Appearance (Powder)	Colorless crystal	Blue Gray	White solid
Size(nm)	Less than 100 nm		
Density (g/cm ³)	2.3	7.5	4.23
Coefficient of Thermal expansion (10 ⁻⁶ /K)	1	10	8.4
Thermal conductivity (W/m.K)	8.4	53	4.8

2.2 Viscosity testing

The physical properties of hybrid nanofluid samples were assessed by measuring their kinematic viscosity and viscosity index (VI). One of the most significant criteria for the role of lubricant is viscosity²²). At 40 °C and 100 °C, the kinematic viscosity of the samples was measured using Viscometer equipment, Viscolite 700, following ASTM D445. The viscosity index (VI) also showed a link between viscosity and temperature²³). The viscosity index (VI) was determined by interpolating the lubricant's kinematic viscosity with the data provided in ASTM D2270. The procedure was repeated thrice, and the average result was obtained.

2.3 Thermal conductivity testing

Thermal conductivity is crucial for nanofluids used in lubricants and metalworking fluids since it significantly impacts heat transfer efficiency. Nanofluids are a combination of nanoparticles dispersed in base fluids that

exhibit enhanced thermophysical properties, particularly heat conductivity. Nanofluids with high thermal conductivity are favoured for enhanced heat transfer at the cutting zone and increased heat dissipation in machining operations, resulting in improved cooling and lubrication²⁴). The thermal conductivity testing was carried out according to ASTM D7896-19. The hotplate was heated until it reached a temperature of 100°C. Once the hotplate stabilized at this constant temperature, the sample was carefully positioned on it. Subsequently, the probe was submerged into the liquid sample, and temperature sensors integrated into the apparatus recorded temperature changes over time. Continuous data collection persisted until the temperature of the sample reached 100°C. The collected data were then subjected to analysis to ascertain the time-dependent temperature variations of the sample. The thermal conductivity of the liquid sample was subsequently calculated using equation 2. The experiment was conducted two times, and the average value of thermal conductivity was calculated.

$$\lambda = \frac{Q}{4\pi m} \quad (2)$$

2.4 Tribological testing

Antifricition and antiwear qualities of each lubricant sample were determined by tribology studies involving four-ball wear tests. In accordance with ASTM D4172, a four-ball wear tribotester (Ducom TR-30 L) was used to evaluate the tribological performance of the samples. The experimental setup employs a chrome steel ball (AISI 52100) with a precise diameter of 12.7mm and a hardness ranging from 64 to 66 HRC. Each experiment included four separate balls. Three stationary balls were placed in the ball pot as seen in Fig. 1, and one spinning ball was held in the collet. The ball pot contained approximately 10 ml of the samples. Subsequently, the ball pot assembly was inserted into the four-ball machine prior to operation. During each set, the upper ball rotates against three fixed balls under a force of 392 N, at a speed of 1200 rpm, and a temperature of 75°C for a duration of 60 minutes. The friction coefficient was automatically calculated by the software. The average lengths of both horizontal and vertical scars were used to measure the mean wear scar diameters (MWSd) of the stationary steel balls, using an image capture tool. The test was repeated twice for each lubricant sample. The worn surface was analysed using a Scanning electron microscope (SEM).

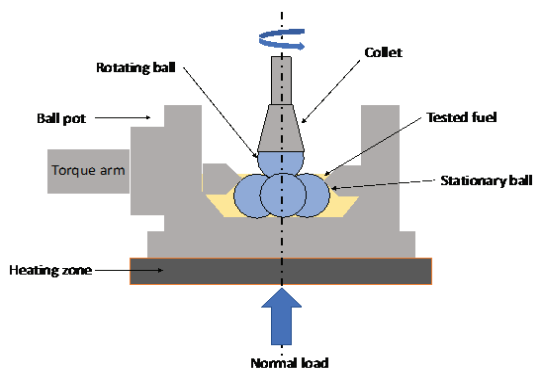


Fig. 1: Diagrammatic schematic for the four-ball test.

3. Result and Discussion

3.1 Kinematic viscosity and viscosity index

Figure 2 displays the kinematic viscosity measured at temperatures of 40 °C and 100 °C, as well as the calculated viscosity index for MRPO, MRPO hybrid nanofluids, and SE. The kinematic viscosity of SE at 40 °C, was significantly higher (23.12 mm²/s) compared to the MJOs samples. The higher viscosity of SE is attributed to the inclusion of a corrosion inhibitor, anti-wear additives, and viscosity improver in the lubricant²⁵. Meanwhile, the lower kinematic viscosity of MRPO samples was related to the chemical alteration of the MRPO composition. Due to a weakening of hydrogen bond intermolecular interactions, the viscosity of MRPO decreased²⁶. In addition, the presence of unreacted FAME also might contribute to the lower kinematic viscosity of MRPOs²⁷.

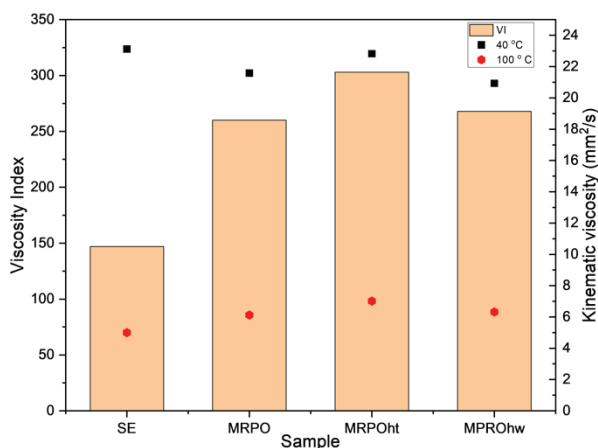


Fig. 2: Test result of kinematic viscosity and VI value calculated for all samples.

Notably, the use of hybrid nanoparticles enhanced the viscosity of MRPO. MRPOht and MRPOhw demonstrate higher kinematic viscosity. MRPOht improved by 2% at 40 °C and 3.1% at 100°C. Meanwhile, MRPOhw improved with 5.4% at 40 °C and 3.1% at 100°C. This trend happens because the presence of nanoparticles creates larger nanoclusters that obstruct the flow of fluid

layers. Therefore, the intermolecular strength become stronger, causing the layers to flow closer to one other, resulting in an increase in viscosity²⁸. In addition, the presence of hBN inside the mixture also contributes to the higher kinematic viscosity. This finding was also proved by Jamaluddin et al.¹²) reported that adding hBN enhances the feedstock's viscosities. MRPOht had the highest kinematic viscosity among the MRPO samples, measuring at 40 °C with 22.82 mm²/s and with 7.01 mm²/s at 100 °C. From this phenomenon shows that TiO₂ plays an essential role in improving kinematic viscosity. Nik Roselina et al.²⁹) discovered a similar result, stating that the inclusion of well-disseminated TiO₂ nanoparticles increases flow resistance resulting in high viscosity of the oil.

SE has the lowest kinematic viscosity at 40°C when compared to MRPOs. However, the kinematic viscosity of SE was the lowest at 100°C, with 5 mm²/s. This discovery demonstrates that the kinematic viscosity of SE decreases dramatically from the greatest to the lowest temperature and hence correlates with a lower viscosity index (VI). The graph shows that MRPO displays a higher VI of 260 than SE. The creation of longer molecular chains and the bulk of TMP ester aid in synthesising higher VI³⁰. MRPOht and MPROhw had higher VI compared to MRPO. Adding hBN nanoparticles significantly improves the VI by about 3 to 16%. hBN exhibited a smaller coefficient of thermal expansion coefficient ($1 \times 10^{-6} \text{ K}^{-1}$), that improved thermal stability of MPRO^{30,31}). Among MRPOs, MRPOht shows the highest VI (303). The coefficient of thermal expansion of TiO₂ being lower ($8.4 \times 10^{-6} \text{ K}^{-1}$) than WS₂ affected the VI³²). Materials having a lower thermal expansion coefficient are desirable while operating at higher temperatures and friction³³). The combination of hBN and TiO₂ produced a smaller thermal expansion coefficient than hBN and WS₂. Thus contribute to higher VI due to the low thermal expansion coefficient. In summary, hybrid MRPOht and MPROhw shows that including the two unique additives enhances the properties of the vegetable-based oil. This finding is corroborated by Moghaddam & Motahari³⁴), who documented that the viscosity of a base oil, when combined with two additives, exhibited a higher viscosity compared to the base oil alone.

3.2 Thermal conductivity

The graph in Fig. 3 shows the thermal conductivity of all samples. The thermal conductivity of SE was lower with (0.14541 W/m.K) than MRPO (0.1570 W/m.K). The incorporation of hybrid nano additives had improved the thermal conductivity of the base oil by 0.6% to 5.4%. In general, the addition of nano additives to the base oil increased the thermal conductivity of the base oil. The thermal conductivity of nanofluids is influenced by Brownian motion, where collisions between nanostructures establish a pathway for heat transmission known as solid-to-solid conduction channel, which may be enhanced by the percolation process³⁵).

MRPOhw show higher thermal conductivity of 0.1580 W.m.K than MRPOht. This was due the incorporation of hybrid nano additives hBN with WS₂ in MRPO. WS₂ had higher thermal conductivity of 53 W/m.K assist in the improvement thermal conductivity of the base oil. In addition, the 2D structures of WS₂ contribute to enhance thermal conductivity of the base oil. The high aspect ratio of tungsten disulphide (WS₂) nano additives allows for heat transmission and long-term colloidal stability of nanofluids. Furthermore, quicker heat transmission across nano additives networks is made possible by the 2D shape of WS₂ nanostructures, which improves the colloidal stability of nanofluids in comparison to spherical nanoparticles^{36,37}. The synergistic effect of hBN and WS₂ on nanofluids improves their thermal characteristics, stability, and heat transmission effectiveness. hBN when mixed with WS₂ enhance the thermal conductivity and stability of nanofluids. These nanofluids have enhanced heat transfer efficiency, making them ideal for applications that demand effective heat dissipation

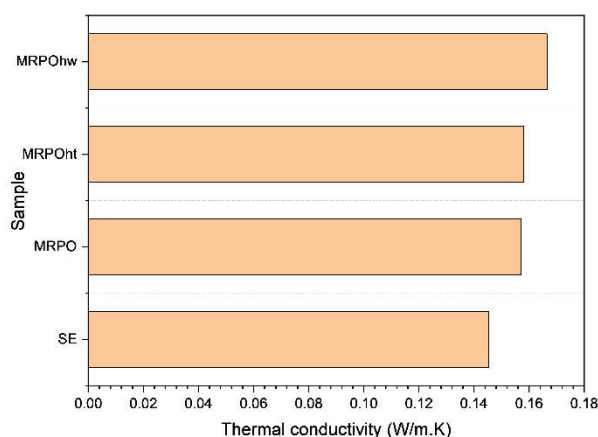


Fig. 3: Test result for thermal conductivity of all samples.

3.3 Mean wear scar diameter (MWSD) and Coefficient of friction (COF)

The graph in Fig. 4 depicts the mean wear scar diameter (MWSD) and friction coefficient (COF) for all samples. SE had higher COF compared to MRPO samples. MRPO, MRPOht and MRPOhw were improved by 6%, 31% and 50%, respectively, in terms of COF. This occurrence implies that a poly branch TMP polyol ester in MRPO improves the tribological performance⁴⁰. Additionally, the fatty acids included in MRPOs have the ability to provide a thin layer of lubrication that firmly attaches to the surfaces in contact. The densely arranged polar carboxyl chains in the fatty acids formed an effective lubricating layer that might potentially reduce friction.⁴¹

Interestingly, adding hybrid nanoparticles improved the COF value compared to SE and MRPO. MRPOhw demonstrates the lowest COF, 0.0662. The inclusion of additives in MRPOs allowed the creation of a thicker lubricating coating on the ball faces, resulting in reduced uninterrupted touch between the metal asperities and, as a

result, lower friction forces, COF, and enhanced lubrication efficacy⁴². Adding hBN fills the surface valleys to form a rolling effect in sliding friction, thus helping reduce the friction⁴³. Moreover, the depositional layer that WS₂ creates, smoothens and evens out sliding friction and surface roughness. As the friction increases with increasing load, WS₂ reacts chemically with the base material of the friction pair to form a lubricating Ferum(II) Sulphide (FeS) layer. It can prevent direct interaction between the regions of friction, hence significantly reducing the coefficient of friction⁴⁴. Creating a stable nanoscale WS₂ tribofilm ensures reduced coefficient of friction values¹³. According to the present result, the incorporation of HBN and WS₂ could drastically improve the COF of MRPO.

From Fig.4, an insignificant reduction in MWSD is shown in MRPO (670 μ m), MRPOht (659.35 μ m) and MRPOhw (656.60 μ m). However, the MWSD for MRPOs was smaller than SE (940.87 μ m) with approximately 30% maximum reduction from the base oil. Excellent lubrication was made possible by developing a TMP triester in the base oil. TMP triester, compared to SE, has a longer fatty acid chain that tended to enhance the thickness of the tribofilm^{30,45}.

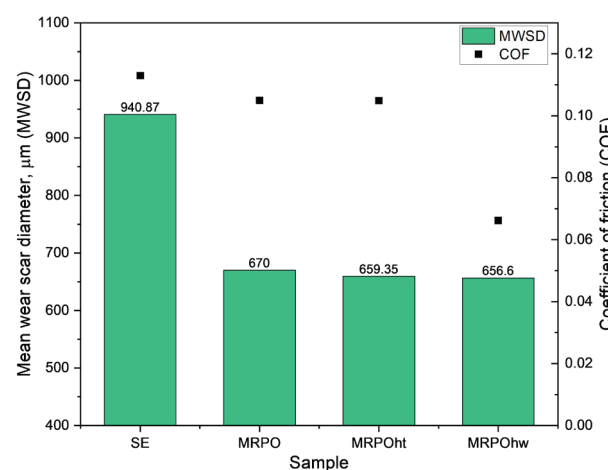


Fig. 4: Test result for mean wear scar diameter (MWSD) and coefficient of friction (COF) for all samples.

3.5 Worn surface analysis

The surface morphology of the worn region is displayed in Fig. 5. Shallow grooves and deep grooves appeared at the worn surface for both MRPOht and MRPOhw. The creation of deep grooves in the center of the surface of Fig.5(a) might be due to the abrasion impact of hBN and TiO₂ nanoparticles entrapped between the balls during the sliding process. In addition, the existence of TiO₂ nano-additives in the friction region might cause the granule abrasions in material⁴⁶. The existence of Titanium (Ti) and Oxygen (O) is confirmed by the EDX analysis of the worn surface in Fig 6(a). From Fig.6, some dark spot was observed at the MRPOht and MRPOhw ball surface. The higher percentage of Boron (B) shown in the EDX

analysis could be related to the presence of agglomeration of hBN at the dark spot.

nanoparticles could improve the worn surface of the steel ball.

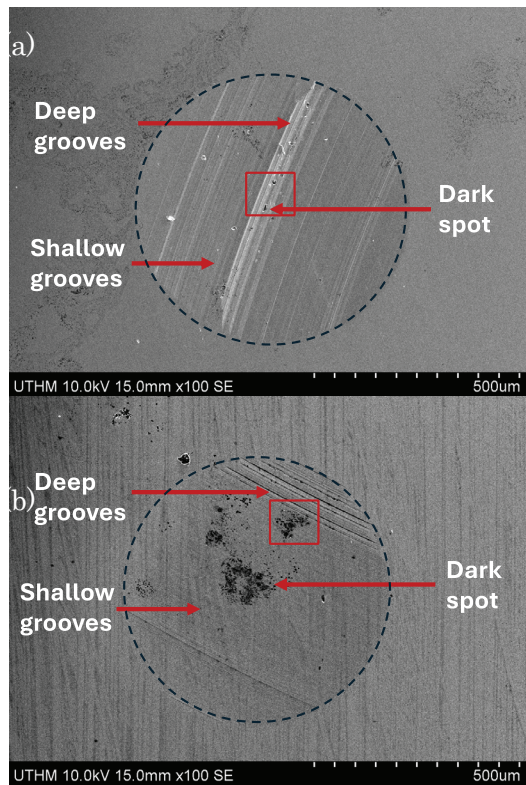


Fig. 5: The worn surface of steel ball for (a) MRPOht and (b) MRPOhw at 100x magnification.

Worn surface MRPOhw shows a darker spot than worn surface MRPOht. During the four-ball wear test using MRPOhw, some black material adhered to the worn areas, which should be the WS_2 nanoparticles. Zhang et al.⁴⁷⁾ also discovered a comparable pattern of the worn surface caused by the addition of WS_2 nanoparticles. WS_2 nanoparticles had darker powder than other nanoparticles, as mentioned in Table 2. The EDX analysis shown in Fig. 6(b) shows the presence of Tungsten and Sulphur at the steel ball worn surface. The size of the black patches is more significant than that of the WS_2 particles, which may be connected to the stacking behaviour of the soft WS_2 particles during the wear tests. During the wear testing, more WS_2 particles may get attached to the worn surfaces. However, the worn surface for MRPOhw appeared to be smoother compared to the worn surface MRPOht. This was due to the presence of WS_2 . They directly fill the grooves, repairing surface damage. This type of tribo-film can enhance wear resistance⁴⁸⁾. An et al.¹³⁾ stated that WS_2 has better antiwear properties. The thermal conductivity of the nanofluids also plays important role. MRPOhw had higher thermal conductivity than MRPOht that resulting in more stable formation of lubricant formation during four ball testing. Thus, the worn surface of steel ball for MRPOhw appear smoother than worn surface surface steel ball MRPOht. Overall, the addition of hybrid

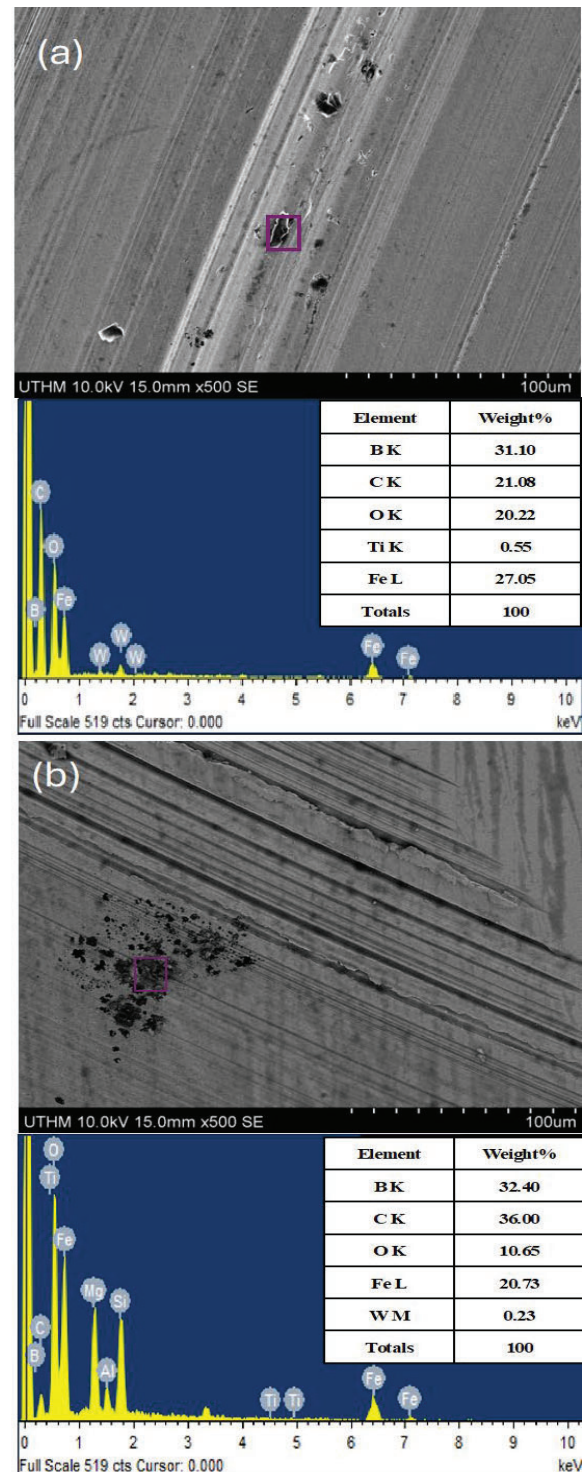


Fig. 6: Chemical analysis on the dark spot by EDX of (a) MRPOht and (b) MRPOhw

4. Conclusion

According to the present research, an evaluation of the comparative impact of each nanofluids as an environmentally friendly MWFs was carried out in this

study, with the results of the tests being compared to commercial SE. After being chemically altered and having hybrid nanoparticles added, it was discovered that the physicochemical and tribological characteristics of MRPO were significantly improved. MRPOht had the highest VI. The viscosity of the lubricant impacts its performance since it modifies the lubrication film's thickness. MRPO with 0.025% hBN and WS₂ (MRPOhw) had higher thermal conductivity of 0.1580 W.m.K and possesses the lowest coefficient of friction (0.0662). MRPOs samples contribute to smaller MWSD compared to SE. The worn surface of MRPOhw was smoother than MRPOht. MRPO with hybrid additives demonstrates remarkable performance in terms of viscosity and tribological characteristics and is appropriate to replace the SE.

Acknowledgments

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Nomenclature

CH ₃ OH	Methanol
COF	Coefficient of friction
FAME	Fatty acid methyl ester
FeS	Ferum sulfide
hBN	Hexagonal boron nitride
<i>m</i>	Slope
MWFs	Metalworking fluids
MRPO	Modified RBD Palm Oil
MRPOht	MRPO+0.025 wt.% of (hBN+ TiO ₂)
MRPOhw	MRPO+0.025 wt.% of (hBN+ WS ₂)
MWSD	Mean wear scar diameter
np	nanoparticles
RBD	Refined, Bleached, Deodorized
SE	Synthetic ester
TMP	Trimethylolpropane
TiO ₂	Titanium dioxide
VI	Viscosity Index
wt.% _{np}	Weight percentage of nanoparticles
wt. _{np}	Weight of nanoparticles
wt. _{bo}	Weight of based oil
WS ₂	Tungsten Disulfide
λ	Thermal conductivity (W/m.K)
<i>Q</i>	Heating Power (W/m)

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