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Investigation of Tribological Properties of Graphene Nanoplatelets in Synthetic Oil

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ABSTRACT

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Lubricants are commonly utilized in industry to minimize friction and wear for tools and components, and additives play indispensable roles in lubricants to attain overall enhanced tribological properties. Because of environmental concerns, the introduction of nanoparticles is regarded as a promising lubricant additive capable of replacing conventional additives and improving lubricant tribological properties. This study investigated the tribological behaviour of 5W30 PAO+ester fully synthetic oil (SO) with and without the addition of graphene nanoplatelets. Besides, this study also focuses on the tribological effect of graphene's concentration (0.01, 0.02, 0.05, 0.1 and 0.2 wt%). The experiments were conducted using a four-ball tester according to ASTM D4172 and surface analysis was done on the worn surfaces using scanning electron microscopy and energy-dispersive x-ray spectroscopy. The results show that the presence of graphene significantly improves the tribological properties. SO enriched 0.05 wt% graphene exhibits the lowest coefficient of friction and wear scar diameter, and the friction and wear were reduced by 33.78% and 34.42%, respectively. The protective film formed on the worn surface is responsible for friction and wear reduction. In addition, the worn surface becomes smoother after being lubricated by nanolubricants, which can be observed through the SEM analysis. EDX analysis revealed the presence of element carbon on the worn surface, implying that nanoparticles had deposited on the worn surface.

1. Introduction

Lubricants are widely used in many sectors such as automotive, transportation, machinery industries and others and remain absolutely necessary. The main function of lubricants is to provide lubrication to reduce friction and prevent wear from the interaction of two contacting surfaces in relative motion. Besides, lubricants also provide corrosion protection on the surface, dissipate heat, act as fluid seal and function as an insulator in transformer applications [1]. Ali *et al.*, [2] reported frictional losses between engine parts to cause total power reduced by 17-19%. The combination effect of friction and wear caused 30% of total energy losses [3]. In this regard, the improvement of

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the lubricant's tribological properties is essential to provide effective lubrication, thus reducing friction and wear. Moreover, additives are important for base oil, especially additives that improve the oil properties. Anti-wear (AW) and extreme pressure (EP) additives are essential in several frictional conditions [4]. However, the conventional AW and EP additives containing harmful compounds such as sulfur, chlorine, and phosphorus cause a negative effect on the environment [5]. Therefore, nanoparticles are considered a promising new class of lubricant additives currently being investigated by researchers. Most nanoparticles are eco-friendly; they can function as AW and EP additives and friction modifiers without an induction period [6, 7].

To date, many studies have been carried out on the application of nanoparticles in the tribology field. The use of nanoparticles as lubricant additives is known as nanolubricants. The addition of nanoparticles has been reported to enhance the tribological performance of lubricants [8-11]. The types of nanoparticles include metal based-nanoparticles, rare earth compounds, and carbon-based nanoparticles. However, friction and wear reduction are based on the nanoparticles' size, shape, and concentration, especially the latter [12]. Currently, the proposed lubrication mechanism of nanoparticles involves enhancing the tribological performance, including rolling effect [13], protective film formation [14, 15], mending effect [16] and polishing effect [12]. Besides, these lubrication mechanisms were classified into two groups. The first group of lubrication mechanisms is the direct action of nanoparticles in lubrication enhancement (rolling effect and protective film formation), and the second group is surface enhancement by nanoparticles (mending effect and polishing effect) [17].

Many researchers are focused on investigating nanoparticles as additives in mineral and synthetic oil-based lubricants. Still, there are few researchers evaluating bio-lubricants. In the present study, bi-synthetic (PAO+ester) oil was selected as base oil because conventional synthetic oil is mostly made of polyalphaolefins (PAO), which category as group IV base oil and the ester oil is under group V base oil [18]. Therefore, the combination of these oils is still undefined. Furthermore, graphene has been chosen as a lubricant additive among all nanoparticles in this study because of its excellent chemical, physical and mechanical properties such as ultrathin structure, high chemical inertness, extreme mechanical strength, good shear capability, and high thermal conductivity [19, 20]. Besides, previous studies for graphene as the lubricants additive are listed in Table 1.

Table 1
 Literature review of graphene as lubricant additive

No.	Types of graphene	Base oil	Concentration	Friction reduction	Wear reduction	Ref.
1	Graphene platelets	350 SN base oil	0.015 to 0.105 wt%	Maximum reduction at 0.075 wt%		[21]
2	Graphene oxide	500 SN base oil	0.02 to 0.08 wt%	~17%	~36%	[22]
3	Graphene oxide	Paraffin oil	0.2 wt%	75%	92.5%	[23]
4	Graphene	SAE 10W-30 oil	0.025 to 0.1 wt%	Maximum reduction at 0.05 wt%		[24]
5	Graphene	Polyalphaolefin-9 (PAO9)	0.01 to 5 wt%	17%	14%	[25]
6	Graphene	Jjoba oil blended with SAE20W40 oil	0.05, 0.075 and 0.1 wt%	Maximum reduction at 0.075 wt%		[26]
7	Graphene nanoplatelets	Palm oil TMP ester blended with polyalphaolefin	0.01 to 3 wt%	5%	15%	[27]

Hence, this study investigates the tribological properties of PAO+ester fully synthetic oil with and without the addition of graphene nanoparticles by using a four-ball tester and focused on the

tribological effect of the graphene's concentration in this lubricant. In addition, the worn surface analysis on the tested sample will be conducted and analysed by scanning electron microscopy (SEM) and energy-dispersive x-ray spectroscopy (EDX).

2. Methodology

2.1 Formulation of Nanolubricants

5W30 PAO+ester fully synthetic engine oil (SO) was used as the base oil to formulate nanolubricants. The appearance and the physicochemical properties of base oil as shown in Figure 1 and Table 2. Graphene nanoplatelets were purchased from Sigma-Aldrich (M) Sdn Bhd, and the properties of graphene nanoplatelets as shown in Table 3.



Fig. 1. The appearance of 5W30 PAO+ester fully synthetic oil

Table 2

The physicochemical properties of SO

Properties	Method	Units	Value
Density @ 15°C	ASTM D4052	g/ml	0.853
Kinematic Viscosity @ 40°C	ASTM D445	mm ² /s	65.05
Kinematic Viscosity @ 100°C	ASTM D445	mm ² /s	11.63
Viscosity Index	ASTM 2270	None	173
Pour point	ASTM D97	°C	-45
Flash point	ASTM D93	°C	232

Table 3

The properties of graphene nanoplatelets

Properties	Description
Appearance (colour)	Black
Form	Powder
Relative density	2.0-2.25 g/cm ³
Thickness	Few nm
Particle size	<2 μm
Surface area	500 m ² /g
Bulk density	0.2-0.4 g/cm ³

Figure 2 shows the morphology and the element composition of graphene nanoplatelets used in this study. The particle size ($<2\ \mu\text{m}$) and structure are shown by SEM micrograph (Figure 2a), and particle size is matched to the details provided by suppliers. The thickness is about 1-10nm (Figure 2b) as determined by high-resolution transmission electron microscopy (HRTEM). Besides, carbon is the main element of graphene nanoplatelets was confirmed through EDX analysis, as illustrated in Figure 2c.

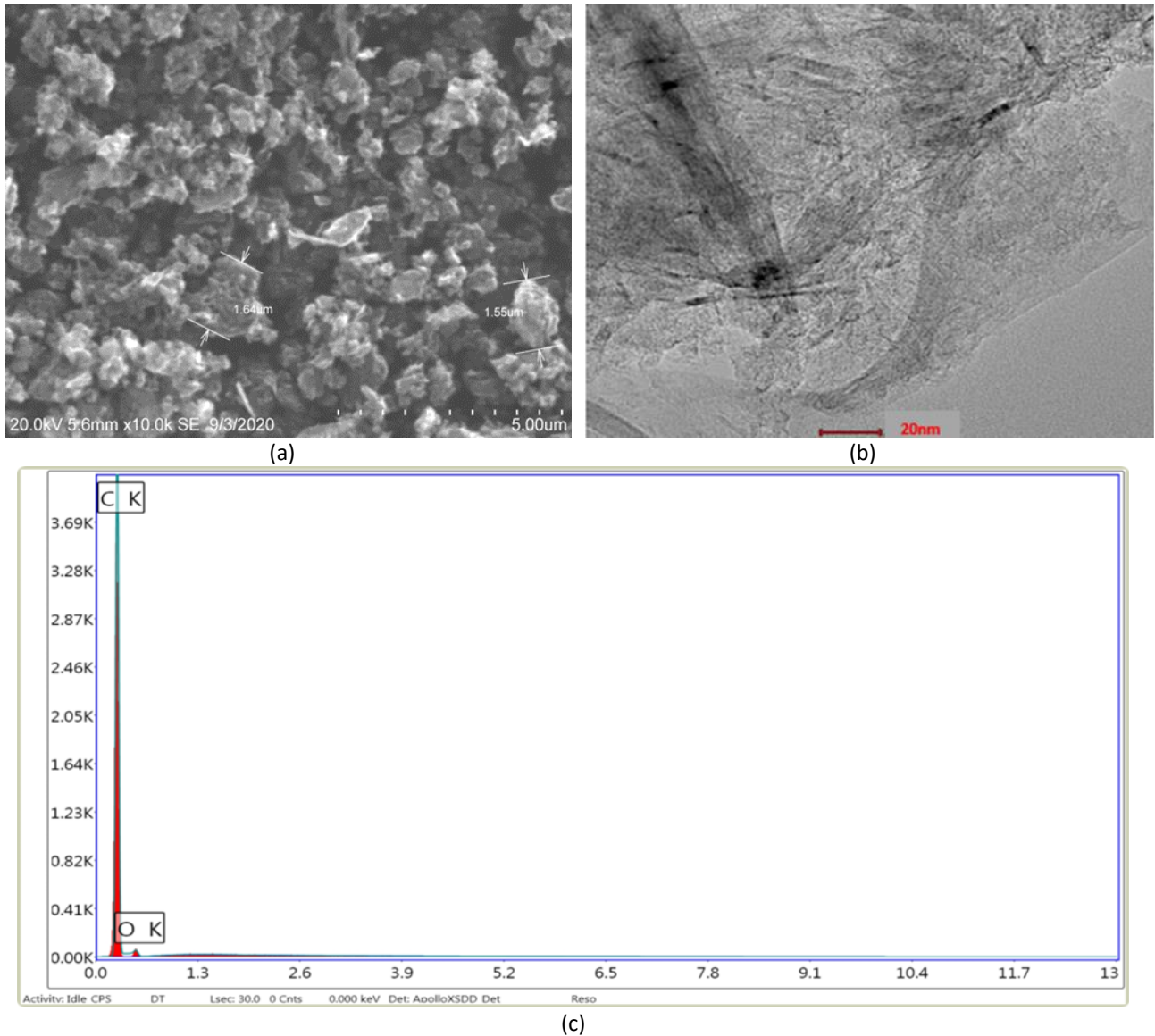


Fig. 2. (a) SEM and (b) HRTEM micrograph of graphene nanoplatelets and (c) EDX analysis of graphene nanoplatelets

The nanolubricants are synthesised based on a weight basis. Therefore, the weight of base oil will be measured to calculate the amount of nanoparticles required. In the present study, graphene nanoplatelets with the concentration of 0.01 wt%, 0.02 wt%, 0.05 wt%, 0.1 wt% and 0.2 wt% will be selected to disperse in SO. The nanoparticles were dispersed in SO by magnetic stirring for 30min (speed: 350-500 rpm; temperature: 60°C) to obtain the homogenous solution. This method was used in previous studies [28, 29]. Besides, the base oil without the addition of nanoparticles will act as the baseline in this study.

2.2 Tribological Test

The investigation of the test lubricant's tribological properties (SO and graphene/SO nanolubricants) was conducted using a four-ball tester (model DUCOM TR30L-LAS) under ASTM D4172 standards and the schematic diagram of the four-ball tester as shown in Figure 3. The test conditions are summarised in Table 4. The balls used were AISI 52100 alloy steel with G25 steel balls, 12.7mm diameter and 64-66 HRC hardness. The steel ball used for the test and ball pot were thoroughly cleaned with acetone before and after each test. Next, three steel balls (bottom balls) were placed and fixed in a steel pot, and a steel ball (rotating ball) was fixed at the chuck. The final step will be to pour approximately 10ml of test lubricant into the steel pot, covering the steel ball to a depth of at least 3mm. Each set of experiment tests will be conducted three times to get an accurate result. After the experiment test, the value of the coefficient of friction (COF) was calculated based on the friction torque (raw data). The wear scar diameter (WSD) of all tested balls (bottom balls) was measured by the image acquisition system (part of the equipment of the four-ball tester). Besides, the details of the apparatus utilized in this study, including accuracy, are listed in Table 5.

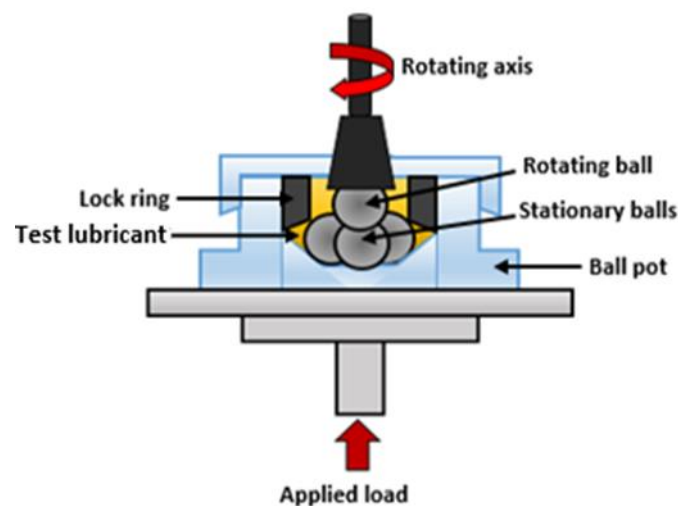


Fig. 3. Schematic diagram of four-ball tester

Table 4

The test conditions of tribological test

Condition	Value
Load (kg)	40 ± 0.3
Speed (rpm)	1200 ± 2
Test temperature ($^{\circ}\text{C}$)	75 ± 2
Test duration (min)	60
Concentration of graphene (wt%)	0.01, 0.02, 0.05, 0.1 and 0.2

Table 5
 Specification of apparatus used in this study [30]

Apparatus	Specification/Properties	Detail	Accuracy
Four-ball tester	Model	DUCOM TR30L-LAS	-
	Speed (rpm)	300-3000	1
	Oil temperature (°C)	Ambient temperature to 100	0.5
	Maximum axial load (N)	10,000	0.5
Optical microscope	Range of scar	100-4000	0.5
	Model	IKA, Oxford C2000	-
Scanning electron microscope (SEM)	Wear scar diameter	-	± 0.01 mm
	Model	Hitachi S3400N	3.0 nm at 30 kV

2.3 Worn Surface Analysis

The worn surface analysis on the tested steel balls was conducted using scanning electron microscopy (SEM) and energy-dispersive x-ray spectroscopy (EDX) to study the tribological effects of graphene nanoplatelets on the friction surface. Besides, the possible lubrication mechanism by nanoparticles may be discovered through this analysis.

3. Results

3.1 Evaluation of Tribological Properties

A series of experimental tests were conducted to evaluate the friction and wear behaviour of graphene nanolubricants using a four-ball tester. In the present study, the obtained coefficient of friction (COF) and wear scar diameter (WSD) result will be under the mixed elastohydrodynamic lubrication regime, in which the COF values are between 0.01 to 0.1 [31]. Figure 4 shows the COF and WSD results from different concentrations of graphene as an additive in 5W30 PAO+ester fully synthetic engine oil (SO). The value-added at the back of the sample represents the addition of concentration in the base oil. An average COF value is as low as 0.049, and the lowest WSD is 492µm, shown by the SO containing 0.05 wt% graphene (SO+0.05), while the highest COF (0.074) and WSD (750.2 µm) exhibited by base oil (SO), as illustrated in Figure 4. Friction behaviour was improved by the addition of graphene from 0.01 wt% to 0.2 wt%, even though COF slightly increased after 0.05 wt%. Several previous studies have found an increase in COF when the concentration continues to increase after the optimum concentration [22, 32]. The friction behaviour of the nanolubricants in this study has a similar trend to those reports. A similar situation also occurs with wear resistance. The wear resistance has been improved by adding the concentration of graphene from 0.01 wt% to 0.2 wt%. However, after the addition of 0.05 wt% concentration of graphene, the wear increased. Thus, the dividing line has occurred between 0.05 wt% and 0.1 wt% in terms of wear and friction. An improvement of 33.78% friction reduction and 34.42% wear protection has been observed for SO+0.05 compared to SO. The friction and wear reduction can be attributed to the lubrication mechanism of nanoparticles, such as forming a protective film on the sliding surface, which avoids surface directly rubbing. Nonetheless, the possible lubrication mechanism may be discussed on the basis of surface analysis.

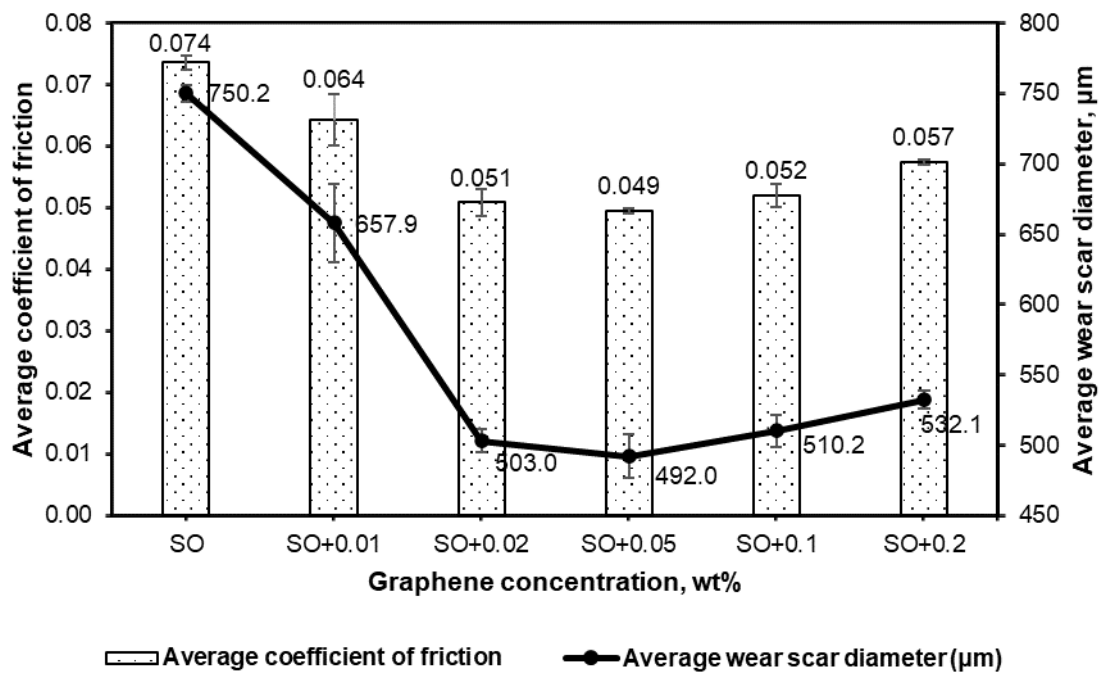
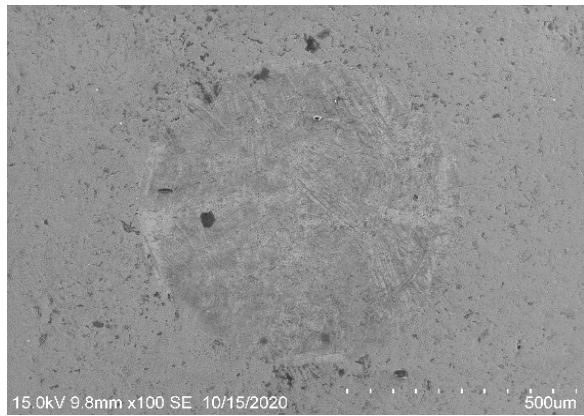


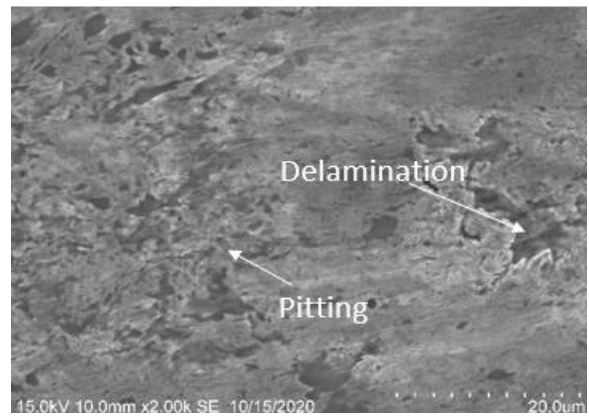
Fig. 4. Friction and wear characteristics of graphene nanolubricants at different concentrations

3.2 SEM and EDX Analyses

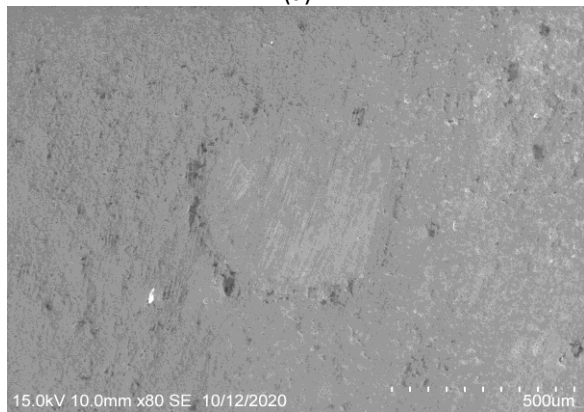
The worn surface of the tested sample will be analysed by SEM and EDX. Figure 5 shows the SEM micrographs of the worn surface on the tested steel ball. Figure 5 (a-b) shows the worn surface of steel ball lubricated by base oil (SO) with 100x and 2000x magnification. The micrograph clearly shows the wear and pits on the surface produced by SO, which should be the adhesive-like wear. After being lubricated with SO+0.01, the pitting surfaces were reduced, indicating that the addition of graphene functions in surface enhancement. The worn surface lubricated by SO+0.02, as shown in Figure 5 (e-f). The worn surface was enhanced compared to the worn surface of SO and SO+0.01. However, a few grooves and pits still occur on the surface. The surface has been further smoothed by the addition of 0.05 wt% graphene, as shown in Figure 5 (g-h). The pits were decreased compared to the previous image, which induces minimum COF and WSD. Figure 5 (i-j) presents the worn surface after lubrication of SO+0.1. Several abrasions were found on the worn surface, and this can be related to the further increment of graphene concentration which causes surface degradation. Further surface deterioration has been observed on the worn surface lubricated by SO+0.2 with the occurrence of cracks, pits, delamination. As a result, it can be concluded that after the addition of 0.05 wt% graphene, further concentration increases may have a negative effect. The smooth surface observed in Figure 5 (g-h), can be attributed to the addition of nanoparticles and their lubrication mechanism: protective film formation and polishing effect. Wang *et al.*, [24] reported the deposition of graphene on the surfaces (mending effect) and reduced surface roughness (polishing effect) which results in friction and wear reduction. The tribofilm formation stabilised by graphene reduces direct surface contact and further minimises the wear reported by Rajan *et al.*, [15]. In this study, two potential lubrication mechanisms (protective film formation and polishing effect) were proposed for this nanolubricant.



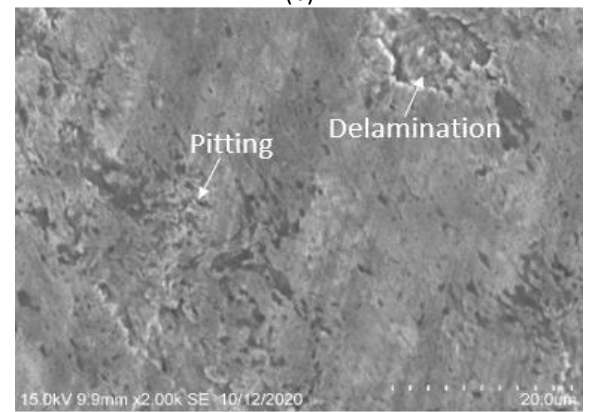
(a)



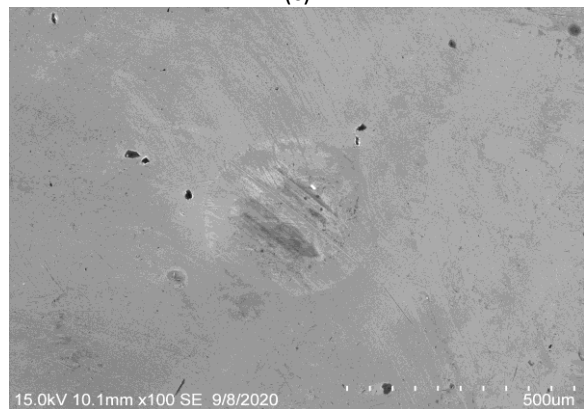
(b)



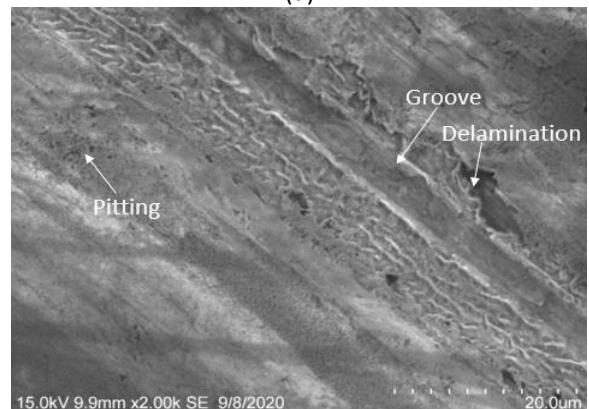
(c)



(d)



(e)



(f)



(g)



(h)

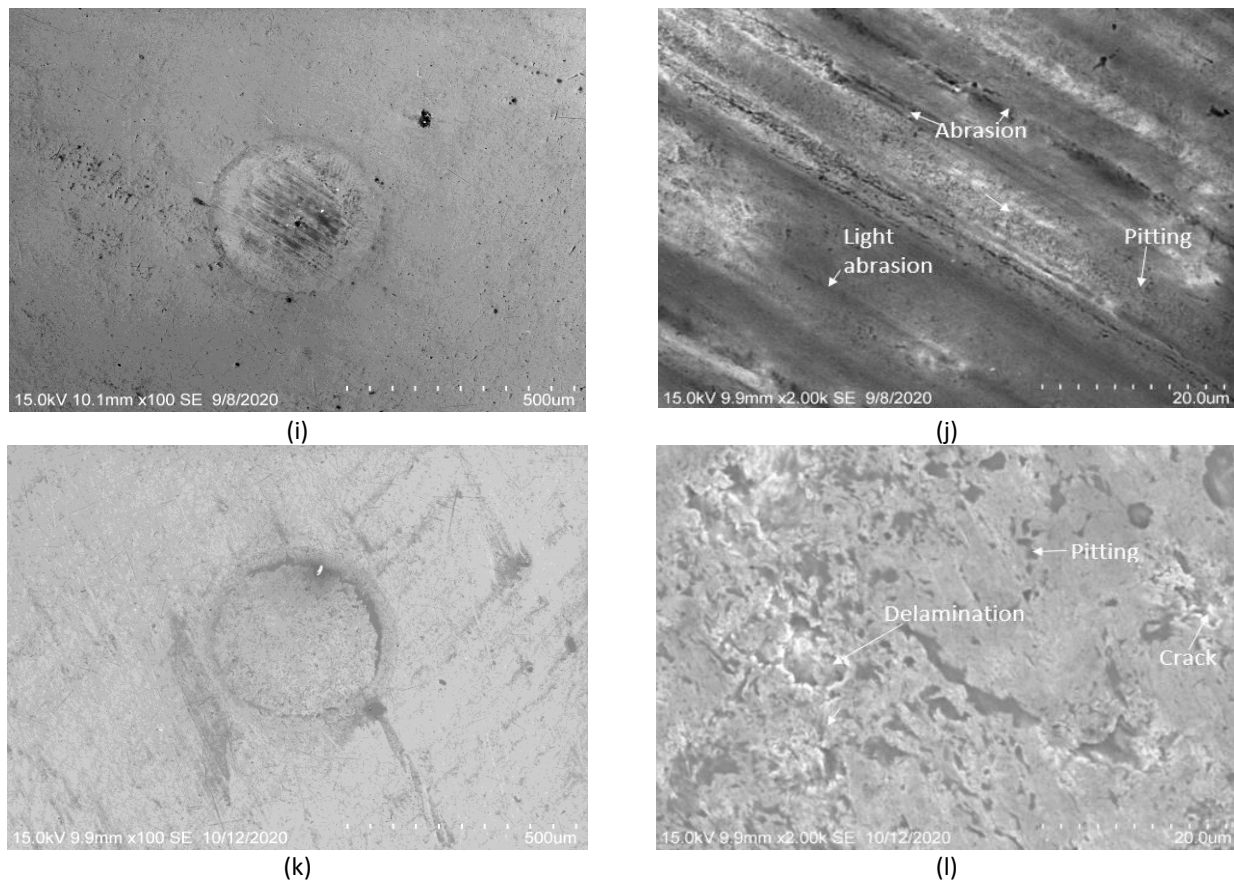


Fig. 5. SEM micrograph of the worn surface on tested steel ball lubricated with: (a) SO (x100), (b) SO (x2000), (c) SO+0.01 (x100), (d) SO+0.01 (x2000), (e) SO+0.02 (x100), (f) SO+0.02 (x2000), (g) SO+0.05 (x100), (h) SO+0.05 (x2000), (i) SO+0.1 (x100), (j) SO+0.1 (x2000), (k) SO+0.2 (x100), (l) SO+0.2 (x2000)

Figure 6 (a)-(f) illustrates the EDX analysis of the wear scars on the tested steel ball, and this analysis was done according to the SEM micrograph (x2000) shown above. A similar element composition was found on all wear surfaces, which were iron (Fe), carbon (C), and oxygen (O). Element iron is the main element composition of AISI 52100 alloy steel balls that contribute to Fe that is shown in this analysis. Element oxygen can be related to the oxide layer forming on the surface during the lubrication process, and this element is the common element. There was a C detection on all surfaces even though with base oil. C is the primary element of graphene, but C was detected on the surface lubricated by SO. This could be the base oil containing undefined additives and surface adhesion due to the oil composition [4]. In contrast, the presence of the C element on the surface lubricated by nanolubricants, can be related to the graphene deposited on the worn surface with the detection of carbon content ranging from 4 wt% to 8 wt% through EDX analysis. Gulzar *et al.*, [33] verified nanoparticles tribo-sintering on the worn surface to provide wear protection through EDX analysis. Jason *et al.*, [34] reported the deposition of nanoparticles on the friction surface with the presence of related elements through EDX. Jamaluddin *et al.*, [35] found similar findings, who reported the deep parallel grooves formed on the surface because of the high content of carbon elements in graphene. Besides, Wu *et al.*, [36] investigated tribological characteristics of graphene as additives and reported the tribo-film is mainly composed of C element through EDX analysis.

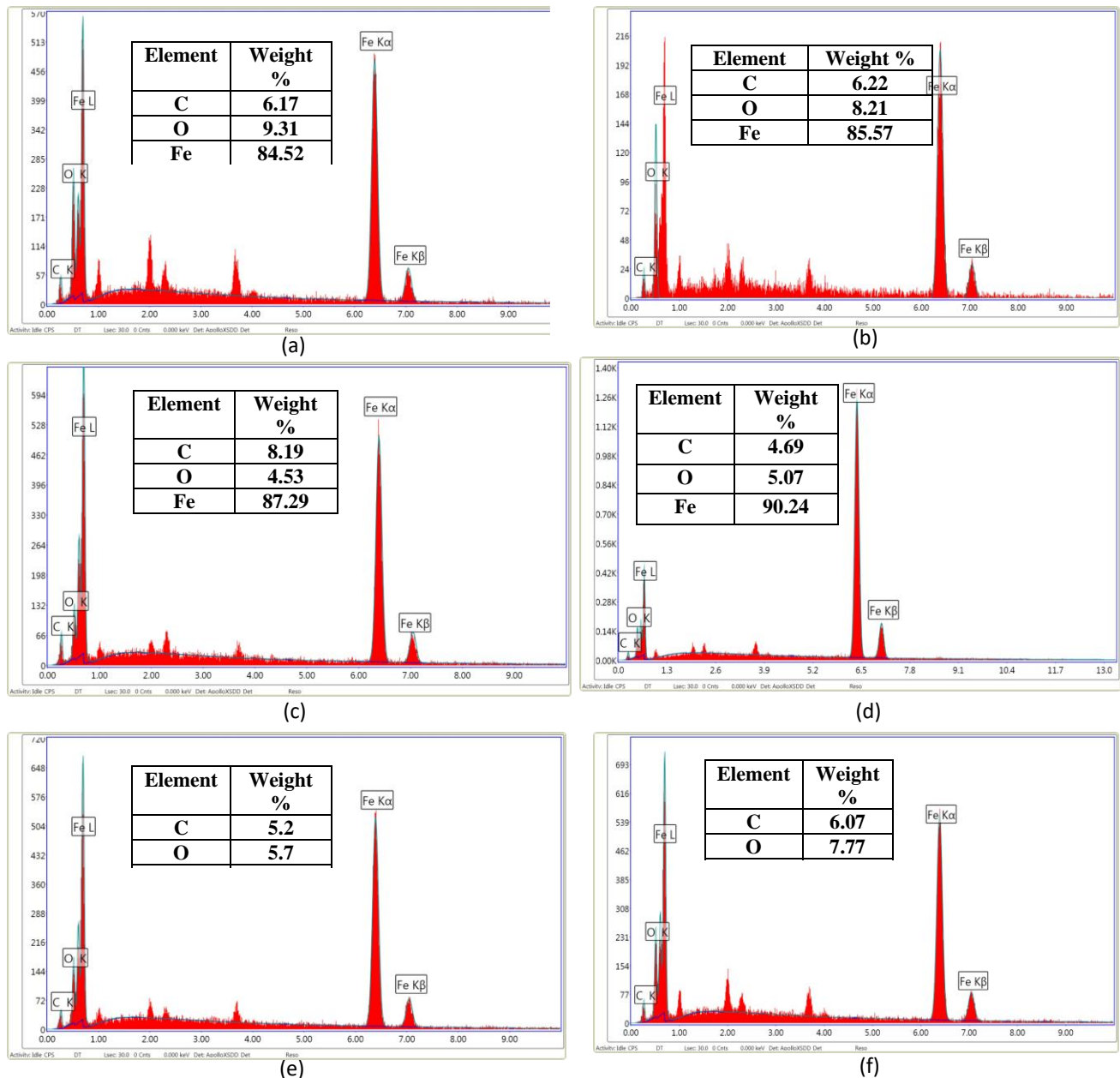


Fig. 6. EDX analysis of the worn surface on tested steel ball: (a) SO, (b) SO+0.01, (c) SO+0.02, (d) SO+0.05, (e) SO+0.1, (f) SO+0.2

4. Conclusions

In conclusion, the addition of 0.05 wt% graphene into 5W30 PAO+ester synthetic oil exhibits the lowest friction and wear. Besides, SO+0.05 shows the improvement of 33.78% friction reduction and 34.42% wear protection compared to SO. The main attribution of friction and wear reduction is related to graphene forming a protective film to reduce surface rubbing. Besides, the addition of graphene smoothed the worn surface; However, at high concentration, it has negative effects, which were evidenced by the SEM analysis. Moreover, the deposition of graphene to the friction surface has been observed through EDX analysis.

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