Comparison on Tribological Behavior of Organic Formulated Carbon Nanotubes and Multi-Walled Carbon Nanotubes in Base Rapeseed Lubricants

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Comparison on Tribological Behavior of Organic Formulated Carbon Nanotubes and Multi-Walled Carbon Nanotubes in Base Rapeseed Lubricants

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Abstract: In this research, the tribological behavior of two distinct carbon nanotubes and the mechanisms influencing tribological performance were investigated. High frequency reciprocating rigs (HFRR) were used for the study, along with rapeseed oil as the base lubricant and commercial SAE 5W 30 as the benchmark. The experiment used particle concentrations of 0.5, 1, and 1.5 wt.% for MWCNTs and EC-CNTs, respectively, as lubricant additives. According to tests on the' viscometric properties, blends of both additives at a concentration of 1 wt.% produced the best results in terms of viscosity index. When compared to base lubricant, 1 wt.% MWCNTs performed better in frictional analysis at 80 N than 1 wt.% EC-CNTs, but less well than SAE 5W 30 benchmarks. In comparison to other evaluated samples, 1 wt.% EC-CNT exhibited remarkable results at higher loads of 100 N. Based on the investigation, EC-CNTs showed accumulation of particles at the front and back of the sliding ball under less operating conditions. This restricts the diffusion of the particles to the contact region, but it disappears at higher conditions. At a load of 100N, MWCNT and EC-CNT reduced friction and wear by 29.3%, 40.9%, and 30.8%, 45.5%, while SAE 5W 30 produced 45% and 36.4%, respectively. The ability of the additives to convert the direct sliding contact into a rolling mechanism and the formation of tribo-film were attributed to their successful performance. The outcomes clearly demonstrate that EC-CNTs behaved similarly to MWCNTs and SAE 5W 30 lubricants, making it appropriate for lubrication application.

Keywords: MWCNTs, EC-CNTs, lubrication, mechanism, friction, and wear

1. Introduction

In mechanical mechanisms, lubrication is one of the most successful ways to reduce energy consumption and increase machine productivity. The improvement of machine performance is due to friction and wear reduction, although the nature of the added additives always depends on the effectiveness of the lubricant[1-4]. The application of nanoparticles as additives in lubricating medium is gaining popularity as nanotechnology advances. Owing to their nano size, surface and contact effects, temperature stability, and variety in particle morphologies, nanoparticles exhibit exceptional physical, chemical, and mechanical features. Several nanoparticles performance in lubrication have been studied in the literature, including graphite[5] polytetrafluoroethylene (PTFE)[6] $MoS2$[7-9] $TiO2$[10, 11], h-BN[12, 13] and Ni[14] as lubricant additives with good remarkable friction and wear characteristics. During the lubrication, observed that rolling effect, protective film by tribo-chemical reactions, ball bearing effect, adsorption on surface to make up for material loss, mending effect, and third body material transfer are a few mechanisms for the outstanding tribological characteristics of nanomaterials.

Perez-Luna[15] affirmed that MWCNTs' distinct physical and chemical characteristics make it more promising for variety of applications. However, it is necessary to investigate MWCNT's benefits in the field of tribology in different operations. Nevertheless, poor solubility and agglomeration of MWCNTs in aqueous solution has been a significant barrier to their use in tribological applications. Agglomerated nanoparticles result in micron-sized particles of uneven shape and size, which wear down the system more quickly than base oil and lower its performance. Depending on the compatibility between the MWCNTs and the base lubricant defined the accumulation effect. However, this agglomeration's impact could be reduced by utilizing an appropriate surfactant, supersonic homogeneous mixer, or sonication technique. Therefore, In this work, an extensive study on the tribological behavior of different carbon nanotubes in base rapeseed oil, together with the impact of sonication on their stability. Kumar et al.,[16] asserted the enhancement of lubricant performance through sonication on the work conducted using TiO2 at different concentrations. If this investigation turns out to be effective, it will be put into practice with the intention of lubricating steel-steel contact.

2. Materials and Method

The MWCNTs NPs, base rapeseed oil, SAE 5W 30 commercial standards, and the chrome ball were provided by Atlas Ball and Bearing Co. Ltd. and acquired from Sigma-Aldrich, respectively. To create the final EC-CNTs, the Department of Tribology at Universiti Teknologi Malaysia used Eichhornia crassipes as a raw material before cyclic heating[17]. Opia et al.,[17] and Xie et al.,[18] describe the process used during formulation of EC-CNTs NPs, employing a ball milling machine and cyclic heating strategy (oven, furnace), with the help of some useful chemicals.

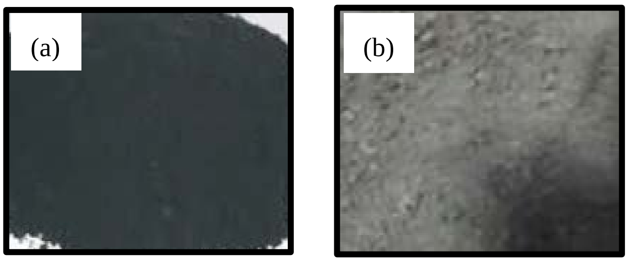


Fig. 1: Images of MWCNTs (a) and the formulated EC-CNTs (b)

2.2. Characterization of MWCNTs and EC-CNT NPs

To examine the morphology and elemental makeup of MWCNTs and EC-CNTs, scanning electron microscopy (SEM) and energy dispersive x-ray (EDX) were used. Using a nanoparticles size machine, the additives size spreading was performed (SU 800; Hitachi Japan). TGA analysis was done on the materials' thermal strength for both the MWCNTs and EC-CNT alone and blends with base lubricant as to understand the thermal resistance. The variety of nanotubes was determined using Raman spectroscopy, which was carried out in the spectral range of 400–2000 cm (Horiba Scientific, UK).

2.3. Viscometric Analysis of MWCNTs and EC-CNTs in Solution

According to ASTM D-445, the viscosity of Rapeseed oil without and with the addition of MWCNTs and EC-CNTs was measured using a viscometer (Cole-Palmer, USA). The testing was conducted at temperatures of 40 °C to 150 °C, using 5-unit intervals. As the temperature increased, the setup measured and tracked the viscosity value via the spindle[19]. Subsequently, 100 ml of basic rapeseed oil (BRO) were subjected to sonication at a speed of 1000 rpm for 30 min while containing 0.5, 1., and 1.5 wt.% of MWCNTs and EC-CNT, respectively. Based on the results of the previous investigation[17], the concentrations were chosen, as well as SAE-5W 30 reference. The MWCNTs and EC-CNTs concentration was selected based on suggestions from prior literature on the ideal nanoparticle’s concentrations in lubricant[20, 15].

2.4.Frictional Analysis

According to ASTM G133-05 standard test procedure, the tribological properties of MWCNTs and EC-CNT were assessed using high frequency reciprocating rig tribo-meter[21]. Strokes of 10 mm, 100 ml BRO, and 5 Hz frequency were utilized during the reciprocating test. The test lab was kept at ambient temperature with a relative humidity of 32 2% to eliminate any outside influences. The effectiveness and behavior of the MWCNTs and EC-CNT particles as lubricant additives to minimize friction and wear were examined in this study, employing different working conditions as presented in Table 1. Before testing, each sample was sonicated to produce the good stability required for good nanofluid, preventing difficulties with sedimentation and agglomeration. Before and after each test, different components for the analysis were cleaned using an ultrasonic cleaning agent such heptane. Since EC-CNTs are amphiphilic, at the proper concentration, can dissolve in lubricant[22, 23].

Table 1 Experiment Parameters

Lubricant samples	Base rapeseed oil Benchmark SAE 5W 30 MWCNTs (0.5, 1 and 1.5 wt.%) EC-CNTs (0.5, 1 and 1.5 wt.%)
Load (N)	60, 80, 100, 120
Temperature (oC)	75
Stroke	10 mm
Operation duration	15 (min)
Friction Pairs	Ball Ra < 20 nm roughness Flat Ra = 200 nm roughness

To guarantee that the trials could be replicated, friction tests were carried out three times for each sample. After the friction research, the lubricated surfaces underwent wear analysis using contact surface profilometer, SEM accompanied by EDX, and X-ray photoelectron spectroscopy (XPS) were employed. Numerous studies are being conducted to identify the constituents in the film created during tribo-chemistry on the substrate, the particle mechanism during operation, and the surface morphology (surface roughness (Ra)/wear radius, wear volume, among others).

3. Results and Discussions

3.1 Samples characterization

SEM images of the MWCNTs and EC-CNT NPs are shown in Fig. 2. The nanotubes' tube-like structure is seen in the pictures, MWCNTs have more pronounced real nanotube structures (Fig. 2(a)), whereas EC-CNTs exhibit thicker nanotube structures. Using EDX and focusing on the white square box, the elements in MWCNTs and EC-CNTs were identified with a high percentage of carbon and oxygen, as indicated in Table 2. This backs up earlier findings on the elemental composition of EC17). When HCl was applied into EC-NPs during the treatment process, the particles shrunk. When examined using size particles, MWCNTs had dark hexagonal forms, whereas EC-CNTs displayed a roughly spherical shape and Metallic Silver color.

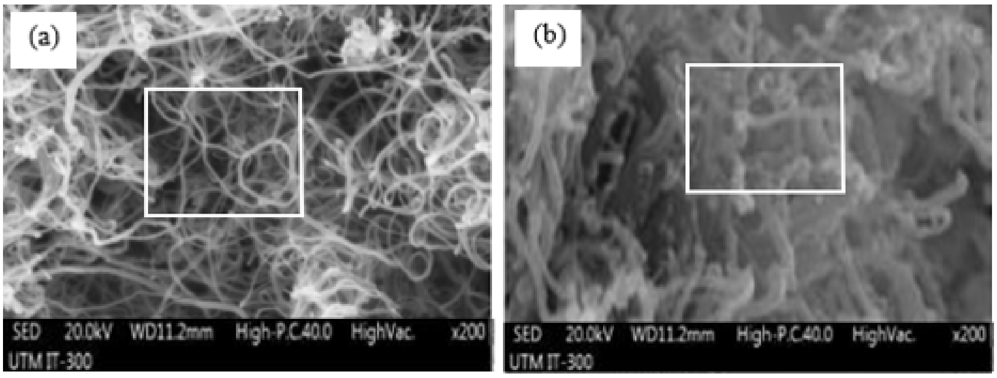


Fig. 2: SEM images of MWCNTs (a) and EC-CNTs (b)

Table 2: Elemental composition in MWCNTs and EC-CNT samples by EDX.

Sample/Element (wt%)	C	O	Si	Mo
MWCNTs	80	20	-	-
EC-CNTs	74.8	18.2	5.1	1.8

Fig. 3, depicts the particle size TGA analysis of MWCNTs (a) and EC-CNTs. The average mean size of MWCNTs and EC-CNTs in solution were 8.5 nm and 88.3 nm, respectively as illustrated in Fig. 3 (a). The TGA results for MWCNTs and EC-CNT NPs are displayed in Fig. 3(b). Derivative thermogravimetric data were used to calculate the weight loss % according to 23). The thermos-gravimetric curve's junction of two tangents, right before the inclination brought on by degradation from different samples, yielded the beginning degradation temperature.

The 38.5% weight loss in rapeseed oil can be attributable to impurities and unsaturated fatty acids that were absorbed during formation. Fig. 3 (b) shows the weight loss after modification with MWCNTs to be 19.3%. (b). The modifications imply that MWCNTs' superior thermal properties are a result of their greater resilience to thermal deterioration. A weight loss of 22.7% from EC-CNTs blends was because of the alteration using EC-CNTs. Weight loss (%) indicated increased thermal resistance.

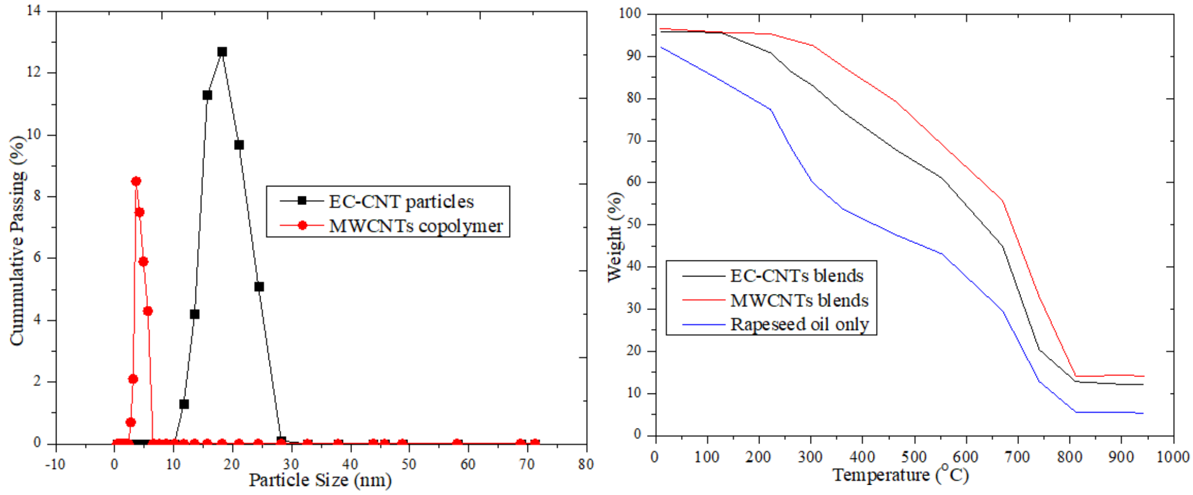


Fig. 3: Particle size distribution (a) and TGA (b) of MWCNTs, EC-CNT NPs and base rapeseed oil samples

Results from an analysis of MWCNTs and EC-CNT NPs using Raman spectroscopy are shown in Fig. 4. In the graphs for the Raman spectroscopy study of MWCNTs and EC-CNT NPs, two peaks were seen at 1375.31 and 1601.37 cm^{-1} (Fig. 4(a)) and 117.9 and 1229.3 cm^{-1} (Fig. 4(b)), respectively. The values on the peaks were believed to be caused by the carbon atom vibrations in the carbon layer. According to the image, EC-CNT had a more dramatic Raman alteration in the G-band and D-band due to the treatment. The weakness in the graphene sheet is clearer in the G-band than the D-band strength[24]. It was evident that in the sample, ID was higher than IG in MWCNTs while vice versa in EC-CNTs.

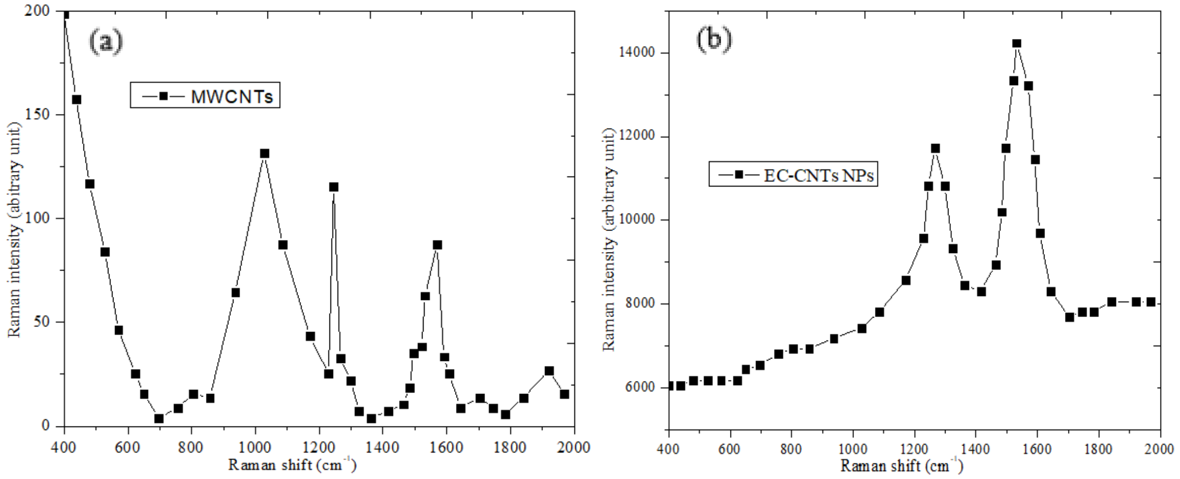


Fig. 4: Raman spectroscopy of MWCNTs and EC-CNTs indicating D and G bands.

The viscometric properties of base oil alone, several additive concentration blends, and the SAE w 30 commercial benchmark investigated in this work are shown in Fig. 5. The test used centistoke viscosity at temperatures between 40 and 150 °C and specific gravities at 25 °C. Table 3 presents the findings of viscosity indices and dynamic viscosities, provide an overview. The findings show that viscosity decreases as temperature rises in all the samples examined, however the trend of base rapeseed oil alone deviates from all additive bends and standard. The two additives' closely related patterns suggested that they are not polymeric by nature, although EC-CNTs demonstrated hydrophobic properties better than MWCNTs. While the performance of viscosity is strengthened by increasing additive concentration, observed that under 1.5 wt.% for both additives showed poor performance and an inconsistent trend, which could have an impact on the lubricant lubricity. Degradation of the lubricant was evident from the decline and the irregular trend effect. The newly created 1 wt.% MWCNTs and EC-CNTs have the best performance compared to previous samples and are significantly similar to the benchmark.

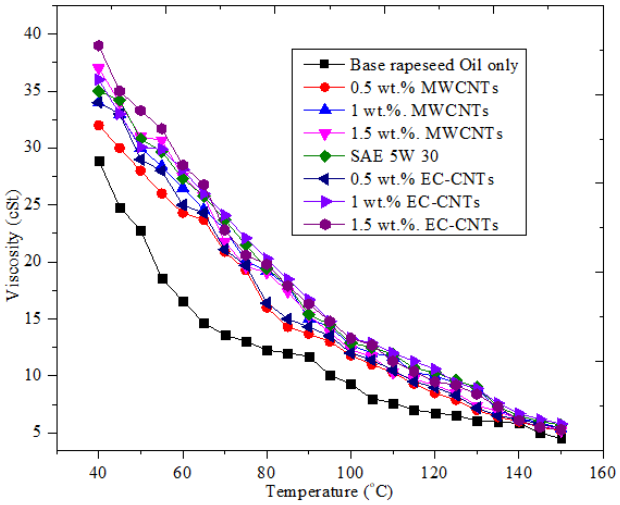


Fig. 5: Samples viscosity versus temperatures (40 – 150 °C) as used in the research.

Table 3. Viscometric characteristics of the samples

Samples	Viscosity (cSt) @ 40°C	 @100°C	 @150°C	Viscosity index
Base rapeseed oil alone	28.9	9.3	4.5	332.3
0.5wt.% MWCNTs	32	11.8	5.2	385.4
1wt.% MWCNTs	34	12.7	5.4	389.9
1.5wt.% MWCNTs	37.1	12.3	5.2	346.5
0.5wt.% EC-CNTs	35	12	5.7	368.3
1wt.% EC-CNTs	34	12.9	5.4	384.6
1.5wt.% EC-CNTs	36.6	12.3	5.8	356.4
SAE 5W 30	37	13.2	5.3	372.3

3.2. Tribological performance of the MWCNTs and EC-CNTs nano particles in solution

Fig. 6 discloses the average coefficient of friction (COF) and wear scar diameter (WSD) of different concentrations of MWCNTs, and EC-CNT nano additive tested. As demonstrated in Fig. 6(a), rapeseed base oil has a greater coefficient of friction than MWCNT and EC-CNT nanofluids, with value of 0.0903. However, with inclusion of the various additives, COF was found to be reduced significantly, but showed poor performance with 1.5 wt.%, revealing that 1 wt.% is the best concentration. The COF reduction under 0.5, 1, 1.5wt.% for MWCNTs and EC-CNTs were 10%, 20.5%, 16.6% and 6.5%, 16.7%,5.2%, respectively, while SAE W5 30 yielded 33.7% which is the best result. It was suggested that MWCNTs' superior performance was due to their lower particle size, which allowed them between the sliding contact, leading in tribo-film formation.

Fig. 6 (b) displays the average WSD variation and wear resistance of the oil samples. According to Fig. 6 (b), all the lubricant samples significantly lower the WSD of the flat steel material. The wear scar diameter variation of the two additives were similar. For the particles, the WSD sharply reduces as the concentration is increased, but the 1.5 wt.% shows no improvement over the 1 wt.%, which reflects the effect on COF results. The WSD of base rapeseed oil was 0.79 mm. With the addition of 0.5, 1, 1.5 wt.% concentration of MWCNTs and EC-CNTs, the WSD were reduced by 17.7%, 40%, 35% and 33%, 44.3%, 27.8%, respectively, while SAE 5W 30 produced 42%. Overall, among all the evaluated samples, 1 wt.% EC-CNTs demonstrated exceptional performance. The reduced friction between the sliding contact was suggested to come translation of direct contact into rolling effect caused by MWCNTs and EC-CNT particles, responsible for the decrease in wear scar diameter. Again, the wear scar diameter gradually reduces as the particle concentration in the base oil increases, bur observed agglomeration which affects the performance of 1.5 wt.% concentrations. In the case of EC-CNTs, the fast formation of tribo-film with rapid mending effect is attributed to its amphipathic tendency.

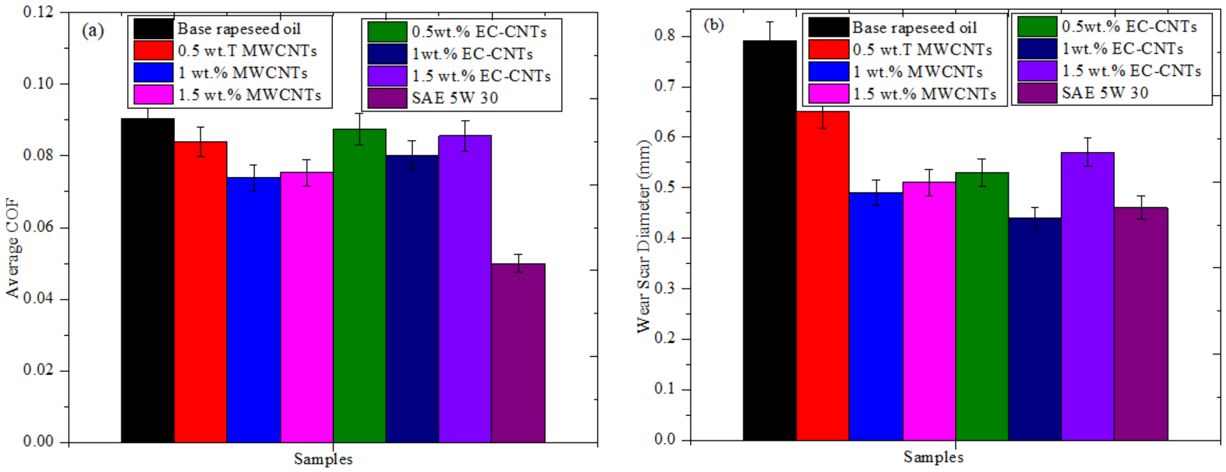


Fig. 6: Variation of average COF (a) and WSD with different concentration of additives in base oil (60 N, 5 Hz, 0.13 m/s, 10 stroke, 15 min).

Fig. 6 (b) displays the average WSD variation and wear resistance of the oil samples. According to Fig. 6 (b), all the lubricant samples significantly lower the WSD of the flat steel material. The WSD variation of the two additives were similar. For the additives, the WSD sharply decreases as the concentration is increased, but the 1.5 wt.% shows no improvement over the 1 wt.%, which reflects the effect on COF results. The WSD of base rapeseed oil was 0.79 mm. With the addition of 0.5, 1, 1.5 wt.% concentration of MWCNTs and EC-CNTs, the WSD were reduced by 17.7%, 40%, 35% and 33%, 44.3%, 27.8%, respectively, while SAE 5W 30 produced 42%. Overall, among all the evaluated samples, 1 wt.% EC-CNTs demonstrated exceptional performance. The reduced friction between the sliding contact was suggested to come translation of direct contact into rolling effect caused by MWCNTs and EC-CNT particles, responsible for the decrease in WSD. Again, the wear scar diameter gradually reduces as the particle concentration in the base oil increases, bur observed agglomeration which affects the performance of 1.5 wt.% concentrations. In the case of EC-CNTs, the fast formation of tribo-film with rapid mending effect is attributed to its amphipathic tendency.

Under temperature of 75 ºC and frequency 5H, utilizing the best concentration (1 wt, %) from the additives, the COF under 100N and average coefficient of friction and WSD under various loads (60, 80, 100 and 120 N) compared to base oil and SAE 5W 30 were conducted as shown in Fig. 7(a, b and c). The outcome revealed that inclusion of additives yielded good COF reduction, while COF from base lubricant was 0.108. The percentage reduction from the different additives and commercial benchmark SAE 5W 30 together with the average COF and WSD from the different conditions are presented in Table 4. The investigation found that the average friction coefficient and flat WSD dropped more sharply compared to the base lubricant as the load increased from 60 to 120N. The use of MWCNTs shows better performance than EC-CNT on COF apart under 120N. but SAE 5W 30 exhibited best in all the conditions tested. The poor performance from EC-CNTs was due to nanoparticles challenges in moving into the contact region as a result of accumulation at the ball front due to the bigger size compared to MWCNTs. At 120 N, due to the frictional energy generated and good tribo-chemistry, the EC-CNTs displayed outstanding performance.

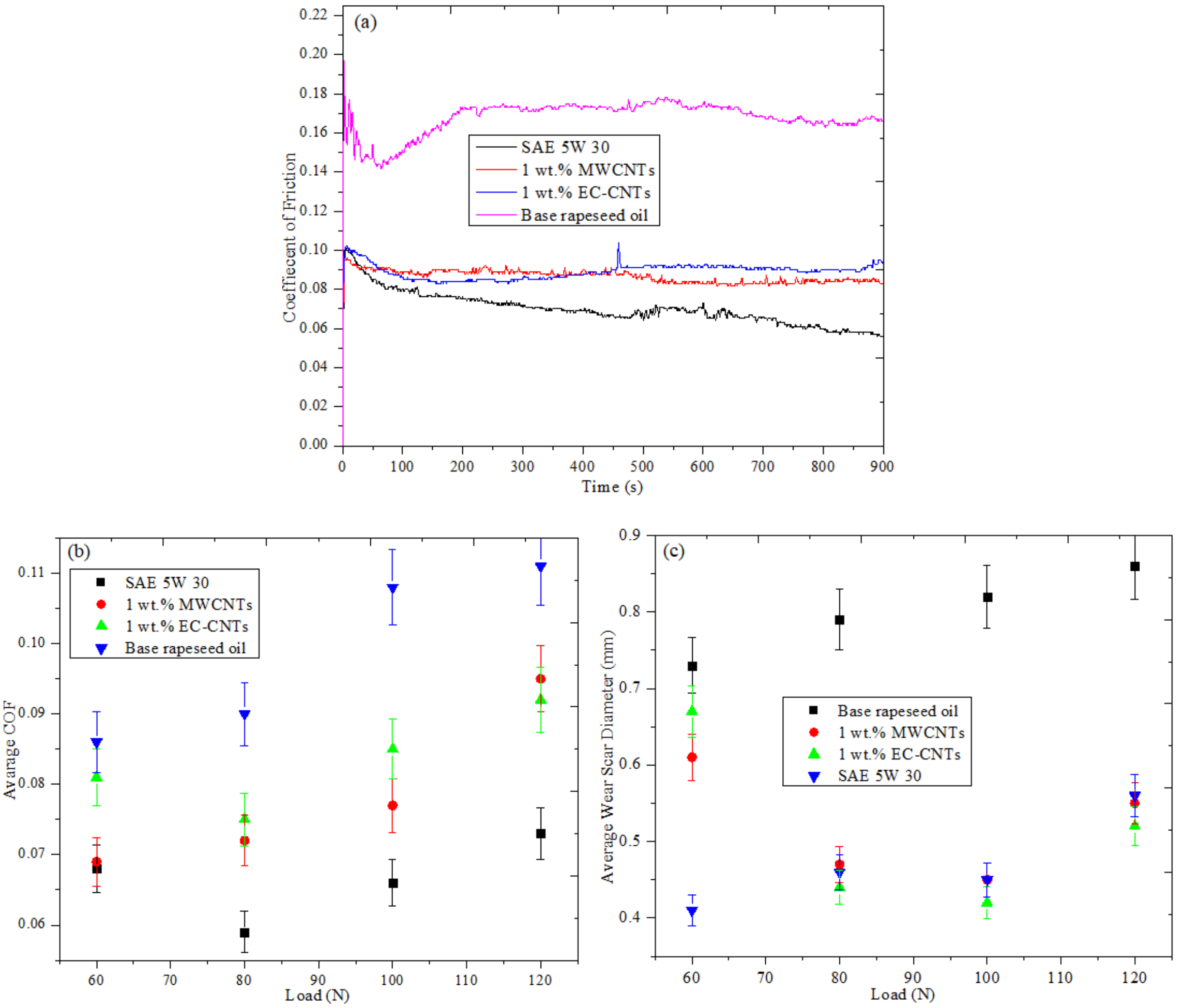


Fig. 7. Variation of average COF (a) and WSD with different concentration of additives in base oil (5 Hz, 0.13 m/s, 75 oC, 10 stroke, 15 min).

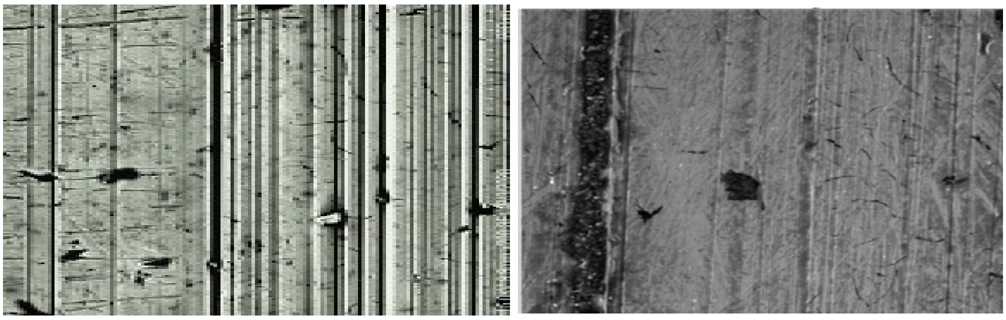
Table 4. Various lubricants COF under 100N, average COF and average WSD under different conditions (60, 80, 100and 120 N).

Sample/ conditions	COF	COF reduction (%)			WSD	WSD reduction (%)		
	Base rapeseed	1wt.% MWCNT	1wt.% EC-CNTs	SAE 5W 30	Base rapeseed	1wt.% MWCNT	1wt.% EC-CNTs	SAE 5W 30
60	0.086	19.7%	15.1%	20.9%	0.73	16.4%	8.2%	43.8%
80	0.090	20.5%	16.7%	33.7%	0.79	40%	44.3%	42%
100	0.108	28.6%	21.3%	38.9%	0.82	45.1%	48.8%	45.1%
120	0.133	29.3%	30.8%	45%	0.86	40.9%	45.5%	36.4%

3.6.SEM analyses of different lubricated surfaces (1wt.% of MWCNTs and EC-CNTs, SAE 5W 30 and base rapeseed oil)

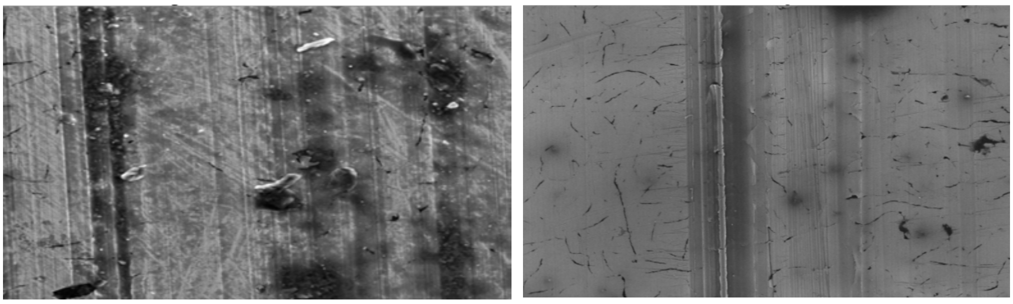
The topography of the worn surfaces can be used to study the tribological outcome of the lubricant additives. Figure 8 depicts the SEM morphologies of the worn surfaces of the steel flat that were lubricated with various additives at a concentration of 1 wt.%. The most significant wear scar can be seen on the worn surface that was lubricated with base rapeseed oil (Fig. 8(a)), which also has numerous grooves, dents, and can be seen to have visible eye. This shows that the operations were on direct contact according to Li et al.,[25]. The worn surface under 1 wt.% MWCNT particle (Fig. 8(b)) and the 1 wt.% EC-CNT particles (Fig. 8 (c)) are lesser than surface lubricated with base oil, relating to WSD information established in Fig. 7. This indicated the presence of third party called film which separates the sliding from direct contact unlike base lubrication.

The SEM findings show that the EC-CNTs and MWCNTs particles can enhance base oil's tribological performance. Although the performance of EC-CNTs at 60 N was observed to be low but outperformed in other conditions. Compared to the nature of the wears, the lubricated additives exhibit a lesser scar that is smooth with shallow furrows, which suggests that the anti-wear property of the used additives. The surface observed mending effect indicating the ability of the lubricants forming healing films on the substrates. This is similar to the observation made in the previous presentation[26]. Also, the effectiveness of EC-CNTs was due to the amphipathic property with high tribo-film formation leading to excellent wear reduction. Table 5, presents the elements discovered at the surface of the lubricated subtracts through EDX analysis.



Base Rapeseed oil only

1 wt.% EC-CNT blends



1 wt.% MWCNTs blends

SAE 5W 30 benchmarks

Fig. 8: Presentation of different worn surfaces under SEM investigation.

Table 5. EDX distribution various lubricated surfaces with base oil only and inclusion of MWCNTs, EC-CNTs, and SAE 5W 30.

Lubricant	Elements								Total (%)
	Fe	C	O	Si	Mo	Mg	Zn	Mn	
Base lubricant	78.3	27.5	5.1	-	-	-	-	0.1	
1 wt.% MWCNTs	42.9	48.2	3.3	0.6	0.3	0.9	-	-	
1 wt.% EC-CNTs	46.9	44.2	6.3	1.8	1.3	0.6	-	0.2	
SAE 5W 30	46.9	3.7	7.3	0.5	2.6	0.2	27.9	-	

3.7. XPS investigations of the lubricated surface with EC-CNTs particles.

According to an EDX study, EC-CNTs is an organic produced anti-wear additive that contains multiple elements like C, O, Si, and Mo. These elements should have effective lubrication mechanisms to generate an adequate tribo-film, which will have a mending effect on the worn surfaces during operation, thus validated using XPS analysis. On the worn surface that has been lubricated with EC-CNTs particles, Fig. 9 displays the XPS spectra of Fe 2p, C 1 s, O 1 s, Mo 3d, and Si 2p. In the spectrum of Fe 2p, two peaks with binding energies of 742.7 eV and 766.5 eV were identified and linked to the chemical state of metallic Fe (Fig. 9(a)). The presence of the carbonyl group was shown by the significant elements C 1 s and O 1 s of EC-CNT, which have spectra binding energies of 1998.3 eV and 960.17 eV, respectively (Fig. 9(b), (c)). This may be clarified by the fact that the material is organic and contains a lot of absorbed carbon As per O1s, the peaks at 960.17 might relate to metal oxides. As seen in Fig. 9(d), Mo 3d has two peaks. Along with the Mo (4 +) component, there is also a component that has the Mo 3d peak at 1273.83 eV, which is associated with the Mo-O bond. Following EC-CNTs additive testing, XPS analysis revealed that molybdenum oxidized, and molybdenum silicate were present on a worn surface. Additionally, another pick was found at 1276.71 eV. This is connected to the Mo (4 +) valence state and originates from the MoSi2 matrix (Mo-Si bonds) due to the heating impact, demonstrating the MoSi2 material's adhesion to the worn track's surface. These results are in line with other investigations into the interactions between nanoparticles and steel surfaces[27]. The outcomes also demonstrate that the nanoparticles of EC-CNTs have been exposed to and involved in the tribo-chemical reaction at the friction surface.

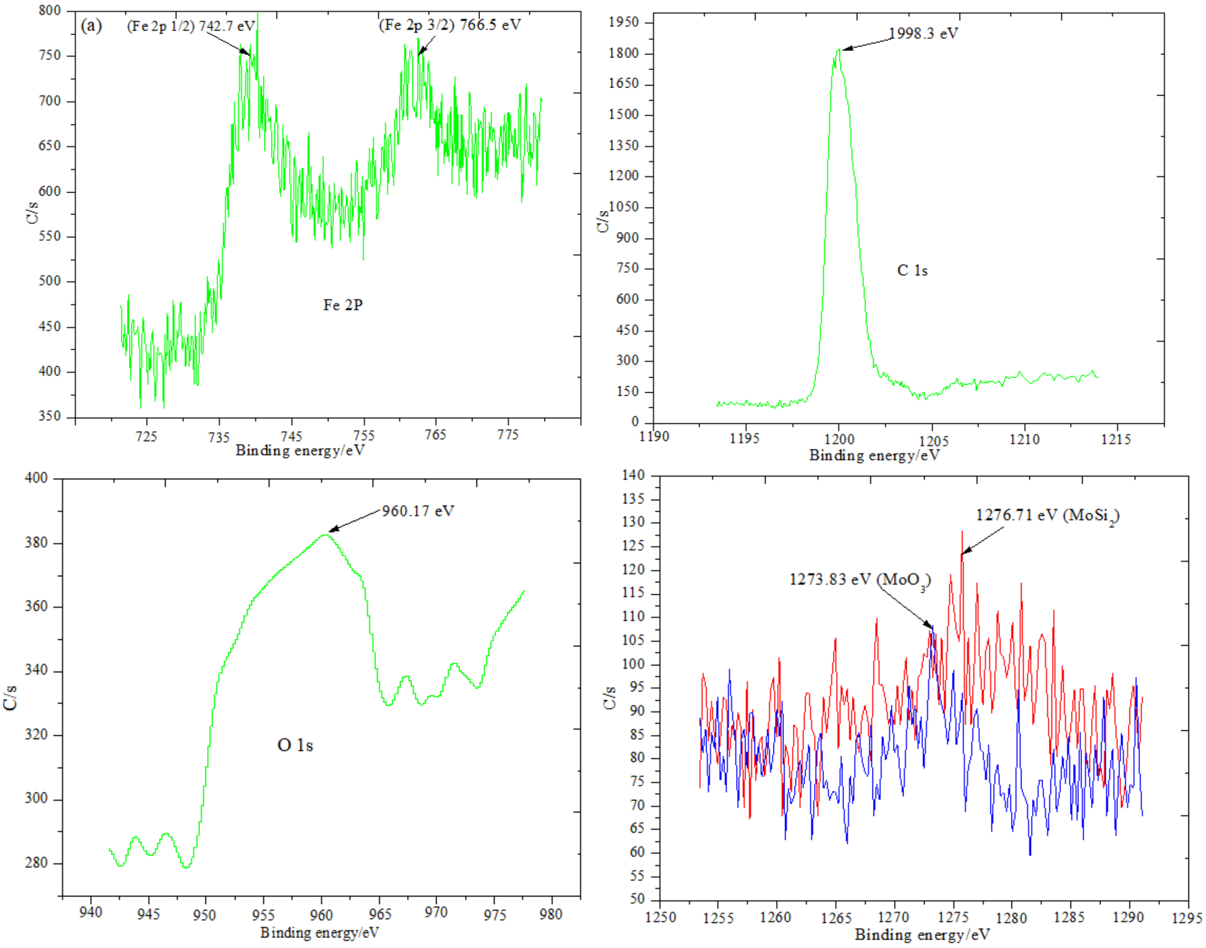


Fig. 9: XPS Analysis of worn steel lubricated surface with EC-CNTs particles blends.

3.8. The tribology model of the MWCNTs and EC-CNTs lubricants

Fig. 10 briefly depicts the schematic of the tribological mechanisms of the MWCNTs and EC-CNT particles in vegetable rapeseed oil. According to Fig. 10 (a), the main lubrication mechanism occurs when MWCNT particles are added to base oil used as a lubricant additive. This relative sliding of the nanoparticles, including EC-CNTs, results in the establishment of the transfer layer. With close investigation, observed that in all working conditions, MWCNTs penetrate the contact surfaces leading to friction and wear reduction but more effective at lower conditions, thus similar to observation by Qu[28]. Conducting the analysis on EC-CNTs as in Fig. 10 (b), observed that at lower working conditions, the EC-CNTs particles were found more at the front and back of the sliding ball in form of agglomeration, suggesting to due to higher size particles. When increase the working condition, the particles were found more diffused at the contact region owing operation, leading to rapid tribo-film formation. According to XPS analysis, the lubricating processes resulted in the development of the transfer film of EC-CNTs particles and the self-repairing property, which can fill the dents on the worn surface. This proves that EC-CNTs nanoparticles can improve the tribological performance of base lubricant unlike MWCNTs and produced results comparatively with SAE 5W30 benchmark. Additionally, the ball bearings' effect on nanoparticles is linked to greater anti-wear properties[29].

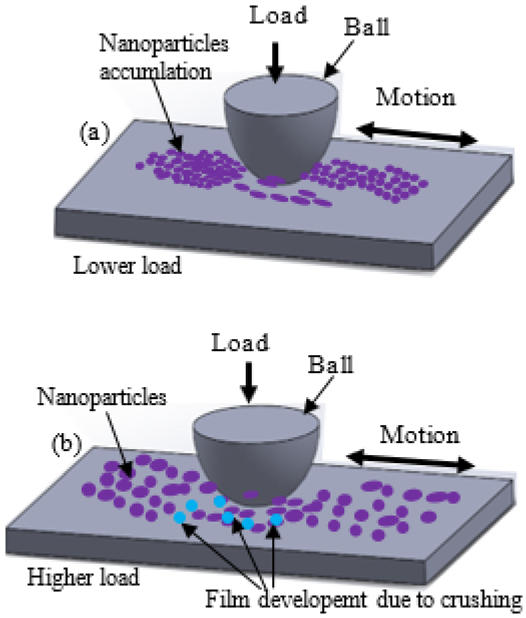


Fig. 10: Tribological mechanism of the additives under low and high working service.

4 Conclusions

The tribological performance of EC-CNT nanoparticles in comparison to commercial MWCNTs was investigated as a lubricant additive base for rapeseed oil. The particles have effective anti-wear properties because of their nanosized, shape, and elemental compositions. The concentration of the additives had a substantial impact on the additives' coefficient of friction and anti-wear effectiveness. All lubricant additives are most effective and had the greatest impact at a concentration of 1 wt.%. Additionally, under more demanding operating conditions, the anti-friction performance of the 1 wt.% EC-CNT nanoparticles outperformed that of commercial SAE 5W 30 and MWCNT nanoparticles. However, at 1.5wt% concentrations of both MWCNTs and EC-CNTs, the friction coefficient and wear values rose. XPS outcome reveals that the good tribological response of the EC-CNTs additives could be from the self-lubricating effect and the tribo-chemical film. The excellent performance of EC-CNTs in reducing wear on the contacting surface was due to amphipathic characteristics, thus leading to fast formation of tribo-film.

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