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Article

Investigations on Tribological Performance of Jatropha Oil Enriched with Polymers under Different Working Conditions

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Abstract

This study examined the effects of polytetrafluoroethylene (PTFE) and Eichhornia crassipes carboxymethyl cellulose (EC-CMC) polymers in base jatropha lubricant and employed commercial shell lubricant as the reference. Wear reduction and coefficient of friction (COF) were measured using 1 mass% polymer concentrations. Characterization analysis showed that the formulation processes required functional groups like carbonyl, alkene, aliphatic CH_2 and CH_3 group that the Fourier Transmission Infrared (FT-IR) studies indicated were necessary for lubrication. According to the frictional analysis, the polymers' tribological performance was influenced by changes in both load and operational speed. At 40 kg and 1200 rpm, PTFE and EC-CMC reduced COF by 53.7% and 49.8%, respectively, compared to base jatropha. However, commercial shell oil produced exceptional performance.

Keywords

PTFE, EC-CMC, tribology, jatropha oil

1 Introduction

The development of advanced industrial machineries and electric vehicles with high speeds, high loads, frequent start-stop processes with components constantly operate in mixed and even boundary lubrication regimes leading to substantial friction generation [1–3]. It has been established that base lubricants lack the potential to provide the necessary service during operations owing to deficiency of substances that promote film (third body) formation [4, 5], especially when subjected at higher working conditions. To accomplish the expectations of lubrication, some quantities of additives were added to the lubricant to reduce friction and wear of metal-metal contacts subjected to boundary and mixed lubrication regimes [6, 7]. The creation of a high-performance tribo-film during the rubbing process was thought to be crucial for enhancing the sliding pair's border lubricity [8]. Though, seizure issues can still arise when metal-metal friction pairs are engaged for an extended interval of time with an unusually high load or low viscous lubricant [9]. Giving the lubricant a suitable viscosity modifier that can replace the original lubricant's reduced viscosity due to internal lubricant degradations is one viable approach for increasing the reliability and lifespan of such motion systems.

In this context, polymer-based materials are being researched extensively as tribo-materials due to their exceptional qualities, such as their low weight and capacity for self-lubrication [10]. Extensive research on the tribological behaviors of polymer-based materials during dry sliding has been done in recent years with huge success on tribo-applications, self-lubrication composites [5, 10]. Studies have been conducted on base lubricated blended with polymer [5, 8] but less compared to the numerous analysis that were done when subjected to dry friction [10]. Notwithstanding, investigation on the tribo-reaction of polymer-based lubricants has not been studied. Although, research showed that the addition of polymeric substances as nano materials with high abrasion resistance considerably improved the wear resistance of epoxy when it was lubricated with oil [11]. It was discovered that the production of a high-performance tribo-film was also of utmost significance for the tribological performance of the polymer additive in lubricant with abrasion resistance [11].

The production of transfer film layers (TFLs) on the counter face, in particular, as a result of tribo-contact, ultimately plays a crucial part in determining the general tribological behavior of sliding materials [5, 12]. As a result, the properties of TFLs by polymers have been regarded as a distinguishing trademark during lubricant formulation [5] as to ascertain its application.

A tribological study of polytetrafluoroethylene in base white oil was conducted by Mengnan et al., [13]. The outcomes demonstrate that the additives can greatly enhance the base oil's anti-friction and anti-wear capabilities. This is owing to the PTFE particles' self-lubricating ability and the creation of tribo-chemical response films. However, the lubrication mechanism of the PTFE was not thoroughly investigated to ascertain the suitable conditions to be used. Opia et al., [11] studied the EC-CMC's tribological responsiveness in base rapeseed oil. When evaluated under pin on a disc tribo-meter, commercial polyphosphate performed better than the application of 1 mass% EC-CMC, which nonetheless produced the best results. The analysis was conducted under less favorable working conditions, however, the tribological mechanism affecting tribological performance was not investigated.

Due to the importance of tribo-chemistry, film creation of polymer-based lubricants during service, and factors influencing film building during tribological operation, it is necessary to understand the mechanism in order to choose where to apply. To understand the behavior of polytetrafluoroethylene (PTFE) and organic polymer of Eichhornia crassipes Carboxymethyle cellulose (EC-CMC) polymer under high-speed operations, this study was designed to examine friction resistance performances. Based on characterizations of worn surfaces and tribo-chemical investigations, the mechanisms of tribo-film production were examined. The study is anticipated to open up possibilities for creating high-performance lubricants based on polymers for industrial utilizations. For this experiment, EC-CMC and PTFE were chosen because of their environmental friendliness [11, 13].

2 Materials and method

Prepared polytetrafluoroethylene (PTFE) powders (40-75 nm) polymer was purchased from Sigma Aldrich firm Malaysia together with base jatropha vegetable oil (BJO) and commercial shell oil (CSO). The jatropha oil used in this is a product from Indonesia, having density of 908 kg/m^3, viscosity of 40.20 mm^2/s, oxidation stability (2.67 h), water content (0.09%), acid value (3.83 mg KOH/g), palmitic acid (15.3%), stearic acid (5.8%), oleic acid (43.5%), linoieic acid (33.7%), found similar with previous study [14], although the properties varies depending on the location of harvest. The organic polymer of Eichhornia crassipes Carboxymethyle cellulose (EC-CMC) of nano size of 53-82 nm was synthesized from Eichhornia Crassipes weed in the production/material laboratory of Universiti Teknologi Malaysia utilizing a ball milling machine for nanoparticle generation [11]. The process of polymer creation employed isobutyl/ isopropyl approach according to Asep Handaya Saputra et al., [15].

2.1 Lubricants preparation

The standard oil (jatropha oil) was used to determine the additive performance and compatibility when used in lubrications with 1% concentrations. Table 1 displays the lubricant-PTFE and EC-CMC nanoparticle mixing conditions. Each formulation was thoroughly mixed using a high-powered sonication machine for 2 hrs:30 min to prevent the agglomeration of nanoparticles and improve stability. In other words, the PTFE- and EC-CMC-modified lubricants were made up of 9% jatropha oil and 1% nanoparticles. The selection of the 1 mass% concentration was chosen based on previous literature recommendation [11, 16], along with their ecofriendly nature.

2.2 Analysis instruments

To conduct investigation of the nature of the materials used and tribological properties, many instruments were utilized on additives, lubricants, and contact surfaces of the specimens. The viscosity of the lubricants was measured using HK-265A apparatus with capillary tube tested at 40°C and 100°C, according to ASTM D 445 standards. To determine the morphology of the nanoparticles, a scanning electron microscope (SEM) (JEOL JSM-6010PLUS/LV) was used. The composition of the elements components on the rubbing

Table 1 Constituents of the solution

Reference lubricant	Additives	Formulations in solution
90% Jatropha oil	1% PTFE	9% jatropha oil + 1% PTFE
90% Jatropha oil	1% EC-CMC	9% jatropha oil + 1% EC-CMC
Commercial shell oil (100%)	-	100% solution shell

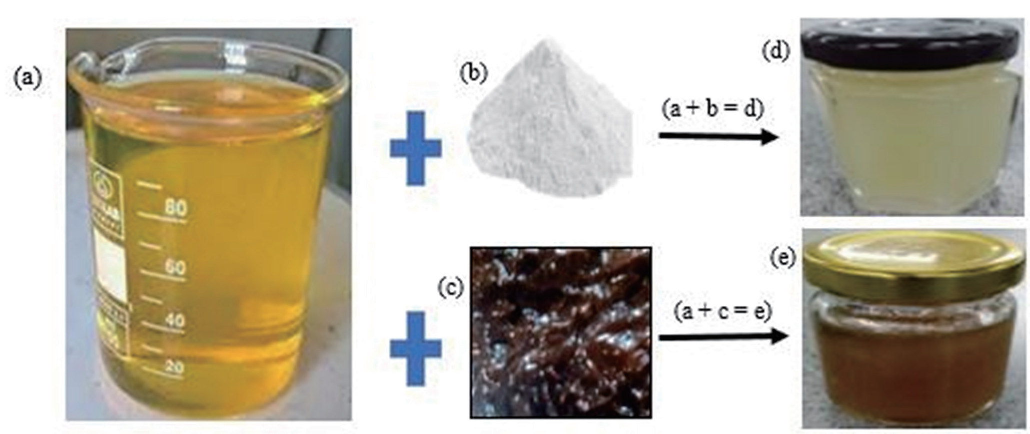

Fig. 1 Lubricant formulation: Base jatropha oil (a); PTFE (b); EC-CMC (c); PTFE blended (d); EC-CMC blended (e)

surfaces was examined using the energy Dispersive X-Ray Spectroscopy (EDS). To investigate the functional groups of PTFE and EC-CMC, the Fourier Transmission Infrared (FT-IR) (Mattson infinite, Model 960 Moog ATI, USA) spectrum was performed. This is to determine whether the functional groups in the formulations are appropriate and comparable to those in lubricants that have been used in the past. Before any testing, 100 scans (background and sample) must be finished, according to Alizadeh et al., [17]. With spacers of 13 mm in diameter and 0.025 mm thick, the test material with a 5 mm thickness was employed. The spectral resolution, in line with the previous statement, was 4 cm in the 400–4000 cm^{-1} frequency range [18]. The spectra were all obtained at room temperature, and an average of 6 to 18 scans were performed [18]. A solution of 150 ml of jatropha oil was blended with 1 mass% of PTFE and EC-CMC separately to create the lubricant (formulation) before testing.

To examine the wear surfaces diameter of the lubricated specimens, an optical microscope (OM) was used. The arithmetic means of the surface roughness, wear volume of the lubricated specimens was measured using optical microscope 3D Surface profilers. To understand the ppm worn on the balls after lubricants lubrication, spectrochemical analysis using rotatory disc spectrometer (Spectroil 100 series) was conducted in accordance with ASTM-D6595 standard, thus ultrasound re-agitation of the samples for 30 minutes was performed prior to the test. A volume of 50 ml of each lubricant (unused and used) was made available to the laboratory in their customized analysis bottle.

2.3 Tribological tests

The four-ball friction tribo-tester was used to investigate materials' capacity for wear resistance and friction reduction in accordance with ASTM D2783 standard as seen in Fig. 2. The internal structure of the four ball tribo-tester machine consisted of an upper moving specimen in contact with a lower specimen that was set in a temperature-controlled oil bath. In this study, the effects of applied load and sliding speed on the tribological properties of polymer materials were comprehensively studied. The detailed test parameters are illustrated in Table 2. The friction coefficient was recorded by a force transducer during the entire sliding test. For a total of 1 hr, each lubricant was tested at a temperature of 75°C. In practical applications, load and speed are important parameters directly influencing the hydrodynamic pressure of the oil film [19], thus could be used in determining the resistance strength of lubricating oil additives in separating body on sliding surfaces.

Table 2 Experiment working conditions

	Parameters	Values
1st	Speed (rpm)	1200
	Temperature (°C)	75
	Load (kg)	15, 20, 30
2nd	Load (kg)	40
	Temperature (°C)	75
	Speed (rpm)	800, 1000, 1200

The upper and lower specimens were made of alloy steel (l2 mm) with Hardness value 59–61 HRC. The surface roughness (Ra) and surface depth (Rq) of ball sample's original surface was ranging between 0.02 and 0.09 μm for Ra, while Rq ranging between 0.03 and 0.08 μm. They were made of GCr15 bearing steel (AISI-52100) with a composition of C, 0.95–1.05 mass%; Si, 0.15–0.35 mass%; Mn, 0.20–0.40 mass%; P, < 0.027 mass%; S, < 0.020 mass%; Cr, 1.30–1.65 mass%; Ni, < 0.30 mass%; Cu, < 0.25 mass%. After each test, tissue paper and degreasing cotton were used to gently wipe away excess oil from the surfaces of the composite sample and counterpart. Additionally, the sample and counterpart were washed in an acetone bath to get rid of any remaining oil.

Wear is the term for the weight loss that occurs as a result of relative motion and friction of the contacting surface. The lower the value of specific wear rate (Ws), the better its wear property. Several factors, like the surface's nature (dry or wet), orientation with relation to the counter face's sliding direction, affect durability of a surface. The wear volume of the material after wear (V), the normal load (FN) that is being applied, and the sliding distance (d) are used to establish the Ws [20–22]. A digital-reading optical microscope was used to evaluate the ball sample wear scar diameter, while using 3-D laser profile-meter, the depth was determined. Using Equation 1, the specimen's volume wear rate, k was estimated.

Volume wear rate,

$$k = \frac{\pi}{3t}\left(R - \sqrt{R^{2} - d^{2}}\right)^{2}\left(2R + \sqrt{R^{2} - d^{2}}\right) \quad (1)$$

In Equation 1, k is the volume wear rate (mm^3/s), t is the duration time (s), R is the ball radius (mm), d is the radius of the wear scar diameter.

3 Results and discussion

3.1 Samples characterization

Using a JEOL JSM-6010PLUS/LV powered by 100V/11 amps,

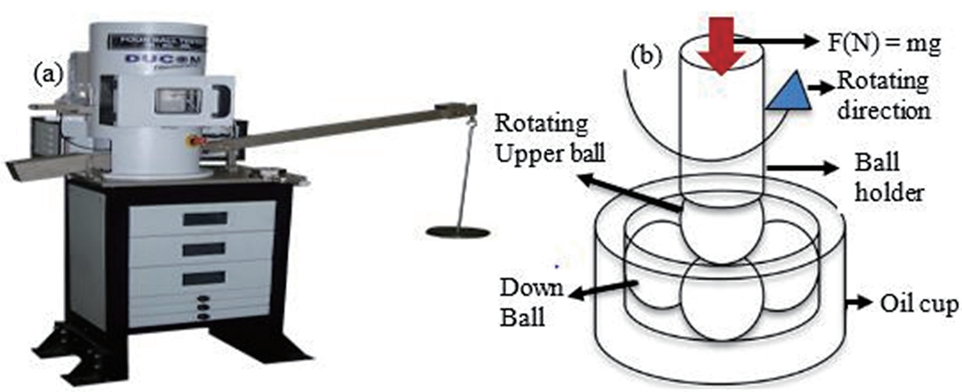

Fig. 2 Four ball tribometer (a) and balls arrangement/mechanism (b)

Fig. 3 shows high resolution (x100) SEM representations of the morphology of PTFE and EC-CMC. In contrast to the EC-CMC sample, which had a gel-like appearance, the image reveals that the aggregated particles were uniformly distributed with the creation of pores, demonstrating the potential to dissolve in lubricant. The findings are consistent with earlier publications [11, 23]. The EDS analysis is shown in Table 1, revealing the presence of many elements in EC-CMC while only carbon and Fluorine while Zirconium (Zr) element was from the coating process. The data from the earlier study was like the elements mentioned [24, 25]. These findings were recommended since water hyacinth has been identified as a viable source for phytoremediation [26].

In the production of lubricating oil, the viscosity of the oil plays a crucial role. At temperatures of 40 and 100°C, the kinematic viscosity density of the lubricating oil was calculated using a concentration of additives of 1 mass% in solutions. The results are displayed in Table 4, where it can be seen that the thickening effect causes the viscosity to drop when the temperature is raised. At 100°C compared to 40°C, this effect is significantly more pronounced.

The viscosity index (VI) was identified as a crucial element in lubricating oil analysis. As seen in Table 4, the viscosity index (VI) rises when polymeric additives are used. This may be because lengthy molecular chains form polymer coils in the oil when the polymer ingredient dissolves in it [27]. The interaction between the polymer chain and the oil molecules (the swelling process) increases as the temperature rises, causing the polymer chains to become more relaxed and tend to extend completely. This increase in volume increases the mixture's viscosity, counteracting the base stock's reduction in viscosity [16]. According to the findings, EC-CMC's viscosity index is higher than PTFE lubricants. This demonstrates the polymer additive's effectiveness in enhancing the base stock's viscosity index. This is similar with literature report [27, 28].

The FTIR spectra of base jatropha oil blend PTFE and jatropha oil blend EC-CMC are displayed in Fig. 4. On the curve bands of the additive EC-CMC sample, the stretching of hydrogen bound O-H (water molecule) was identified as the cause of the peak at 3449.1 cm^{-1}. The broad absorption band (3010–3700 cm^{-1}), which is present in the formulation and coincides with the -OH groups in aliphatic or aromatic alcohol,

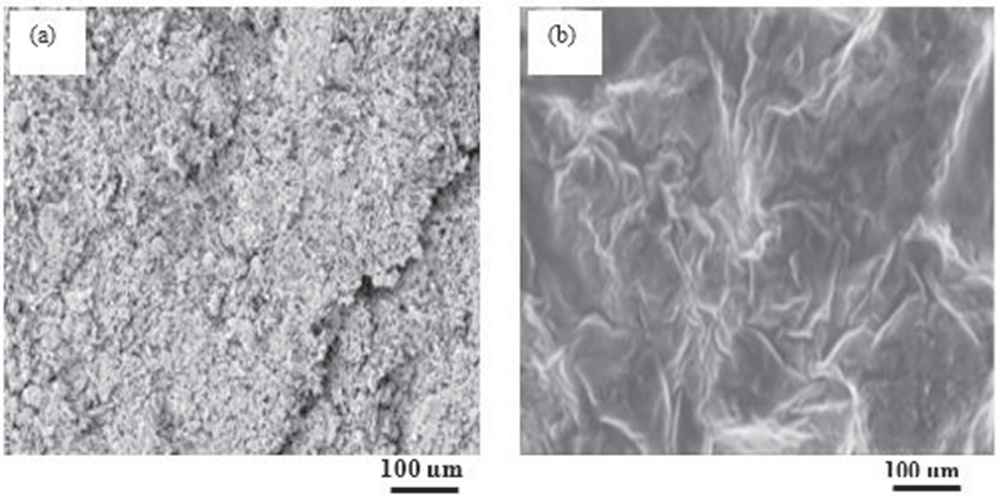

Fig. 3 SEM images of PTFE and EC-CMC polymers

Table 3 Elements found in the polymers according to EDS report

Material/Elements (mass%)	C	O	Mo	Ca	F	Na	Cu	K	Cl	Al	Mg	Zr
PTFE polymer	10.8	-	-	-	57.15							32.04
EC-CMC polymer	56.2	20.1	3.4	3.1	-	8.3	1.3	1.8	2.1	2.2	1.3	-

Table 4 The samples kinematic viscosities (cSt), densities and viscosity index at different temperatures

Samples/ properties	Density @15°C (g/cm^3)	Error (±)	Viscosity @40°C	Error (±)	Viscosity @100°C	Error (±)	Viscosity Index VI	Flash Point (°C)	Error (±)
Base Jatropha	0.9036	0.004	53.71	0.71	10.83	0.18	115.02	226.01	0.03
1 mass% PTFE	0.9151	0.002	88.93	0.52	14.91	0.36	176.45	219.33	0.05
1 mass% EC-CMC	0.9183	0.004	89.15	0.27	16.64	0.41	202.47	231.05	0.03
Shell oil	0.9007	0.003	83.89	0.35	13.61	0.25	165.86	230.11	0.07

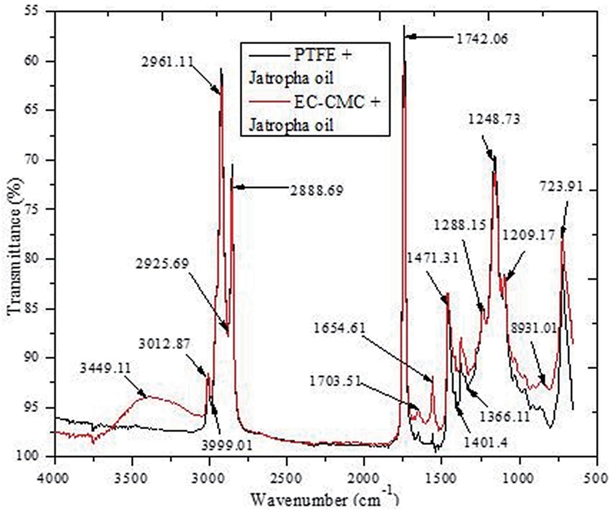

Fig. 4 FT-IR of Jatropha oil, PTFE + Jatropha and EC-CMC + Jatropha oil

demonstrated the hydrophilic propensity of EC–CMC. This and the spectroscopic analysis performed by Manun et al., [29]. For both samples, the spectrum showed the cis-double bond's (=CH) stretching vibration at a wavelength of 3012.8 cm^{-1}. At 2961.1 and 2854.5 cm^{-1}, the aliphatic CH_2 group's sharp symmetric and asymmetric stretching vibrations were detected. The samples' spectra also reveal symmetric and asymmetric stretching bands stretching the vibration shoulder of the aliphatic CH_3 group at 2888.6 cm^{-1} and 2557 cm^{-1}.

The samples showed ester triglycerides carbonyl functional groups at 1742 cm^{-1}, while free shoulders of fatty acids and C=C stretching vibration of cis-olefins appeared at 1703.5 cm^{-1}. The pronounced peak at 1654.6 cm^{-1} was attributed to the vibration of OH group found in jatropha blend EC-CMC. Nevertheless, the bond building was much with EC-CMC due to the large amount of carbonyl, alkene group. Aliphatic CH_2 and CH_3 group vibrations at 1471 cm^{-1}, cis-disubstituted CH bonds at 1401.4 cm^{-1}, plane CH_2 group vibrations at 1366.1 cm^{-1}, and CH_2 group ending vibrations at 1337.5 cm^{-1} were discovered to be in the region of bonding distortions and alteration. This findings are similar with observations in literatures [12, 30]. At 1288.1 cm^{-1} and 1209.1 cm^{-1}, fingerprint regions in the spectrum were found, revealing C-O ester groups. The final overlap of the cis-disubstituted olefines' out-of-plane vibration and CH_2 rocking was attained at 723.9 cm^{-1}, although the EC-CMC still indicates further vibrations that indicate greater amount of carbonyl constituents. According to the investigation, the spectra trend, functional groups, breaking, and bond formation of PTFE and EC-CMC in jatropha oil, respectively, showed high compatibility and expected to enhance lubricity of jatropha lubricant.

3.2 Frictional analysis

3.2.1 Influence of loads on the polymers tribological performance

Figure 5(a-c) depicts the characteristics of the coefficient of friction under operating speed of 1200 rpm at different loads (15 kg, 20 kg and 30 kg) for the various lubricants. Figure 5(d) displays the average wear scar diameter (WSD) of the lubricants under varied loads. It was discovered that the applied loads had a substantial impact on COF performance of lubricants, which had an impact on wear behavior. The study found that, out of all the conditions evaluated, PTFE proved to be the lubricant that reduced friction more effectively. Under 15 kg working conditions, base jatropha, 1 mass% PTFE, 1 mass% EC-CMC, and reference commercial shell oil, produced COF values of 0.0745, 0.0655, 0.1126, and 0.0711, respectively. This yielded COF reduction by 12.1% and 4.6% for 1 mass% PTFE and commercial shell, while –51.1% was recorded under 1 mass% EC-CMC showing increment on COF against base jatropha lubricant. According to a viscometric investigation, the 1 mass% EC-CMC's poor performance was thought to be caused by its very viscous nature since high viscosity causes high COF.

When the load was increased to 20 kg, all of the tested lubricants had an increase in COF, producing 0.1063, 0.0672, 0.1319, and 0.1064 for base jatropha, PTFE, EC-CMC, and commercial shell lubricants, respectively. The PTFE behavior, however, correlated with the tests done with 15 kg. Apart from EC-CMC, which produced 0.1239 in comparison to the result of 20 kg, all lubricants produced a marginal increase at the 30 kg testing. Base jatropha had a COF of 0.1093, which was lower than the values for EC-CMC and shell lubricants. This resulted in a COF reduction of 28.6% for PTFE, while EC-CMC and the reference shell lubricant witnessed –13.4% and –12.3, signifying COF increment, respectively compared with base jatropha oil. The experiment revealed that EC-CMC performance could be enhanced at higher load. The findings are similar to previous literature outcome [5]. The study also showed that as working conditions increase, more frictional energy is produced, which improves tribo-chemistry and results in the creation of films that reduce wear and COF, thus supported by Singh et al., [22], findings.

The wear resistance and variation in the average wear scar diameter of the oil samples are depicted in Fig. 5(d). When compared to the graph trend, it can be shown that the wear scar diameter has a close link with both the polymers and the commercial shell oil. Base jatropha lubricant exhibits significant WSD, indicating a lack of separating film between the sliding bodies. The wear scar diameter for PTFE and EC-CMC particles marginally rises as working conditions increase. Commercial shell oil displayed excellent results despite PTFE's ability to reduce WSD was superior to EC-CMC due to the presence of an anti-wear constituent. Base jatropha yielded WSD of 725.17 mm, 792.31 mm, and 847.11 mm under 15 kg, 20 kg, and 30 kg, respectively. With a 15 kg load, PTFE and EC-CMC reduced WSD by 33.37% and 28.52%, respectively. At 20 kg, these reductions were 34.15% and 27.46%, and at 30 kg, the reductions were 36.09% and 33.46%, respectively. This demonstrates that WSD reduction against base jatropha oil increases as load increases. The detected functional groups and tribo-chemistry during the PTFE and EC-CMC particles lubrications may be responsible for the reduction in wear scar diameter, yet commercial shell oil achieved excellent results. Due to the lack of anti-wear elements in the formulations, PTFE and EC-CMC were thereafter not recommended under high operations unlike shell lubricants for wear reduction.

3.2.2 Influence of sliding speeds on the polymers tribological performance

The friction coefficient evolutions of the various polymers versus sliding time under 40 kg at different sliding speeds are shown in Fig. 6(a-c), while the wear scar diameter is shown in Fig. 6(d). During the investigation of the lubricants, the applied loads and sliding speeds significantly influenced the results. In the research, decreasing patterns were observed under polymer

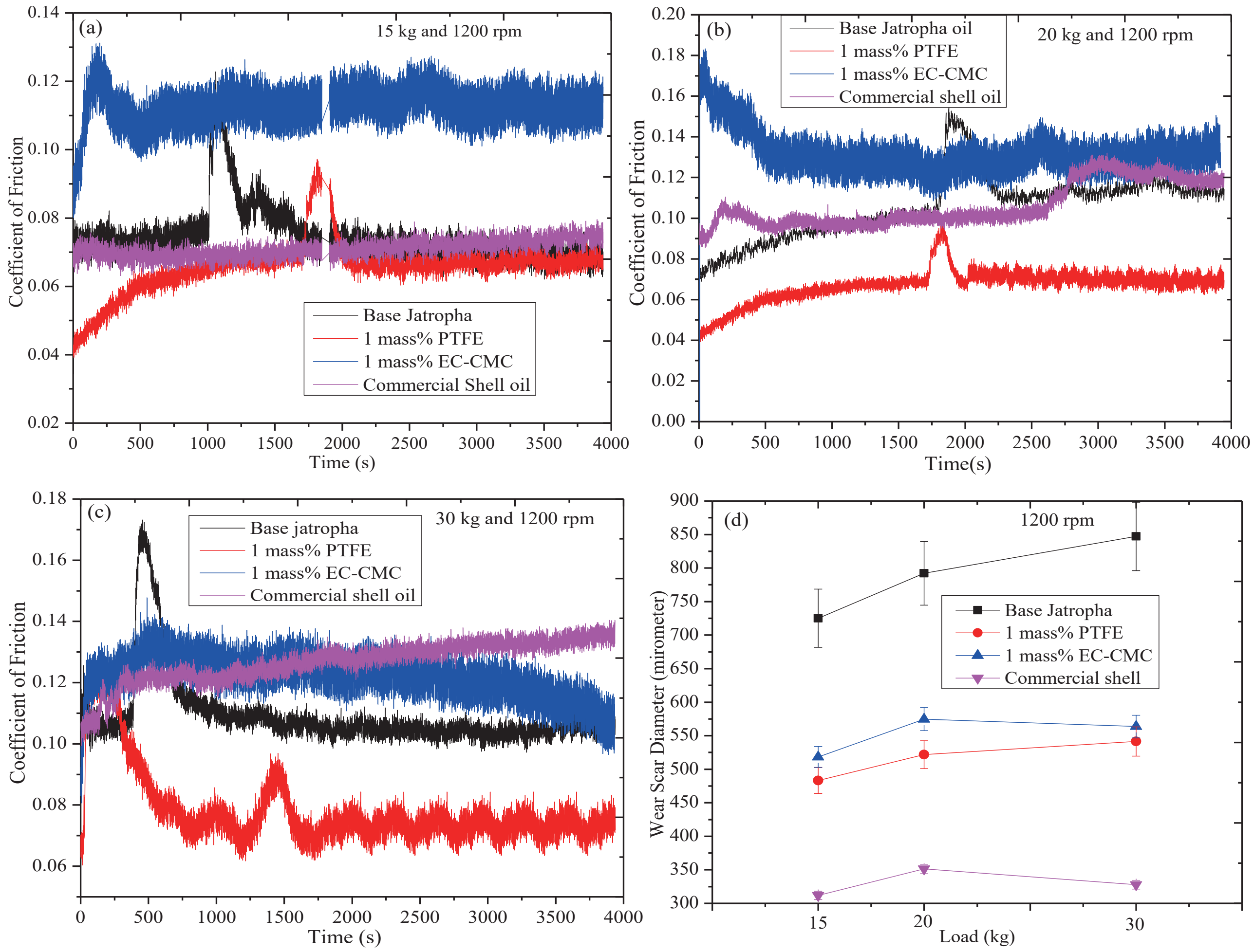

Fig. 5 Evolutions of coefficient of friction against sliding time at 15 kg (147 N) (a); 20 kg (196 N) (b); 30 kg (294) (c); and specific wear rates (d) of the various lubricants at operating speed of 1200 rpm and 75°C

additives and commercial shell showcases alternating, base jatropha lubricant increases with increasing sliding speed. For 800, 1000, and 1200 rpm, base jatropha produced 0.2018, 0.2201, and 0.2431, PTFE produced 0.1675, 0.1132, and 0.1125, while EC-CMC produced 0.1869, 0.1331, and 0.1218. Higher COF values were shown by the commercial shell lubricant, which were 0.1769 for 800 rpm, 0.2135 for 1000 rpm, and 0.2106 for 1200 rpm, respectively. Base jatropha and shell oil both behaved similarly in terms of COF since the quantity of COF obtained was more than that of the polymers and so showed a similar curve.

The operations under 800 rpm, 1000 rpm and 1200 rpm yielded COF reductions of 16.9%, 48.5%, 53.7% for 1 mass% PTFE, while 7.4%, 39.5%, 49.8% were generated from 1mass% EC-CMC when compared to base jatropha lubricant. Again, with the same working conditions, COF reductions of 5.3%, 46.9%, 46.5% were obtained from 1 mass% PTFE, while 1 mass% EC-CMC recorded increase on COF by 5.6% under 800 rpm, but reduction by 37.7% under 1000 rpm and 42% at 1200 rpm compared with commercial shell lubricant. According to the analysis, the tested polymers' COF decreases in comparison to base jatropha oil when working conditions increase due to ability to generate frictional energy and have good tribo-chemistry, which results in desirable film formation. These findings revealed that viscous modified lubricant is appropriate for yielding a better lubricant film between surfaces during sliding contact at higher working conditions. This observation was similar with conducted research by Singh et al., [22]. The outcome of the tested polymers shows good tribological performance in lowering COF within the conducted conditions. This was suggested to come from the good functional groups found in the samples under FT-IR as presented in Fig. 4, thus provided needed film formations at the contact surface.

Figure 6(d) depicts the mean wear scar diameter for samples under 40 kg at varied sliding speeds. With the use of a microscope machine, the wear scar diameter was measured. At higher loads, compared to Fig. 5(d), a larger wear scar diameter was achieved. More wear scars are produced by the increased pressure on the contact surface according to Zhao et al., [5]. At the sliding speed of 800 rpm, 100 rpm, 1200 rpm, the base jatropha recorded a substantial rise in wear scars as the sliding speed increased, yielding values of 831.55 μm, 885.13 μm and 935.01 μm, whereas a modest increase was seen with commercial shell lubricant. With regard to the sliding speed, a decrease and increase were noted with polymers values of mean wear scar diameter.

When operating at 800 rpm with 40 kg, PTFE and EC-CMC produced WSD reductions by 46.95% and 43.11%; 50.99% and 50.62% at 100 rpm; and 36.23% and 31.95% at 1200 rpm respectively when compared to basic jatropha oil. It was observed that the use of the polymers was able to generate tribo-film together the generated frictional energy between the sliding body contact leading to body separations. The base

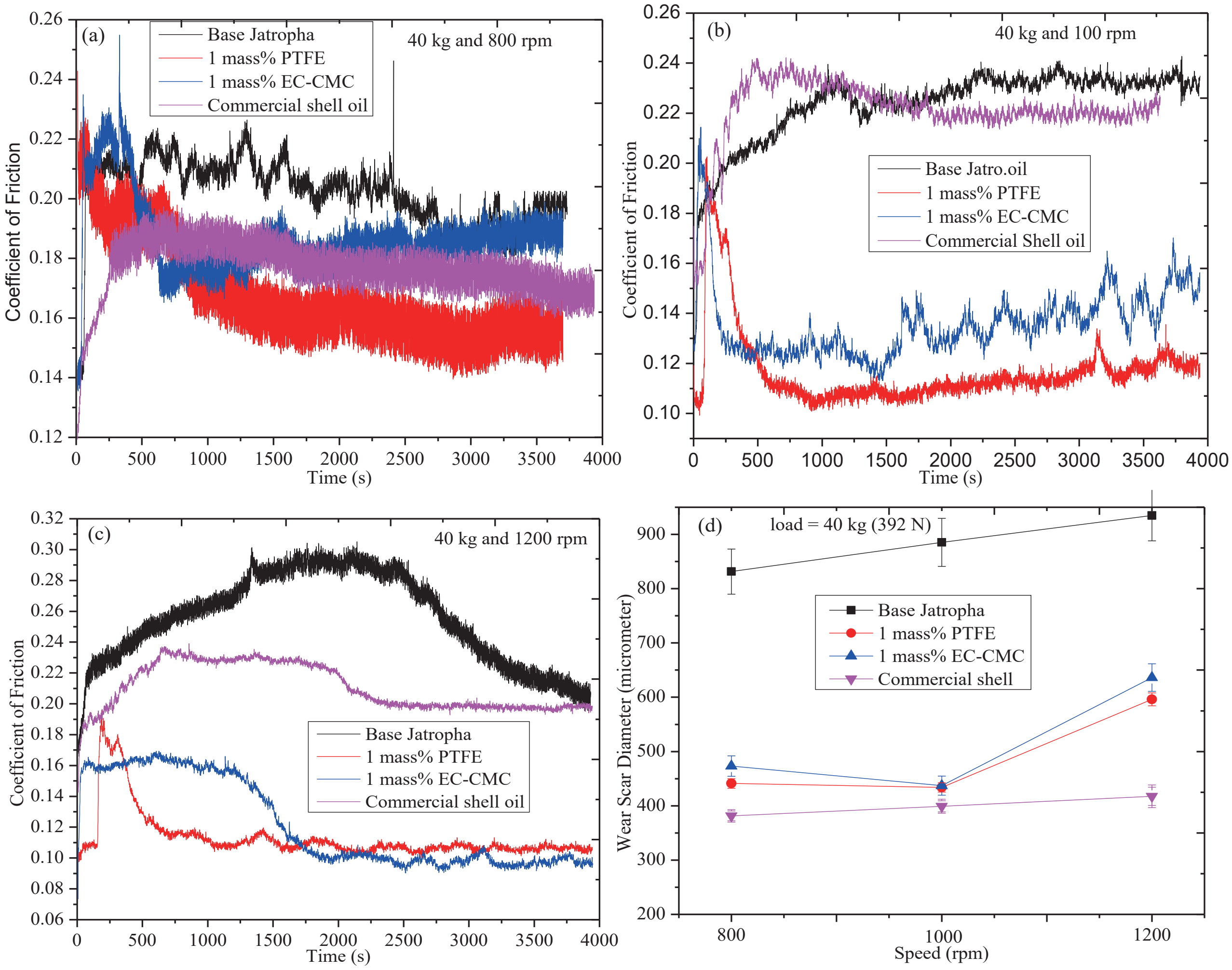

Fig. 6 Evolutions of coefficient of friction against sliding time under speed of 800 rpm (a); 1000 rpm (b); 1200 rpm (c) and specific wear rates (d) under a specific load of 40 kg (392 N) and 75°C

jatropha lubricant operation experiences direct sliding contact during operation leading to high WSD. The poor reduction on the WSD under polymer additives was due to lack of anti-wear additive in the formulation unlike commercial shell counterpart. According to the research done by Abdollah et al. [31], there is also a significant impact of the parameters taking into account load and sliding speed.

3.3 Tribo-pair volume wear rate

Figure 7 displays the volume wear rate of the samples tested at various loads and sliding speeds. Figure 7(a) showed the wear rate for loads of 15 kg, 20 kg, and 30 kg when operating at 1200 rpm, whereas Fig. 7(b) showed the wear rate for loads of 40 kg when operating at 800 rpm, 1000 rpm, and 1200 rpm. According to the research as in Fig. 7(a), observed similar wear rate graphs among all the samples except commercial shell oil. However, EC-CMC and PTFE displayed a high wear rate apart from base jatropha oil, owing to the lack of anti-wear constituent in the formulation unlike commercial shell lubricant which exhibited excellent wear characteristics. The commercial shell oil contains anti-wear elements that shield the surfaces of elements during service, leading to wear reduction according to literature [32]. The specific wear rate of base jatropha oil is increased excessively with increasing the applied load, which correlates with COF results. According to the study, a change in load shows an impact on wear rate. The findings were consistent with previous research [33]. The study suggested that inclusion of anti-wear additive in EC-CMC and PTFE will enhance their tribological properties and will be comparable to commercial shell counterpart.

As shown in Fig. 7(b), the lubricants had various behaviors in terms of volume wear rate when subjected to a load of 40 kg at speeds of 800, 1000, and 1200 rpm. When compared to other samples, the application of base jatropha lubricant yielded a high-volume wear rate. The volume wear rate was found to be lowered while using 1 mass% PTFE, but it showed an increase with increased speed, whereas 1 mass% EC-CMC had the reverse result, showing a decrease in wear rate with increased operating speed. Under 800 rpm, 1 mass% PTFE and 1 mass% EC-CMC reduced volume wear rate by 75.78% and 41.90%, respectively. At 1000 rpm and 1200 rpm, the reductions were 52.11% and 49.27%, and 59.05 and 53.52%, respectively when compared to base jatropha lubricant. Commercial shell performance produced excellent results, with values remaining consistent as speed increases. This was due to the shell lubricant's effective anti-wear attributes. The results of the volume wear rate indicated that incorporating an anti-wear additive to the newly developed polymer lubricants would improve their anti-wear qualities and make them more comparable to shell lubricant.

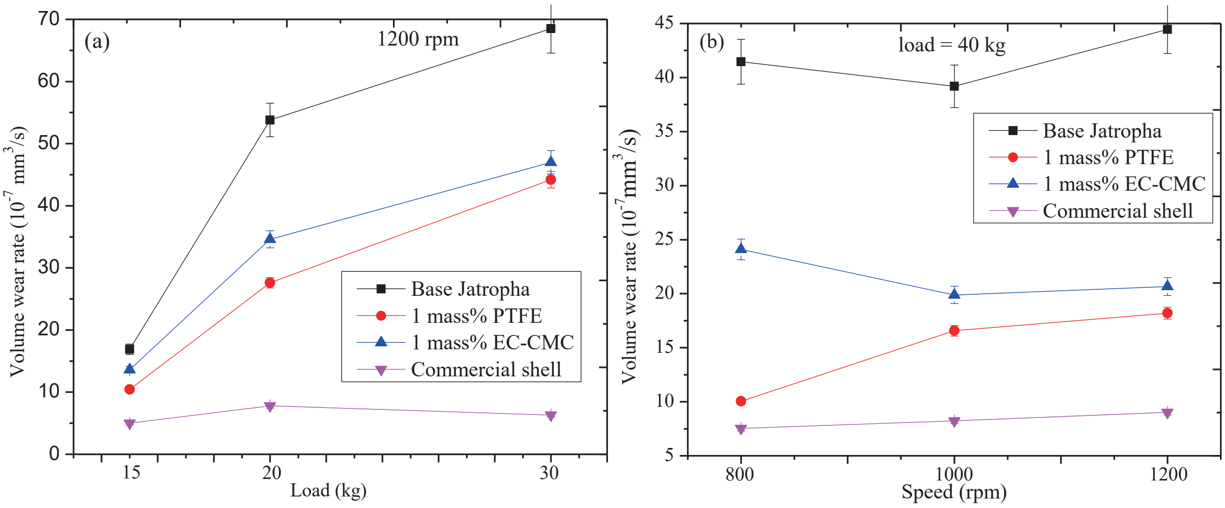

Fig. 7 Volume wear rate of the lubricants tested under different loads (a) and speed (b) at 75°C

4 Worn surface analysis

Figures 8 and 9 depict the worn surface morphology and 3D topography of the samples taken under an optical microscope, highlighting the wear zone and surface roughness. In Fig. 10, the SEM surface images, and EDS spectrum were displayed. This was taken from the operations at a speed of 1200 rpm with a load of 40 kg, which produced the best COF performance. All the tested samples, apart from the commercial shell lubrication sample, showed uneven and grooved wear on the worn surfaces when the lubricated surfaces were closely examined, indicating the commercial shell reference lubricant's superior protective abilities. As shown in Fig. 8(a), base jatropha produced more abrasive and deeper parallel grooves wear on the surface due to direct contact on the tribo-pairs, leading to high COF and WSD. PTFE produced less WSD (Fig. 8(b)), though EC-CMC produced smoother surface (Fig. 8(c)), aside from commercial shell, which gave excellent performance in terms of wear protection with

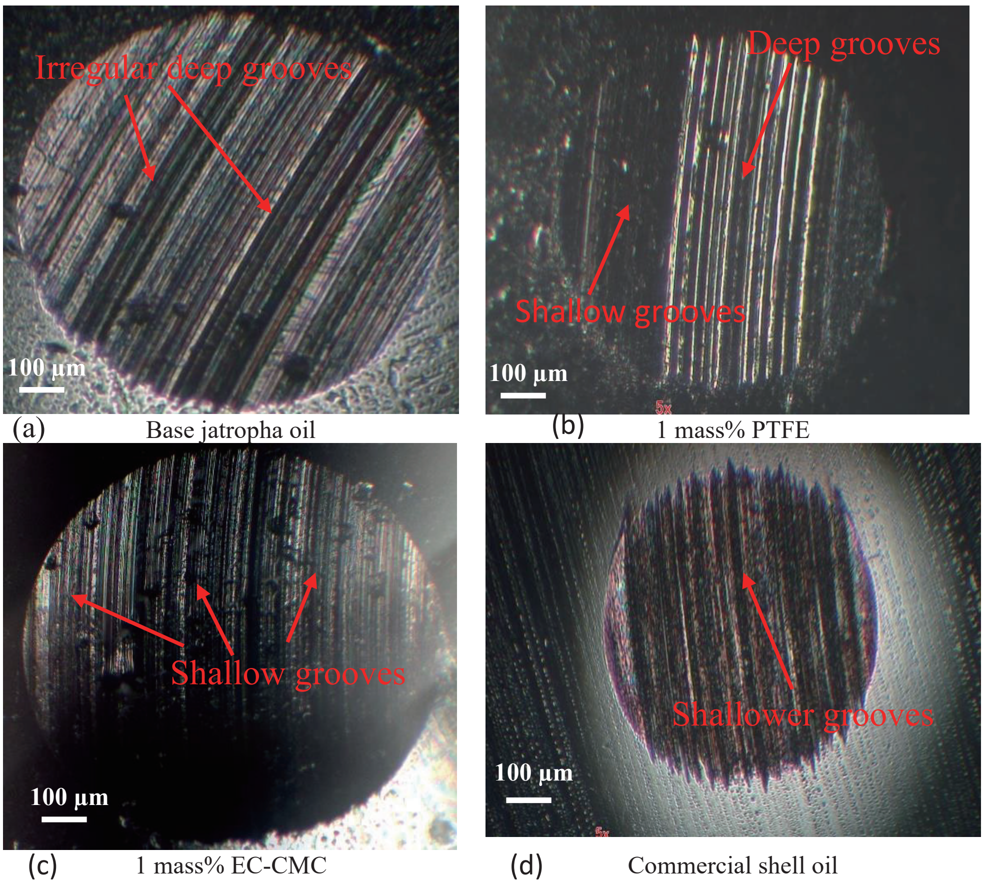

Fig. 8 Microscopic images presenting the worn surfaces under various lubricants: Base jatropha (a); 1 mass% PTFE (b); 1 mass% EC-CMC (c); Commercial shell lubricant (d)

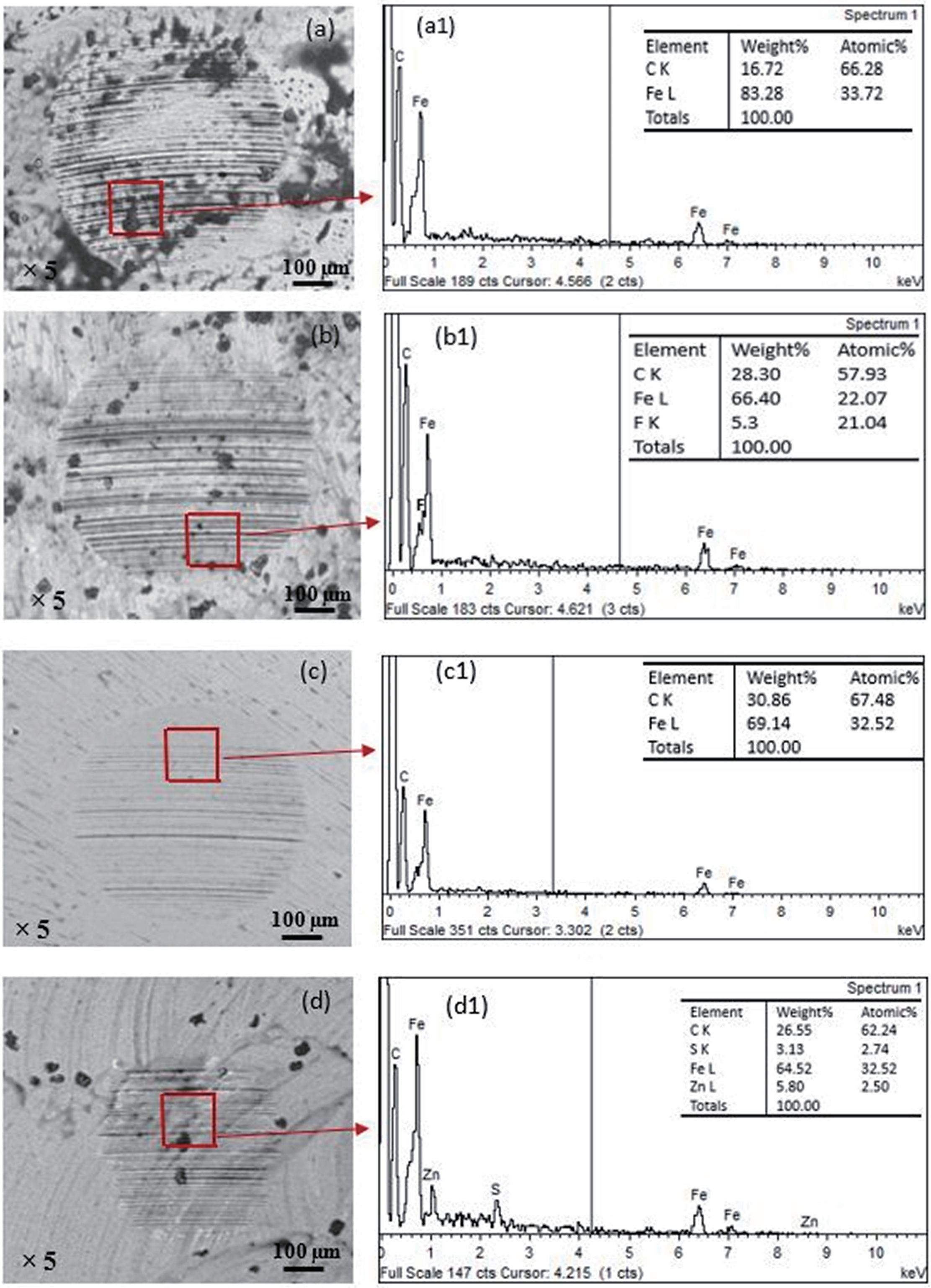

Fig. 9 SEM images (a-d) and the corresponding EDS (a1-d1) of the tested samples; Base jatropha oil (a-a1); 1 mass% PTFE (b-b1); 1 mass% EC-CMC (c-c1); Commercial shell oil (d-d1)

lowest WSD (Fig. 8(d)).

The analysis proceeded further and conducted more surface research to determine the efficacy of the lubricants towards surface wear protection to support the optical surface morphology on the samples. The surface Ra and Rz for each of the various surfaces were examined under surface profilometer. The mean average surface roughness was determined via three separate lines during the Ra and Rz investigation. According to investigations, base jatropha exhibits wider wear with the highest Ra and Rz values, which are 2.266 µm and 10.533 µm, respectively. When PTFE and EC-CMC were analyzed, the processes produced Ra and Rz of 1.565 µm, 7.633 µm and

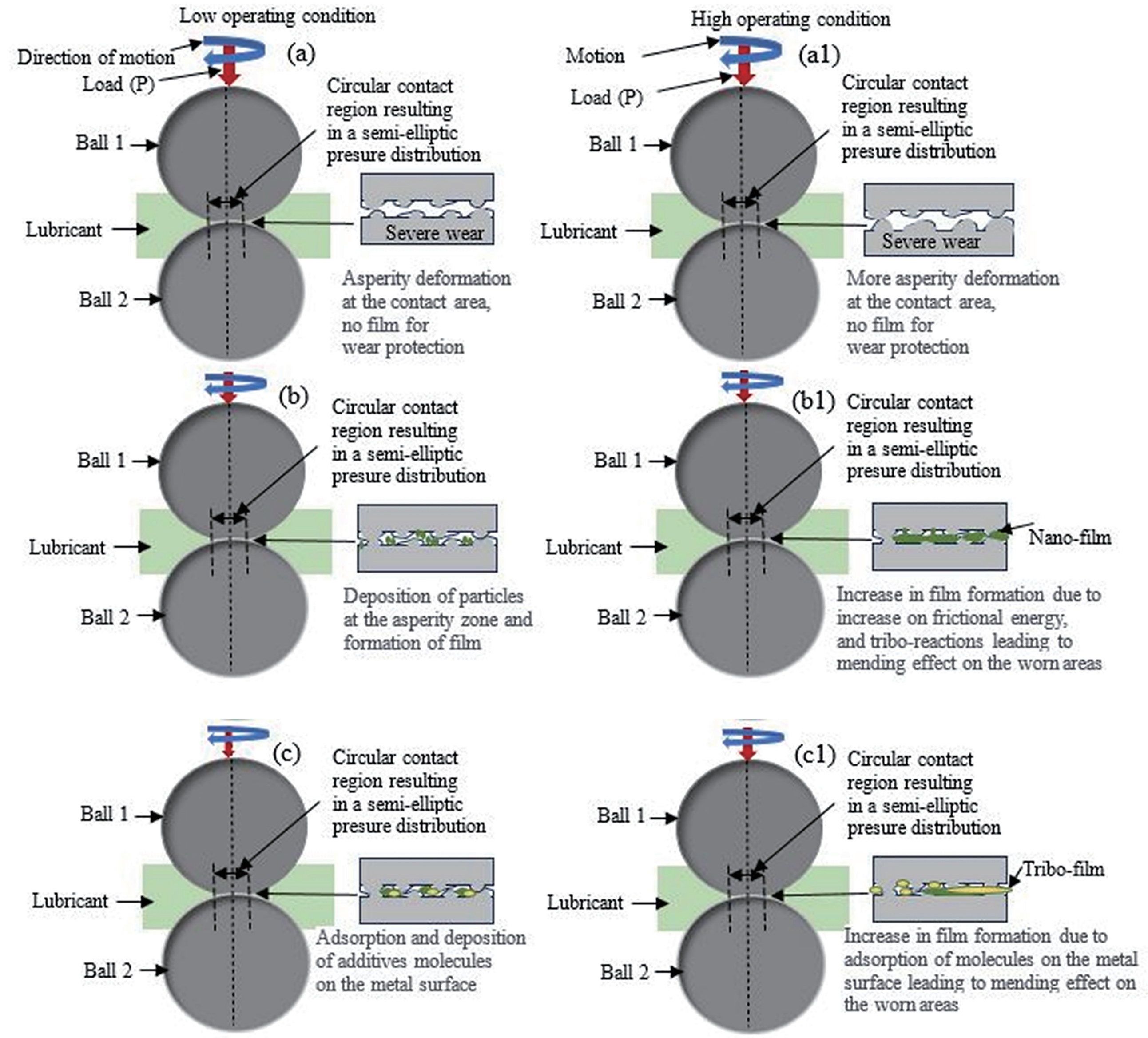

Fig. 10 Lubrication mechanism at lower operation (a-c) and higher operation (a1-c1) of the lubricated surfaces: Base jatropha oil (a-a1); 1 mass% PTFE (b-b1) and 1 mass% EC-CMC (c-c1) under applied load of 40 kg at sliding speed of 1200 rpm for duration of 1 hr

1.516 μm, 6.901 μm, respectively. Commercial shell showed outstanding performance on Ra (1.104 μm) and Rz (3.033 μm), superior to all other evaluated lubricants.

Further experimental proof of the tribo-film on sliding contact interfaces under different lubricants is provided in the study as presented in Fig. 9. The EDS element maps, as indicated by the red boxes in Fig. 9(a), revealed the presence of iron (Fe) and carbon (C) on the rubbing surface wear the base jatropha lubricant, but of very high of Fe. The increase in Fe was due to direct contact between the sliding tribo-pairs, owing to the lack of film during the operation. Order lubricants identified the existence of F in addition to Fe and C, which was caused by the PTFE additive used (Fig. 9(b)). In contrast, EC-CMC created a large proportion of C with produced Fe (Fig. 9(c)). The presence of C as revealed by EDS was due to high amount of carbon in EC-CMC additive as indicated in Table 2, thus was the reason for the C found lubricated surface with EC-CMC as confirmed by EDS analysis. This promotes in the production of tribo-films, which reduce wear and friction but do so by activating frictional energy under increased operating conditions. By diffusing into the sliding contact and limiting direct contact between the tribo-pairs elements, the nano particles (EC-CMC and PTFE) contribute in wear-reducing mechanism. This is similar with previous observations during polymer lubrication operations [10, 34]. Several elements were discovered using commercial shell lubricant (see Fig. 10(d)). According to the study, the presence of the identified components in CSO significantly enhanced wear reduction.

Figure 10 briefly explains the schematic of tribological mechanisms of the base jatropha, EC-CMC and PTFE nanoparticles in base jatropha oil, as well as commercial oil. When only base jatropha was used, substantial wear was observed as working conditions increased, as shown in Fig. 10(a), showing lack of third body or film. The main lubrication process when EC-CMC particles were added to base oil as a lubricant additive was deposition of nano particles in the worn areas and creation of the transfer film as third body due to the relative sliding of the nanoparticles, leading to mending effect as shown in Fig. 10(b), thus supported by EDS results. A similar mechanism was seen when PTFE was used (see Fig. 10(c)). Due to the rise in tribo-reactions and frictional energy, when operating conditions increase, film formation improves. Better anti-wear properties were found under PTFE and were linked to the substrate surfaces' absorption of formulation additives, which lessened the effects of friction. The findings were in line with previous work conducted on chitosan as a bio-lubricant additive in paraffin oil [35].

The study concluded on mechanism of operation that the tribological performance of the employed additives was significantly impacted by the size of the additives and the working conditions. As working conditions are increased during testing, an active tribo-film (third body) can be created. This third body protects the material surface from direct contact, which would otherwise cause excessive wear and reduce COF. The higher the operation, compared to EC-CMC, the faster the nanoparticles penetration at the contact zone owing to PTFE nano-sized nature. This phenomenon was reported in previous literature [36].

5 Spectrochemical analyses of the lubricants

In Table 5, the lubricant's elemental chemistry was shown both before and after the operations. To perform this, the amount of an element detected in the lubricant was measured in particles per million (ppm). It serves as an indicator to determine the degree of ball wear following lubricating operations. This analysis' concept might be applied to lubricant diagnostics services. According to Abere et al., [37] assessments, the lubricant's wear protection decreased with high amount of steel ball components detected after service. A number of elements, including pure metals, alkaline earth metals, and metalloids, were found in the lubricants when they were tested. When analyzing the metal elements in the lubricants before and after service, two main sources were considered, including the tribo-pairs and the additives used. Most of these elements came from an alloy of steel, although some were also found in lubricants.

Nevertheless, additional elements in CSO were identified in larger ppm at pure state and believed to have been added as additives during formulations. It was found that the alloy steel tribo-pair's under base lubricant created higher levels of several elements, which indicates the ball's surface wasn't adequately protected during lubrication, due to lack of additive tribo-film (third-body as seen in Fig. 10(a). According to the results of the lubricant analysis, reduction on the Fe, which was the greatest element in the steel ball, was found to be reduced under PTFE and EC-CMC compared to the BJO, although the CSO produced the lowest value of Fe after the operations. However, some components were seen to be present in greater amounts under EC-CMC, confirming their inclusion in the formulation and supporting effectiveness in phytoremediation, as reported by Li et al., [38].

6 Conclusions

This study examined the impact of PTFE and EC-CMC nanoparticles on the tribological properties of base jatropha oil under sliding test conditions. The findings came to the

Table 5 Lubricants chemical composition before and after used

Elements (ppm)	Unused lubricants (Before use)				Used lubricants (after used)			
	BJO	1 mass% PTFE	1 mass% EC-CMC	CSO	BJO	1 mass% PTFE	1 mass% EC-CMC	CSO
Iron (Fe)	0	0.50	2.03	2.05	25.38	7.38	9.25	3.16
Phosphorous (P)	0	0	0	660.6	45.60	9.86	789.60	539
Copper (Cu)	0.05	0.06	3.93	0.05	0.26	0.03	5.92	0
Silicon (si)	0.16	3.77	9.35	2.65	10.89	0.17	8.75	6.77
Aluminum (Al)	0	0	3.65	0.27	0	0	0.88	0
Zinc (Zn)	0.32	0.76	1.96	906.9	34.76	1.60	3.77	871.9
Sodium (Na)	0.15	3.40	468.26	1.13	1.94	0.27	24.41	18.9
Molybdenum (Mo)	0	0	0	13.88	0	0	13.07	0
Manganese (Mn)	0	0	0.64	121	0	0	0	0
Barium (Ba)	0.19	0.26	1.11	0.39	0.96	0.21	0.71	1.23
Calcium (Ca)	0	0.23	11.38	1248	40.62	0.14	1261	2.01
Nickel (Ni)	0.49	0.23	0.17	1.06	0.15	0.04	0.54	0
Potassium (k)	0.10	0.77	24.56	0.11	0.12	0.09	1.79	2.63
Magnesium (Mg)	5.18	2.53	14.89	32.6	2.84	3.40	31.13	14.9
Silver (Ag)	0	0.5	0.02	0.05	0.08	0.09	0.03	0.02
Lead (Pb)	0	0	0	0	0	0	0	3.02
Titanium (Ti)	0.39	0.29	0.41	0.18	0.07	0.33	0.43	0.83
Hydrogen (H)	1226	1280	1399	1408	1084	1210	1402	6771
Carbon (C)	8903	7721	7463	8410	9202	8998	8162	2363
Chromium (Cr)	0	0.18	0.05	0.02	0.21	0.18	0.37	0
Boron (B)	0	0.28	0.37	0.93	0.30	0.40	1.11	0.18
Lithium (Li)	0.01	0	0	0	0	0	0.02	23.71

following conclusions:

- The research revealed that the incorporation of different polymers increased the lubricant's viscosity and VI during viscometric investigations. The formulation confirmed the presence of functional groups needed for lubrication, including aliphatic CH_2 and CH_3 groups, carbonyl, and alkene groups, using FT-IR analysis.
- The nanofluid tribological characteristics of PTFE and EC-CMC particles strongly depend on the system operational conditions including the sliding speed and working load. With three different loads (15 kg, 20 kg, and 30 kg) working at 1200 rpm, PTFE gave good COF results while EC-CMC and commercial shell oil performed poorly in comparison to base jatropha oil. However, when the load was increased to 40 kg and the speeds were varied (800, 1000, and 1200 rpm), all of the tested lubricants outperformed the base jatropha, but PTFE stands out for its exceptional performance.
- In comparison to base jatropha oil, PTFE and EC-CMC exhibit some degree of wear protection under wear-resisting conditions, whereas commercial shell lubricant offered outstanding wear reduction in both WSD and volume wear rate. When PTFE and EC-CMC were used at speeds of 800 rpm and 40 kg, respectively, they reduced WSD by 46.95% and 43.11%, while 50.99% and 50.62% at 1000 rpm, and 36.23% and 31.95% at 1200 rpm, indicating comparable performance between the two polymers.
- The EDS analysis of the elements on the worn surfaces revealed that nanoparticles were involved in tribological operation, resulting in the detection of element F under PTFE and an increase in C under EC-CMC with a decrease in element Fe for both, showing the formation of third bodies at the contacting region, reducing friction and wear. A reduction in the Fe ppm under applications of commercial shell lubricants and polymers was shown by spectrochemical analysis, supporting the wear protections from lubricants.

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