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# Tribological behavior of organic formulated polymer concentration effect in bio-based lubricant using high-frequency reciprocating rig

Anthony Chukwunonso Opia [1*], Abdul Hamid Mohd Kameil [1], Samion Syahrullail [1], Stanley Chinedu Mamah [2], Mazali Izhari Izmi [1], Che Daud Zul Hilmi [1], Abd Rahim Abu Bakar [1], Abdelgader Agilah Saleh [3]

[1] School of Mechanical Engineering, Faculty of Engineering, Universiti Teknologi Malaysia, 81310 UTM, Skudai, Johor, MALAYSIA.
[2] Department of Chemical Engineering, Alex Ekwueme Federal University, P.M.B. 1010, Abakaliki, Ebonyi State, NIGERIA.
[3] Bright Star University El-Brega, P.O Box 858, Mersa-El-Brega, LIBYA.
[*]Corresponding author: chukwunonso@graduate.utm.my

KEYWORDS

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Characterization
Base rapeseed oil
Lubrication
Tribological properties

ABSTRACT

This study developed Eichhornia Crassipes Carboxymethyle cellulose (EC-CMC) organic polymer and evaluate its concentration effect as additive with base rapeseed oil (rap. oil). The developed EC-CMC was characterized using SEM, Raman spectroscopy, FT-IR to know its nature and compatibility with base oil. The experiment was conducted in terms of coefficient of friction (COF), load carrying capacity, surface roughness (Ra) and wear scar diameter (WSD). In the area of COF, addition of EC-CMC shows significant reduction, against base oil. Under load of 100 N, the COF recorded from various concentration 0.5, 1 and 1.5 wt.% EC-CMC were 0.0699, 0.0559, and 0.0866 respectively compared to base oil with 0.0891, while commercial SAE 5W-30 has COF of 0.0547. This is 37.3 % reduction against base oil but increased by 2.6 % compared to SAE 5W-30. The load capacity of the blended lubricants revealed that higher loads provide better COF than lower load. Analyzing Ra, samples with EC-CMC yielded smoother surface than lubricated surface with base oil. Sample with 1wt.% EC-CMC yielded 47.1 % reduction wear scar dimeter against pure rap. oil. The good performance from EC-CMC is attributed to their functional groups detected by FT-IR analysis. The outcome of this will contribute to utilizing organic polymers in lubrication so as reduce emissions generated from inorganic polymers.

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## 1.0 INTRODUCTION

Lubricant's enhancement has received several attentions for past decades now. As lubrication is the application of substances to maintain the smoothness of two of more surfaces that are in motion, it was observed that additives are the only material that can provide such properties which polymers is one of them (Bovington 2010)(Jason et al., 2020). Reduction of friction ad wear through lubricants are strongly depended on formulated film (Chauveau et al, 2012). Ability of lubricant to flow is one of the most essential property of fluid, however, strongly influences by polymer (Ghaednia et al., 2013) (Jeffreys et al., 2019). As a result, between the sliding surfaces, a thin layer of lubricant prevents wear and reduces power loss (Jeffreys et al. 2019). One of the essential lubricant additive is the polymer, which serve as viscosity modifier or viscosity index improver (VII) (Martini, Ramasamy, and Len 2018). Change in viscosity due to temperature variation is detected through viscosity index (VI). Low-temperature viscosity is unaffected by these improvers (Martini et al. 2018). Viscosity index improvers are used to limit the rate of change of viscosity with temperature(Mohamad et al., 2012). These improvers have little effect on viscosity to low temperature. However, as the oil heats up, the enhancers cause the oil viscosity to rise only within limits set by the additive form and concentration (Martini et al. 2018).

The studies conducted using different polymer to improve lubricant lubricity in order to control friction and wear (Dandan et al., 2019; González et al., 2011; Huang, Li, & Cheng, 2019; Shara, Eissa, & Basta, 2018; Tohyama et al, 2009) yields positive results. According to (González et al. 2011) Self-lubricity, lower density than metallic materials, resistance to tribo corrosion or general oxidation, non-toxic design, and potential processing to final form, are all benefits of using polymeric materials (polymers, blends, and composites). However, slight change in operational conditions like temperature, pressure, speed and temperature might negatively alter the tribological performance mostly in the areas of friction and wear rate (Reeves et al., 2015)(Libor et al., 2014). The use PTFE polymer yielded high wear rate and Low friction (Mukesh Kumar et al., 2016). Also, application of Epoxy and phenolic polymers as binder agents, provides many advantages like, reduce high friction, reduces wear rate due to brittleness and micro-detaching particles (Adriana et al. 2010). The effect of lignin from hardwood and softwood biomass has been studied as a lubricating additive to ethylene glycol (Mu et al., 2018). It demonstrates the results of anti-wear property enhancement from its superior metal surface adhesion potential and superior lubrication film strength (Mu et al. 2018). The expected function of this EC-CMC polymer is to serve as viscosity improver in the base oil during lubrication. The service from this polymer would help to reduce operational friction as well as contribute in wear reduction through formation of some tribofilm on the sliding contact. Also, reduction in lubricant degradation and lifespan elongation of lubricant can achieve from use of EC-CMC polymer. Therefore, it is important to conduct tribological analysis using bio lubricant (bio-additive and bio-oil) which will be beneficial to the industry in solving the emission challenges from the use of fossil materials. This present study formulates polymer (Carboxymethyle cellulose (CMC)) using Eichhornia crassipes (EC) bio-material and tested its tribological effects on different concentration with base rapeseed oil under sliding contact. Selection of vegetable rapeseed oil in this study was based on literatures on the compatibility among bio-materials in lubrication (Beard et al. 2017; Liew Yun Hsien 2015; Opia et al. 2019, 2020; Wu et al. 2013). The use of commercial synthetic light engine oil SAE 5W-30 was employed as to ascertain the level of tribological enhancement from EC-CMC addition into base rapeseed oil.

## 2.0 MATERIALS AND METHODS

### 2.1 Materials

*EC* aquatic plant were harvested from Nigeria. Other materials needed for the formulation of carboxymethyle cellulose polymer are sodium chloride (NaCl2), sodium hydroxide (NaOH), diluted acetic acid or ethanoic acid (CH3COOH), Toluene 20 ml, ethanol (100%) 400 ml per run, isobutyl-isopropyl alcohol, Sodium monocholoacetic (NaMCA) and distilled water.

### 2.2 Synthesis Preparation of Carboxymethyle Cellulose (CMC) Polymer From EC-NPs

The nominated stem of EC was exposed to direct sunlight for about 21 days, and further subjected to 150 $^{0}$C in a sensitive memmert oven for about eight (8) hours for drier. Prior to nanoscale of EC, the size was reduced using electric dry blender. Thereafter, planetary ball mill machine (PM100) (Retsch Germany) (250mL tungsten carbide (WC) grinding jar was carefully loaded with about 70% by volume WC mill balls of 3 mm in diameter together with 33.3% by volume of dry grinded *EC* (approximately 78 mL) for production of nanoparticles for about 4 hours at a programmed speed of 300 rpm as in Figure 1.

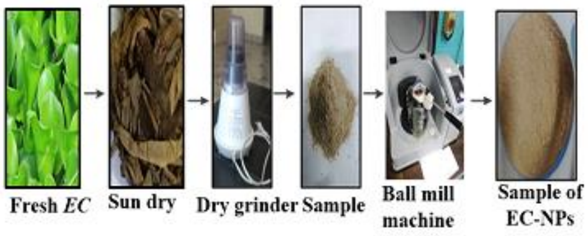


Figure 1: Preparation of EC-NPs for CMC polymer.

The *EC-NPs* is set for Carboxymethyle cellulose (CMC) polymer formulation. The cellulose constituent of the generated EC-NPs was first isolated. This stage is called pre-treatment and was done as to remove other non cellulose materials (hemicellulose, lignin, protein, etc), thereby makes the cellulose molecules accessible during the chemical treatment for hydrolysis strength (Neto 2017), known as alkali step, done to solubilize and depredate the remaining impurities and was down with aqueous solution of NaOH. 15% at room temperature followed by other steps as seen in Figure 2. However, the steps should be carefully done in order to avoid breakdown of cellulose chains during formulation of pure cellulose.

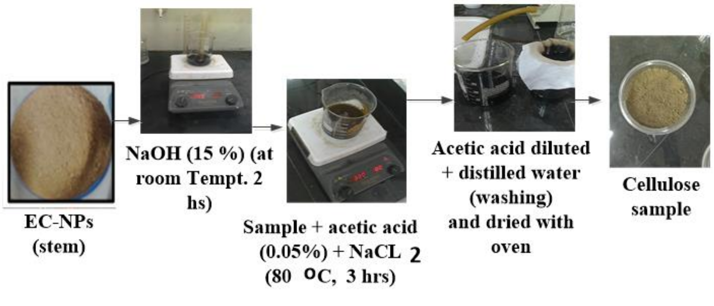


Figure 2: Preparation of cellulose for CMC polymer.

The hemicellulose remaining content was removed by purification using 170g of NaOH for two hours at room temperature, followed by other steps till the last EC-CMC product as illustrated in Figure 3.

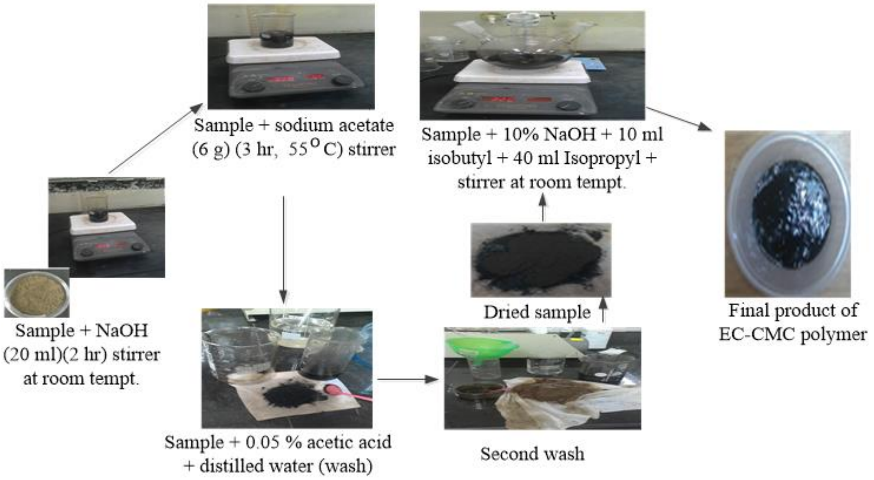


Figure 3: Schematic diagram for final CMC Polymer formulation.

### 2.3 Carboxymethyle Cellulose Polymer Sample FESEM and EDX Characterization

Field Emission Scanning Electron Microscopy (FESEM) fitted with EDX was used for the EC-CMC morphology and elemental composition respectively. The particle size analysis was analysed using Zetasizer Nano ZS; Malvern instrument, United Kingdom.

### 2.4 FT–IR Spectroscopy

The application of Infrared spectrum was employed on the selected samples (mineral base oil with and the EC-CMC formulation. The method used by Alizadeh Asl et.al., (Alizadeh etal., 2017) was applied in this analysis. The investigation on the functional group of EC-CMC and base mineral oil was conducted using infrared spectroscopy (EQUINOX 55, BRUKER Germany), carried out under wave numbers of 4000-400 $cm^{-1}$ under room temperature. Carrying out the analysis, little quantity of the samples was carefully placed on the diamond ATR accessory surface. The analysis was on spectra wave number range of 4000-700 $cm^{-1}$ with 100 scans (background/sample) at a 4 $cm^{-1}$ spectral resolution. Analysing operation data were collected using OPUS Ver. 6.0 software in ASII- computable format. After each sample analysis, the ATF diamond surface was thoroughly cleaned with acetone chemical to avoid contamination.

### 2.4 Determination of Kinematic Viscosity and Viscosity Index of EC-CMC Blended

The kinematic viscosity of Rapeseed oil without and with EC-CMC polymer additive was tested using a Cole-Palmer viscometer in accordance with ASTM D-445 and ASTM D-446, with readings taken at 5 unit intervals from 40 $^{0}$C to 100 $^{0}$C. Separately, 0.5 wt. %, 1 wt. %, and 1.5 wt. % EC-CMC polymers were added to 100 mL of rapeseed oil and thoroughly mixed for 20 minutes using an IKA T25 digital ultra Turrax homogenizer mixer set to a programmed rotational speed of 13x1000 rpm (Shara et al. 2018).

## 2.5 Experiment Procedure

The friction and wear tribological properties investigations on EC-CMC were tested on a sliding contact between a high chromium steel flat (AISI 52100) and an aluminum ball using a high-frequency reciprocating rig (HFRR) as shown in Fig 4. The experiment was conducted under low and high loads at temperature of 75 °C to ascertain EC-CMC tribological effect. The formulated EC-CMC samples of different concentrations (0.5 wt. %, 1 wt. % and 1.5 wt.%) was tested on their viscosity using viscometer while the working conditions were listed in Table 1. Before the test, samples of EC-CMC sample (0.5, 1 and 1.5 wt%) were carefully dispersed in the chosen base lubricant (50 ml) with the help of ultrasonic probe for 25 minutes. The steel plate was cleaned with neat acetone at the end of the experiments for various chemical analyses. The test was conducted with pure base oil without and EC-CMC inclusion, however, commercial SAE 5W-30 with viscosity index of 333 as stated by the manufacturer was used as a second control to compare the enhanced EC-CMC blended. The base rapeseed oil and SAE 5W-30 lubricants were purchased from Sigma-Aldrich.

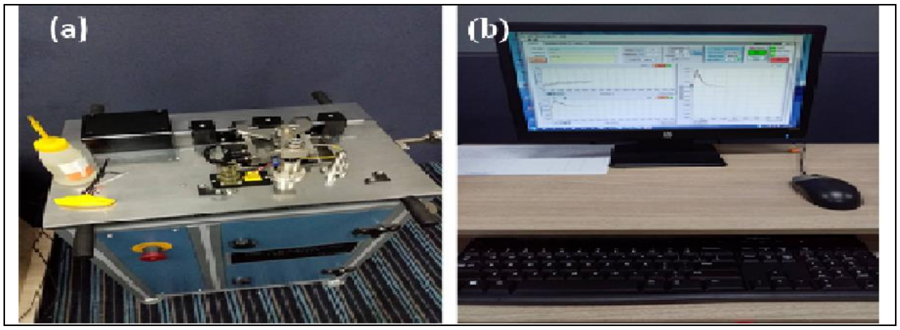


Figure 4: Machine set up (a) HFRR machine (b) system monitor.

Table 1: Operation parameters.

| Components | Properties |
|---|---|
| Flat steel plate | 570-750 Hv AISI 52100 (40*40 mm) |
| Ball aluminium | 190-210 Hv (10 mm) |
| Frequency | 5 Hz |
| Temperature | 75 °C |
| Stroke | 10 mm |
| Time duration | 10 min |

## 2.6. Friction and Wear Study

The study was carried on the sliding using the materials and conditions listed in Tab.1. Applied loads of 40 N and 100 N were used in the first stage to ascertain the various lubricant tribological enhancement. The balls used had a diameter of 12 mm and a roughness of approximately 20 nm, while the flat had a 40 mm by 40 mm diameter and a surface roughness of Ra =300 nm. The use of WINDOCUM 2010 was empolyed for the calculation COF. The study was first conducted on base rap. oil, followed the oils blended with EC-CNT (0.5, 1 and 1.5 wt %) before testing with SAE 5W-30 to compare the enhancement level of EC-CMC polymer. Further investigation was done using normal applied loads of 25 N, 40 N, 60 N and 80 N to analyse the load carrying capacity of EC-CMC concentrations and to confirm the best candidate. The use of SEM, surface profilo-meter to determine the surface roughness Ra and Wear scar diameter (WSD) of steel plat.

## 3.0 RESULTS AND DISCUSSION

### 3.1 FESEM and EDX characterization

Figure 5 displays SEM of the EC-CMC polymer, showing its gel-like surface morphology. The network structure of the gel was seen with the structure of pores. The EDX outcome, listed numerous elements as shown in Table 2.

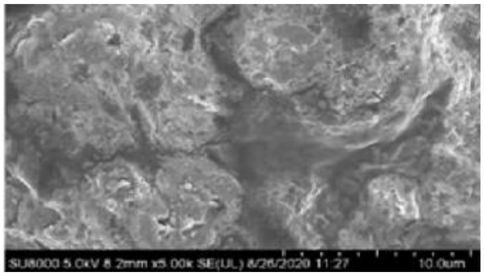


Figure 5: SEM image of EC-CMC polymer.

Table 2: Elements in EC-CMC using EDX.

| **Elements** | **C** | **O** | **Si** | **Cl** | **Mg** | **Ca** | **S** |
|---|---|---|---|---|---|---|---|
| Percent. (wt.%) | 54.3 | 12.7 | 8.5 | 6.6 | 4.0 | 3.9 | 0.2 |
| Elements | K | Fe | Al | Mo | Na | P | |
| Percent. (wt.%) | 2.6 | 2.6 | 1.7 | 1.3 | 0.8 | 0.2 | |

### 3.2 FT-IR Spectroscopy Analysis

Figure 6 shows the functional groups present in EC-CMC polymer, base rap. oil, in combination and when heated using FT-IR analysis. The samples spectrum reveals their aliphatic stretching bands ($CH_3$ + $CH_2$) at 2907.13 and 2799.74 $cm^{-1}$ with methyl and methylene ($CH_3$ + $CH_2$) functional group deformation bands at 1699.14 and 1480 $cm^{-1}$ (Ali et al. 2018). Comparing behaviors and functional groups in the various samples spectra, a large and strong band at almost 3345 $cm^{-1}$ was detected in the EC-CMC and EC-CMC blended spectra due to the O-H/N-H group (Alizadeh Asl et al. 2017). The new EC-CNT +oil (heat) band at 3328 $cm^{-1}$ lower than EC-CMC and EC-CMC + oil was observed attributed to loss of hydroxyl group owing to the heating effect (Shara et al. 2018). However, the base oil spectrum shows rocking vibration connected to the hydroxyl group (OH) at 3720 $cm^{-1}$ band. The EC-CMC polymer, EC-CMC + base oil and when heated displays more of absorption bands with stretching aliphatic bands (CH + CH) at 2907.13 and 2799.74 $cm^{-1}$ spectrum. This is because the propagation of oil monomers constituents inside the polymer chains is regarded a physical phenomenon at an ambient temperature when no external influence, therefore slight variation among the structure was observed (Shara et al. 2018). The stretching vibration of the carbonyl group C=O group in the additives was noticed with formation of peak at 1800 $cm^{-1}$ for base oil (Alizadeh Asl et al. 2017). However, a significant shift in the EC-CMC, EC-CMC blended and EC-CMC + oil (heat) sample was observed around 1593 $cm^{-1}$ due to the low concentration of COO-group. Within 1470 cm$^{-1}$, a developed peak was discovered among EC-CMC and EC-CMC + oil, which was attributed to the weak C=C group. At that region with EC-CMC (heat), slight new peak was formed, attributed to drop in C=C group due to the heating effect. The vibration of the N-H group and sharing vibration with the C-H group (Asep Handaya Saputra 2015), leads to development of peaks at 1098 cm$^{-1}$ for EC-CMC and new peak at1255 cm$^{-1}$ for EC-CMC + oil (heated) respectively. Similar spectrum trend was noted in the mixture of base rap. oil and EC-CMC + oil (heated). The new developed spectra of base oil + EC-CMC heated at 1555 cm $^{-1}$ and that of new peaks observed on EC-CM + oil elucidate the compatibility among EC-CMC and base oil lubricant, also heated sample new band at 3435 cm$^{-1}$, shows the polymer molecules expansion due to micelle increases in bulk, thus serves as viscosity improver. The observation is the same with presentation by Shara (Shara et al. 2018).

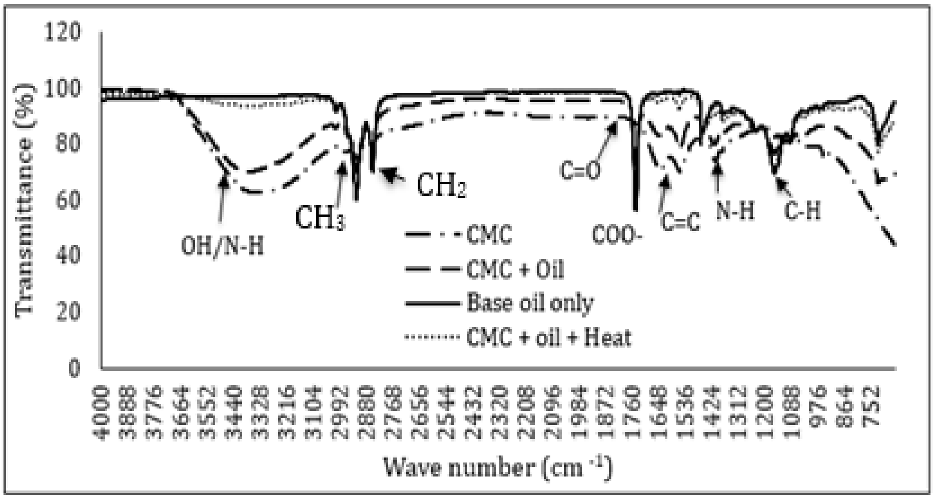


Figure 6: FT-IR analysis of base rapeseed oil, EC-CMC polymer (1 wt. %) and oil + EC-CMC (heat).

Figure 7 shows the viscometric properties of rapeseed oil and the blended 0.5 wt.%, 1 wt.%, 1.5 wt. % EC-CMC polymer tested in this study under specific gravities at 25°C, viscosity in centistoke at 40°C, 100°C, and 150°C. The viscosity indices of the samples are summarised in Tab.3. The results indicate that viscosity decreases as temperature increases. The effect is much higher at 100 °C than that at 40 °C. The viscosity index (VI) value increases as polymers are mixed into lubricating oil according to the research. Since the viscosity of lubricating oil decreases as the temperature increases, the polymer molecules expand(Alizadeh Asl et al. 2017). As a result, the micelle increases in bulk. The increased micelle size offsets the decreased viscosity of the polymer doped lubricants oil (Alizadeh Asl et al. 2017). This behavior shows that the EC-CMC exhibits the characteristics of viscosity improver polymer capable of enhance the tribological property of base oil when subjected to heat.

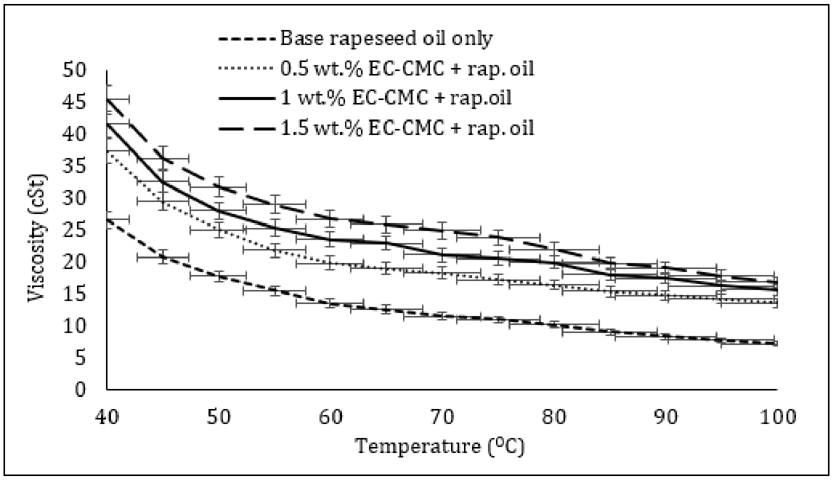


Figure 7: Lubricants viscosity versus temperatures as used in the study.

Table 3: Viscometric properties of the lubricants.

| **Lubricant** | **Specific gravity @ 25$^0$ C** | **Viscosity** | | **Viscosity Index (VI)** |
|---|---|---|---|---|
| | | **@ 40$^0$ C** | **@ 100$^0$ C** | |
| Base Rapeseed oil (BRO) | 0.890 | 27.02 | 9.48 | 368 |
| 0.5 wt.% EC-CMC + BRO | 0.985 | 38.10 | 16.71 | 448 |
| 1 wt.% EC-CMC + BRO | 1.035 | 42.70 | 17.13 | 425 |
| 1.5 wt.% EC-CMC + BRO | 0.997 | 47.04 | 21.92 | 466 |

### 3.3 Friction and Wear Characterization of EC-CMC Polymer

Figure 8, shows the COF of the different concentration of the formulated EC-CMC in base oils, base oil only and commercial SAE5W-30 under applied load of 100 N and time duration of 10 min. Under a higher load (100 N) and a frequency of 5 Hz, the friction reducing effects produced by the addition of EC-CMC can be seen more clearly. All concentrations (EC-CMC) in base oils have lower coefficients of friction compared to base oils, though 1.5 wt.% EC-CMC COF is close to that of base oil. The COF under 1 wt.% EC-CMC was 0.0559 while base oil and SAE 5W-30 were 0.0891 and 0.0547 respectively. This is 37.3 % reduction when compared to that of base oil. The COF generated by 0.5 wt.% EC-CMC was 0.0699 with COF reduction of 27.5 % against base oil. It was observed that the COF of all the EC-CMC concentration were of similar trend decreases as time increase. In the case of base oil, the COF decrease at the start and increase around 110 s till the end. The result revealed that with 1.5 wt.% EC-CMC was poor due to some aggregates recorded. The reduced COF from the EC-CMC samples could be from formation of smoother sliding surface through developed film leading to friction reduction. Moreover, the EC-CMC polymer's good performance was caused by the frictional energy produced from the sliding mechanism. The effect of the EC-CMC indicated that the optimal outcome concentration was 1 wt.% EC-CMC. This finding is similar to the presentations on performance of polymer by Tohyama et al., (Tohyama et al. 2009).

Figure 9 shows the effect of EC-CMC concentration under load 40 N and frequency of 5Hz. The results depict nearly opposite effect to the operation under 100 N as shown in Figure 8. Improvement in COF was only shown with 0.5 wt.% EC-CMC with value of 0.0699, compared to base oil of 0.0848. However, with 1 and 1.5 wt% EC-CMC, gives higher results of 0.1126 and 0.1317 respectively. The result with 0.5 wt.% EC-CMC yielded 17.6 % reduction in COF while 1.5 wt.% EC-CMC increased by 55.3 % %. The findings seem to be similar to Young et. al., (Xu et al., 2015) report on average COF against testing temperature at small load and sliding speed. The poor performance from 1.5 wt.% EC-CMC could be from insufficient frictional energy and poor dispersion of EC-CMC, leading to high agglomeration effect. This could be because frictional energy was a key factor in enhancing the tribological activity of base lubricant with additives through tribo-chemistry leading to thr formation of tribo-film that reduces friction and wear at the sliding interface and possibly achieved through high load and frequency (Aldana et al. 2014).

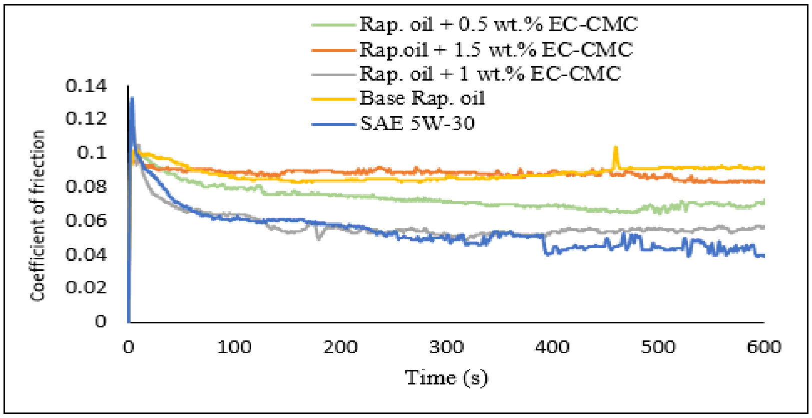


Figure 8: Friction reducing effects from different lubricants under 5 Hz and 100 N.

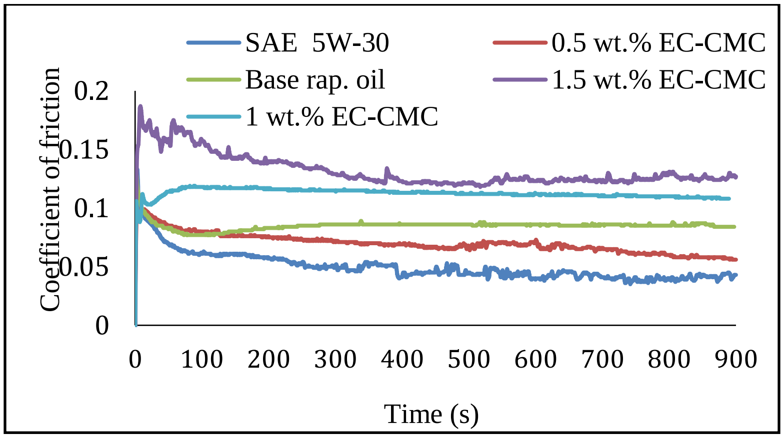


Figure 9: Effects of various lubricants under 5 Hz and load of 40 N.

### 3.4 Surface Roughness (Ra) and Wear Surface Diameter (SWD) Analysis

The tribological characteristics of the lubricants are not determine by the COF only but also with morphology of the lubricated surfaces (surface roughness and wear scar diameter). Figure 10 shows the Ra of the lubricated surfaces under 100 N with frequency of 5 Hz. The results indicated smooth surfaces from the use of EC-CMC. In Figure 10 under load of 100 N, 1 wt.% EC-CMC blended show good result with smoother surface with lower result compared base oil lubricated surface. The surface roughness reduction between 1 wt.% EC-CMC bended oil and base oil was 27 %. Surface lubricated with 0.5 wt.% EC-CMC show highest surface roughness among all the concentrations with Ra of 0.593 µm. The observations and the behavior of the formulated polymer in operation is similar with the previous presentation (Shara et al. 2018).

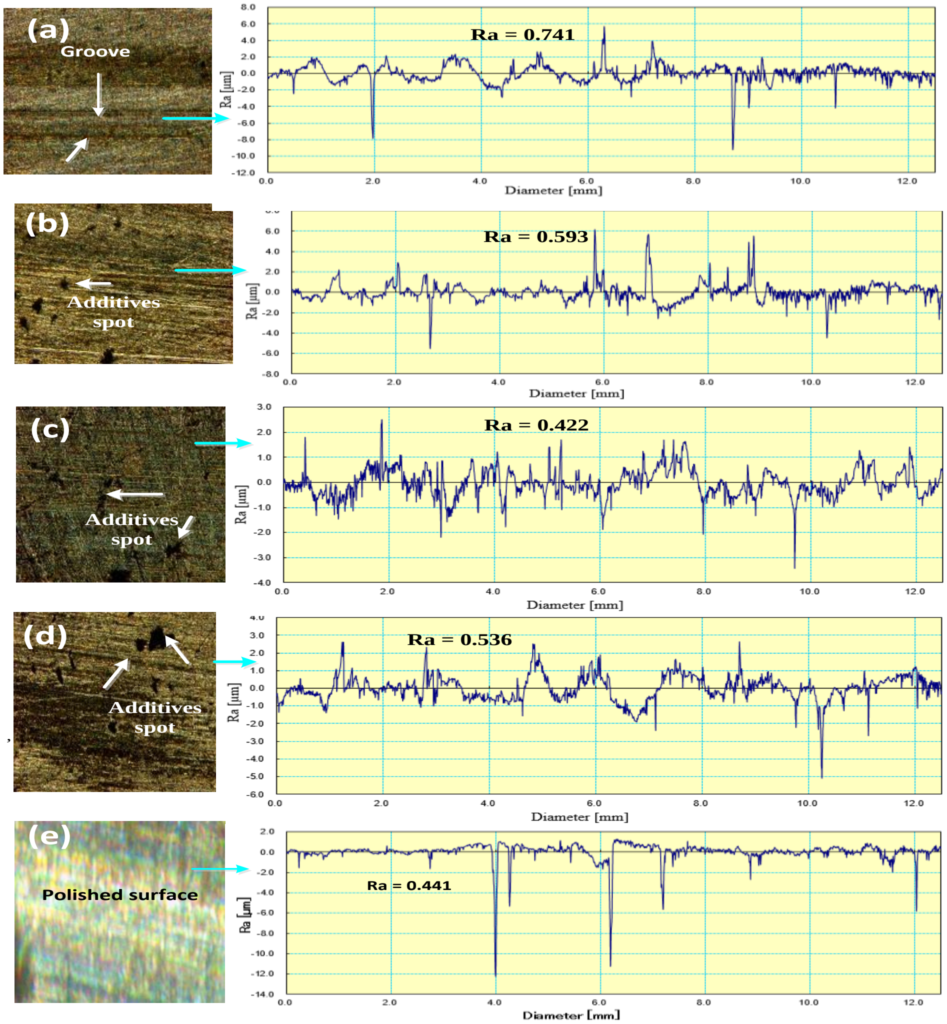


Figure 10: Surface roughness of the lubricated surfaces of various samples under 100 N and frequency of 5 Hz (a) base rap. oil only, b) Rap. oil + 0.5 wt.% EC-CMC, c) Rap. oil +1 wt.% EC-CMC, d) Rap. oil +1.5 wt.% EC-CMC, e) SAE 5W-40) .

Figure 11 shows the lubricated surfaces of SAE 5W-30, base oil without and with EC-CMC (0.5,1 and 1.5 wt. %) under load of 40 N and frequency of 5 Hz. As previously reported, lubricant samples with EC-CMC 1 and 1.5 wt.% under 40 N yielded increase in COF compared to base oil but here, gives smoother surfaces. The sliding shows Parallel grooves with numerous light scratches effect. Although much of the grooves were recorded on samples of rap. oil without EC-

CMC, but the present of EC-CMC formulate smoother sliding mechanism. The Ra reduction with 0.5 wt.% EC-CMC inclusion was 102.8 % thus yielded the best operation. The inclusion of polymers does not affect the bulk rheological properties of their blend always, but can as well develop films at the lubricating boundary which eventually leads to low friction and wear, according to previous studies (Müller et al., 2006)(Ohno 2010).

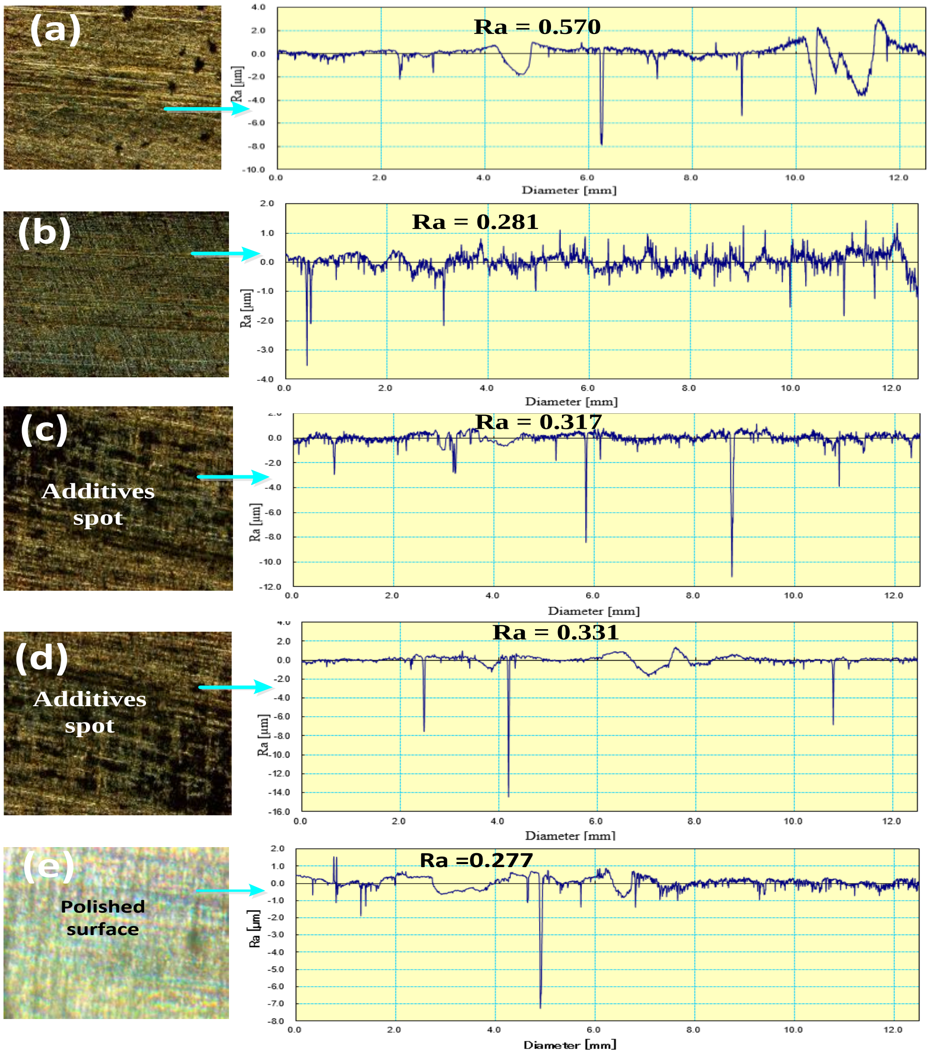


Figure 11: Surface roughness of the lubricated surfaces of various samples under 40 N and frequency of 5 Hz (a) base rap. oil only, b) Rap. oil + 0.5 wt.% EC-CMC, c) Rap. oil +1 wt.% EC-CMC, d) Rap. oil +1.5 wt.% EC-CMC, e) SAE 5W-30).

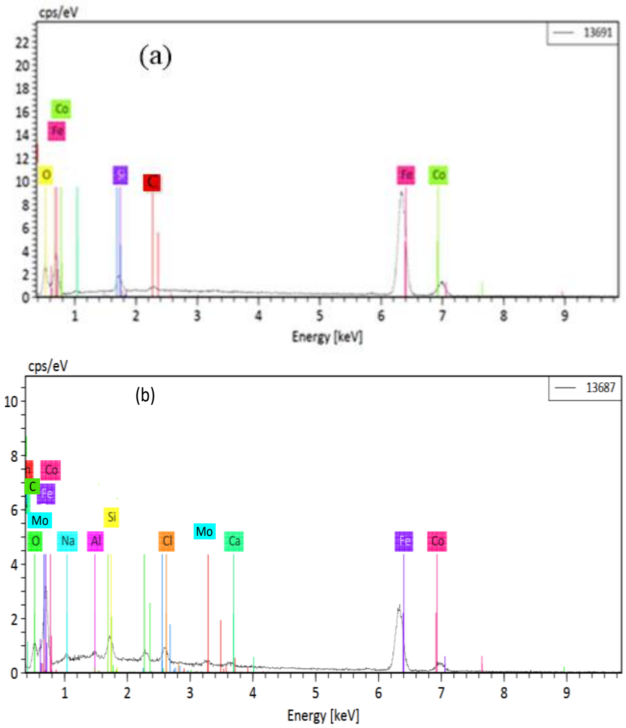
Figure 12 and Tab. 4 gives the elementary features of contact surfaces lubricated with different lubricants. Figure 11 (a) shows pronounced of Fe constituent on the surface lubricated with base rap. oil, also presence of C and O. were observed probably due to organic nature of the product. However, more of elements were observed on lubricated surfaces with EC-CMC (Figure 12 (b), (c), (d). All the graphs show element of Fe, thus generated from the steel material used. More so, surfaces of EC-CMC polymer show more elements like Mg, Si, Co, Ca, apart from O and C. Presence of Mg, Si, Co, Ca, Cl and other element on the graphs are generated from the additives. The results obviously indicate occurrences of some chemical reactions leading to formation of tribo-film for surface separation. The development of tribofilm comes from frictional energy generated from the interactions between surfaces and the additive of EC-CMC.

Figure 13 (a) and (b) shows the WSD of SAE 5W-30, rap. oil without additive and with EC-CMC additives (0.5, 1, and 1.5 wt.%) under 100 N and 40 N respectively. Rap. oil blended with EC-CMC shows significant improvement in WSD reduction. The reduction was much pronounced under 100 N than 20 N (says so due to the load difference), although the values from 40 N operation were smaller. In Figure 13 (a), 1 wt.% EC-CMC yielded the best WSD result when compared to base oil lubricated surface. Under EC-CMC concentration of 0.5, 1 and 1.5 wt.% yielded WSD of 0.049, 0.039 and 0.061*10 $^{-3}$ mm$^3$ respectively, while lubricant of SAE 5W-30 and base rap. oil gives WSD of 0.037 *10 $^{-3}$ mm$^3$ and 0.07 *10 $^{-3}$ mm$^3$ respectively. The reduction effect on WSD using 1 wt.% EC-CMC was 47.1 %, approximately the same with SAE 5W-30, compare to base oil, while 0.5 and 1.5 wt.% EC-CMC shows WSD reduction values of 48.9% and 19.7 % respectively as presented in Figure 13 (a). Figure 13 (b) shows little WSD from the lubrication. Though small WSD was recorded but inclusion of EC-CMC polymer gives better result apart from 1.5 wt.% EC-CMC. The EC-CMC concentration of 0.5, 1 and 1.5 wt.% EC-CMC gives WSD values of 0.031, 0.035 and 0.059*10 $^{-3}$ mm$^3$ respectively. The use of 0.5 wt.% EC-CMC provides the best result with SWD reduction of 12.9 % against base oil under 40 N but has lower wear reduction to that of SAE 5W-30 with value 32.3 %. However, 1.5 wt.% EC-CMC gives the worst result with increase on WSD to about 2%.

### 3.5 Load Carrying Capacity Of The Various Lubricants (base rap. oil without and with 0.5, 1, 1.5 wt.% EC-CMC) Under Different Load.

The load carrying strength of base rap. oil without additive and with different concentration of EC-CMC and SAE 5W-30 under 25, 40, 60, 80 and 100 N was conducted as shown Figure 14. The operation indicated that substantial reduction in COF and WSD were recorded with addition of EC-CMC polymer in the base rapeseed oil compared to pure base rapeseed oil. From the results, sample of 0.5wt.% EC-CMC, 1 wt.% EC-CMC and SAE5W-30 show more of decrease in terms of COF and WSD, with an increase on applied load, while samples of 1.5 wt.% EC-CMC and base rapeseed oil exhibited some levels of poor lubricity as shown in Figure 14. The study observed that additives of 0.5 wt.% EC-CMC, 1 wt.% EC-CMC, 1.5 wt.% EC-CMC, exhibits good lubricating performance in base oil under various loads. Although, some poor results were recorded under 1.5 wt.% EC-CMC operation. During the lubricants operation, the average COF of 0.5, 1 wt.% EC-CMC and SAE 5W-30 follows the same trend in terms of variation, while base oil and 1.5 wt.% EC-CMC maintain the same behavior. Testing 0.5 wt.% EC-CMC, 1 wt.% EC-CMC and 1.5 wt.% EC-CMC additives for COF and WSD. Under a load of 25 N, the average tribological properties were decreased by 17.5 %, 21 %, -2 %, 38 % for COF and 20 %, 28 %, -3 %, 33 % for wear scar diameter respectively, followed by 19.7 %, 24.1 %, 1 %, 37.5 for COF and 28 %, 34 %, 5.7 %, 46 % for wear scar diameter under 40 N. At higher load, yielded 11.2 %, 39 %, 10 %, 40.8 % for COF and 32.3 %, 38.7 %, 18.6 %, 52.4 % for wear scar diameter under 80 N, while 100 N yielded 9.3 %, 28.5 %, -4 %, 35.6 % for COF and 27 %, 47.1 %, 12 %, 47.4 % respectively against base oil. The negative value indicate increase in COF or WSD pointing at the level of poor tribological effect on the testing.

Preferable change in the additives performance was observed at 40 N and 80 N application, showing no negative value from any of the condition. This shows that load 80 N gives the best value in terms of friction and wear reduction, thus recommend the best load usage for EC-CMC polymer as shown in Figure 14 (a). The values recorded from 1 wt.% EC-CMC is close to that of SAE 5W-30, showing better performance both for low load and high load. With these obtained results, we can confidently say that 1 wt.% EC-CMC polymer and rapeseed oil performs well at low and high load and compatible. The study discovered that the COF reduction is by tribo-film formation and is a function of energy built during sliding process (Ali et al., 2018). Apart from viscosity improver operation of the EC-CMC in base oil, the inclusion of EC-CMC particles significantly reduced the wear diameter as well as surface roughness (Figure 10, 11). This could be because of the rolling behavior in separating the contact surfaces. Also the excellent performance of EC-CMC is attributed to its nano scale, thereby diffused into the worn valleys and friction zone, thus contribute during tribo-chemistry operation together the sliding surfaces to generate active tribo-film (Kalin et al. 2012; Xu et al. 2015). Further revealed that agglomeration of nanoadditives affects the friction reduction performance of lubricant's than wear scar diameter regardless of the hardness of EC-CMC polymer.



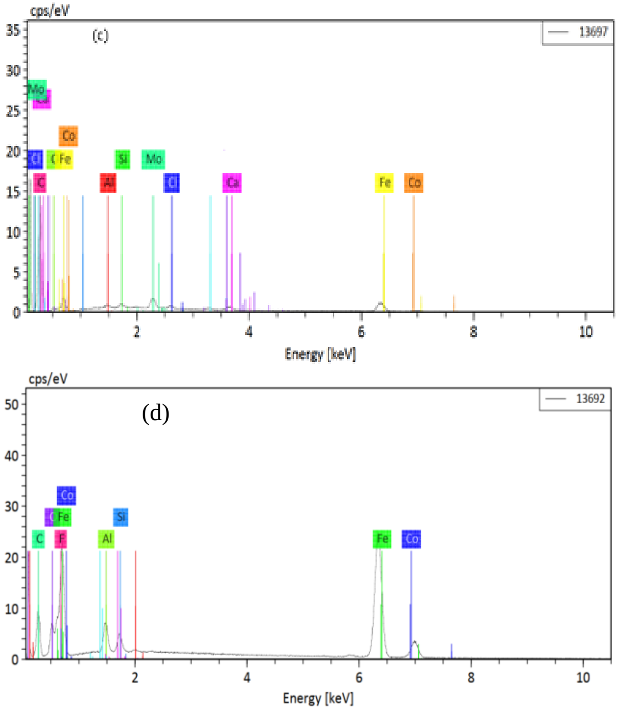


Figure 12: EDS graph of lubricated surfaces (a), Base rapeseed oil, (b) 0.5 wt.% EC-CMC, (c) 1 wt.% EC-CMC and (d) 1.5 wt.% EC-CMC under load of 100 N.

Table 4: EDS analysis of the lubricated surfaces from the different lubricants (%).

| | Fe | O | Si | Ca | C | Al | Co | Mo | Mg | Na | Cl |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Base rap. oil | 95.03 | 2.70 | 1.03 | | 2.71 | - | - | - | - | | - |
| 0.5 wt.% EC-CMC | 73.51 | 8.81 | 4.09 | 2.63 | 12.09 | 0.15 | 2.51 | 1.07 | - | 0.10 | - |
| 1 wt.% EC-CMC | 68.19 | 11.07 | 5.71 | 2.01 | 14.51 | 1.30 | 1.97 | 1.19 | 0.12 | - | 0.17 |
| 1.5 wt.% EC-CMC | 87.11 | 7.07 | 3.71 | - | 15.37 | 0.10 | 1.97 | - | - | - | - |

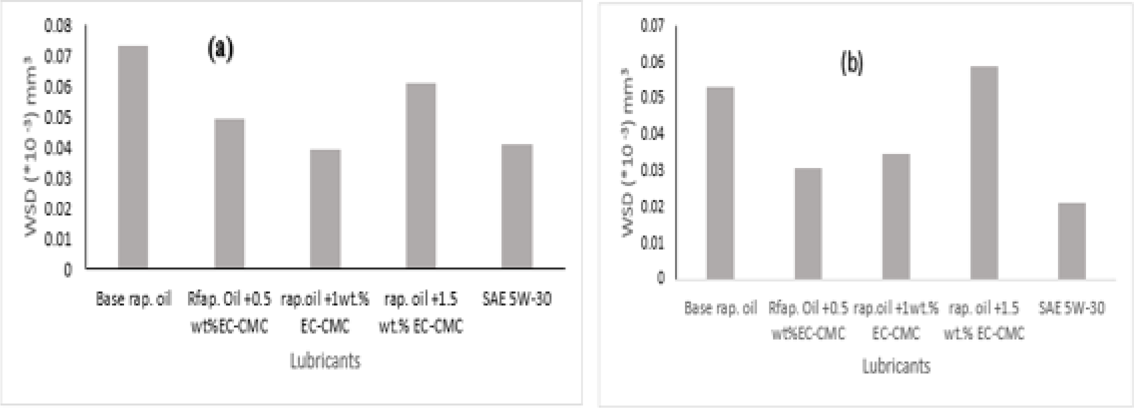


Figure 13: WSD of the lubricated surfaces (SAE 5W-30, rape. Oil without and with 0.5, 1 and 1.5 wt.% EC-CMC) under 100 N (a) and 40 N (b) with frequency of 5 Hz.

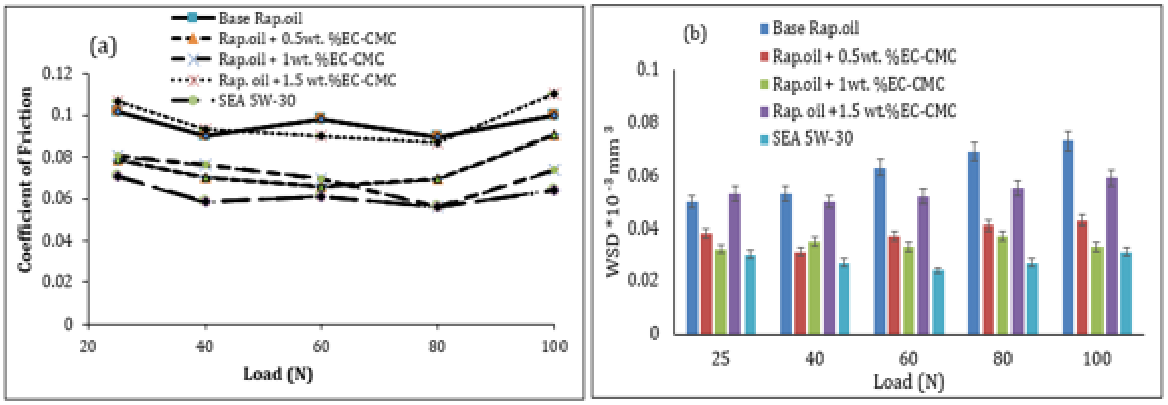


Figure 14: Load carrying capacity of the various lubricants; a) Coefficient of friction, b) Wear scar diameter (under 75 $^0$C, 5 Hz, stroke of 10 mm).

## 4.0 CONCLUSION

The results from the Experiment indicated that the use of the EC-CMC polymer as a lubricant additive substantially increased the polymeric properties of the lubricant. The tribological properties revealed that all lubricant samples enriched with ECMC polymers show superior COF reduction at high loads in 5 Hz compared to rap. oil sample. Under 100 N, 1 wt.% EC-CMC inclusion show the best effect both in COF, Ra and WSD. However, application of 40 N load, shows some poor results in COF with 1 and 1.5 wt.% EC-CMC. The use of 1 and 1.5 wt.% EC-CMC significantly affected the tribological performance on COF reduction but enhances the Ra and WSD both at low and high operating conditions. It was proposed in the surface analysis that the inclusion of 1wt. % EC-CMC polymer helps to establish smoother contact surfaces to avoid the phenomenon of serious wear and large grooves. Also, that better performance of EC-CMC required enough frictional energy, thus facilitate tribo-chemistry operation together the sliding surfaces to generate active tribo-film.

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