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Article

Effect of Surfactants on the Tribological Behavior of Organic Carbon Nanotubes Particles Additive under Boundary Lubrication Conditions

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Abstract

The tribological enhancement of nano-lubricant that consist of formulated Eichhornia Crassipes carbon nanotubes (EC-CNTs) dispersed in base rapeseed oil using sodium dodecyl sulfate (SDS) and oleic acid (OA) as surfactants was conducted in this study. The experiment was done using high frequency reciprocating rig, applying 0.5 mass%, 1 mass% and 1.5 mass% EC-CNTs alongside 2% and 5% of the various surfactants and sonicated for 25 minutes and 50 minutes respectively. The use of surfactants and sonication was to reduce agglomeration. The outcome showed that SDS yielded better results than OA, further revealed that the use of 2% SDS under sonication time of 50 minutes reduced the solution agglomeration compared to 5% SDS and sonication time of 25 minutes. The tribological test conducted on EC-CNTs concentration effects was based on friction and wear reduction, load carrying ability, lubricant film stability, wear scar micrograph and mechanism of particles. The results indicated that inclusion of nanoparticles substantially enhanced the tribological characteristics. However, pronounced enhancement was recorded with 1 mass% EC-CNT compared to counterpart lubricants. Friction and wear reduction by 1 mass% EC-CNT were 65.4% and 63.6% respectively against base oil. The nanoparticles exhibited excellent mechanism for which the tribological enhancement was achieved.

Keywords

EC-CNTs nanoparticles, surfactants, rapeseed oil, lubrication mechanism, friction and wear

1 Introduction

Environmental concerns over inorganic additives in lubrications have prompted researchers to look into using renewable and degradable additive materials in lubricants formulation. Researchers have recently focused their attention on Eichhornia Crassipes because of the large amount of cellulosic substrates that are being investigated for many engineering formulated products [1–3]. It has been reported that biomaterials with a high cellulose content are suitable for the development of carbon nanotubes due to the carbonaceous abundance [4, 5]. More so, because of their excellent mechanical characteristics, such as high elastic modulus, tensile strength, and strong thermal stability, carbon nanotubes (CNTs) have attracted a lot of interest for industrial uses [6]. An active lubricating system contribute an essential service in mechanical operation, since about 80% of mechanical failure are from friction and wear [7, 8]. Basically, lubrication is centred on two principles like; development of fluid pressure to avoid direct contact through separation and formulation of sacrificial surface tribo-films to prevent the surfaces against abrasion and shear [9, 10]. Also, fluids due serve in creating hydrodynamic pressure and hydrostatic pressure as to support working load. However, during high operating condition, boundary tribo-film generated from lubricant additives operate as protective layer or film to avoid certain asperity contacts [10]. To improve lubricants properties towards minimising wears and friction, researchers have reported that blending base lubricants with suitable nanoparticles can enhance tribological characteristics of the lubricant such as ZnO and WS_2, [11, 12], ZnO [13], titanium dioxide [14], CNT [6, 15], CuO and graphite nanoparticles [16], Al_2O_3 and TiO_2 [17]. However, some factors like size, concentration, material of the nanoparticle and shapes, due influence the expected behavior of the lubricant performance

[12, 18–21]. Wang et al., [22] conducted study of the effect of different size of nano serpentine powders (NSPs) in paraffin oil towards friction and wear reduction. The results indicated that 0.5 mass% yielded the optimal performance with coefficient and wear reduction of 22.8% and 34.2% respectively. Literature have it that smaller size of nanoparticles preferably from 25-250 nm in diameter provides more effective friction and wear reduction [23–25]. In the analysis of nanoparticles, emphasis had made that spherical shaped nanoparticles shows excellent behavior through rolling in the contact area thereby contribute in friction and wear reduction [26, 27].

On the other hand, additives concentration seriously affects the lubricant performance, suggesting that within 1 mass% additive concentration gives the best result in application [18, 28]. Investigations indicated that nanoparticles agglomeration occurs if the concentration in base oil is not proportional thus poorly dispersed in the oil leading to poor lubrication [27, 29–31]. To avert this unwanted situation, surfactants are applied in solving the problems [11, 32]. Many studies have reported about lubricant particles stability enhancement through application of surfactant and sonication technique in order to eliminate occurrence of particles agglomeration [11, 33, 34]. With this method, lubricant lubrication performance will be improved leading to friction and wear reduction [11, 32], during internal combustion engine operation. The reduction in agglomeration will aid in the prevention of breakages in oil lines and machine orifices. Adequate surfactant decreases the attractive pull among the nanoparticles thereby minimises degree of agglomeration through reduction in surface energy [35]. In this present study, organic anti-wear Eichhornia Crassipes carbon nanotubes (EC-CNTs) additive of three different concentrations were used. The study centred on the application of different dispersion approach via sonication time and surfactants to determine their dispersion stability effect as well as the lubricants tribological behavior under boundary conditions.

2 Materials and method

2.1 Samples preparation

The EC-CNTs was prepared in Universiti Teknologi Malaysia, in the department of mechanical engineering, option of tribology [36]. The formulation adopted approach of cyclic heating after been reduced into nanoscale using ball milling machine [36, 37]. The laboratory analysis was conducted on the developed anti-wear additive [36, 38]. The base oil applied in the analysis was vegetable rapeseed oil, produced under manufacturer specification of 38.2 cSt at 40°C, density of 0.73 at 15°C. The two surfactants used were sodium dodecyl sulfate (SDS) and oleic acid (OA). Both the base oil and surfactants were purchased from Sigma-Aldrich company. The chosen surfactants SDS [34] and OA [11, 39] have been used in lubricant enhancement, while the choosing vegetable oil was because of the recommendation on good relationship between bio-material (bio-rapeseed oil and bio-EC-CNT). In the area of their source, they are organic products with long hydrocarbon chain belonging to polar group. However, they possessed long chains are nonpolar and plays the function of dispersability and solubility of the nanoparticles in the lubricant, hence absorbed by generated surface energy by the nanoparticles. The properties of the EC-CNT nanoparticles, according to the conducted analysis are stated in Table 1, while the SEM image is shown in Fig. 1, with low resolution indicating agglomeration in size distribution and Table 2 listed the elements in EC-CNT.

This study uses EC-CNT concentration of 0.5 mass%, 1 mass% and 1.5 mass% according to the previous literatures [18, 28, 39], with base oil of 100 ml and surfactants value of 5 and 2 mass% against every EC-CNTs concentration used. Furthermore, sonication technique for lubricant stability was introduced for 25 minutes and further tested using the best candidate for another 50 minutes for all the EC-CNTs concentrations together the 100 ml base oil before the main tribological analysis. Figure 2 shows the processes for the nanofluids preparation with the nano surface modification using surfactant and sonication method.

2.2 Method

Since surfactants were blended as to enhance the stability of the lubricants. Analysis was conducted as to ascertain the particle size distribution and dispersed in the two surfactants using dynamic light scattering (DLS). During the operation,

Table 1 Lubricants properties (Rap. oil without and with different concentration of EC-CNTs)

Properties	**Base rapeseed oil**	**Rap. oil + 0.5 mass% EC-CNT**	**Rap. oil + 1 mass% EC-CNT**	**Rap. oil + 1.5 mass% EC-CNT**
Density at 15°C (g/ml)	0.730	0.873	1.083	1.103
viscosity at 40°C (cSt)	38.79	68.03	72.5	74.29
Viscosity @100°C	8.4	14.5	15.3	15.7
Specific gravity @15°C [ASTM D287]	0.93	0.95	0.97	0.92
Viscosity index [ASTM D2270]	224	172	181	173
Pour point [ASTM D97]	-18	-21	-19.8	-25
Flashpoint [ASTM D92]	320	230	229	237

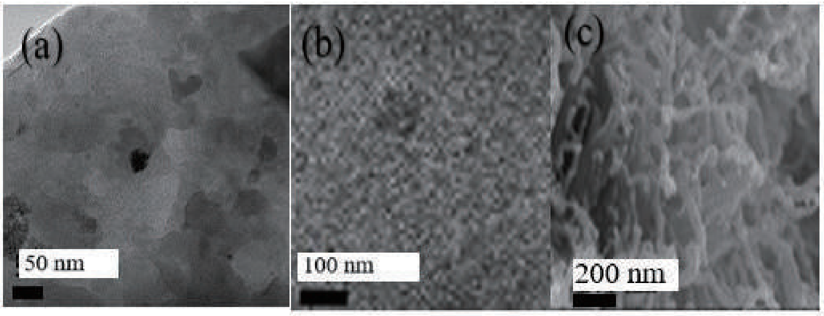

Fig. 1 SEM images of EC-CNT additives (a) low (b) medium and (c) high resolution

Table 2 EDS for elemental composition in EC-CNT Samples

Sample/Element (mass%)	C	O	Si	K	Al	Cl	Co	Ca	Mo	Mg	Cu	Ni
EC-CNT	70.4	15.4	5.3	1.8	1.6	1.5	1.3	1.3	0.5	0.4	0.3	0.2

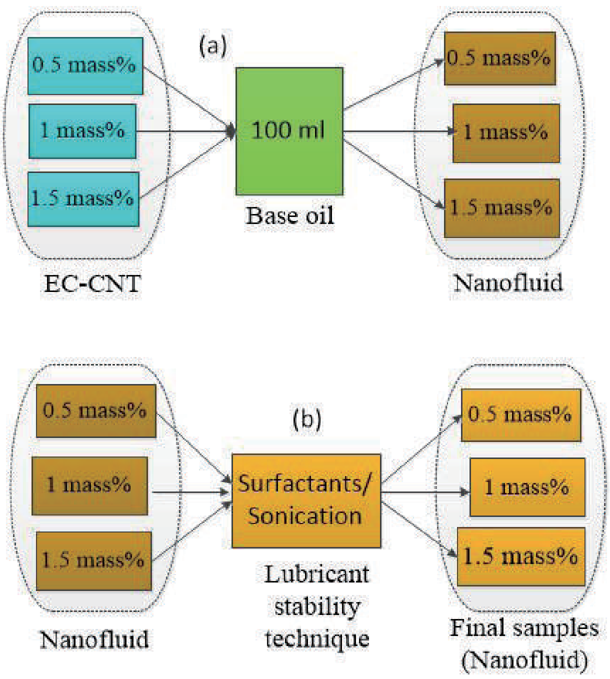

Fig. 2 Schematic diagram of lubricant samples preparations (a) blending EC-CNT with base oil (nanofluid) and (b) sonication of the various Nanofluids

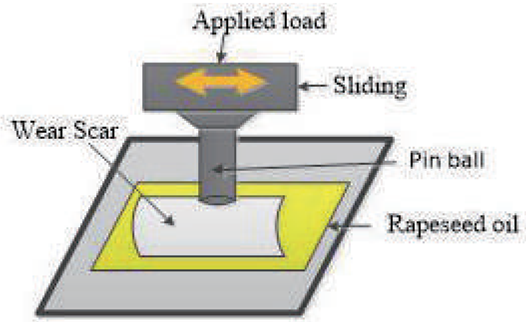

Fig. 3 Skeletal diagram of HFRR sliding section

the DLS data were generated for over some hours for individual sample formulated. The displayed data's stands for hydrodynamic particles diameter distribution in solution. Testing for friction and wear effect from the various formulated lubricants was done using high frequency reciprocating rig (HFRR) otherwise known as ball-on-flat was used as shown in Fig. 3.

The pin ball and flat for the tribo-test (12 mm and 40*40 mm) were made from AISI 52100 steel, with surface roughness (Ra) approximately 0.03 μm and 0.06 μm as calculated by profile-meter Zygo Newview 7100. The experiment was conducted under humid ambient air (28–32 RH) and room temperature (25°C). Applied load of 100 N, frequency of 5 Hz, stroke of 10 mm and temperature of 75°C for time duration of 15 min. Various tests were carried out by holding ball with ball holder, placed directly and attached with the system load reader which record the operation frictional forces as the system sets into operation. During the reciprocating test, the flat top was field with 100 ml lubricant to ascertain the tribological effect of the metal surface. Before each test, various elements for the analysis were ultrasonically washed applying cleaning agent like heptane for 7 min, followed ethanol and hot air for final dry, thus performed before and after each run. After the friction study, wear analysis was conducted on the lubricated surfaces using surface profile-meter 150 stylus, SEM incorporated with EDX and atomic force microscope (AFM). These analyses are to confirm the Ra/wear volume, the elements in film developed during tribo-chemistry on the lubricated surfaces as well as the particle mechanism during lubrication.

3 Results and discussions

In this study, the candidates with the best performance after sonication is used for the subsequent analysis. However, data's generated are further tested on their friction and wear resisting ability. The most enhanced samples were again conducted test; effects of different load on coefficient of friction, effects of temperature on the lubricants, lubricant film stability, Wear scar volume micrographs and mechanism of operation by the nanoparticle.

3.1 Dispersion stability study using surfactants and sonication method

Figure 4 depicts the average particles of various EC-CNTs

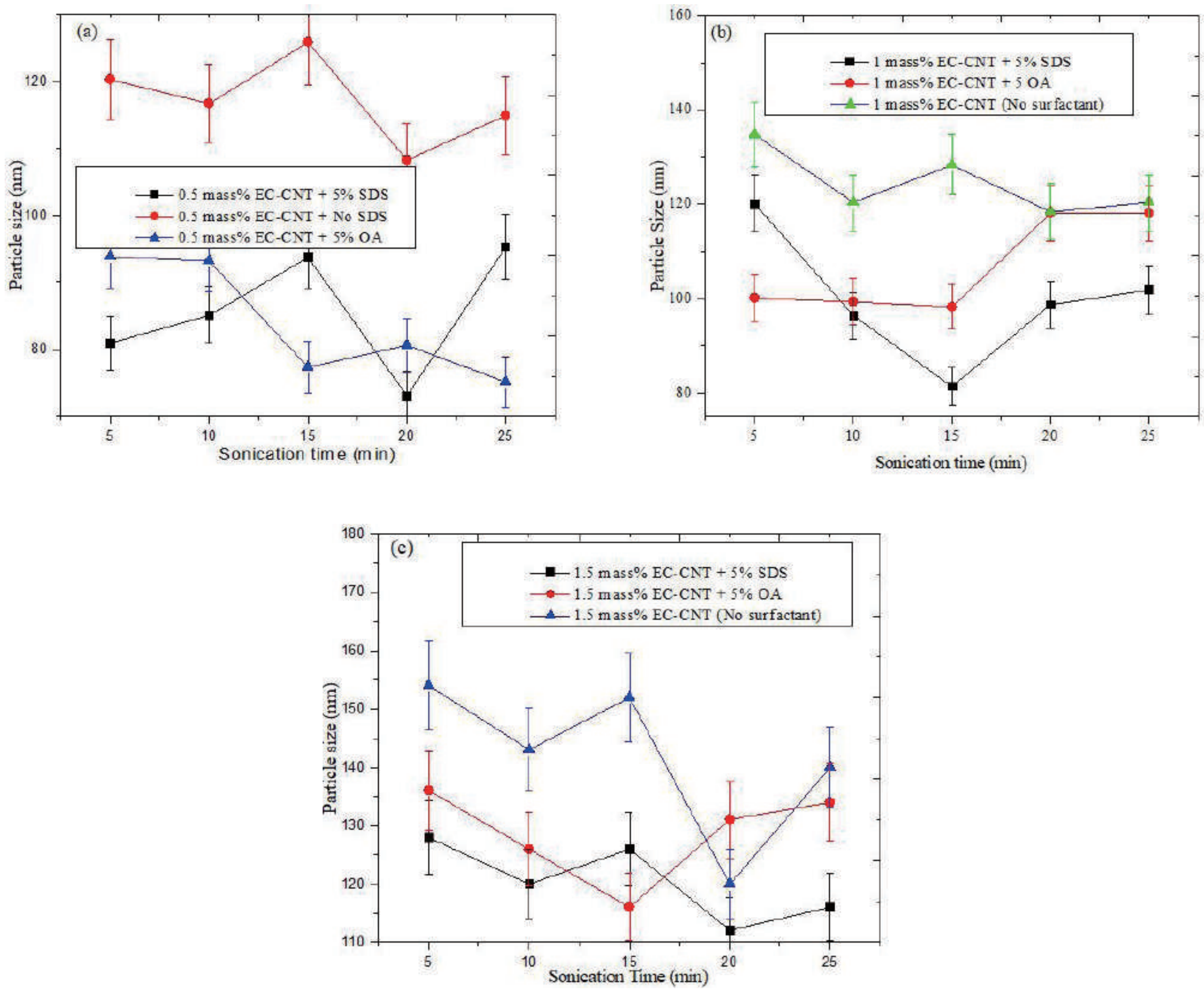

Fig. 4 Average hydrodynamic diameter of different concentration of EC-CNTs (0.5, 1, 1.5 mass%) particles in base rap. Oil with and without surfactants against sonication time (25 minutes), using DLS

(0.5, 1, 1.5 mass%) with and without surfactants (5 mass%) in rapeseed oil against sonication time of 25 min, determined through DLS machine. Surfactants of OA and SDS were used in the analysis. The results from dynamic light scattering shows that average particles of different concentration of EC-CNTs in rapeseed oil both with and without surfactants range from 83 nm to 131 nm for 1 mass%, 79 nm to 128 nm for 0.5 mass% and 153 nm to 101 nm for 1.5 mass%. The variation on the particles detected shows agglomeration occurrence in all the concentrations, however, 1.5 mass% EC-CNT exhibited greatest agglomeration effect. The manifestation of the particle size variation via agglomeration suggests to occur from different physiochemical properties of the EC-CNTs mostly the surface energy possessed [32, 40]. Comparing the samples with and without surfactants, inclusion of surfactants significantly reduces the average particle sizes and the operational effects depends on the relationship between the EC-CNTs and surfactants. In the behavior of SDS with 0.5 mass% and 1 mass% EC-CNTs yielded functional effect, thereby reduces the particles approximately from 117-78 nm with SDS against 110-98 with OA, while 1.5 mass% reduced from 127-101 nm with SDS compared to 139-109 with OA respectively. The observation suggests that operation with SDS reduces the Van der Waals attractive pull between the EC-CNTs particles compared to OA counterpart, resulting to reduced EC-CNTs aggregates in rapeseed oil, according to previous investigation [41]. The lowest value (78 nm (0.5 mass%), 98 nm (1 mass%), and 101 nm (1.5 mass%) observed during the study suggested that SDS performed best.

For the side of 1.5 mass% EC-CNTs, higher particle size were recorded which could be as a result of higher concentration with high surface energy [40]. More so, size variation could be from surfactants addition as they may attach to the surface of the nanoparticle thus increase the diameter. This finding is true owing to the particle size results variations. With the results, the SDS surfactants provides the minimal particle size thus should be applied in the subsequent analysis as to examine the further effects of surfactants on EC-CNTs concentration with another chosen sonication time. The best sample result of 0.5 mass% EC-CNT with 5 mass% SDS will be use to compare the effect from SDS 2 mass% under sonication time of 25 minutes and 50 minutes respectively.

Figure 5 shows the various particle size recorded by DLS from different EC-CNT concentrations in base rapeseed oil

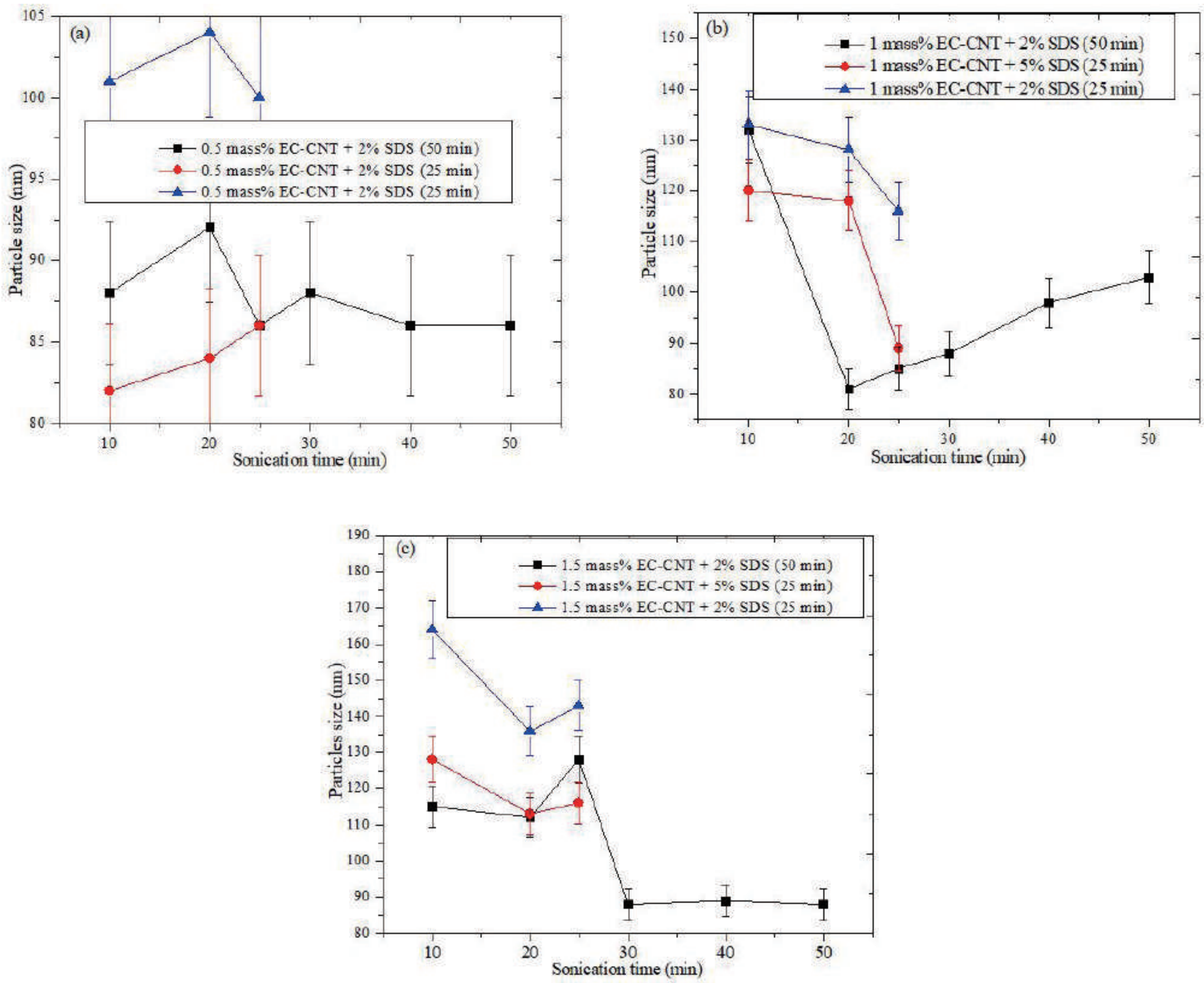

Fig. 5 Average hydrodynamic diameter of different concentration of EC-CNTs (0.5, 1, 1.5 mass%) particles in base rap. oil with different concentration of surfactant against sonication time (25 and 50 minutes) using dynamic light scattering

using best candidate of 25 minutes' sonication and SDS of 2 min for different 25 minutes and 50 min. When 0.5 mass% EC-CNT in rapeseed oil was blended with new amount of SDS (2 mass%) and sonicated for 25 min (same the previous time used) the average particle for the first 10 min was 102 nm and increased to 107 nm at 20 min and finally end at 25 min with 99 nm all with sonication. In the area of 0.5 mass% EC-CNT with SDS (2 mass%) dispersed in rapeseed oil with full sonication, average particles size was 88 nm at first 10 min, increases to 97 nm at 20 min and ended with 92 nm at 50 min time as shown in Fig. 5 (a). Comparing the recorded results with 0.5 mass% EC-CNT with SDS (5 mass%), shows low average particle size with 81.8 nm at 10 min and increased to 83.7 nm at 20 min, still increased to 85.5 nm at 25 min.

Dispersing 1 mass% EC-CNT with different amount of SDS (2 and 5 mass%) in rapeseed oil at different sonication time. For sample of 1 mass% EC-CNT with 2 mass% SDS for 25 min' sonication time. At first 10 min, the particle was 132 nm, reduced to 124 nm at 20 minutes, still reduced to 114.7 nm at 25 min of sonication. On the other hand, with 1 mass% EC-CNT with 2 mass% SDS under 50 minutes of sonication. The graph shows that the particle was 132 nm at 10 min, decreases to 80 nm at 20 min' time of sonication and ended at 50 minutes with 105 nm. When comparing with 1 mass% EC-CNT with 5 mass% SDS for 25 min' sonication, shows that the outcome was better than the counterpart (1 mass% EC-CNT + 2% SDS (25 min) as shown in Fig. 5 (b).

On dispersing 1.5 mass% EC-CNT with SDS (2 and 5 mass%) in rapeseed oil and sonicate for time of 25 and 50 min respectively. All the samples display similar pattern of fluctuation in size particles. Sample of 1.5 mass% EC-CNT with 2 mass% SDS for 25 min, started with 164 nm at 10 min and ended with 145.3 nm at 25 min, while 1.5 mass% EC-CNT with 2 mass% SDS for 50 min started with 114 nm at 10 min and decreases to 85.6 nm at 50 min. In comparing with 1.5 mass% EC-CNT with 5 mass% SDS for 25 min, at first 10 min the average particle was 127 nm and decreases to 141.7 nm at 25 min. The performance of SDS in this analysis is excellent supporting some previous presentations [34]. The result of 1.5 mass% EC-CNT with 5 mass% SDS is close to the sample with 50 min sonication but better than 1.5 mass% EC-CNT with 2 mass% SDS for 25 min as shown in Fig. 5 (c). With the overall observations in all the samples, different EC-CNT concentration dispersed in base rapeseed oil with 5 mass% SDS for 25 min' sonication and 2 mass% SDS for 50 min' sonication time exhibits best performance with lower particle sizes and fairly stable values, thus be considered in the further tribological analysis. The appearance of the solutions is shown in Fig. 6.

3.2 Friction and wear study

Figure 7 shows the COF obtained from different EC-CNTs concentration dispersed in base oil together the chosen SDS percentages, compared with base lubricant blended SDS and base lubricant alone. The results under 25 min sonication are shown in Fig. 7 (a), while 50 min sonication is shown in Fig. 7 (b). Addition of SDS into base oil yielded additional lubricity with COF reduction of about 4.7% against base oil. It clearly shows that lubricant (base oil + EC-CNT) without surfactant (SDS) provided good reduction in COF with addition of 0.5, 1 and 1.5 mass% EC-CNTs with values of 16.8%, 27.1% and 10.3% respectively, compared base lubricant alone. The higher COF reduction on 1 mass% EC-CNT, suggests to be from its optimum dispersion (Fig. 5 (b)). This outcome is similar to the previous study presentation on nanoparticle attributes in lubrication [42]. With inclusion of 2 mass% SDS under 25 min sonication significantly reduced the COF in all the concentration. The reduction was 41%, 62.6% and 38.3% for 0.5 mass%, 1 mass%, and 1.5 mass% EC-CNT respectively compared base oil. Again, under 25 min sonication using 5 mass% SDS on different EC-CNT provided COF reduction similar to 2 mass% SDS results as shown in Fig. 7 (a), but 0.5 mass% EC-CNT show more enhanced performance with 57% reduction against 41% recorded with 2 mass% SDS. This evidently revealed that application of 5 mass% SDS under 25 min sonication did not show good enhancement on friction with EC-CNTs, except little improvement on 0.5 mass%. Therefore, is wise to say that 2 mass% SDS is more effective with 1 mass% EC-CNT particles in lubrication. This could be due to better solution formulation and less particle agglomeration during lubrication.

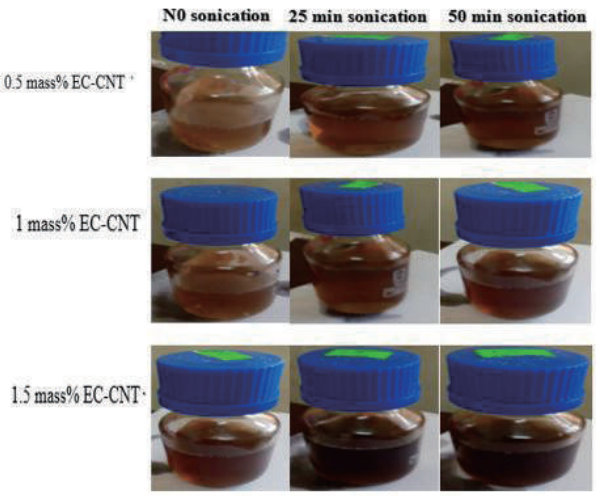

Fig. 6 Images of different EC-CNTs concentration dispersed in rapeseed oil, showing the nature of dispersion together with 2 mass% SDS surfactant and sonication times

Figure 7 (b) gives the COF against different EC-CNTs concentration under 50 min sonication time using 2 mass% SDS, compared with the base lubricant. The results reveal additional enhancement owing to the increase on sonication time. The COF reduction with 0.5 mass%, 1 mass% and 1.5 mass% EC-CNT were 59.8%, 65.4% and 56.1% respectively compared to base oil. The generated results from 50 min sonication using 2 mass% SDS gives clear expectations on the analysis, thus minimised the agglomeration effects of nanoparticles. Although, some good friction coefficient due obtained under samples with agglomeration (2 mass% SDS under 25 min), thus suggests that no much agglomeration effect from nanoparticles during lubrication on this testing. The enhanced results from 2 mass% SDS with 1 mass% EC-CNT both in 25 min and 50 min sonication was due to homogeneous solution and less agglomerations during operation.

The average wear scar diameter (WSD) recorded from base lubricant alone, base oil with SDS and different EC-CNT concentration dispersed with different SDS percentages under sonication period of 50 min compared to base lubricant are shown in Fig. 8 (a). As reported in previous presentations [16, 22, 43] that inclusion of nanoparticle additives enhances wear resistance. Lubricated surface with base oil blended SDS gives poor protection of -4.2% against base lubricant WSD of 11.8 *(10^{-3}) mm. This could be from poor tribo-chemistry

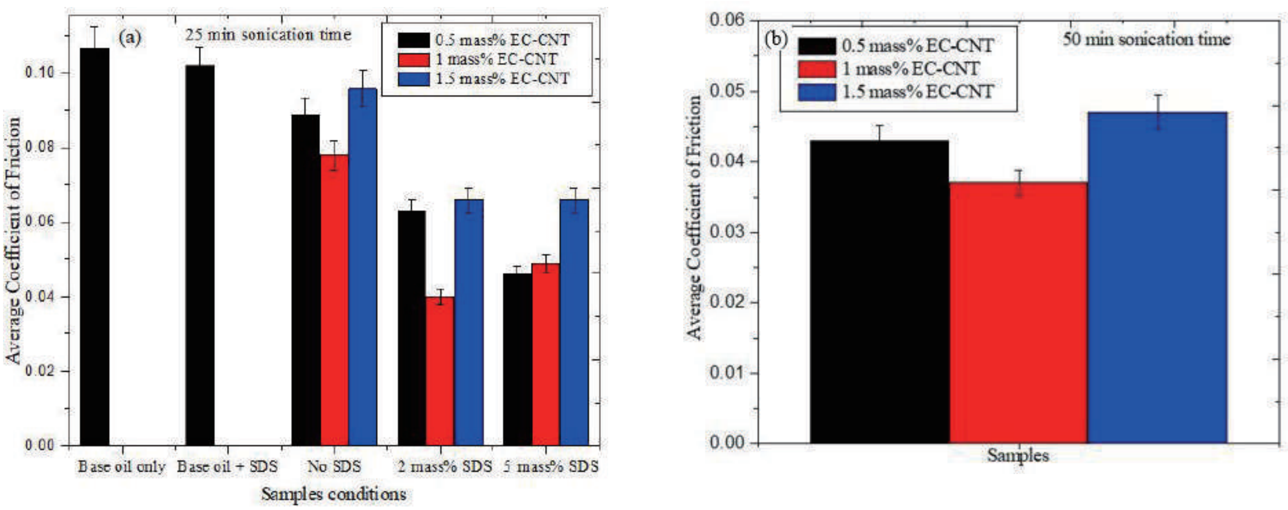

Fig. 7 Reduction in COF from various EC-CNTs dispersions in rapeseed oil with and without SDS percentages for (a) 25 minutes and (b) 50 minutes

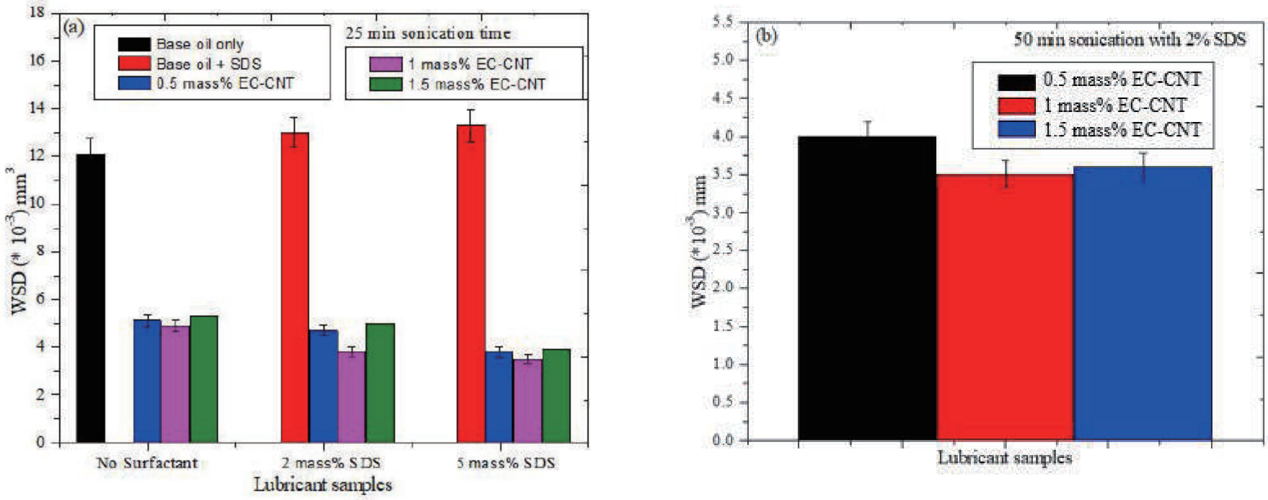

Fig. 8 Reduction in WSD exhibited by various EC-CNTs dispersions in rapeseed oil with and without SDS percentages for (a) 25 min and (b) 50 min sonication period

on the sliding surface leading to surface deformation. All the EC-CNT samples decreased the wear effects. The enhanced performance was much 1 mass% EC-CNT without SDS than other concentration under 25 min sonication. This revealed that EC-CNT has high tendency of forming film layer in protecting the sliding surfaces. With 1 mass% EC-CNT without SDS gives wear reduction of 49.2%. Inclusion of 2 and 5 mass% SDS yielded significant improvement on wear reduction to about 50%, 60.2%, 48% with 2 mass% SDS and 48.6%, 59% and 43.5% with 5 mass% SDS for 0.5 mass%, 1 mass% and 1.5 mass% EC-CNT respectively under 25 min sonication as shown in Fig. 8 (a). If not for the short sonication duration, leading to low dispersability of particles with tribo-chemistry reaction, the results of lubricants with 5 mass% SDS would have shown greater enhanced performance. The behavior appears similar to the observation in previous presentation [44].

Figure 8 (b), depicts the average wear diameter reduction from different EC-CNT nanoparticles under 2 mass% SDS and sonication time of 50 min. Various EC-CNT additives exhibited excellent wear resistance ability with values of 57.6%, 63.6% and 60.3% for 0.5 mass%,1 mass% and 1.5 mass% EC-CNT respectively. The tested lubricants with 2 mass% SDS under 50 min sonication yielded excellent results more than the samples under 2 mass% and 5 mass% under 25 min sonication time, therefore, will be applied in the subsequent analysis till the end. This has proven EC-CNT nanoparticle additive good for anti-wear application in lubrication.

3.3 Load carrying capacity and temperature effect

Figure 9 (a) presents the COF results generated from base oil blended with SDS and different EC-CNT concentration under different load. The results show that inclusion of EC-CNTs decrease the COF. Further revealed that the effect of nanoparticles is more pronounced at higher loads. The COF at 0.5 mass%, 1 mass% and 1.5 mass% EC-CNT decreases from 0.076 at 40 N to 0.041 at 120 N, 0.069 at 40 N to 0.031 at 120 N and 0.076 at 40N to 0.047 at 120 N respectively, while base oil with SDS reduces from 0.103 at 40 N to 0.073 at 120 N. Apart from the base oil with SDS, other lubricants work within the boundary lubrication regime. This is to say that the lubricant film thickness was observed to be smaller than the sliding surface roughness [45]. As a result, much contact is found between the sliding elements but still separated by the lubricant film generated. More energy is generated in boundary lubrication, ie, working condition has significant impact on the energy generation during lubrication [46, 47]. With low applied load, sliding friction produces less energy resulting in poor tribo-chemistry process between the nanoparticle and sliding surface. In this condition, there is an increase in possibility of nanoparticles engagement in filling the worn areas thus perform great operation in separating the surfaces, thus particles are more active in performing lubrication service in boundary lubrication condition [48, 49].

Figure 9 (b) described the steady condition of temperature of the various lubricants at the end of each load testing. In the samples of EC-CNTs particles, lower COF results lead to less heat pronouncement thus observed less temperature at the end of experiment. According to the graph, lubricant with 1 mass% EC-CNT has showcase good behavior not only in COF but in thermal property, thus influences the thermal characteristics of the nanofluid. Therefore, the use of EC-CNT substantially reduces the temperature of the lubricant compared to non-particle lubricant.

3.4 Lubricant film stability analysis

Tribological analysis was carried out on the selected canditate as to explore the lubricant film stability using load of 40 N and sliding frequency of 5 Hz, for 15 min as shown in Fig. 10. In using base lubricant alone, under the mationed working condition, the lubricant was observed to display unsteady film with decrease from the start till about 90 s, and rise again to about 200 s due to increase in friction then stabilized till the end. With addition of SDS into base oil, the lubricant shows unsteady lubricant film from the start to about 100 s before stabilizes with decrease in friction to about 400 s and maintain the same level till the end as presented in Fig. 10 (a). Addition of 0.5 mass% and 1 mass% EC-CNT exhibits the same film stability from the beginning with decrease in friction to about 120 s before stabilizes till the end. Sample with 1 mass% EC-CNT shows similar to 0.5 mass% and 1.5 mass% EC-CNT but had film breaking at about 350 s to 400 s and stabilizes till the end as shown in Fig. 10 (b). The unstable film time during lubrication is referred to as the lubricant film stability time breaking. During this film breaking, the COF's trend path

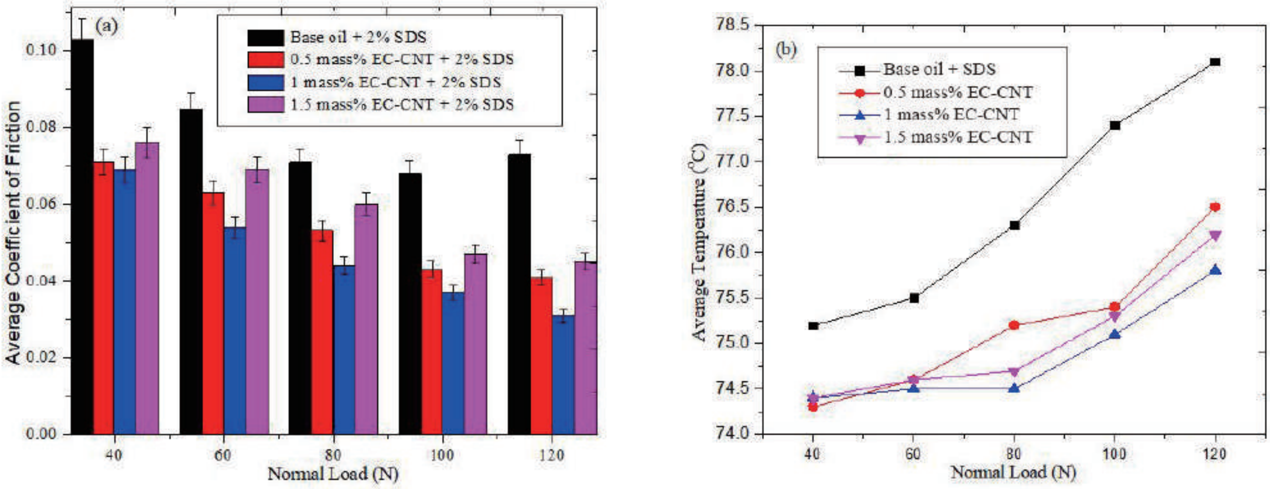

Fig. 9 Average COF (a) and temperature (b) exhibited by base oil and various EC-CNTs nanoparticles dispersions in rapeseed oil with 2 mass% SDS for 50 min sonication period

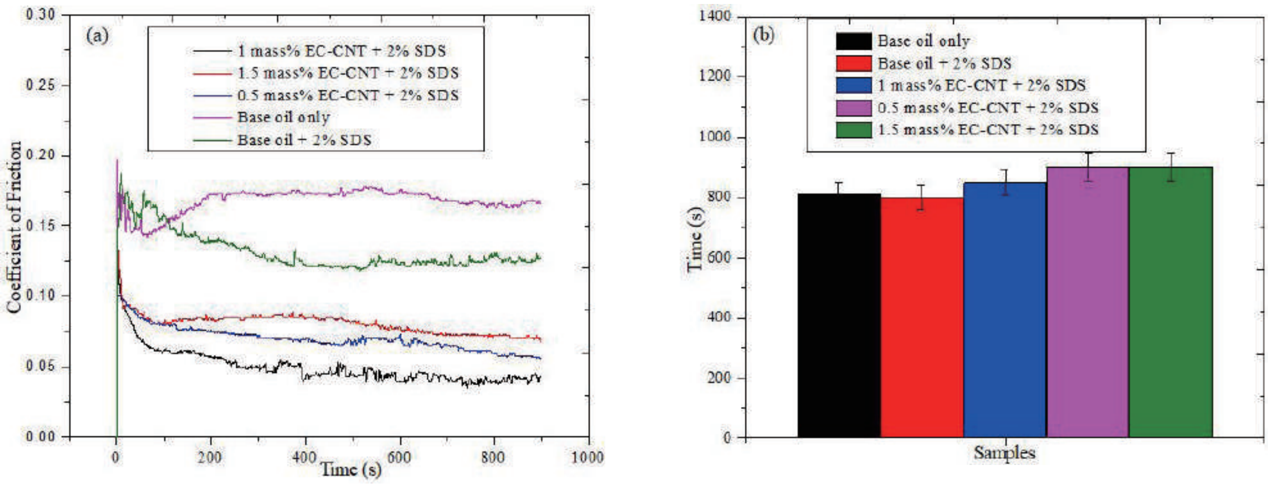

Fig. 10 Film stability graph (a) and lubricant film average time breaking (b) (40 N, 5 Hz, 75°C and 10 mm stroke)

shows an up and down pattern. This exhibits similar behavior with previous presentation on 1.0 mass% MoS_2 and 0.7 mass% SiO_2 film analysis [47]. The outcome from the results show that lubricant with EC-CNT particles exhibit good lubricity through maintaining the formulted lubricant film in terms of tribo-film stability during sliding compared to the ones without EC-CNT.

3.5 Wear scar micrographs and nanoparticle mechanism

As to ascertain the nanofluid lubricating mechanism, the lubricated worn flat surface was analysed using SEM and AFM machine. Figure 11 shows the SEM images from different lubricated surfaces (base oil only, base oil + SDS, 0.5 mass% EC-CNT, 1 mass% EC-CNT and 1.5 mass% EC-CNT) under 40 N load, 5 Hz and stroke of 10 mm. As shown in Fig. 11 (a), well pronounced grooves can be observed, similar to that of Fig. 11 (b), indicating that wear behavior was abrasive. The surface lubricated with 0.5 mass% EC-CNT appears shallower and smoother when compare to base oil and base oil + SDS as shown in Fig. 11 (c). Some healing effects on the grooves were observed, which are from the tribo-chemistry between the EC-CNT and sliding surface. The red boxes (Figs. 11 (c), (d), (e)) indicates the point of healing by the nanoparticles and was selected during EDX inspection. In the area of lubricated surface of 1 mass% EC-CNT, provide the smoothest surface as shown in Fig. 11 (c). However, the worn surface seems healed by EC-CNT inclusion. The surface lubricated with 1.5 mass% EC-CNT, shows healing also, but show some furrows. This results are similar to the previous nanoparticles analysis on steel contacts [47].

The anti-wear property improvement from EC-CNT particles were also demonstrated thorough EDS showing elemental distribution on the various lubricated surfaces as listed in Table 3. This evidently proves that EC-CNTs particles embedded on the contact surfaces, thus generates tribo-film leading to friction and wear reduction.

The 3D topographical images of the lubricated surfaces with their corresponding wear volume (Wv) are shown in Fig. 12. The analysis was conducted on base oil only and EC-CNTs nano-lubricants (0.5 mass%, 1 mass% and 1.5 mass% EC-CNT) using AFM machine. According to the figures, the base lubricant (without EC-CNT) shows higher wear volume value. Surface lubricated with 1 mass% EC-CNT gives the smallest wear volume, followed by 0.5 mass% EC-CNT before surface lubricated with 1.5 mass% EC-CNT. Also, the results revealed

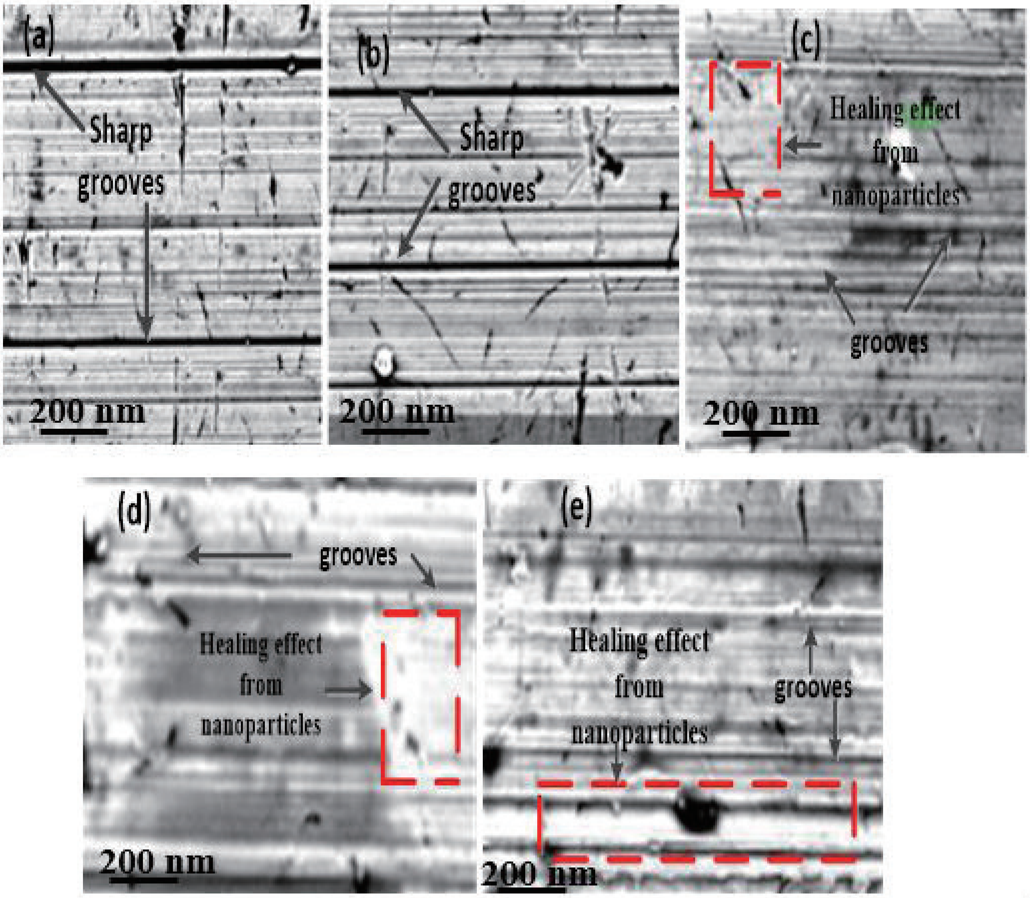

Fig. 11 SEM images of wear scars of various lubricated surfaces, (a) base oil, (b) base oil + 2 mass% SDS, and ((c) 0.5 mass% EC-CNT, (d) 1 mass% EC-CNT and (e) 1.5 mass% EC-CNT all with 2% SDS)

Table 3 EDX measurement of lubricated surfaces (base oil, base oil + 2% SDS, 0.5 mass% EC-CNT, 1 mass% EC-CNT and 1.5 mass% EC-CNT) (all EC-CNT were blended with 2% SDS)

Samples/elements (At. %)	Fe	C	O	Cr	Mn	Si	Ca	Al	Co	Na	Cl	Total (%)
Base rap. oil	95.23	1.36	1.05	0.51	0.6	0.09	-	-	0.7		-	100
Base oil + 2% SDS	84.38	1.28	3.78	1.09	0.07	0.07	-	-	0.43	7.90	-	100
0.5 mass% EC-CNT	68.63	14.06	5.41	0.57	1.70	1.96	4.58	0.86	0.97	2.03	0.23	100
1 mass% EC-CNT %	63.85	16.01	6.54	1.82	1.04	2.67	2.91	1.13	1.15	2.05	0.83	100
1.5 mass% EC-CNT	74.18	11.00	3.67	0.84	0.93	2.07	4.03	-	0.68	1.77	1.03	100

that average roughness decreases with addition of EC-CNT with 1 mass% EC-CNT showing the best performance. The inclusion of EC-CNT particles significantly reduced the wear volume as well as surface roughness. This could be because of the rolling behavior towards separation of the surfaces. Also the excellent performance of EC-CNT are attributed to its nano scale, thereby diffused into the worn valleys and friction zone, thus contribute during tribo-chemistry operation together the sliding surfaces to generate effective tribo-film.

Several mechanisms of nanoparticles in nanofluid towards friction and wear reduction have been proposed in literatures like; mending/healing effect, rolling effect, thermal stability improvement, polishing and viscosity alteration performance. In this study, based on the friction and wear performance of the nanoparticle (EC-CNT), polishing effect was not observed from the nanofluid lubrication. The mechanism of healing and rolling effect of the nanoparticle (here is EC-CNT) are modelled in Fig. 13. This is because the elements of EC-CNT were found in all the surfaces lubricated by EC-CNT nanofluid, thereby reduces the value of Fe element of steel. Therefore, mending/healing and rolling mechanism are the main operation by EC-CNT during lubrication. On the other hand, the wear reduction during the sliding was by rolling mechanism exhibited by the nanoparticles thus, separate the two bodies from direct contact as shown in Fig. 13 (b). The result was confirmed due to the reduction on the size of the furrows images observed on the surfaces lubricated with EC-CNT additive. More so, the substrates revealed wear healing owing the filling of the valleys by the EC-CNTs particles during lubrication. The tribological enhancement from EC-CNT lubrication is proposed as follows;

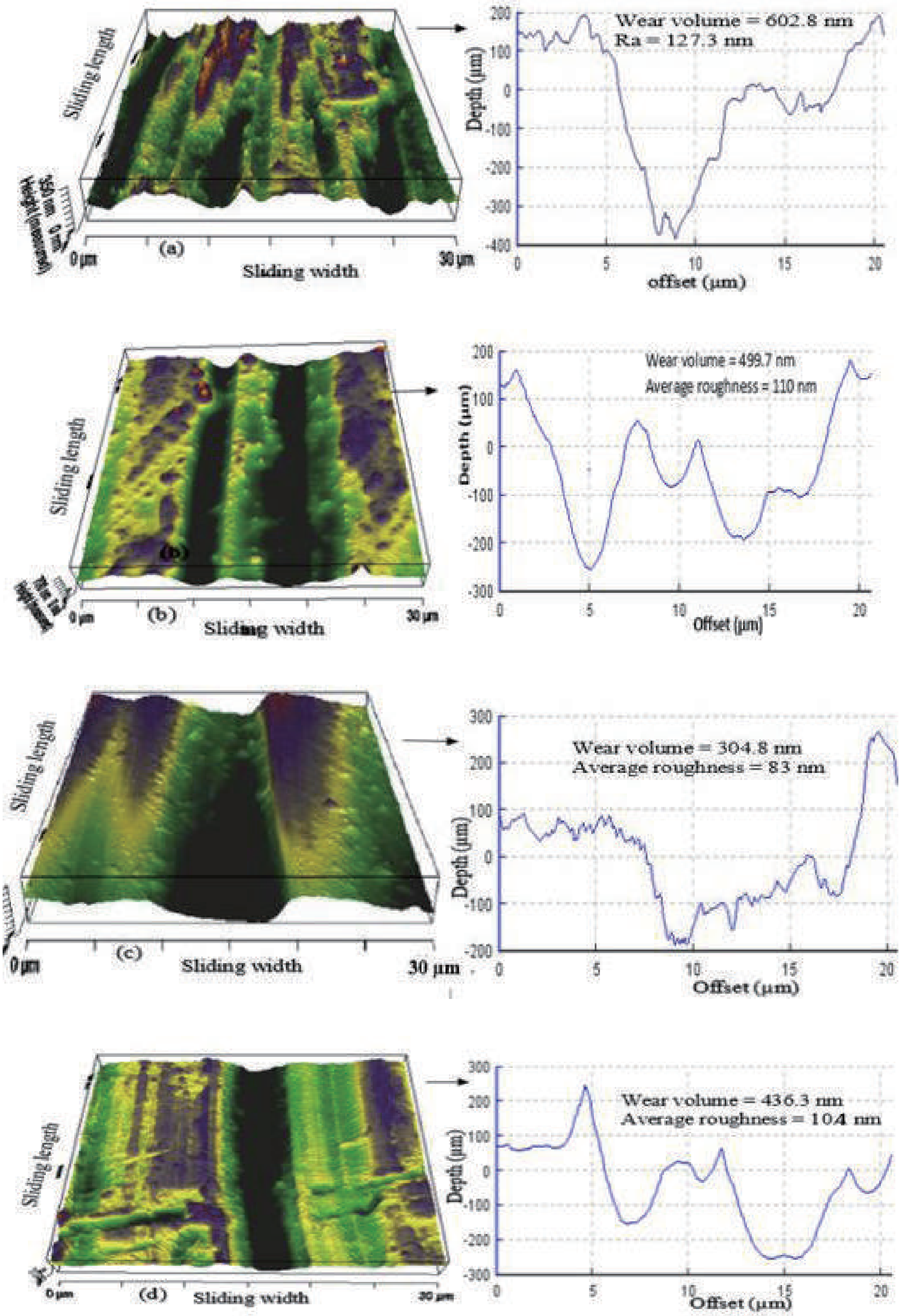

Fig. 12 3D topography images of various lubricated surfaces with profiles section of their wear tracks both wear volume and Ra after tests; (a) Base lubricant, (b) 0.5 mass% EC-CNTs nano-lubricants, (c) 1 mass% EC-CNTs nano-lubricants, (d) 1.5 mass% EC-CNTs nano-lubricants (all blended with 2 mass% SDS and sonicated for 50 min) (under 40 N load, 5 Hz, 10 mm stroke)

according to model developed by De-Xing Peng [50], stated that at high sliding contact, the nanoparticles penetrate into valleys of the sliding surfaces and stick leading to the film formation with the help of tribo-chemistry reaction. Due to activated energy on the sliding interface by the frictional force, the EC-CNT together the lubricant develops film through the tribo-chemical reactions on the substrates surface as illustrated in Fig. 13 (b). The formulated film prevents direct contact, serve as stress distributor, and load carrying bearing [51].

In this study, the minimum film thickness h_{min} observed can be predicted using Hamrock-Dowson equation for elastic lubrication [52, 53] as given in Eqs. (1) and (2).

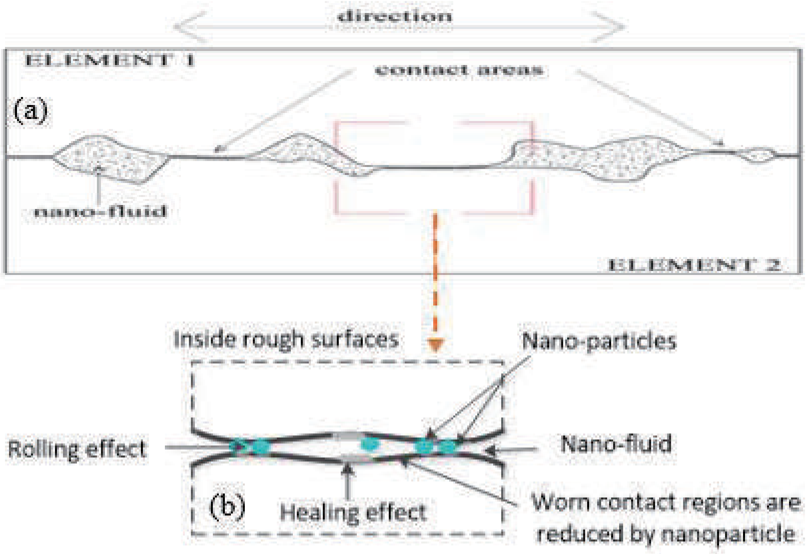

Fig. 13 Schematic description of proposed mechanism by the nanoparticles for wear reduction

$$h_{min} = 2.8\, R \left(\frac{\eta U_e}{ER}\right)^{0.65} \left(\frac{W_y}{ER^2}\right)^{-0.21} \qquad (1)$$

$$\frac{1}{E} = \frac{1}{2}\left(\frac{1 - V_{ball^2}}{E_{ball}} + \frac{1 - V_{ball^2}}{E_{flat}}\right) \qquad (2)$$

Where η is dynamic viscosity, R is the radius of the ball (12/2 = 6 mm), W_y is the normal load, U_e is the sliding speed, V_{ball} and E_{ball} are the Poisson's ratio and elastic modulus of the stell ball (AISI 52100) while E_{flat} and V_{flat} stands for the Poisson's ratio and elastic modulus of the flat steel plate (AISI 52100). Taking 1 mass% EC-CNT for the analysis and applying the appropriate values. The calculated film thickness of the lubricant (1 mass% EC-CNT) at different contact were 57 nm, 52, nm, 48 nm, 45 nm and 41 nm for 40 N, 60 N, 80 N, 100 N and 120 N respectively. With these values, the analysis shows that film thickness in the contact decreases with load increment as observed on load carrying capacity (Fig. 9 (a)).

4 Conclusions

As regard to the analysis conducted using surfactants and sonication to enhance EC-CNTs nanoparticles dispersion stability in base oil for friction and wear reduction in sliding contact surfaces, the study drawn the conclusion with the following.

(1) Application of surfactants and sonication technique enhances the dispersion and stability of EC-CNT nanoparticles in base lubricant. Surfactant of SDS yielded better results compared to OA counterpart. Under the two sonication time selected for different EC-CNT concentration dispersed in base rapeseed oil, samples of 5 mass% SDS for 25 min sonication and 2 mass% SDS for 50 min sonication time exhibits best performance with lower particle sizes and fairly stable values.

(2) Under friction and wear analysis, better results were achieved on 50 min sonication with 2 mass% SDS for all the EC-CNTs concentration compared to 25 min sonication time with 5 mass% SDS. However, 1 mass% EC-CNT shows the best performance in both friction and wear reduction.

(3) Ability to carry load by lubricant can be enhanced through inclusion of nanoparticles. EC-CNTs exhibited excellent performance on carrying different capacity of load, showing that under high load and contact during sliding, more energy is generated for film formation leading to friction and wear reduction.

(4) In the area of generated lubricant film stability, the base oil alone and base oil with SDS shows distortion on the formulated film. At the early stage of sliding at about 90 s then affected by temperature with increase in friction at about 200 s and stabilizes till the end for base oil alone while base oil with SDS formulated film waved at early phase at about 100 s, friction decreases gradually till about 400 s and stabilizes till the end. With addition of EC-CNT, 0.5 mass% and 1.5 mass% gives the best results with uniform decrease in friction from the beginning and stabilizes at about 100 s without film break down compared to 1 mass% which shows film break down at about 350 s to 400 s before stabilizes till the end. Though 1 mass% EC-CNT yielded best COF.

(5) In terms of mechanism, EC-CNT nanoparticles show rolling and healing effects. This is proven due to elements of EC-CNT detected on the lubricated surface by EDS analysis. The deposited particles on the worn or pits/valleys contributes in reducing friction and wear because they help in tribo-chemistry with sliding surface thereby formulate effective and workable tribo-film that is capable of separating direct contact.

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