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The effects of additives on anti-wear properties of lubricating grease formulated from waste engine oil ☆

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ABSTRACT

The goal of this study was to study the anti-wear properties of grease formulated from waste engine oil (WEO) when additives are added. There were two types of grease formulated namely sodium and fumed silica (FS) greases. The greases were formulated using a weight percentage ratio with and without the addition of additives before the attributes of consistency, FTIR (Fourier transform infrared spectroscopy), and anti-wear analysis were conducted. Results showed that the addition of additives did not affect the properties of the formulated grease except for that of sodium grease as demonstrated by the FTIR result. Sodium grease produced a spectrum with a peak in the region < 600 cm^{-1} when analyzed using FTIR. The corrosiveness of the grease toward the copper strip was also low, as determined by class 1 corrosiveness. The addition of additives resulted in no improvement in the anti-wear properties of the grease as the coefficient of friction (COF) was low for the grease without additives than for the grease with additives. However, the addition of additives can reduce the wear scar diameter of the greases. Based on the findings of this investigation, it was found that the addition of additives did not alter the properties of the greases or improve the anti-wear properties of the greases except for the wear scar diameter.

1. Introduction

Grease, according to NLGI, is a mixture of three main elements namely base oil, thickener, and additives. The base oil is usually the primary component of grease, which can either be petroleum-derived or synthetic oils. The thickener helps to maintain the grease in a semi-solid state and sets the grease apart to form a liquid lubricant. As for additives, they are only present in a small number inside the grease, which helps to enhance the performance of the grease.

During the formulation process, the thickener produces entanglement networks that trap the oil while also contributing to the proper function of the grease. The composition and microstructure of the grease influence the performance of the lubricating grease during the production process [1,2]. Sodium stearate and fumed silica were used in the study due to their resemblance to soap grease and non-soap grease, respectively. As sodium is the most common soap grease produced, the data is abundant. Previous research on the production of greases has been focusing on determining their properties. Each study took on a different angle of perspective. The findings have confirmed the properties of sodium soap grease, such as low or non-resistant to water as found by Akumefula et al. (2019) and Buhlak et al. (2014) [3,4]. Apart from that, Abdu Rahman et al. (2019) and Iheme, Chukwuma, et al. (2014) have found that the thickener used has a significant impact on the grease consistency [5,6]. Fumed silica was selected due to the current trend in non-soap grease production, which uses fumed silica (FS) as a thickener. This material has a very large surface area due to its extremely small particle size with a strong thickening effect. Furthermore, there has been no report of swelling and chemical inertness in FS, making it an effective thickener. FS grease showed great stability with less oil bleeding and oil separation as stated by A. Japar et al. (2019) and (Razali et al., 2017) [7,8]. Furthermore, Zakani et al. (2018) found that the structure of the grease is also different from soap grease as FS grease falls under the non-soap grease category [9].

Additives are essential for increasing the quality of grease. To obtain grease with optimum quality, 2–8 percent of additives are often included in grease formulation. A well-made grease has features that are appropriate for its use, such as consistency, thermal stability, oxidation, wear protection, corrosion resistance, and so on [10]. In this proposed study, PTFE and graphite were considered in the formulation as they are excellent at decreasing friction coefficient and wear rate [11,12]. PTFE particles have a shock-

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absorbing effect that will help to keep the surface apart while in graphite, carbon molecules are attached to just three other carbon particles to frame solid, two-dimensional layers that are amazingly steady. These properties help to improve the tribological performance of the grease.

Wear caused by a lack of lubrication is one of the most common problems with machinery. Wear is defined as an occurrence when two surfaces in persistent contact cause damage, gradual removal, and displacement of material on a solid surface. Wear in the machinery will almost certainly result in reduced functionality, which may lead to eventual machine failure. Wear differs from friction in a way that friction is the force created when two surfaces come into contact, whereas wear is the occurrence of chemical and physical degradation that affects the quality of the substances in contact [13,14]. The use of appropriate additives can help to reduce machine wear.

A previous study revealed that it is possible to formulate grease without the addition of additives when WEO is utilized. The resulting grease has qualities that are similar to those seen in commercial grease. The goal of this research was to see how additives can affect the anti-wear capabilities of the grease prepared with WEO and the improvement that the additives add to the grease properties.

2. Experimental work

2.1. Grease formulation

The first step in making sodium grease was to remove any remaining moisture from WEO by heating it to 120 °C for at least one hour. During the healing phase, continuous stirring was done to guarantee that all of the moisture inside the WEO evaporated into the air. After one hour, the temperature was raised to 180 °C while the stirring continued. When the temperature hit 180 °C, a thickening was generated by gradually adding sodium stearate. A stator-rotor homogenizer running at a minimum speed of 4000 rpm was used to homogenize the mixture for at least 3 h. The homogenization process was carried out until the sample paste was completely smooth. The additive was added after the thickener was thoroughly incorporated into the mixture. The grease was allowed to cool to ambient temperature before being stored in an enclosed container after homogenization. To ensure that the grease was entirely cool, it was let cool for two days.

Abdulbari et al. (2008) developed the process for generating FS grease. WEO was heated to 120 °C for at least one hour before being used to make FS grease. Stirring was also done regularly to ensure that all of the moisture in the oil was evaporated. Before adding the fumed silica, the temperature of the WEO was lowered down to 80–90 °C. The addition of FS was done in stages to ensure perfect homogenization [15].

The homogenization continued for the next three hours at a minimum speed of 4000 rpm using the homogenizer. The homogenization process was done when the mixture formed a gel-like consistency. The additives were added 30 min before heating and heated until fully homogenized. The homogenization was continued for an additional one hour without the heating process. Additives were added at the final concentration of 2 % in this study to investigate the possible changes in the grease properties. The ratio of ingredients used in the grease formulation is shown in Table 1 after the best SG and FS compositions were determined.

Table 1
Grease formulation ratio.

Grease	Additives (g)	WEO + Thickener: 100 %	
		WEO (g)	Thickener (g)
SG	–	282	18
FS	–	249	51
Grease	**Additives (g): 2 %**	**WEO + Thickener: 98 %**	
		WEO (g)	**Thickener (g)**
SG + PTFE	6	279.18	17.82
SG + Graphite	6	279.18	17.82
FS + PTFE	6	246.51	38.61
FS + Graphite	6	246.51	38.61

2.2. The base oil and grease analysis

The physicochemical properties of the WEO, treated WEO, and FEO were studied and analyses of the consistency, FTIR, and anti-wear properties of the grease were carried out.

3. Result & discussion

This section addressed the findings of this investigation. Table 2 and Table 3 show the results obtained from the analysis of the grease properties.

3.1. Oil physicochemical

Table 2 shows that the untreated WEO turned black after being used in the field. Pre-treatment of WEO resulted in improvement in the properties of the WEO also with some of the oil properties being restored. However, the appearance of the WEO remained black even after the treatment. It was also found that the treated WEO has a higher VI than FEO. It is known that many factors can contribute to the increase in VI in WEO, such as contamination and oxidation. The degradation of FEO after usage led to a high VI of WEO due to changes in the molecular structure of the oil. This molecular change, however, cannot be corrected as the changes include polymerization, evaporative losses, oxidation, as well as the formation of carbon and oxide soluble. Despite the changes in molecular, the contamination that presents in WEO can be removed [16]. On the other hand, there was no significant difference recorded between the density of the FEO, untreated WEO, and treated WEO oil.

3.2. Grease consistency

The greases that are the most grease-like and functional are NLGI with grade 2–3 consistency or 220–295 mm/10 penetration.

Table 2
The physicochemical properties of base oil.

Base oil	Test method	Fresh Engine Oil (FEO)	Untreated WEO	Treated WEO
Appearances	Visual	Clear & bright	Black	Black
Kinematic viscosity: @ 40 °C,	ASTM D445	103.31	–	95.653
mm^2/s @ 100 °C, mm^2/s		13.655	–	13.788
Viscosity index (VI)	ASTM D2270	132.04	–	146.21
Density, g/mL	Gas pycnometer	0.8554	0.8751	0.8640

Table 3
The properties of the greases with and without additives.

Properties	SG	SG + PTFE	SG + Graphite	FSG	FSG + PTFE	FSG + Graphite
Appearance	Normal grease	Normal grease	Normal grease	Normal grease	Normal grease	Normal grease
Consistency (NLGI)	2–3	2–3	2–3	2	2	2
Coefficient of Friction (COF)	0.053	0.062	0.063	0.11	0.09	0.08
Wear Scar Diameter (WSD), µm	451.44	415.65	432.78	774.61	760.68	787.05

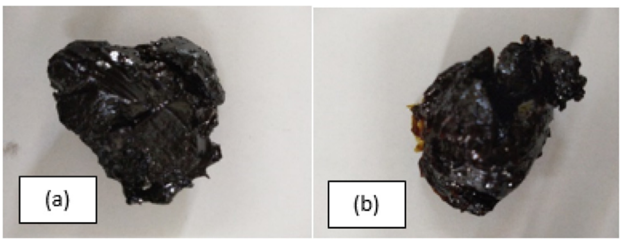

Fig. 1. The appearances of WEO-based sodium (a) and FS (b) greases.

[17,18]. A texture that is too soft may cause the grease to migrate away from the intended site, whereas a texture that is too stiff may cause failure in the migration of the grease to the intended location.

As indicated in Fig. 1, the production of sodium and FS grease resulted in grease with a black appearance. This is because WEO emits a dark color. Except for the addition of additives, the characteristics of the greases were not different from those of existing greases due to the same formulation used. Because the additives are designed to improve a specific aspect of the grease, they are unable to change the whole quality of the grease. As a result, the best grease produced has the same quality as that of NLGI grade, as shown in Table 3.

3.3. FTIR analysis

As previously mentioned, the properties of sodium and FS greases did not change following the incorporation of the additives except for a small alteration. This is evidenced by the spectrum in Fig. 2, which shows a comparable peak produced by sodium and FS greases. The only differences can only be seen in the spectrum of the sodium grease at the region < 600 cm^{-1} (fingerprints region), whereby the grease without additives produced a lot of small peaks. This region indicated that there was an S—S stretch in the sodium grease with the addition of the additives [19]. Based on these spectra, it can be concluded that the additives caused a slight

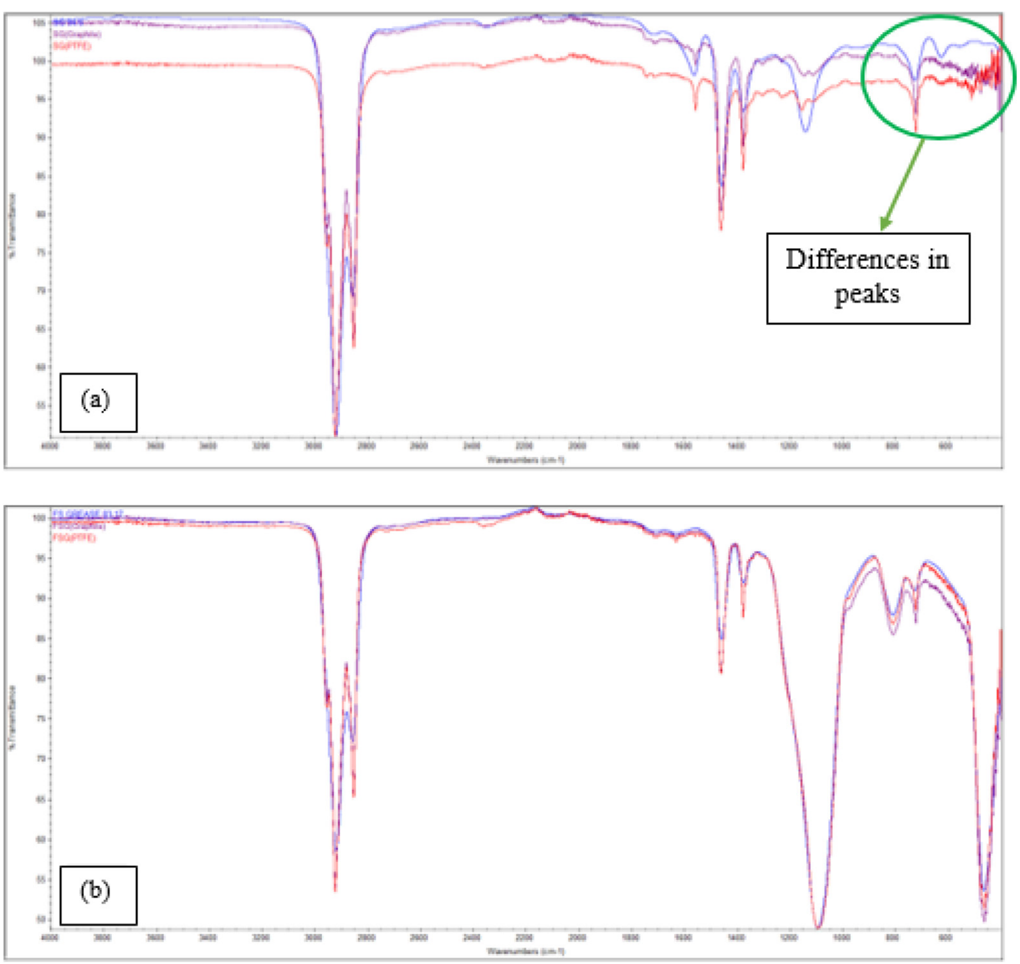

Fig. 2. The spectrum of (a) sodium & (b) FS grease with and without additive.

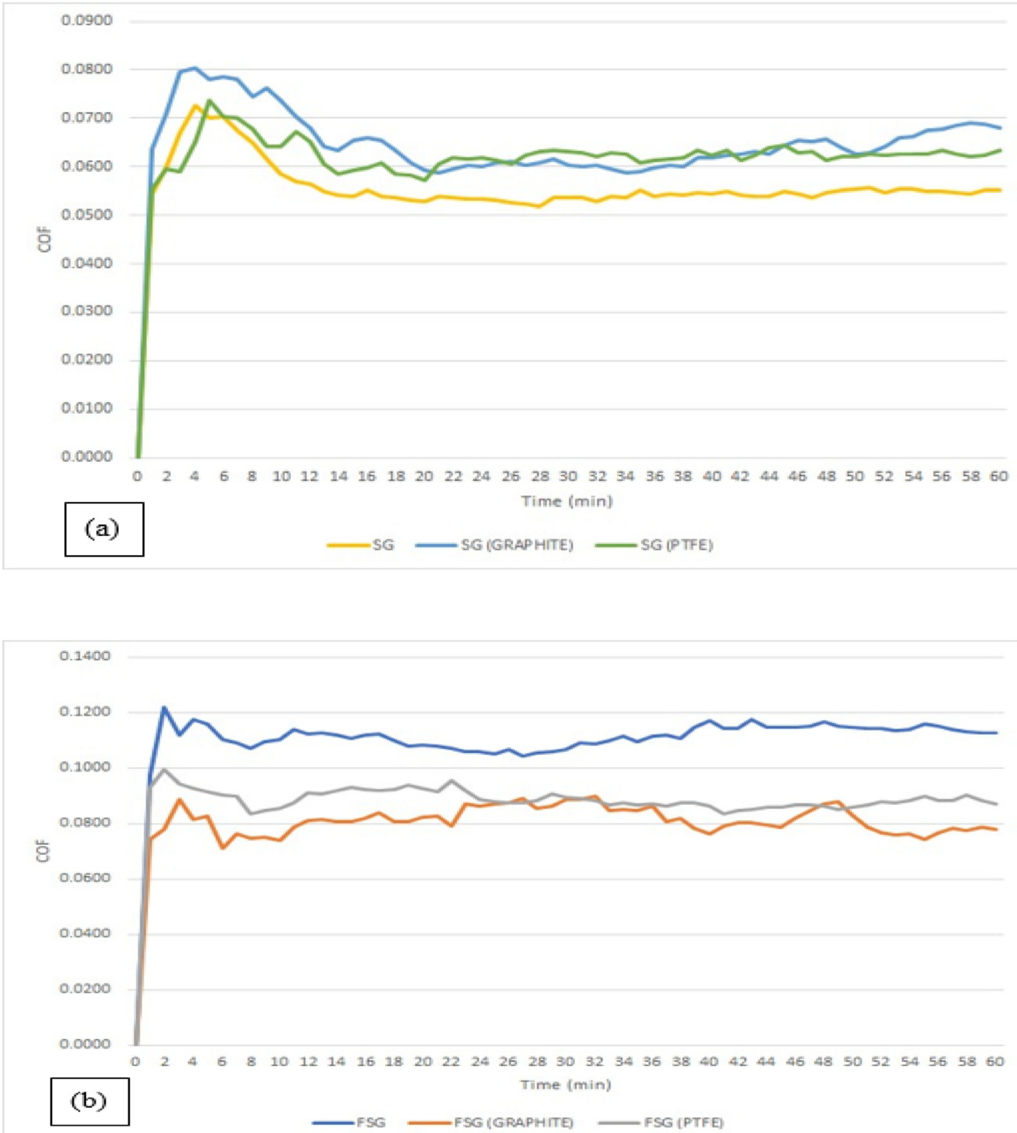

Fig. 3. Friction coefficient (COF) of the (a) sodium & (b) FS greases.

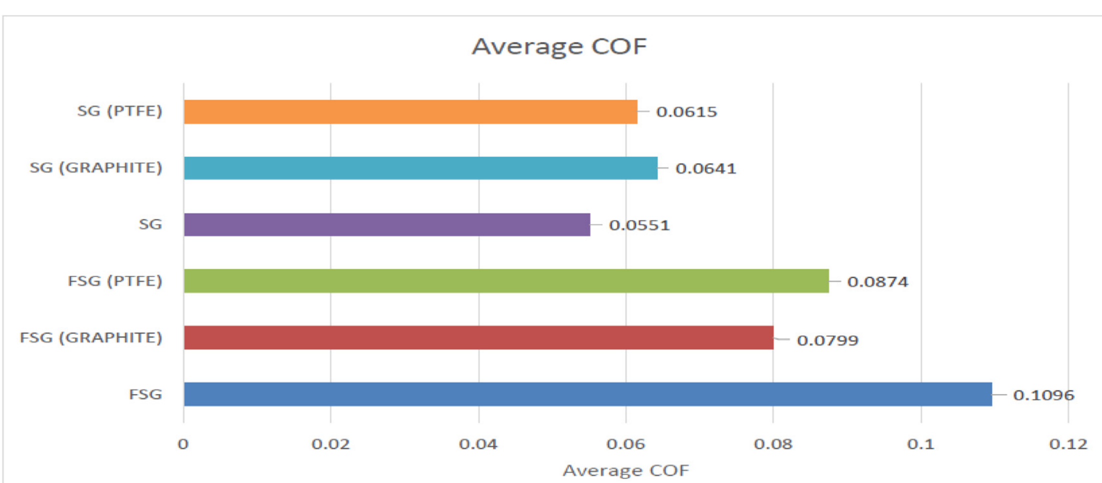

Fig. 4. Average COF of greases.

change in grease properties as intended. However, these changes did not happen to FS grease, as the spectrum for FS grease remained similar with and without additives. Such observation implies that the small difference in the FTIR spectra might also be due to the use of additives in small quantities.

3.4. Anti-wear

Since PTFE and graphite were used as additives to reduce friction between moving parts, tribological research was conducted to compare the greases with and without additives.

3.4.1. Friction test

For a period of 60 min, COF was measured in sodium and FS grease with and without additives. Fig. 3 depicts the COF of the grease, as well as the fluctuation. As soon as the test began, both greases produced a peak and fluctuation. In sodium grease, the variation lasted for 18 min before stabilizing. Such observation differs from that of FS grease in a way that the fluctuation lasted only for 6 min before stabilizing in the latter. Until the end of the test, the fluctuation remained stable.

The average COF of the sodium greases without additives was 0.055, which was lower than the average COF of sodium grease with PTFE and graphite, with a value of 0.061 and 0.064, respectively. The results corresponded with the FTIR analysis of the grease, which showed that the addition of additives to the sodium grease resulted in a peak shift at < 600 cm^{-1}. Based on this information, it can be concluded that the additions did not affect the anti-wear properties of the grease. Instead, the changes could be due to a chemical reaction between grease ingredients, causing the additives to lose their capacity to lower COF.

On the other hand, the addition of additives to FS grease was able to lower the COF of the grease. With an average COF of 0.11, FS grease without additive recorded the highest COF. With an average of 0.087, FS grease with PTFE recorded the second-highest COF, followed by FS grease with graphite with an average of 0.08. The results demonstrated that the grease can perform its role by boosting the grease's anti-wear effect. Fig. 4 shows the average COF for various greases.

Overall, the lowest COF was recorded for sodium grease without additives (Fig. 4). When comparing sodium to FS greases, low COFs were recorded for all sodium greases, with or without additives. The findings demonstrated that sodium grease provides greater lubrication than FS grease as the former has a lower COF than the FS grease. In addition, the lower COFs recorded for the sodium grease without additives than that with additives indicated that there is no need to incorporate additives into the grease. The results demonstrated that the lubrication of sodium grease without additives is superior to that of sodium grease with additives.

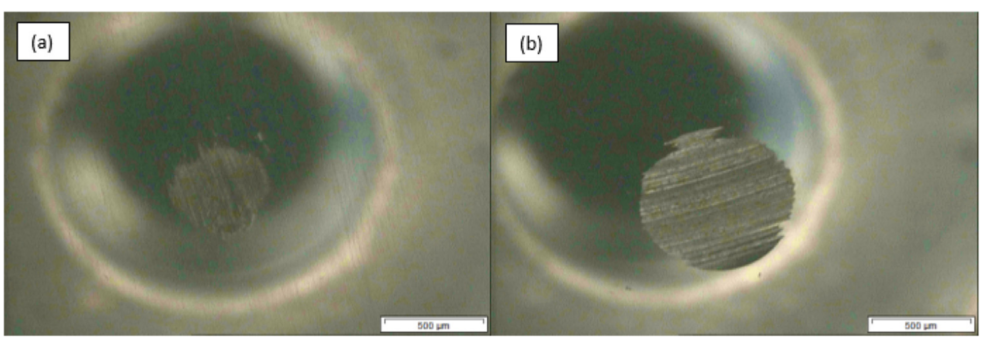

Fig. 5. WSD of (a) sodium (b) FS grease without additive.

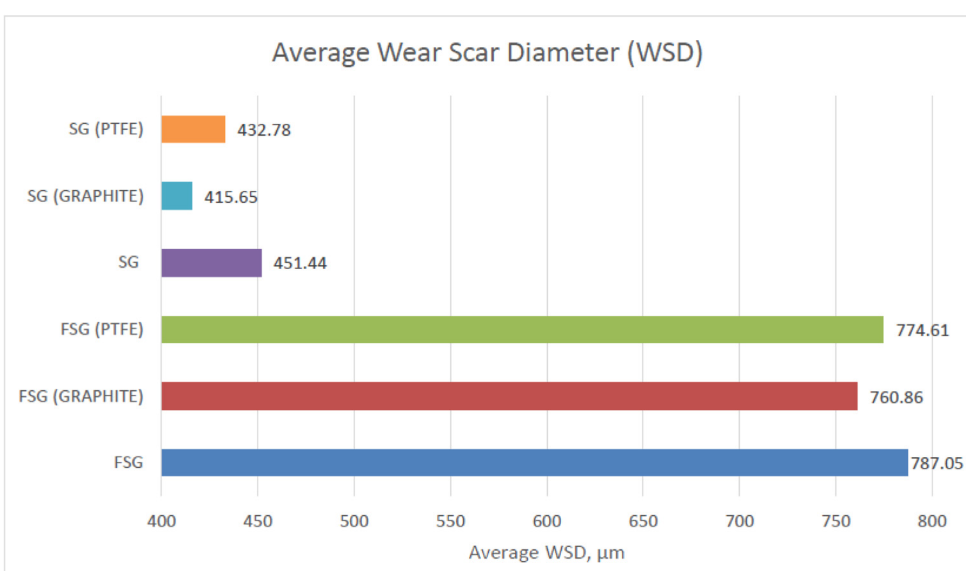

Fig. 6. Average WDS of sodium and FS grease.

3.4.2. Wear scar diameter (WSD)

To lower the WSD on the equipment using lubricating grease, ensuring the stability of the lubricating film under pressure is critical. Lubricating grease is thought to reduce WSD by reducing friction between the surfaces of the equipment [20]. Fig. 5 depicts the WSD of sodium and FS greases in the absence of additives. When compared to FS grease, the sodium grease resulted in a low WSD.

The average WSD for sodium grease was 451.44 μm, while the WSD for sodium greases added with PTFE and graphite was 432.78 μm and 415.65 μm, respectively. In contrast to the COF value, the lowest WSD was recorded for the sodium grease added with graphite while the highest was recorded for sodium grease without any additives. The presence of saturated nanoparticles on the surface of the grease caused this condition. An excess of nanoparticles will create a big cluster that eventually destroys the surfaces of the equipment. Three-body abrasion is the term given to this phenomenon [21]. This number, however, posed no threat to the quality of the grease because it is still lower than that of the commercial sodium grease.

In the case of FS grease, the average WSD of FS grease without additive was 787.05 m, while FS grease with PTFE and graphite has a WSD of 774.61 m and 760.86 m. These results are comparable to sodium grease, with the lowest WSD obtained from FS grease with graphite and the highest WSD without additives. Based on the overall WSD data, graphite was found capable of lowering the WSD of lubricating grease. Due to the presence of nanoparticles in the grease, the grease without additives has a high WSD rating. Fig. 6 shows the average WSD for every grease.

4. Conclusion

The grease made from WEO demonstrated good properties with or without the addition of additives. Based on the results, the FTIR evaluation of the grease revealed that the addition of additives did not change the spectral profile of FS grease while a slight change in the fingerprint region of the spectrum was observed with the use of sodium grease. On the other hand, the addition of additives did not improve the anti-wear ability of the grease as the grease without additives showed a low value of COF when compared to grease with additives. However, the addition of additives could reduce the wear scar diameter of sodium and FS grease. Although the additives were not able to reduce the COF of the greases, the obtained COFs are still considered low as compared with the grease available in the industry. Further research is recommended to learn more about the quality of the grease.

Declaration of Competing Interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: The authors wished to acknowledge the financial support from the Ministry of Higher education, Malaysia, through an internal grant (RDU190398), Universiti Malaysia Pahang.

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