



## Tribological performance of novel modified Calophyllum Inophyllum nano-fluid from Pahang, Malaysia

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KEYWORDS	ABSTRACT
Calophyllum inophyllum Nanoparticles Tribology Friction Wear	This study aims to examine the performance of modified Calophyllum Inophyllum oil (MCIO) added with activated carbon made from the Calophyllum Inophyllum (CI) fruit shells as nano additives to enhance the oil performance as lubricants on friction and wear characteristics. This eco-friendly nanofluid is formulated by combining the nano-activated carbon with MCIO using homogenization and sonication techniques. These nanofluid samples are subjected to physical analysis according to ASTM standards to determine their kinematic viscosity and viscosity index. A four-ball wear test is conducted for investigating the fluid's tribological performance. It was revealed that the formulation of MCIO containing 0.025 wt. % nano-activated carbon has the lowest coefficient of friction of 0.0551, with wear scar diameter of 724.2 $\mu\text{m}$ and wear rate $5.91 \times 10^{-7}$ mm. The novel MCIO with activated-carbon nano additive demonstrated superior lubricant performance compared to synthetic ester, neat MCIO, and MCIO + 0.025 wt. % NC based on the tribological performance shown.

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## 1.0 INTRODUCTION

The increasing concern for sustainability and environmental issues has resulted in the substitution of bio-based metalworking fluids for mineral oil-based metalworking fluids. Metalworking fluids (MWFs) are generally used in machining applications such as drilling, turning, cutting, and milling (Park et al., 2011). MWFs are commercially formulated with various types of additives to promote a good lubricant layer that acts when there are movements between two or more contact surfaces. This thin lubricant film between the contact surface can create a gap that helps reduce friction and wear. Recently, biobased MWF from vegetable oil stock has received attention as it is characteristic that high in fatty acids has led to high viscosity index (VI) and high flash point value compared to mineral-based oil MWF (Fernando et al., 2019). This natural characteristic of vegetable oil has helped it to perform better as a lubricant and excelled in reducing friction and wear (Heikal et al., 2017). However, in regard to its excellent performance as a lubricant, bio-based MWFs need a few modifications in their formulation to ensure the capabilities of the MWFs to perform at various temperature levels. In this study, the bio-based MWFs derived from *Calophyllum inophyllum* fruits have been formulated with the addition of additives. Commercial MWFs usually comprise 70 to 90% of base oil while the remains are composed of hydrocarbons and non-hydrocarbon molecules (Bart et al., 2013). The main function of additives in lubricant is to improve the properties of the base oil under different conditions of operating temperature and to fill the needs of high requirements of any machinery operating system. Additives in lubricant can be categorized into several kinds based on their general roles in the improvement of tribological performance and tool life extension.

The first category of additives imparts new properties to the base oil which are known as surface protective additives. Examples of these additives are additives that act as anti-wear additives, extreme pressure additives, corrosion inhibitors, detergents, and dispersants. The second category is additives that kind of enhancing the properties that already exist in the lubricant. These additives are known as performance additives and the additives that fall into these categories are viscosity index improvers, friction modifiers, viscosity modifiers, and pour point depressants. The third category of additives is known as lubricant protective additives. The additives that fall into this category are antifoams and antioxidants. Nanoparticles are additives that belong to the first and second categories of additives, where they act as anti-wear additives, viscosity index improvers, and viscosity modifiers (Shahnazar et al., 2016).

While several studies have also been conducted to determine the influence of the size, type, shape, and concentration of nanoparticles as additives in lubricants (Oginni et al., 2019). Spherical nanoparticles have better dispersion stability than rod and tube nanomaterials, while nanomaterials with a high aspect ratio (length/diameter) are simpler to aggregate. For instance, due to low dispersion stability, it is challenging for 1D CNTs to create homogenous tribofilms on the friction surfaces to efficiently provide lubrication meanwhile 3D graphite is easily degraded during mechanical shearing, and its lubricating effectiveness and tool life are decreased (Mistry et al., 2015). Due to their high surface ratio and great van der Waals bond, graphene nanosheets generally aggregate, which has restricted the range of applications for them (Lv et al., 2020). The protective film must be replaced between consecutive contacts since it gets eliminated during the sliding operation. If the pace at which the corrosive film forms is higher than what is necessary to regulate adhesion, material loss will occur (Chemistry and Technology of Lubricants, 2010). The selection of the types of additives and their concentration is a crucial step in ensuring the tribological properties are improved as certain additives are very reactive at their optimum temperatures and increase the risk of wear. If the concentration of additives is excessively high,

the lubricant can be led to severe corrosion, however, if the concentration of the additive is insufficient to support film formation, excessive adhesive wear will happen (Dai et al., 2016).

The study by (Guimarey et al., 2021) revealed that the addition of 0.25wt. % graphene nanoplatelets (GNP) in trimethylolpropane trioleate synthetic base oil (TMPTO) has the lowest coefficient of friction compared to samples that contain 0.5wt. % and 0.1wt. %. In another study, the addition of nano zirconia oxide (ZnO<sub>2</sub>) with 0.5 wt. % with an average size of 19.45nm in Canola oil methyl ester as the base oil (COME) average frictional torque was found to be 37.2% lower than ester metalworking fluid which is a commercially used metalworking fluid while the addition of 1.0 and 1.5 wt. % of ZnO<sub>2</sub> has resulted in 6.1% and 19.4% of reduction compared to COME.

In another study (Omrani et al., 2019), the ability of porous micro and nano carbon spheres (MCNS) to absorb and store the oil was beneficial for the long-lasting development of the oil film on the sliding surface. The carbon sphere with pores acts by releasing the lubricating oil when there is compressed stress from the two-contact surface. The connection between polar and non-polar adhesion is strengthened by the well-structured porous structure and surface chemical capabilities. In this paper, the tribological performances of bio-based lubricants (MCIO) blended with nano carbon and nano-activated carbon were compared with the synthetic ester (SE, Ucut Jinen MQL) as the benchmark oil. The additives were mixed into the neat MCIO at 0.025wt. % for each additives sample respectively. To experimentally evaluate the effectiveness of these types of additives when blended in the modified Calophyllum inophyllum (MCIO) based lubricant, rheological properties (kinematic viscosity and viscosity index) and tribological properties (four-ball wear test) were conducted.

## **2.0 METHODOLOGY**

### **2.1 MCIO Sample Preparation**

The crude Calophyllum inophyllum oil (CIO) was produced locally and in-house at the materials testing laboratory located at Faculty of Manufacturing and Mechatronic Engineering Technology, Universiti Malaysia Pahang. At the first stage of chemical modification, fatty acid methyl ester (FAME) was created by chemically altering the CIO via a two-step acid-based transesterification technique. Modified Calophyllum inophyllum oil (MCIO) was created during the final transesterification process, which involved reacting FAME with Trimethylolpropane (TMP) ester and sodium methoxide (NaOCH<sub>3</sub>) at a molar ratio of 3.5:1 as depicted in Figure 1 respectively.

### **2.2 Nano-Carbon Production Process**

The main material used is the Calophyllum inophyllum (CI) shells collected after the CIO extraction. The shells were cleaned and charred at 450°C for 2 hours in the furnace to turn them into charred shells. The charred shells are crushed by using a pestle and mortar and sieved with a filter size of 30µm. These carbons powder undergo a size reduction process by using the ball-milling method for 72 hours at 3500rpm to obtain a fine nano-carbon (NC) size under 100nm.

### **2.3 Nano-Activated Carbon Synthesis Process**

The activation process was carried out by using a chemical activation process with sodium hydroxide (NaOH) as the activation agent. 15 grams of ground carbon powder were soaked in the

NaOH solution for 24 hours at ambient temperature to impregnate the NaOH into carbon powder. After the impregnation process, these powders undergo an activation process in the furnace for 2 hours at 450°C. This carbonization process helped the penetrating process of the chemical agent into the carbon powder which will react the pores to open. After the activation process, the activated sample was allowed to cool down at room temperature. Then, the activated sample was firstly washed with distilled water, then with hydrochloric acid (HCl), and finally with distilled water again until the pH of the carbon reaches 7. This washing step was needed to remove the metallic sodium, excess hydroxide, and soluble impurities in the char (Byamba-Ochir et al., 2016). The activated carbon (AC) was dried in the oven for 24 hours at 100°C to remove moisture and the excess hydroxide. To obtain a nanoparticle under 100nm, these activated carbons were milled for 72 hours using the ball-mill machine with a speed of 3500 rpm. Nano-activated carbon (NAC) samples were characterized by using field emission scanning electron microscopy (FESEM) to obtain a high-resolution image of the NAC sample to observe the change in NAC structure morphology before and after the activation process.

#### 2.4 MCIO Nano-Fluid Production Process

The acquired NAC and NC powder were dispersed into MCI0 lubricant with a concentration of 0.025 wt. % respectively. In this study, the additives were dispersed into the lubricant sample, where the initial dispersion of nano-additives was done using a homogenizer with the speed of 15000 rpm at room temperature for 2 hours and final dispersion using an ultrasonic machine (Branson 3800) with a frequency of 40 kHz at 60°C. Figure 2 shows the first dispersion process by using a homogenizer to combine the additives with lubricants while Equation 1 represents the calculation used for the nano-fluid preparation process. The rheological properties of MCI0 with NAC and NC powder were tested according to ASTM D445 (kinematic viscosity) and ASTM D2270 (viscosity) while its tribological properties were tested according to ASTM D4172 (four-ball wear test). The oil undergoes a stability test by using a storage method with a duration of 6 months to test the homogeneity of the oil with nano-additives added. After three months of storage, there was no sedimentation observed. However, after six months of storage time, slight sedimentation was observed under the bottom of the bottle.

$$\text{Concentration of particles(wt.\%)} = \frac{\text{Weight of solute(g)}}{\text{Weight of solution(g)}} \times 100\% \quad (1)$$

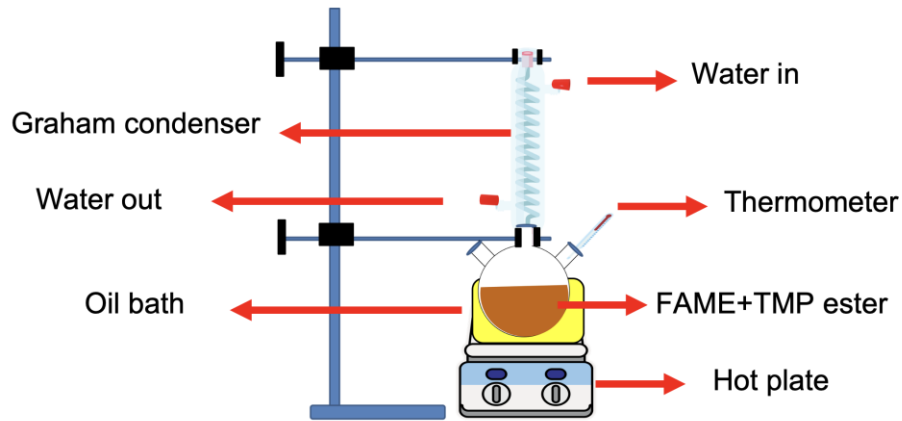


Figure 1: MCI0 transesterification process.

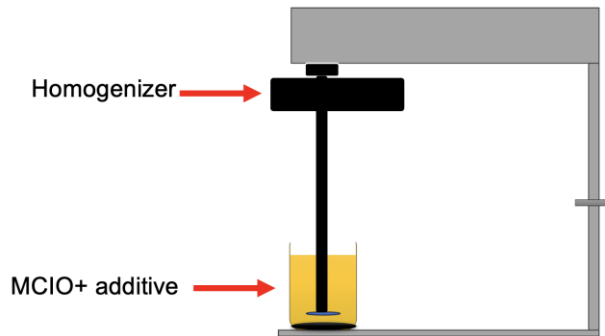


Figure 2: Additives mixing process.

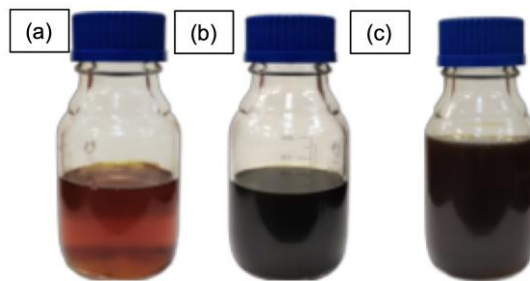


Figure 3: MCI0 sample of lubricants; (a) MCI0, (b) MCI0+0.025 wt% NC and (c) MCI0+0.025 wt% NAC.

## 2.2 Tribological Test

Figure 4(a) depicts the DUCOM TR-30 L four-ball tribo tester employed in this study's tribology test, which was conducted per ASTM D4172 standard procedure. The ball used in this test is made of chrome steel (AISI 52100), has a 12.7 mm diameter, and has a hardness rating between 62 and 66 HRC. The test rig is attached with a thermocouple for measuring the temperature. Next, the balls were prepared for testing by washing the balls with acetone to rid of the contaminants and

dust on the ball surface. The pot assembly and the lock rings were also washed with acetone to remove the lubricant residue after each test was done. Then in the ball pot assembly, three stationary steel balls were joined together, connected with lock rings, and coated with 10 mL of lubricant as shown in Figure 4(b). The rotating ball, the fourth ball, was tightened to the spindle and secured at the collet. After that, 392 N load is applied slowly on the ball pot in the machine to avoid shock from the load to the machine. The specimen was then heated to 75°C and set to run at 1200 rpm for one hour. After one hour of testing period, the lubricant sample was drained from the ball pot, and the steel balls were cleansed with acetone. The scar diameter was observed under an optical microscope and SEM to analyze the surface morphology. The diameter of each ball was measured three times to ensure the accuracy of the measurements. The procedures were repeated for other samples of lubricant. Following the test, Equation 2 was used to determine the coefficient of friction for each concentration where  $T$  is equivalent to frictional force (Nm),  $W$  represents the load(N) and  $r$  is the distance between the center of the contact surface on the lower balls to the axis of rotation.

$$\text{Coefficient of friction, } \mu = \frac{T\sqrt{6}}{3Wr} \quad (2)$$

Table 1: Four-ball wear test parameter.

Parameters	Value
Load (N)	392
Temperature (°C)	75
Spindle speed (rpm)	1200
Duration, (Hour)	1

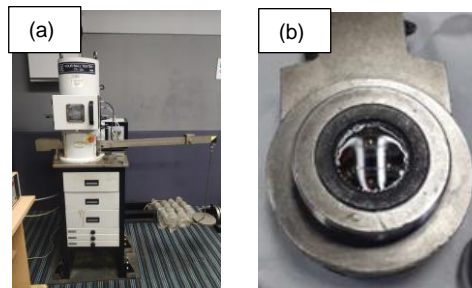


Figure 4: Four-ball wear test; (a) DUCOM TR-30L machine and (b) Ball pot containing stationary steel balls with lubricant.

### 3.0 RESULTS AND DISCUSSION

#### 3.1 Activated Carbon Surface Morphology

The activated carbon particles' chemical composition and surface morphology is characterized by using field emission scanning electron microscopy and energy dispersive x-ray (FESEM-EDX). Table 2 shows the chemical composition of the nano-activated carbon after the activation process. The activation process can be defined as a process where the carbon dioxide molecules are

diffused into the carbon particles, hence increasing the carbon dioxide reaction will create more pores in the activated carbon (Talib et al., 2021). Based on Table 2 below, the highest element concentration detected contains 56.02 % carbon followed by 18.94 % oxygen and 13.58 % sodium. The element presence in the activated carbon proved that the activation process has successfully been implemented on the carbon particles. Figure 5(a) shows the field emission scanning electron microscopy (FESEM) images of the non-activated carbon particles and Figure 5(b) shows the pores of activated carbon after 72 hours milling process. The pores formation on the NAC powders were round and large in shape indicating their high porosity and large surface area. This pore developed during the activation process which is an important step to enhance the surface area of the nano-carbon particles and the pore volume of the activated carbon (Khoo & Aziz, 2021).

Table 2: The chemical composition of activated carbon.

Element	Chemical composition (%)
Carbon	56.02
Oxygen	18.94
Sodium	13.58
Carbon	56.02

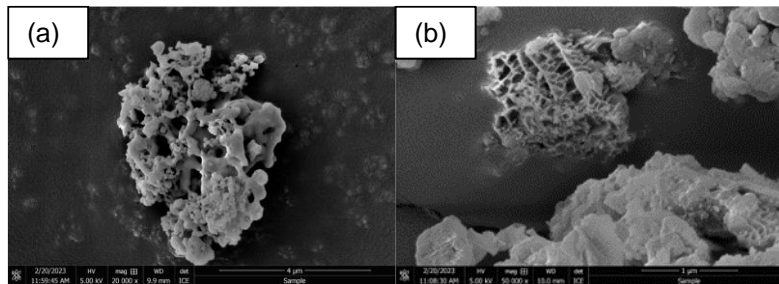


Figure 5: SEM images of; (a) Nano-carbon particles (NC) and (b) Pores on nano-activated carbon (NAC).

### 3.2 Rheological Properties of The Lubricant

Table 3 presents the rheological properties of the lubricant samples. From the table, the kinematic viscosity of benchmark oil (SE) at 40 °C and 100 °C are 24.30 mm<sup>2</sup>/s and 5.20 mm<sup>2</sup>/s respectively. The kinematic viscosity of MCIO+0.025 wt. % NAC is 26.44 mm<sup>2</sup>/s at 40°C and 6.25mm<sup>2</sup>/s at 100°C while the kinematic viscosity of MCIO+0.025 wt. % NC is 27.18 mm<sup>2</sup>/s at 40°C and 6.20 mm<sup>2</sup>/s at 100°C. The presence of 0.025wt. % nano-additives has significantly improved the viscosity index of MCIO by 20.52% for MCIO+0.025 wt. % NC and 24.96% for MCIO+0.025 wt. % NAC compared to SE respectively. The increments in the viscosity of the oils are due to the high bonding of non-polar functional groups in nano-carbon particles that exhibit high thermal stability compared to polar functional group nanoparticles in synthetic ester (Talib et al., 2022). Consequently, the presence of non-polar functional groups in bio-carbon nanoparticles, along with their high surface area of pores has contributes to the increased thermal stability of lubricating oil (Pownraj & Valan, 2020).

Table 3: The rheological properties of lubricants.

Rheological properties	Temp (°C)	SE	MCIO+0.025wt. % NC	MCIO+0.025 wt. % NAC
Kinematic viscosity (mm <sup>2</sup> /s)	40	24.30	27.18	26.44
Viscosity index	100	5.20	6.20	6.25
	-	150.21	188.98	200.17

### 3.3 Wear Scar Diameter of The Steel Balls

The wear scar formed on the ball is measured based on ASTM D4172 standard while Figure 6 shows the measurement for wear scar diameter observed under the optical microscope and the results for the wear scar diameter (WSD) and the wear rate of the ball are presented in Table 4 respectively. The diameter of the wear scar for MCIO+0.025wt. % NC has increased by 5.4% compared to SE, while the wear scar diameter (WSD) for MCIO+0.025 wt. % NAC has reduced by 7.6 % when compared to SE. This is due to SE being packed with other additives such as anti-wear additive, extreme pressure additive and surface modifier additive. While in MCIO+NAC, the smaller wear scar formed is due to the NAC porous structure that plays an important role in NAC adsorptive characteristics. The porous structure of NAC is the active site that helped to form a strong interfacial bond between carbon and metal pores (Abdul Sani et al., 2023). Generally, a material with strong interfacial bonding has high resistance to kinetic energy, leading to great adsorption force between the metal pores and nano-activated carbon pores. However, in NC structure, there were less porous structures due to its non-activated state. When there is less porous structure, the agglomeration between the particles tend to happen due to Van der Waals effect that take place during the collision of the particles with steel balls surface (Birleanu et al., 2022). This will increase the shear force acting on the steel balls contact surface and causing increment in contact area. High surface area of abrasion on the surface will occur thus resulted in high wear rate and wear scar diameter (Jason et al., 2021).

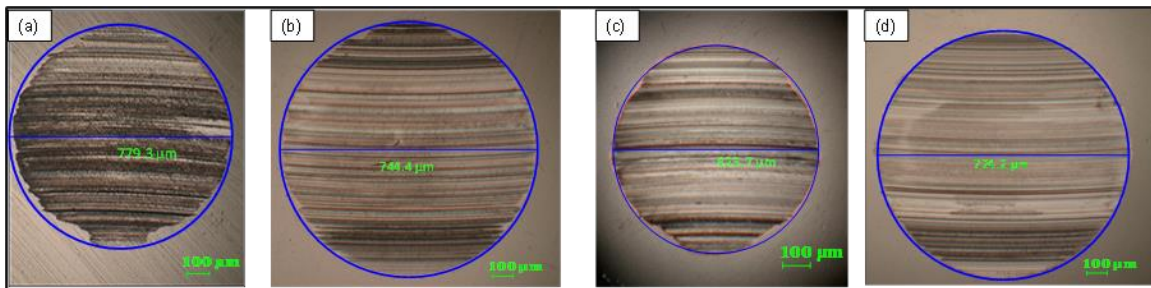


Figure 6: Wear scar diameter measurement for lubricant samples ;(a) SE,(b) MCIO,(c) MCIO+0.025wt.% NC and (d) MCIO+0.025 wt.% NAC.

Table 4: Wear scar diameter (WSD) and wear rate of the four-ball wear test.

Lubricant	WSD in mm	Wear rate in 10 <sup>-7</sup> mm
Synthetic ester (SE)	0.7793	7.92
MCIO	0.7444	6.45
MCIO+0.025 wt% NAC	0.7242	5.91
MCIO+0.025 wt% NC	0.8237	9.91



### 3.4 Coefficient of Friction (COF) For Steel Balls

Based on the COF value from Table 5, for MCIO+ 0.025 wt. % NAC and MCIO+0.025 wt. % NC, the value of COF was improved by 46.60% and 32.04% compared to SE. The coefficient of friction value from the tribological test suggested that there was presence of abrasive materials in SE that led to high coefficient of friction. Coefficient of friction (COF) is a dimensionless number that can be used to define the ratio of the friction force and normal force existence. Materials that exhibit low COF generally known as lubricous material which is less abrasive and based on the viscosity of the lubricants sample, SE exhibits the lowest viscosity index and kinematic viscosity followed by MCIO+NC and MCIO+NAC. The viscosity index of a lubricant can determines the resilience of the lubricants to maintain the tribo-film at various temperature ranges. Furthermore, the ball-bearing effect that exists has changed the sliding effect of the contact surface between the steel balls and as a result, it acts as a barrier between metal surfaces, keeping them from clashing which contributes to lower COF value for MCIO+NAC and MCIO+NC(Jun Liew et al., 2021).

Table 5: The tribological performances for each lubricant sample.

Sample	Coefficient of friction (COF)	Wear scar diameter (WSD)( $\mu\text{m}$ )
SE	0.10272	779.3
MCIO	0.05551	744.4
MCIO+0.025 wt% Nano-carbon(NC)	0.07000	823.7
MCIO+0.025 wt% Nano-activated carbon (NAC)	0.05514	724.2

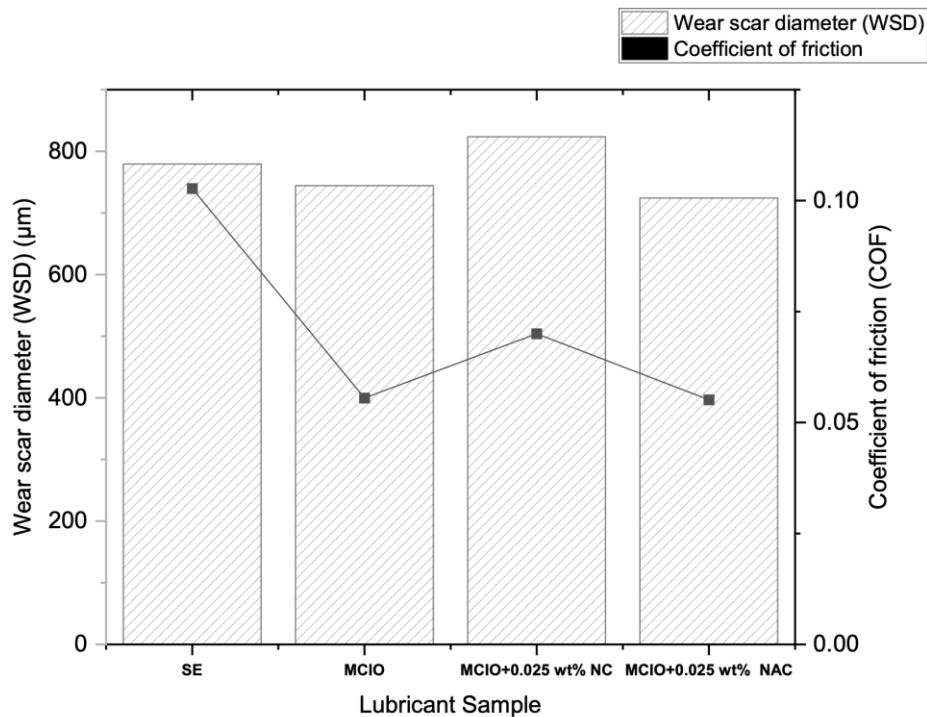


Figure 7: Graph of friction coefficient and wear scar diameter for all samples.

### 3.5 Surface Morphology Analysis

Figure 8 depicts the surface morphologies of the worn surface of the steel balls used in evaluating the lubricant sample. The examination of worn scar surfaces is essential for detecting metallic failures induced by friction and wear between contact surfaces. The formation of wear on the steel ball surface lubricated with SE shows a visible image of adhesive wear and surface oxidation on the ball as shown in Figure 8(a). There is material detached from the worn steel ball cavity and deep grooves formation due to the unstable friction coefficient behavior and lack of lubricating film coating between the interface of the balls. Balls coated with MCIO lubricant as depicted in Figure 8(b) have lower formation of deep grooves due to linoleic acid and oleic acid contents as well as the presence of TMP ester that helped sustain the formation of protective film during the test. The fatty acids in the vegetable oils act as natural tribofilms enhancers that reduce the gap between the balls during the collision. Figure 8(c) proved that MCIO-based lubricant with 0.025 wt% NAC has the lowest wear formation than neat MCIO, MCIO+0.025 wt% NC, and SE. due to the development of a lubricating film between the rubbing surfaces and the nanoparticles that serve as ball bearings between these surfaces, thus reducing the wear and the friction coefficient of the lubricating oil. Based on previous research by (Mujtaba et al., 2021), the interaction between the treated material and the additive in the given condition leads to the formation of a tribo-film and the most important features that need to be considered are the stability and dispersion of nanoparticles will contribute to better tribological properties than the other parameters such as nanoparticles' structure, size, and shape. Lubricants with MCIO+0.025 wt% NAC showed better tribological results compared to MCIO +0.025 wt% NC because of its better dispersion stability. The addition of nanoparticles based on a previous study by (Jun Liew et al., 2021) reported that the nano-additive has filled the deep groove which can enhance the wear protection capability. This act is also known as the mending effect as it also contributes to another formation of protection layer on the ball surface and improves the surface by giving a smoother worn surface.

The EDS analysis for balls coated with MCIO+0.025 wt% NAC shows the presence of carbon content at the ball surface which validates the protection layer form during the tribological test is conducted. The elements of Cr, Fe, C, and O which originated from the ball material and a thin film of nano-fluid deposited on the contact surface were detected by using energy dispersive X-ray spectrometer spectrum (EDS) as shown in Figure 9.

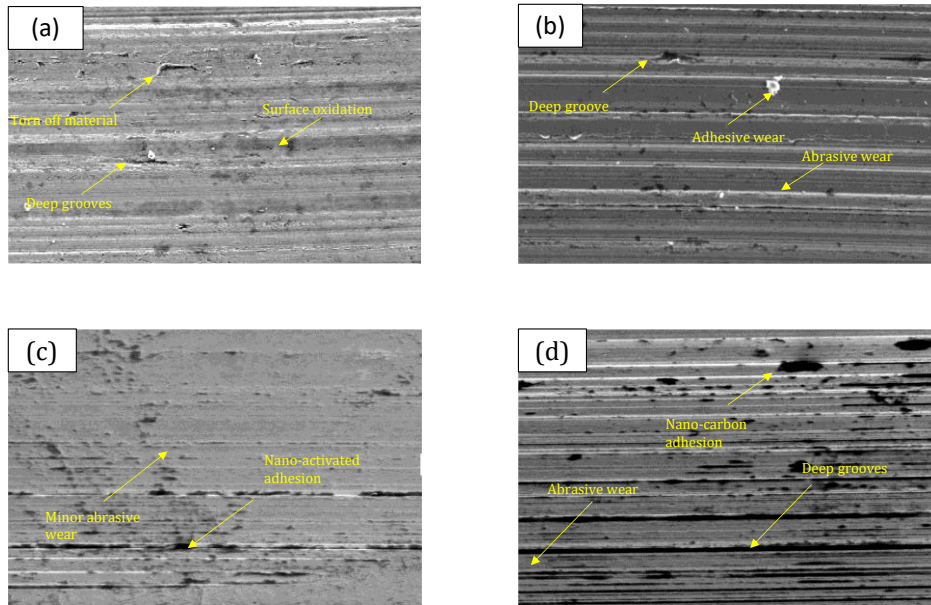


Figure 8: SEM images of worn formation on the steel balls after tribological testing (a) SE, (b) neat MCI0, (c) MCI0+0.025 wt% NAC, and (d) MCI0+0.025 wt% NC.

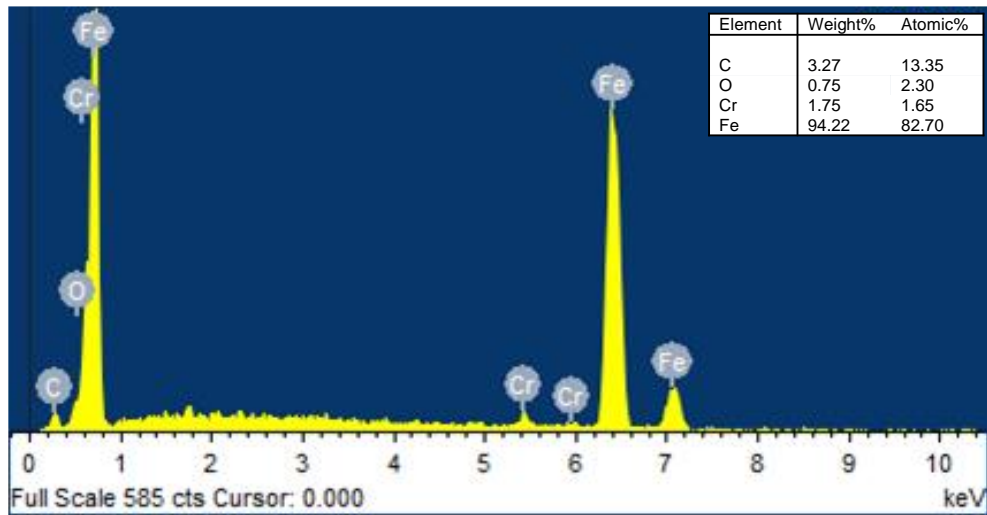


Figure 9 EDX analysis for the ball coated with MCI0+0.025wt% NAC lubricant.

## CONCLUSIONS

In conclusion, the presence of NAC, TMP ester, and natural acids of MCIO has shown a significant impact in terms of wear and friction reduction compared to a synthetic ester (SE). This modified Calophyllum Inophyllum oil (MCIO), MCIO+0.025 wt. % NAC shows better results in a coefficient of friction and wear scar diameter compared to a synthetic ester (SE) and MCIO+0.025 wt. % NC because of its porous structures. The addition of NAC particles with optimum concentration to MCIO has improved its lubricity and rheological properties by providing an excellent tribological film at the interface contact as it provides a sufficient adhesive force with the metal pores (Mat Tahir et al., 2016). By utilizing in-house made Calophyllum inophyllum nano-activated carbon and modified Calophyllum inophyllum oil, decrement in waste disposal and cost has proven that MCIO combined with nano-activated carbon can be the substitute for the existing mineral oil-based metalworking fluid.

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