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Article

Tribological Performance Evaluation of Blended Lubricants Incorporated with Organic Polymer

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Abstract

Eco-friendly lubricants have drawn a lot of interest in the lubrication industry as a way of promoting global sustainability in response to the growing environmental pollution danger posed by the use of petroleum-based lubricants. As a result of these, developing lubricants with organic additives stands as a promising technique in solving the environmental challenges caused by non-degradable materials. This research investigates the effect of bio-based water hyacinth (Eichhornia crassipes) (EC) carboxylmethyl cellulose (CMC) polymer in different base lubricants as well as under different volumetric blend to determine their compatibility effect on lowering friction and wear using base rapeseed oil (BRO) and mineral oil (MO) as a base lubricant sample. High frequency reciprocating rig tribo-tester machine was used in the experiment, followed by substrate surface analysis via energy dispersive x-ray spectroscopy. The additives were evaluated for their potential to improve tribology in terms of friction, surface roughness and wear reduction, load-carrying capacity, and mechanism of repair. Testing the additive concentrations, produced recommendable result at 0.8 mass% EC-CMC. The best performance was obtained when BRO70/MO30 was blended with 0.8 mass% EC-CMC. When compared to base BRO and MO lubricants under 80 N, 0.8 mass% BRO70/MO30 reduced the coefficient of friction and wear scar diameter by 44%, 32%, and 33%, 21% respectively. However, it was shown that nanoparticles had greater tribological performance at higher working capacities owing to the rapid and active tribo-film formation.

Keywords

EC-CMC polymer, mineral oil, rapeseed oil, mechanism, lubrication, friction, and wear

1 Introduction

Concerns over the use of petroleum-based lubricants and inorganic additives and their effects on the environment in recent years have pushed researchers to create ecologically friendly lubricants as a new substitute. Statistics show that between 5 and 10 million tonnes of petroleum products are released into the environment each year through spills, industrial waste, and marine engine exhaust emissions [1]. With these, if nothing is done to conserve our globe, these problems will have a negative effect on our environment and result in a devastating calamity. Responding to these, researchers have concentrated on the likely alternative strategies since conventional lubricants are frequently combustible and the contaminated waste that results from their disposal from end products contains dangerous materials [2, 3]. As a result, there are severe regulations implemented by environmental authorities compelling manufacturers to take the environmental impacts into consideration as a novel and important issue [4, 5]. In this regard, it is considered that vegetable oil derived from agricultural feedstocks would provide a very attractive replacement for the traditional lubricating oil made of petroleum. This is owing to their biodegradability, low toxicity, superior lubricity, high viscosity index (VI), and high flash point, vegetable oils have demonstrated a significant promise for use as lubricants [6, 7]. To make the formulation greener, researchers also suggested using bio components in both the base lubricant and the additives [8, 9].

Vegetable oils have been the subject of numerous research regarding their use in lubricating processes, however not deemed acceptable for direct use as lubricants due to a variety of drawbacks, such as poor thermo-oxidative stability and low-temperature reactivity [7, 9, 10]. Previous researchers have conducted extensive research on ways to mitigate the limitations of vegetable oils by modifying them chemically and adding chemical additives such as antioxidants [11], pour point depressants [12], and viscosity modifiers [13]. Zulhanafi et al.,[14], conducted enhancement of performances of vegetable lubricant using Tertiary-Butyl-Hydroquinone (TBHQ) additive. The findings showed that the inclusion of TBHQ reduced the coefficient of friction and produced a surface roughness that was smooth. Hassan et al., [15] conducted lubrication investigation on blended lubricants using deodorized (RBD) palm olein and mineral oil. The research found that the RBD palm olein blend (E53.11/RB46.89) performed admirably as a bio-lubricant and might potentially serve as a complete bio-lubricant due to the lack of detrimental effects on wear.

In tribology, viscosity is regarded as a crucial characteristic of lubricating oil. It may have an impact on the quantity of film thickness produced between two moving contacts, which may have an impact on the level of wear and friction. A lubricant's inability to produce a thick enough film due to its low viscosity can result in certain asperities from two moving surfaces coming into contact, which will increase friction and wear as well as fuel consumption [16]. Dandan et al.,[17] employed ethylene-vinyl acetate copolymer (EVA) as viscosity improver in base lubricant. The results of the experiments showed that 4% of EVA copolymer is the best concentration for increasing the friction-reducing qualities and anti-wear performance since film thickness is formed between two surfaces in contact.

Although research has already been conducted to assess the viability of viscosity modifiers for enhancing lubricants viscosity characteristics [17]. However, its implications on the tribological performance of lubricating oil in blends with organic polymer [18] has not been fully investigated. Therefore, the purpose of this research is to examine the tribological effectiveness of Eichhornia crassipes carboxymethyl cellulose (EC-CMC) polymer as a viscosity improver in blended base lubricants. Adoption of this EC-CMC was based on the previous response in lubrication [19], and its ecofriendly nature, expected to contribute in solving global pollution effects.

2 Materials and method

The bio-polymer utilized in this analysis was formulated in Universiti Teknologi Malaysia in the Department mechanical Engineering, Tribological option [20] using bio-plant of Eichhornia Crassipes aquatic plant obtained from Nigeria, West Africa. Other materials used were pure base rapeseed oil (BRO), pure white mineral oil (Dchemie product), tribo-pairs (steel flat and ball and flat). Chemicals like; NaCl (25%), deionized water, 0.05% acetic acid, aqueous NaOH solution (5%), isobutyl, and isopropyl were also utilized, thus acquired from Sigma Aldrich company in Malaysia. Due to its direct refinement from crude oil with a Sulfur content of only 0.03 % and a viscosity index range of 80 to 120, the mineral oil belongs to Group II.

2.1 Biopolymer and lubricant formulation

A ball milling device (Retsch, PM 100, Germany) was used during the development of the EC-CMC polymer to bring down the EC material into nanoscale size, employing the EC plant's root (25 g) and stem (25 g) in a 1:1 ratio to create EC-NPs. To prevent contamination during the EC-CMC polymer synthesis, non-cellulose components like lignin and hemicellulose were removed from the EC-NPs. This was accomplished by blending 25 g of EC-NPs stem and root with 15 ml of 15% aqueous NaOH solution and letting the mixture sit at room temperature for one hour. As a result of the impurities being eliminated, the cellulose molecules can correctly combine during the successive hydrolysis strength treatment (breaking a link and adding the hydrogen cation and the water's hydroxide anion).

Following the procedure described by Opia et al., 2021), the dry produced sample was mixed with 10% NaOH, 5 ml of isobutyl, and 20 ml of isopropyl [21], and stirred for 6 h at room temperature, thus, EC-CMC polymer product was subsequently created. The image of EC-CMC polymer is shown in Fig. 1. To determine their tribological strength with organic polymer, the base lubricants were prepared as follows: neat base rapeseed oil (BRO) alone, pure base mineral oil (MO) alone, blends of EC-CMC polymer with various base lubricants, and blends of EC-CMC polymer with the base lubricants at different volumetric mixing blending ratios (30%, 50%, 70%). These blends are shown in Table 1 with the various volumetric ratios and formulated EC-CMC polymer presented in Fig. 2. The lubricant formulation with EC-CMC indicated a homogeneous

Fig. 1 Image showing the nature of formulated EC-CMC polymer used in the study

Table 1 Lubricant samples under different blended ratio

No	Lubricant type	Lubricant code	Blending ratio (%)
1	Pure rapeseed oil	BRO100	Mineral oil (0%) + BRO 100%
2	Pure mineral oil	MO100	BRO (0%) + MO 100%
3	Blend	BRO70/MO30	BRO (70%) + MO (30%)
4	Blend	BRO50/MO50	BRO (50%) + MO (50%)
5	Blend	BRO30/MO70	BRO (30%) + MO (70%)

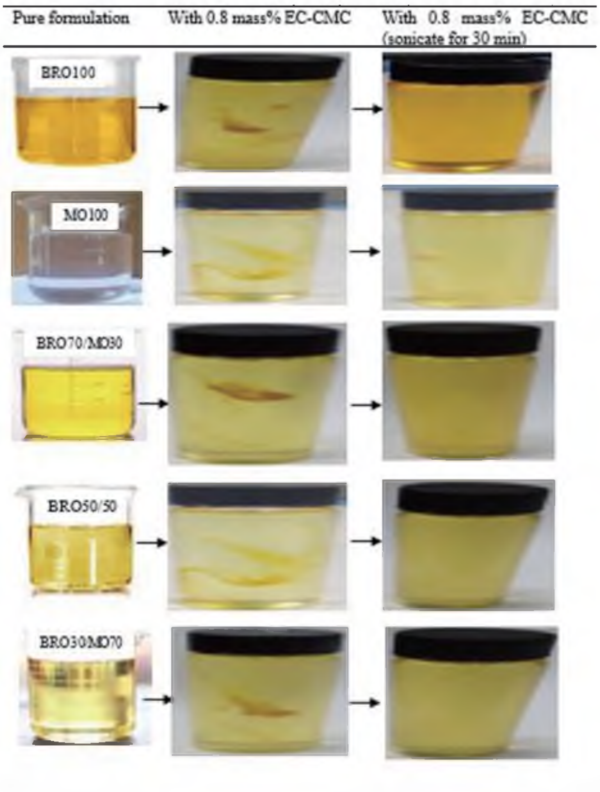

Fig. 2 Images of the various lubricants used in this study

solution when sonicated for 30 min. According to Kumar et al., [22] sonication technique contribute in lubricant performance enhancement, thus enables lubricants to achieve good stability for long period of time without particles accumulations.

2.2 Analytical tools

Using a Transmission electron Microscopy (SEM, JEOL-6100, JOEL Co., Japan) incorporated with EDX (EDX, Energy 350, Oxford, UK), energy dispersive X-ray spectroscopy, accordingly for detection of the morphology and elemental components of EC-CMC polymers were examined. A dynamic light scattering (DLS) particle size analyzer was used to measure the particle size distribution (Zetasizer Nano ZS; Malvern instrument, United Kingdom). A functional group analysis using Fourier-transform infrared (FT-IR) spectroscopy was conducted to determine the functional groups and the compatibility of the EC-CMC polymer and used lubricant. The TGA analysis of EC-CMC, BRO + EC-CMC, BRO + EC-CMC and MO oil was carried out to evaluate the thermal strength or changes in samples material mass. This was done over time at a range of defined temperatures depending on sample decomposition using a predetermined heating rate in a controlled setting. The study used tribo flat mass change before and after testing to determine wear loss. At the conclusion of the testing, the wear scar volume and depth are provided using an atomic force microscope (AFM) (Park NX 10, USA), while the chemical composition at the worn surfaces, an X-ray photoelectron spectroscopy (XPS) machine was employed.

2.3 Viscometric analysis of the various lubricants

A viscometer (Cole-Palmer, USA) was used to test the kinematic viscosity of rapeseed oil without and with the addition of EC-CMC in accordance with ASTM D-445 and ASTM D-446. Measurements were made at 5-unit intervals from room temperature (RT) 40°C to high temperature (HT) 150°C. A viscometer with a heater and spindle was put into the lubricant under test according to Salaji & Jayadas, 2021). As the temperature increased, the setup measured and tracked the viscosity value via the spindle [12]. Separately, 80 ml of BRO and pure MO were combined with 0.3 mass%, 0.6 mass%, 0.8 mass%, and 1 mass% EC-CMC. This mixture was then thoroughly mixed for 30 minutes using ultra-homogenizer (IKA 25, ULTRA-TURRAX, USA) blender set to a programmed rotating speed of 1500 rpm to ensure homogeneous solution.

2.4 Frictional analysis

A ball on flat tribo-tester in agreement with ASTM G133-05 (see Fig. 3) was used to examine the anti-wear behavior of lubricant compositions under various sliding situations. Investigations were done using varying viscosity modifier concentrations (0.3, 0.6, 0.8 and 1 mass%), before finding the optimal concentration. The effectiveness of EC-CMC in base rapeseed and mineral oil as lubricant additives to minimize friction and wear were examined in this study, employing different working conditions as presented in Table 2. Before testing, each sample was sonicated to produce the good stability required for homogeneous solution. Before and after each test, different components (tribo-pairs) for the analysis were cleaned

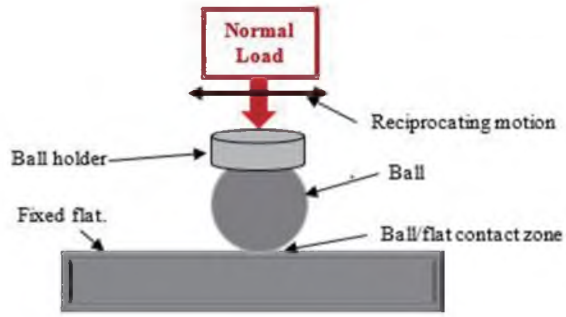

Fig. 3 Schematic presentation of mechanism of high frequency reciprocating rig tribo-tester

Table 2 Experiment parameters

Parameters	Name/value
Lubricant samples	Base rapeseed oil
	Pure mineral oil
	BRO70/MO30 BRO50/MO50 BRO30/MO70
Additive concentrations	0.3, 0.6, 0.8 and 1 mass%
Load (N)	40, 60, 80, 100
Temperature (°C)	75
Stroke	10 cm
Operation duration	15 (min)
Friction Pairs	Ball Ra < 20 nm roughness Flat Ra < 200 nm roughness
Frequency	5 Hz
Ball	Steel ball
Flat	stainless steel flat
Contact pressure	798 MPa
Ball size (mm)	12
Flat mm	40 by 40

using an ultrasonic cleaning agent such heptane. Since EC-CMC are amphiphilic, at the proper concentration, can dissolve in lubricant [23].

The coefficient of friction (COF) was recorded by the tribometer (Windocum 2010) instrument during testing and extracted using Excel software. Friction tests were carried out three times for each sample to ensure that the trials could be reproducible, and the mean average values were taken for graph plotting. After the friction research, the lubricated surfaces underwent wear analysis using the surface profile-meter 150 stylus before applying other machines as stated in section 2.3 (analytical tools).

3 Results and discussion

3.1 Formulations morphology and particles size characterization

Figure 4 displays SEM representations of the EC-CMC's morphology of high resolution. The image shows that the EC-CMC sample had a gel-like appearance. This outcome suggests that the formulas were successful. The elements identified in the samples in Table 3 are shown by the EDX. The data from the earlier study was like the elements mentioned [24, 25]. These outcomes were supported since water hyacinth has been identified as a viable source for phytoremediation [26].

Figure 5 displays the distribution size particles under a dynamic light scattering (DLS) particle size machine. The results demonstrate that the EC-CMC polymer disperses most effectively in polar and non-polar N-Methyl-2-pyrrolidone solutions, with average mean diameters of 82.7 nm, respectively.

3.2 FT-IR spectroscopy and TGA analysis

Using FT-IR analysis, Fig. 6 illustrates the functional groups present in the EC-CMC polymer, BRO blended BRO, MO, and BRO + MO + EC-CMC. The spectra of the samples show aliphatic stretching bands CH_3 and CH_2 at 2907 cm^{-1} and

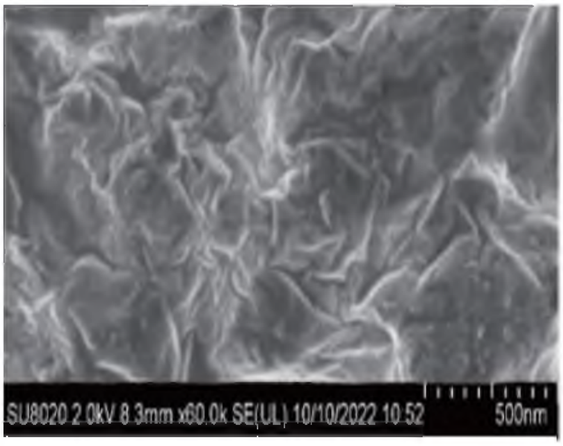

Fig. 4 SEM analysis of EC-CMC polymer

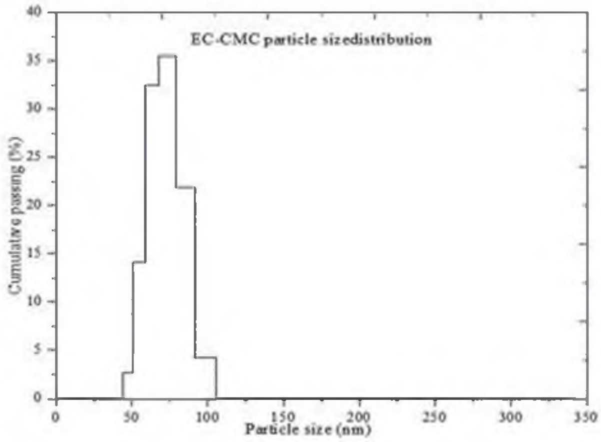

Fig. 5 Particle size analysis of the EC-CMC additive

Table 3 Elemental distribution in EC-CMC polymer under EDX analysis

Material/Elements (mass%)	C	O	Mo	Ca	Si	Cu	K	Cl	Al	Mg
EC-CMC polymer	56.2	20.1	3.4	3.1	8.3	1.3	1.8	2.1	2.2	1.3

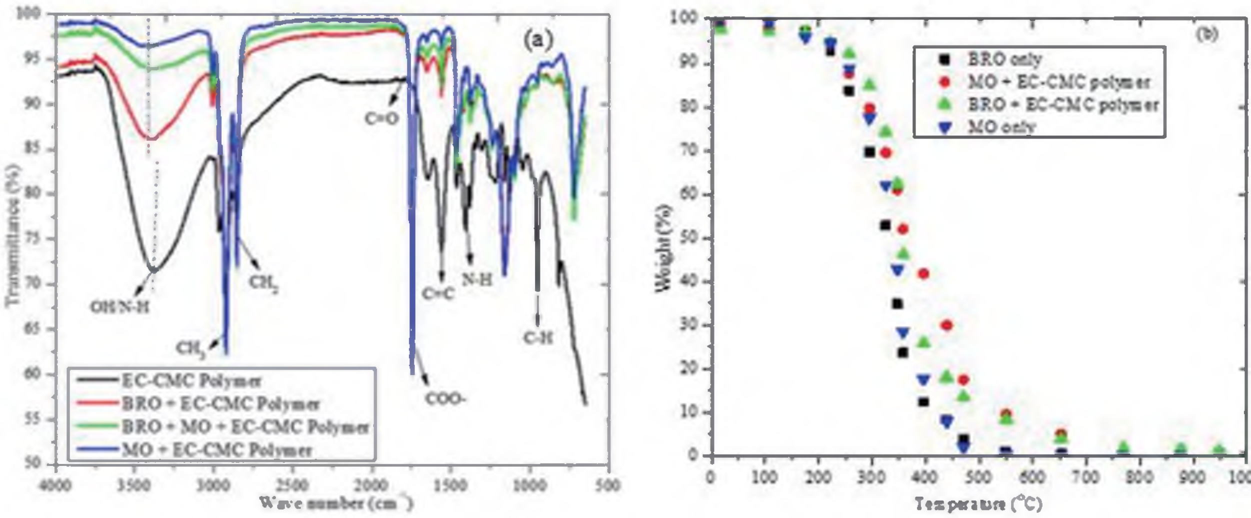

Fig. 6 Presentation of FT-IR analysis (a) and TGA (b) of the EC-CMC blended lubricants

2799 cm^{-1} and functional group deformation bands for methyl and methylene CH_3 and CH_2 at 1699 and 1480 cm^{-1}. When comparing the behaviors and functional groups in the spectra of the different samples, an enormous and powerful band at about 3345 cm^{-1} was found in the EC-CMC and close to sample of BRO mix EC-CMC spectra because of the O-H/N-H group. The investigation noticed shrink when using BRO+ MO and EC-CMC blend MO. The hydroxyl group (OH) is associated with a rocking vibration in the EC-CMC spectra at the 3720 cm^{-1} band, which was insufficient for other samples and caused peak shift. The EC-CMC blended lubricants exhibit greater stretched aliphatic bands CH_3 + CH_2 at 2907 cm^{-1} and 2799 cm^{-1} in their absorption bands. This is because, at ambient temperature and without external influence, the propagation of oil monomers components inside the polymer chains had a little difference in the structure [27].

All samples showed a stretching vibration of the carbonyl group C=O on only the EC-CMC sample, clearly displayed the curve at 2187 cm^{-1}. But every other sample showed a considerable shift, which was attributed to a decrease in the C=C group brought on by the introduction of base lubricants and the identification of various molecular components. Peaks at 1338 cm^{-1} and 997 cm^{-1} were developed because of the N-H group's high vibration and its shared vibration with the C-H group when using EC-CMC, although other samples showed strong shifts indicating the generation of new bound in diverse novel formulations. The newly developed spectra from those samples and those of newly discovered peaks that are comparable to one another clarify the compatibility between EC-CMC and base oil lubricant. Besides, it demonstrates the polymer molecules expand as the bulk of the micelles rises, which improves viscosity. The observations were like previous presentation reports [27].

Using TGA analysis as presented in Fig. 6 (b), the EC-CMC's efficacy was evaluated. This is done to determine the thermal resistance of polymers when added to lubricants. The values were taken from the pronounced point of weight loss (%). During the TGA calculations on the samples, temperatures of 200°C and 350°C were taken based on the pronounced degradation effect on the curve. Weight loss of 38% and 27%, respectively, were shown by the examination of the base of BRO and MO that was chosen. The weight loss was considerably reduced when combined with EC-CMC, falling to 21%, 16.6%, and 18.1% with EC-CMC + MO, EC-CMC + BRO, and EC-CMC + BRO + MO, respectively. When compared with pure lubricants, the drop changes indicate that the inclusion of EC-CMC positively reduced the temperature effect on the formulated EC-CMC blended lubricants. Compared with EC-CMC blended MO, the weight loss of EC-CMC blend BRO is visibly lower, which may be attributed to the organic product showing good compatibility leading to improved thermal properties. The similar analysis and observation was found by Bhadra et al., [28].

3.3 Viscosity properties

Figure 7 (a) contrasts the kinematic viscosities of BRO and MO with various additive concentrations at temperatures ranging from 40 to 100°C, and Fig. 7 (b) displays the corresponding viscosity index. The image serves as an example of how rising temperatures cause the kinematic viscosity of all the tested lubricants to decrease. Additionally, other researchers achieved a similar result involving viscosity reduction through temperature increases [29, 30]. In addition, the findings suggest that under greater temperatures, a comparison of all oils' viscosity amounts reveals to be all equivalent to one another. The maximum kinematic viscosity value at 40°C was 43.3 cSt, obtained for BRO + 0.8 mass% EC-CMC, compared with BRO at 38.8 cSt, while the highest under the MO mix was 35.2 cSt, obtained for MO + 0.8 mass% EC-CMC, compared with MO at 28.4 cSt. When evaluating viscosity index, it could be seen that the highest value under MO and BRO blends was 441.6 and 399.3 which was found with 0.8 w% EC-CMC, respectively, as against MO of 231.7, and BRO of 197.3. With values of 197.3 and 231.7, respectively, BRO and MO produced the lowest overall viscosity index (VI). This is obviously correlated with the findings of the kinematic viscosity performance investigation, which demonstrate that the optimal concentration of EC-CMC is 0.8 mass% producing more homogeneous solution than 1.0 mass% EC-CMC.

3.4 Friction and wear reducing characteristics

Reduction in friction and surface roughness influence of the EC-CMC additive (0.3 mass%, 0.6 mass%, 0.8 mass%, and 1 mass%) compared with base MO and BRO lubricants were

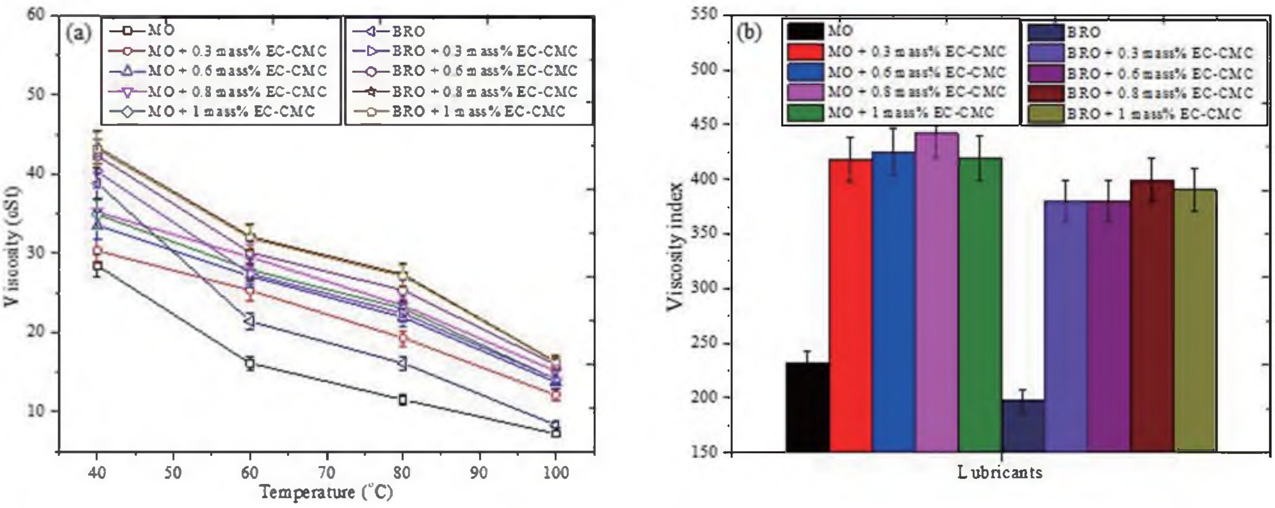

Fig. 7 Variation of viscosity against temperature (a) and viscosity index form the various lubricants (b) (40-100°C)

studied at different loads under a steady temperature to gain more insight into the additive behavior during testing, as demonstrated in Fig. 8 (a and b). The analysis found that the average COF and flat WSD increased but reduced significantly in comparison to both base lubricants results when the load increased from 40 to 100 N. Nevertheless, the reduction in COF and WSD under the additive inclusion alternate in nature depending on the concentration. The study shows that the MO lubricants revealed a similar trend while BRO yielded similar behavior as shown in Fig. 8 (a).

The investigation observed that BRO + 0.8 mass% EC-CMC produced the smallest COF value (0.07731), while MO + 0.3 mass% EC-CMC yielded the highest COF value (0.1200) under 80 N and 100 N, respectively. The study discovered complexities of nanoparticles dispersing into the contact zone under low operating condition because of inability to generate frictional energy leading to high COF. At loads of 40, 80 and 100 N the friction coefficient of base MO and BRO were 0.099, 0.120, 0.126 and 0.120, 0.121, 0.124, respectively. This yielded reductions of 5.8%, 24.3%, 21.5% and 16.1%, 36.3%, 29.2%; under 0.8 mass% EC-CMC as the best concentration. The poor reduction recorded with MO could be due to incompatibility between the additive molecules, possibly having high molecular weight when blended with MO, resulting in poor tribo-film formation and a high COF.

A lubricant's efficiency is determined not only by its low COF when in use but also by its capacity to reduce surface roughness (Ra) under profilometer equipment. The lower steel flat, which was lubricated with several lubricants, was subjected to analysis. With varying concentration of additives against base oil, the wear surface roughness reduction was found between 33.5% to 7.5%. As concentration and load increase, performance improves in comparison to base lubricants, as seen in Fig. 8(b).

However, 0.8 mass% EC-CMC produced the greatest performance for both MO and BRO blends. The calculated Ra under loads of 40, 60, 80, and 100 N for MO and BRO were 0.797, 0.809, 0.861, 0.918 μm and 0.688, 0.703, 0.771, 0.893 μm, respectively. Lack of EC-CMC polymer's anti-wear properties could be the cause of the development of poor reduction values. As a result, the study determined that a concentration of 0.8 mass% EC-CMC was the best for minimizing friction and wear. It was chosen as the best candidate lubricant additive in this investigation and will be used in further analysis.

Applying the optimal concentration of 0.8 mass% EC-CMC, a comparison for reduction on friction coefficient values and wear scar diameter for blended BRO and MO under different volumes was conducted as shown in Fig. 9 (a and b). Based on Fig. 9 (a), it can be observed that COF of the additives blended samples decreases as time of operation increases. Due to the bio-lubricants' long chain fatty acids [12] and esters, which are described as surface active materials, the blending lubricants lowered the friction coefficient value more than the neat rapeseed and mineral lubricants.

As the loads are increased from 40 to 100 N, the oil film's hydrodynamic lifting effect becomes more significant. At this point, the body to body sliding contact is minimized, experiencing thin film formation, thus similar to previous conducted research [31]. The blended lubricants exhibited similar trends compared with pure tested lubricants. The COF for BRO was 0.121, while MO yielded 0.120 under 80N and 75°C. Under application of 0.8 mass% additives, the research observed improvement in COF reduction as the load increases compared with base lubricants as shown in Fig. 9 (a). The analysis revealed that BRO70/MO30 (0.062) yielded the lowest COF followed by BRO50/MO50 (0.068) before BRO30/MO70 (0.087). This reduction COF by 48.9%, 43.4%, 28% against BRO and 48.4%, 43%, 27.4% against MO, respectively.

Analyzing the surface protective impact of 0.08 mass% EC-CMC on the substrate, as presented in Fig. 9 (b). The investigation revealed that inclusion of additive gives a significant reduction on the wear on the lubricated surface but varies on concentrations. The WSD from BRO was 673 μm while MO recorded 597 μm. The study shows that additive wear reduction under BRO were higher than that of MO, suggesting fatty acid presence leading to faster surface mending than MO. This proves the analysis conducted under vegetable over fatty acids activities on film formation (Aravind et al., 2015; Kumar et al., 2018; Lundgren et al., 2018). The wear scar reduction for BRO70/MO30, BRO50/MO50 and BRO30/MO70 were 36.6%, 29.1% and 27% against BRO, whiles 28.5%, 20.1% and 17.8% were produced against MO. This demonstrated that the application of 0.8 mass% BRO70/MO30 gives maximum element protection when used in lubrication.

Figure 10 depicts the average COF and wear loss under different loads tested using temperature of 75°C. Testing with

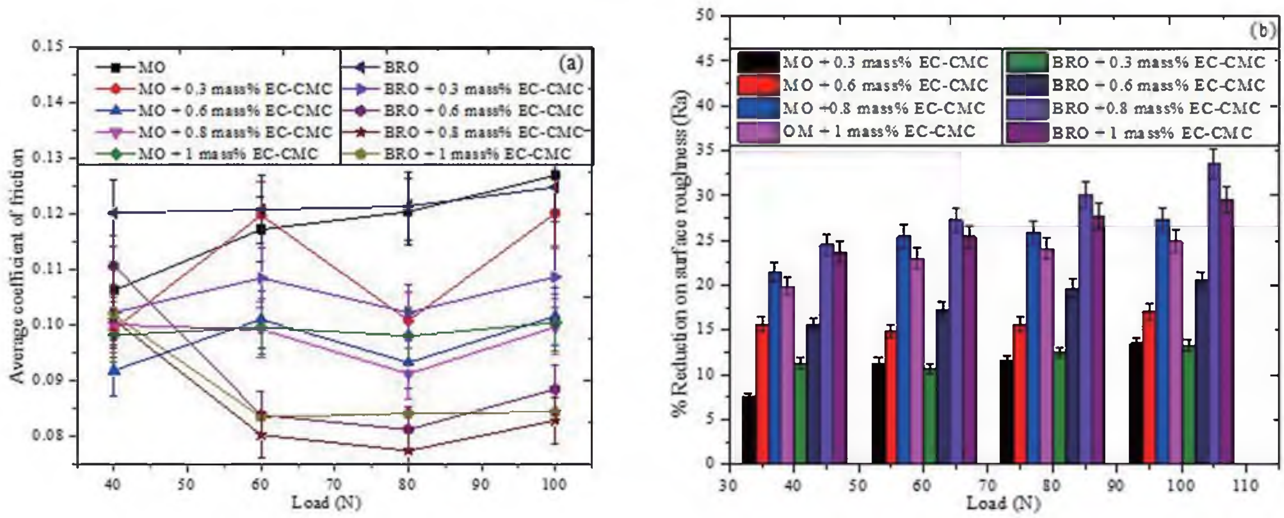

Fig. 8 Average COF (a) and surface roughness reduction (%) (b) (under 40-100 N, 75°C)

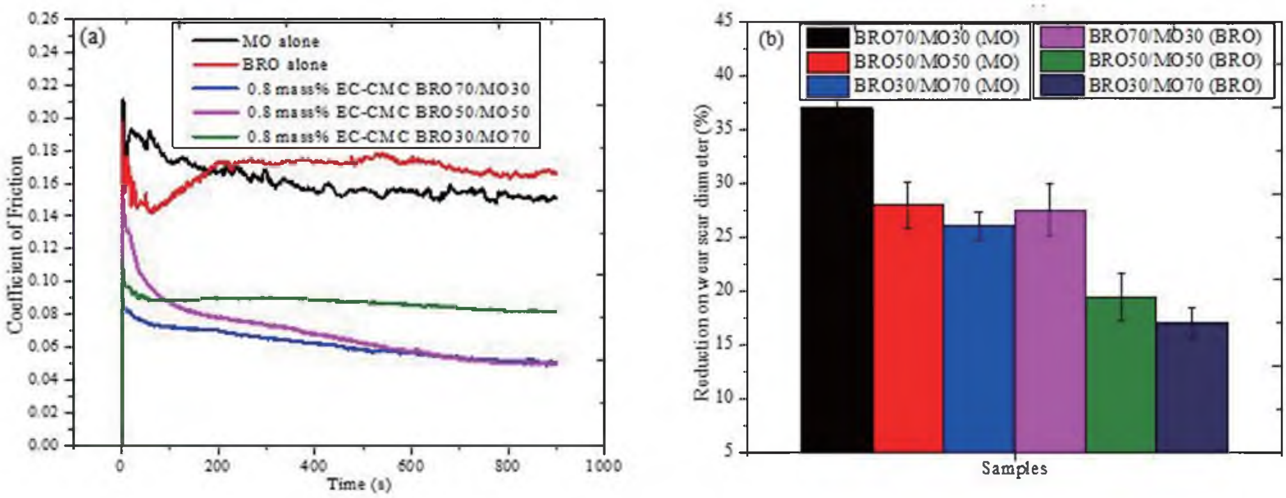

Fig. 9 Reduction in COF (a) and wear scar reduction (%) (b) (under 80 N, 75°C)

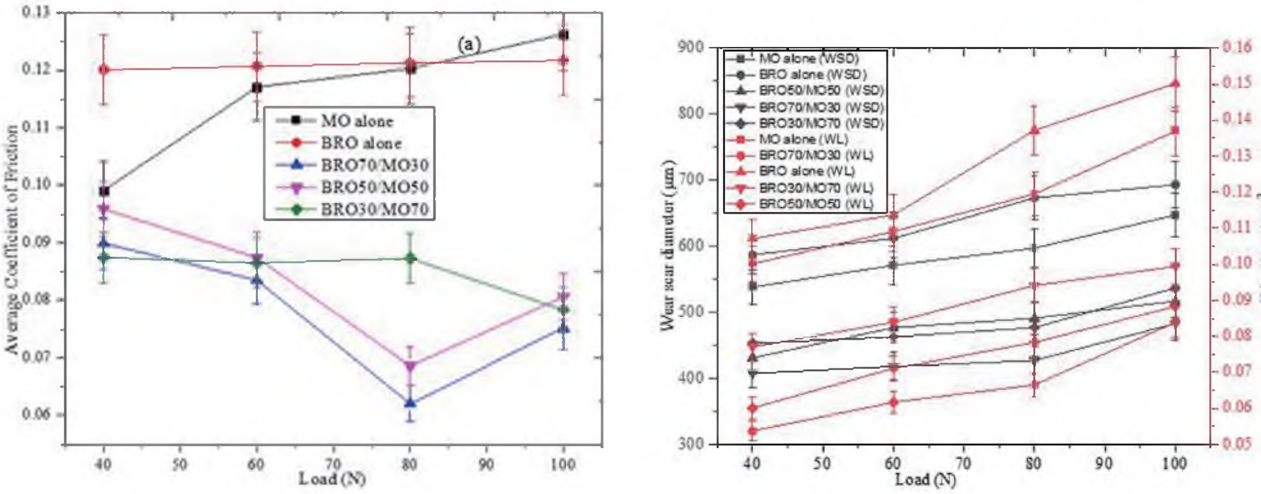

Fig. 10 Average COF (a) and wear loss reduction (%) (b) (under 40-100 N, 75°C)

BRO and MO produces the maximum COF in all the conditions examined, showing substantial reduction in COF with usage of 0.8 mass% EC-CMC additive as illustrated in Fig. 10 (a). The graph of the average COF under additive blended lubricants demonstrated the same trend decreases as load increases but showcase opposite at the highest load of 100 N, except that of BRO30/MO70. This shows that with much rapeseed oil with EC-CMC additive, rapid and active tribo-film could be generated leading to friction reduction. The outcome of the various lubricants revealed that 0.8 mass% BRO70/MO30 under 80 N yielded the best result. This result correlates with previous results (Fig. 8), revealing that maximum frictional energy with best tribo-chemistry due generated under 80 N application. The various average percentage COF reduction are presented in Table 4.

Under wear scar diameter analysis as shown in Fig. 10 (b), application of EC-CMC demonstrated surface protective effect, however, the effect was higher when used with BRO, indicating good tribo-chemistry leading to formation of active film. The wear values from the base lubricants as well as the percentage reductions from the use of various formulations are presented in Table 4. In conducting analysis on wear loss (WL) from the plat sample used shows that utilization of EC-CMC significantly protects the material surfaces from abnormal wear as presented in Fig. 10 (b). As shown in graph Fig. 10 (b), the wear loss from pure lubricants was far from additive blended samples indicating direct contact between the sliding tribo-pairs. The graph with additives gives a similar trend, revealing the protective effectiveness of the EC-CMC. The various percentages of wear losses from the different lubricants are presented in Table 4.

3.5 Wear surface study of flat samples

Figure 11 shows the SEM morphology of flat specimens of various volume blends of rapeseed and mineral oil. The image shows different abrasive wear forms, such as varying scar depths and uneven grooves found on the tested flats' surfaces. The inclusion of 0.8 mass% EC-CMC reduced the diametric wear scar value more than the neat BRO and MO. The image shows diverse abrasive wear forms, including varying scar depths and imbalanced grooves that were found on the surfaces of the tested flats. Based on Fig. 11 (a), the wear scars for base lubricants demonstrated higher visible scars but more pronounced under BRO. Again, while mineral oil had a wear scar, but more noticeable than BRO, which suggests a very little presence of tribo-film throughout the operation. The tribo-

Table 4 Average percentage reduction (%) of the various lubricants on COF, WSD and WL

Tribological parameters	Samples	Reference parameter	Load (N)			
			40	60	80	100
COF	BRO		0.1201	0.1207	0.1213	0.1217
	MO		0.0991	0.1171	0.1203	0.1263
COF reduction (%)	BRO70/MO30	Against BRO	25.0%	30.8%	48.9%	38.2%
	BRO50/MO50		20.0%	27.6%	43.4%	33.8%
	BRO30/MO70		27.2%	28.3%	28%	35.7%
	BRO70/MO30	Against MO	9.3%	28.6%	48.4%	40.5%
	BRO50/MO50		3.2%	25.3%	43.0%	36.2%
	BRO30/MO70		11.8%	26.1%	27.4%	38.0%
WSD (μm)	BRO		587 μm	612 μm	673 μm	693 μm
	MO		538 μm	571 μm	597 μm	647 μm
Wear scar reduction (%)	BRO70/MO30	Against BRO	30.7%	31.7%	36.6%	30.3%
	BRO50/MO50		26.6%	22.1%	27.0%	25.4%
	BRO30/MO70		22.8%	24.3%	29.1%	22.5%
	BRO70/MO30	Against MO	24.0%	26.8%	28.5%	25.3%
	BRO50/MO50		19.9%	16.5%	17.8%	20.1%
	BRO30/MO70		15.8%	18.9%	20.1%	17.0%
Wear loss (mm^3)	BRO		0.107 mm^3	0.113 mm^3	0.137 mm^3	0.150 mm^3
	MO		0.100 mm^3	0.109 mm^3	0.119 mm^3	0.136 mm^3
Wear loss reduction (%)	BRO70/MO30	Against BRO	49.0%	45.7%	51.5%	44.1%
	BRO50/MO50		43.8 %	37.5 %	42.9 %	41.1%
	BRO30/MO70		28.0%	26.2 %	31.4 %	33.8%
	BRO70/MO30	Against MO	46.4%	43.4%	44.4%	38.7%
	BRO50/MO50		39.9%	34.8%	34.5%	35.5%
	BRO30/MO70		22.9%	23.1%	21.6 5%	27.4%

film mending effect from EC-CMC is demonstrated in Fig. 11. As shown in Fig. 11 (c), (d), and (e)), respectively, the optimal performance was under the conditions of BRO70/MO30, followed by BRO50/MO50, and then BRO30/MO70. Because of the rough particulate material that resulted in abrasion wear, parallel grooves may be seen alongside various depths on the roughened surface [34].

According to the viscometric study (Fig. 7 (b)), the notion that BRO created larger wear scars was caused by direct contact that was detected since tribo-film development was not possible. The reduction on the use of EC-CMC additives shows the tribo-film formation ability of EC-CMC leading to the separation of the sliding body from direct contact unlike outcome of pure BRO and MO. Also suggested to be from the excellent value of viscosity index under 0.8 mass% EC-CMC blends, and above that, the fatty acid which could cover the sliding contact region fully. The lowest amount of the diametric wear scar was 408.4 μm identified in BRO70/MO30, followed by BRO50/MO50 of before BRO30/MO70 compared with pure BRO of 660.8 μm and MO of 546.6 μm.

By using EDX analysis, it was discovered that the lubricated surface with BRO and MO included less elements, demonstrated the absence of tribo-film formation during operation according to Abdel-Rehim [35]. Lubricated subtract revealed more components and decreased the Fe percentage as seen under pure lubricants lubrications when evaluated with 0.8 mass% EC-CMC blended BRO/MO. More elements meant that the substrate surfaces were protected from direct contact, lowering COF and WSD, which is consistent with the findings on Figs. 9 and 10. In Table 5, the EDX outcomes from the various lubricants are displayed.

The AFM 2D images a, b, c, d, e and the surface graph profile (a1, b1, c1, d1, e1) were utilized to examine the wear effect on the tested substrate surface lubricated with different formulations under 80 N and tempt. of 75°C, as demonstrated in Fig. 12. The morphology of 2D surface pictures defined the lubricant's effectiveness to protect the surface throughout lubrication.

The application of 0.8 mass% EC-CMC reduced wear compared with base lubricants. In Fig. 12 (a, b and a1, b1), the wear images of the base lubricant was rougher with numbers of ploughing tracks and abrasive wear process was observed compared with tests conducted using 0.8 mass% EC-CMC BRO/MO. The AFM revealed that the BRO shows similar features with that of MO, however, the grooves were shallower with MO. Nevertheless, 0.8 mass% EC-CMC BRO70/MO30 exhibited lowest wear depth followed by BRO50/MO50 before BRO03/MO70 as presented in Fig. 12 (c-c1, d-d1, e-e1). As a result, the lubricated surface with base lubricants wear depth of 199.7 nm (BRO) and 183.5 nm (MO) when calculated from the zero reference point as shown with arrows, thus presented in Fig. 12 (a1-e1). This resulted in wear depth reductions from BRO70/MO30, BRO50/MO50, BRO30/MO70 by 82.1%, 53.3%, 39.2%, and 80.5%, 49.2%, 33.8% against BRO and MO, respectively. The findings evidently strengthen the result of Fig. 8, discovered more film on the lubricated surface under 0.8 mass% EC-CMC blend BRO.

4 Lubricated surface chemistry analysis

The chemical composition of the worn surface was identified using XPS analysis in order to investigate the tribo-chemistry mechanism of EC-CMC as an additive in base lubricant towards surface protection and coefficient reduction. The XPS spectra of Fe 2p, C1s, O1s, Si2p, and Mo 3d on the worn surfaces are shown in Fig. 13, after 15 min of lubrication using 0.8 mass%

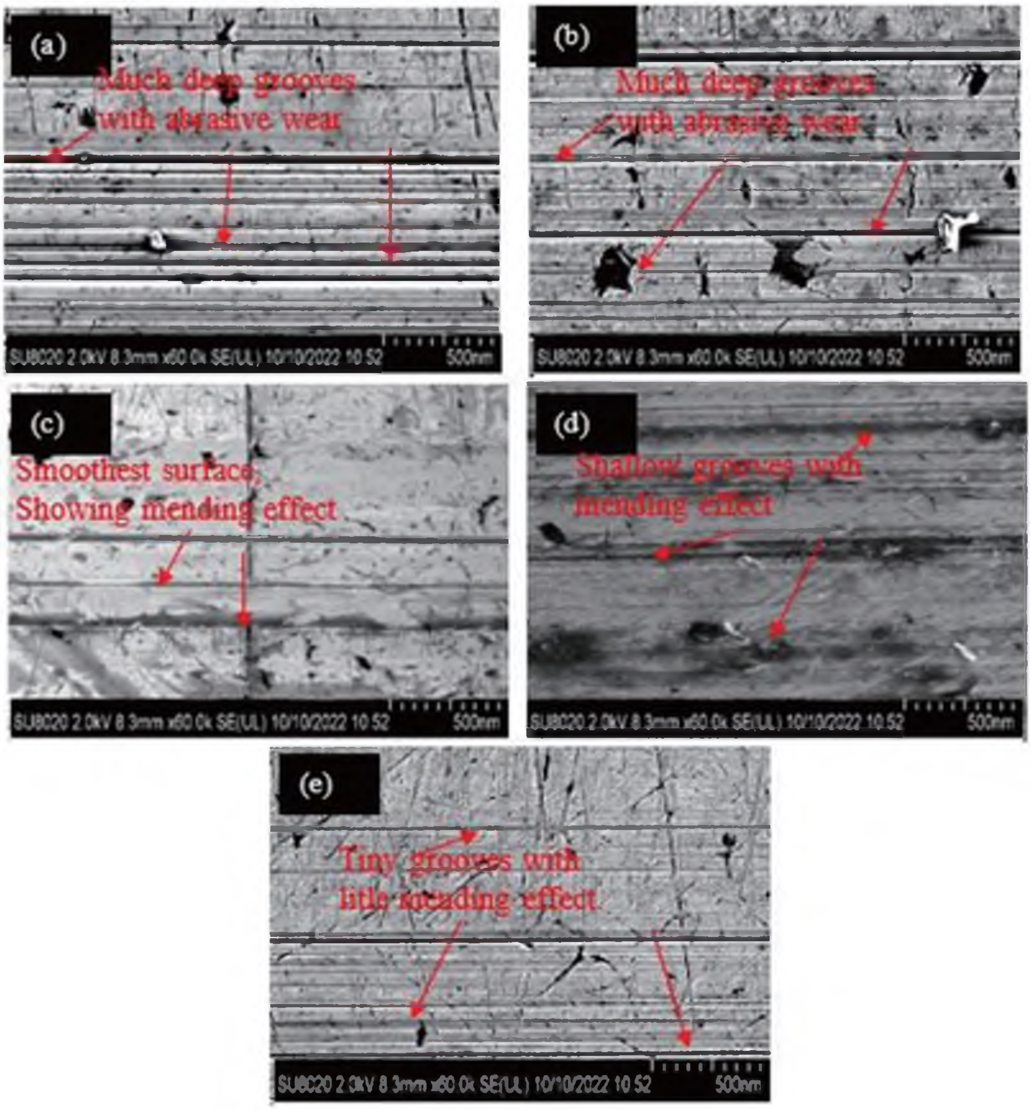

Fig. 11 SEM morphology of the various lubricants lubricated surfaces; BRO, (a); MO, (b); 0.8 mass% EC-CMC blended (BRO70/MO30 (c); BRO50/MO50 (d) and BRO30/MO70 (e)) under applied load of 80 N, 5 Hz, 15 min, 75°C

Table 5 EDX demonstrations on the elements found on different lubricated surfaces

Lubricant samples	Elements									Total (%)
	C	O	Fe	Si	Mo	Ca	P	Na	Mn	
BRO	6.8	5.9	86.7	-	0.3	-	-	-	-	100
MO	6.3	5.4	88.3	-	-	-	-	-	-	100
0.8 mass% EC-CMC BRO70/MO30	38.9	5.1	51.4	1.5	1.3	0.2	0.2	1.1	0.3	100
0.8 mass% EC-CMC BRO50/MO50	34.3	6.3	54.1	2.3	0.9	0.3	-	0.6	0.1	100
0.8 mass% EC-CMC BRO30/MO70	31.9	5.7	59.3	1.6	0.6	0.4	0.1	0.4	-	100

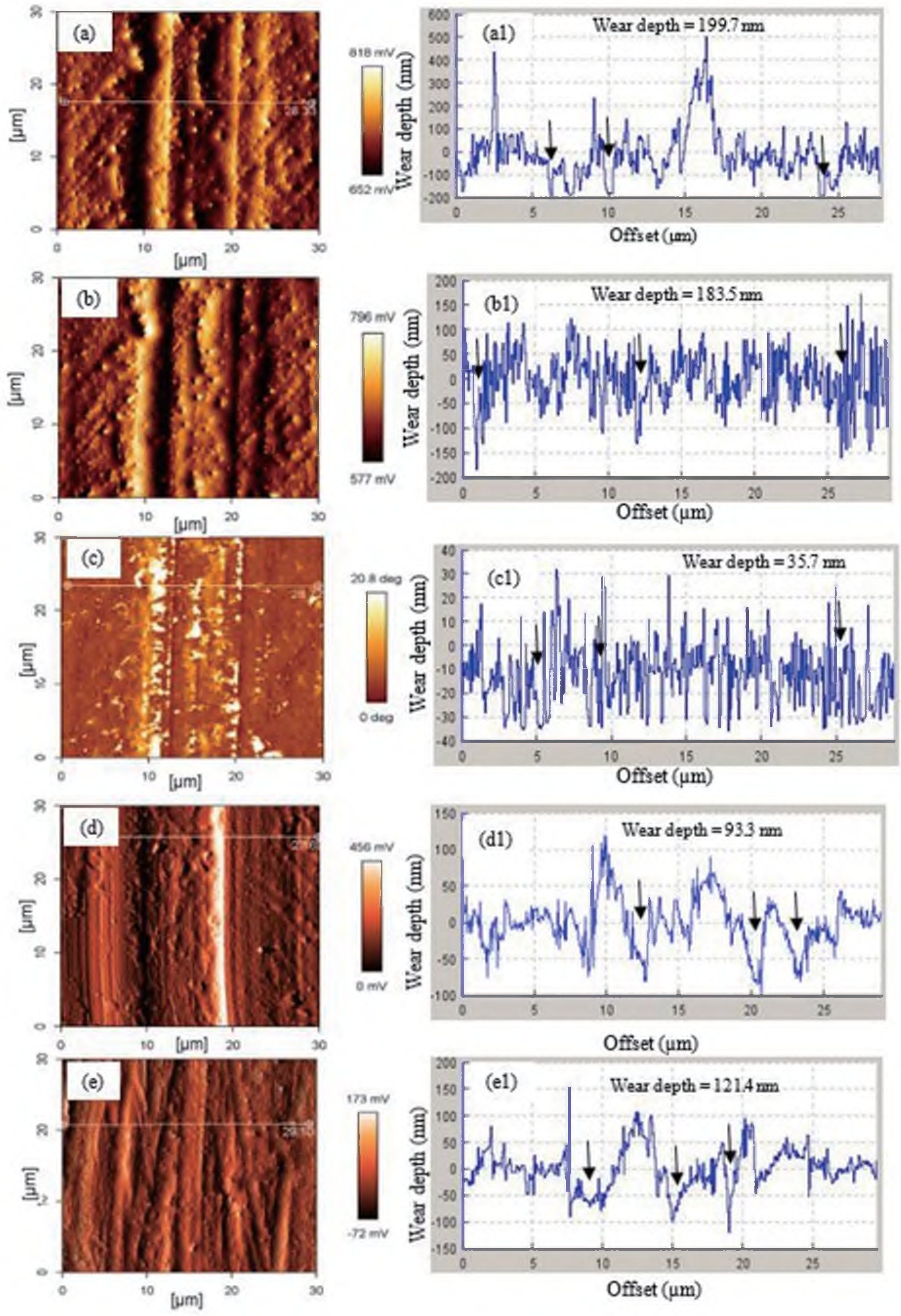

Fig. 12 2D images (a-d) with corresponding micrographs (a1-d1) of various worn surfaces (BRO (a-a1); MO (b-b1) and 0.8 mass% EC-CMC blended (BRO70/MO30 (c-c1); BRO50/MO50 (d-d1) and BRO30/MO70 (e-e1)) under applied load of 80 N, 5 Hz, 15 min, 75°C

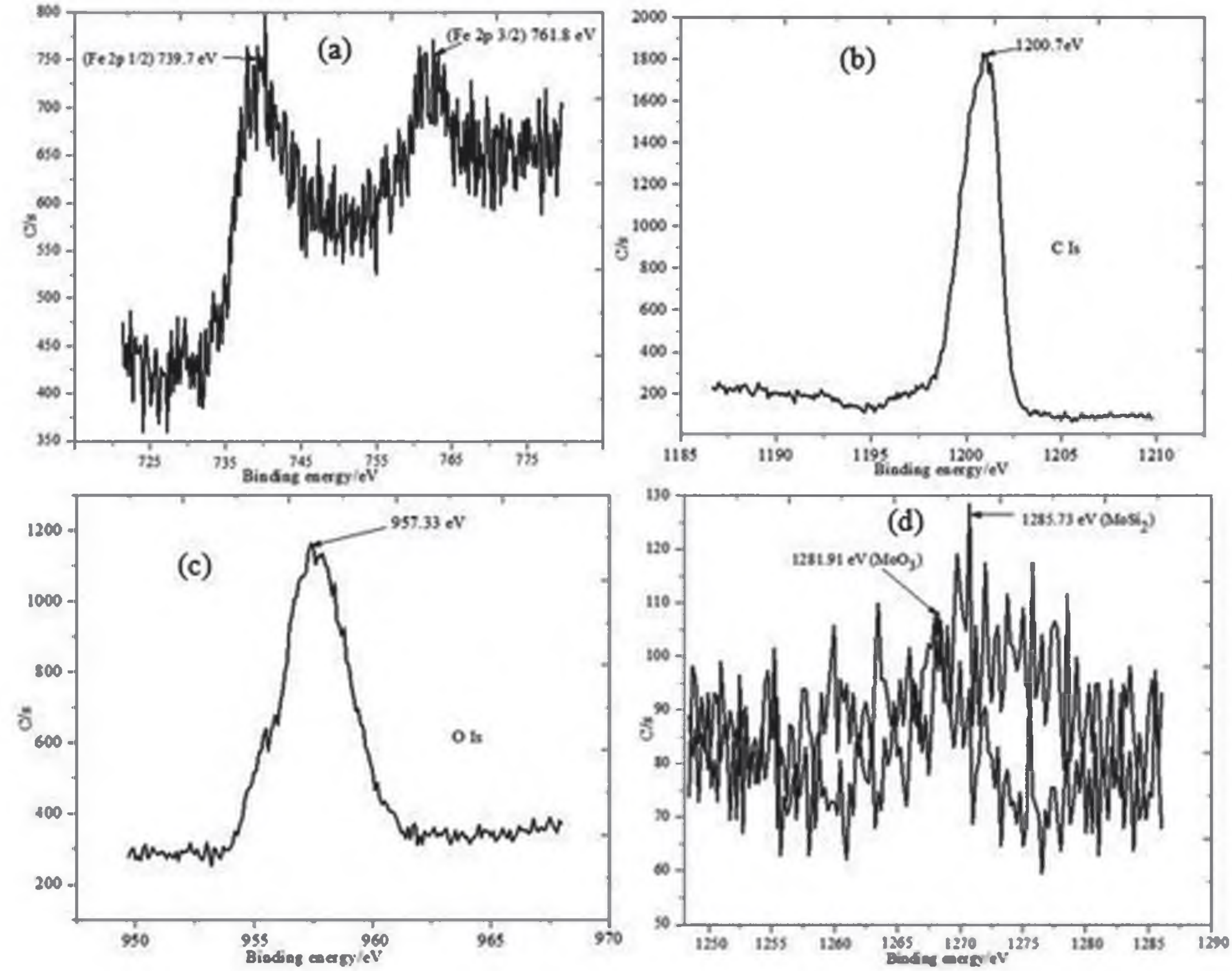

Fig. 13 XPS analysis of worn plat lubricated surface with 0.8 mass% EC-CMC BRO70/MO30

EC-CMC BRO70/MO30 under 80 N. Utilizing XPS research to identify the chemical composition on the worn surface, the tribo-chemistry mechanism of EC-CMC as an addition in base lubricant towards surface protection and coefficient reduction was explored. Figure 13 shows the XPS spectra of Fe 2p, C1s, O1s, Si2p, and Mo 3d on the worn surfaces following 15 min of lubrication with 0.8 mass% EC-CMC BRO70/MO30 under 80 N. The NIST XPS Database was used to find the binding energies of the chemicals [36].

During the analysis of the lubricated substrate, Fe 2p of two peaks with binding energies of 739.7 eV and 761.8 eV were detected and associated with chemical composition of metallic Fe (Fig. 13 (a)). The spectra at 739.7 eV, which correspond to Fe $2p^{1/2}$, were discovered in the spectrum of Fe 2p, suggesting the presence of FeOOH, which is derived from the hydroxyl group discovered in EC-CMC according to FT-IR analysis. The second spectra peak, which is Fe $2p^{3/2}$ and indicates Fe_2O_3, was discovered at 761.8 eV, and may have been produced by combining FeO and the hydroxyl ion (OH-) of water present in the lubricant. However, the significant elements C 1s and O 1s of EC-CMC, with spectra binding energies of 1200.7 eV and 975.3 eV, respectively, demonstrate the presence of the carbonyl group (Fig. 13 (b), (c)). This is owing to the organic nature of the additive with substantial carbon and, the high oxygen content could relate to metal oxides, as suggested by previous work [37].

As shown in Fig.13 (d), two peaks of Mo 3d were detected. The spectra have Mo 3d peak at 1281.9 eV and is connected to the Mo-O bond is present in addition to the Mo (6+) component, however, observed noise at the peaks. After EC-CMC additive testing, an XPS study of a worn surface showed that molybdenum oxidized, and molybdenum silicate ($MoSi_2$) were present. A second selection was discovered at 1285.7 eV as well. This shows the $MoSi_2$ material's adherence to the worn track's surface and is related to the Mo (6+) valence state. It also comes from the $MoSi_2$ matrix (Mo-Si bonds) as a result of the heating impact. These findings concur with those of prior studies on the interactions between nanoparticles and steel surfaces [36, 38]. The results also show that the tribo-chemical reaction at the friction surface has revealed and is suspected of being involved the nanoparticles of EC-CMCs.

5 Tribo-chemistry mechanism of EC-CMC polymer

Friction test results show that the EC-CMC additive possesses friction and wear reducing abilities during lubrication application. XPS analysis indicates that the presence of effective elements in the tribo-film on the rubbing surface could be the main reason for the tribological performance. It was observed that under smaller load, the operations lacks adequate frictional energy for the generation of sacrificial film for contact separation but achieved it at higher load. It was proven through the reduction on COF and wear under 80 N than 60N as presented in Fig. 8. The study demonstrated starvation of lubricants at the front and back of the ball during operation under lower load thereby making it difficult for the particles to diffuse in between the rubbing region as shown in Fig. 14 (a). This observation was not noticed under higher load (80N) as presented in Fig. 14 (b) but starts demonstrating tribo-film formation and becomes more pronounced as operation continues as illustrated in Fig. 14 (c).

As operation attains frictional energy, molecules from tribo-film begin to be absorbed (silicon, carbon, and molybdenum) on the substrate surfaces thus, mending or healing effect occurs. Although, more of friction reducing effect was noticed from EC-CMC than anti-wear ability. In conclusion, the variances between the generated adsorption film and the tribo-film on the rubbing surface, which depends on system working conditions, are the fundamental determinants of the tribological performances of EC-CMC polymer samples, thus similar to previous study [39].

6 Conclusion

The effectiveness of using bio-based additives as an alternative to inorganic based additives in lubricant formulations for industrial application has been investigated (quantity depletion and environmental impact). This is because of global sustainability initiatives that emphasize the usage of resources with no negative social impact. To determine their enhancing strength, EC-CMC polymers were suggested, produced, characterized, and used in various lubricants. Tribological enhancement employing EC-CMC was comprehensively evaluated in terms of coefficient of friction, surface roughness, wear scar diameter, and wear loss and compared under vegetable and conventional lubricants as well as blended condition. From the results of the work, the following conclusions were drawn.

1. When the effectiveness of EC-CMC blended with base rapeseed oil and mineral oil was tested, obviously produced good tribological performance when compared to BRO and MO under different working conditions. During the research, it was found that tribo-film was produced by the nano lubricant interaction with the substrate via tribo-reaction. This interaction is more pronounced when employed on BRO, and it was therefore determined that EC-CMC was responsible for the reduced friction coefficient and wear roughness. Among all the concentrations tested, 0.8 mass% demonstrated the optimal performance both on BRO and MO.
2. Under various lubricant blending, application best additive candidate (0.8 mass% EC-CMC) with BRO70/MO30 demonstrated outstanding performance followed by BRO50/MO50 before BROMO30/MO70. The findings of the research revealed that although EC-CMC polymer enhances tribological performance of the formulated lubricants the effect was better on COF reduction than anti-wear operation. This was due to rapid tribo-film formation on the substrate surface acting as shield against direct contact as revealed by the EDX and XPS analysis.
3. The analysis also observed that the formulations produced better results at higher working (80 N) conditions than lower conditions (60 N). This was due to the ability of the lubricants undergoing tribo-chemistry for the development of tribo-film due to the generated frictional energy. Furthermore, in mechanism of EC-CMC towards friction and wear reduction, the investigation noticed difficulties by the particles entering the contact region, accumulating at the front and back region of contact region of the ball thereby causing starvation of lubricant, thus mostly observed at lower working conditions.

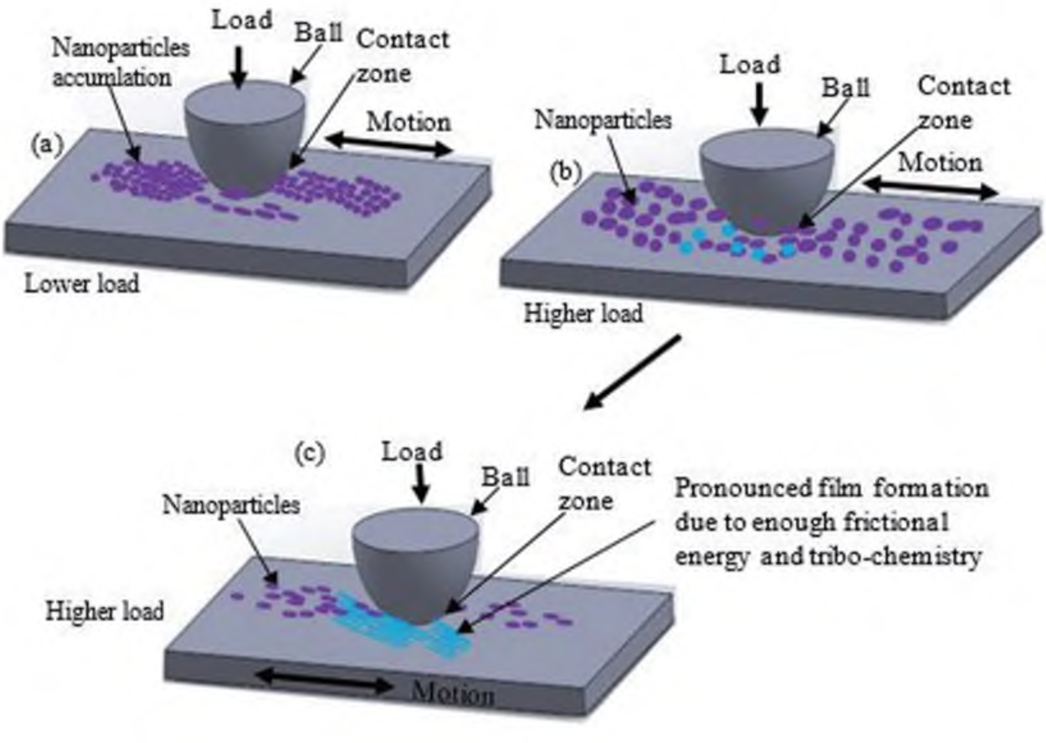

Fig. 14 Illustration on the tribological mechanism of EC-CMC polymers during lubrication

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