



## Experimental investigation on stability, tribological, viscosity, and thermal conductivity of MXene/Carboxymethyl cellulose (CMC) water-based nanofluid lubricant

Dieter Rahmadiawan <sup>1,2\*</sup>, Shih-Chen Shi <sup>1</sup>, Zahrul Fuadi <sup>3</sup>, Hairul Abral <sup>4</sup>, Nandy Putra <sup>5</sup>,  
Ridho Irwansyah <sup>5</sup>, Dedison Gasni <sup>4</sup>, Andhy M. Fathoni <sup>5</sup>

<sup>1</sup> Department of Mechanical Engineering, National Cheng Kung University (NCKU), TAIWAN.

<sup>2</sup> Department of Mechanical Engineering, Universitas Negeri Padang, INDONESIA.

<sup>3</sup> Department of Mechanical and Industrial Engineering, Universitas Syiah Kuala, INDONESIA.

<sup>4</sup> Department of Mechanical Engineering, Andalas University, INDONESIA.

<sup>5</sup> Department of Mechanical Engineering, Universitas Indonesia, INDONESIA.

\*Corresponding author: dieter@ft.unp.ac.id

KEYWORDS	ABSTRACT
MXene Nanofluid Water-based lubricant COF CMC	This study examines the effect of MXene/carboxymethyl cellulose (CMC) water-based nanofluid applied as high performance and eco-friendly lubricant. The formulation is a two-step method by dispersing deionized water with 0.4. wt% CMC powder using sonication. The CMC solution was mixed with MXene powder in a variation of 0.35 and 0.7. wt%. Stability, thermal conductivity, viscosity, and pin-on-dis tribometer were conducted. The potential test reveals that CMC can be a better surfactant than cetrimonium bromide (CTAB). Moreover, the CMC enhances the viscosity of the base fluid by up to 46% compared to pure water. In terms of tribological properties, it has been proven that the inclusion of Carboxymethyl Cellulose (CMC) in lubricants leads to improved friction coefficients. Among the tested samples, the MXCMC0.7 exhibited the most favorable average friction coefficient (0.45), which is 25% better than water in a 4-hour tribological test. Thus, the water-based nanofluid formulation of MXene/CMC demonstrates promising performance as an eco-friendly lubricant and proves to be suitable for machining applications.

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## 1.0 INTRODUCTION

In the present era, biomaterials play a pivotal role in driving sustainable advancements in modern technology (Rahmadiawan, Abrial, et al., 2022). The world's oil reserves might run out within the next 50 years, according to the current reserve to production ratio for fossil-based oil (Ritchie et al., 2022). Furthermore, the use of fossil fuels typically results in the release of hazardous compounds into the environment, which poses a risk to sustainability. Therefore, in recent years, experts have noticed the need for a less expensive, renewable, more environmentally friendly, and more effective substitute for petroleum oils for machine lubrication (Mansor et al., 2021). Water is being increasingly considered as a potential substitute lubricant. Yet, water properties are very much different than conventional lubricant. It has low boiling point, viscosity, and thermal properties (Rahmadiawan, Fuadi, et al., 2022).

Water-based nanofluids lubricant, mixture of nanoparticle and water-based lubricant, are the most studied and utilized class of nanosuspensions in lubricant applications due to water's higher thermal conductivity and heat capacity than other base fluids (vegetable oil, ethylene glycol, etc.) (Rubbi et al., 2021). Water-based nanofluids are particularly well-suited for use as a lubricant due to their advanced thermal properties, relatively good thermal stability, and sustainable nature. It is also suitable for low-temperature manufacturing applications up to 90 °C, such as metal forming, hot or cold rolling, turning, and milling (Huang et al., 2021a)(Rahman et al., 2021). However, the high friction caused by the workpiece and tool during the process significantly increases energy losses (Xie et al., 2021). Moreover, water could also corrode metal, leading to corrosive wear. Previous research revealed that adding nanoparticles to the base fluid effectively solved this problem (Khan et al., 2020; Samylingam et al., 2018; Talib et al., 2022; Wickramasinghe et al., 2020; Yunus et al., 2020).

MXene is a new-class nanoparticle that offers exceptional promise in various fields, including energy storage, biology, and medicine (Rosenkranz et al., 2022). It has been proven that MXene nanoparticles increase thermal conductivity by up to 30.6% compared to base fluid (Mao et al., 2022). It is also preferred to be applied as heat transfer fluid in PV or thermal solar system. It is reported that MXene increased the PV system thermal efficiency by up to 98% (Abdelrazik et al., 2020). However, research regarding understanding the tribological characteristics of MXene nanosuspension is still scarce. One work reported that MXene water-based nanofluid successfully decreased the friction coefficient and the wear rate up to 20% and 48%, respectively (Nguyen & Chung, 2020). This is because the direct contact between the ball and the disk is hindered by  $Ti_3C_2$  flakes or particles that could provide a protective film or mending mechanism (Nguyen & Chung, 2020). However, the based fluid used is water which is low in viscosity. The most crucial property of the lubricant with a nanoparticle as an additive is viscosity and stability. If the lubricant is too thin and moves too freely or quickly, it does not form a good film to separate moving parts, wearing out machines more quickly. It is important to add proper additives to increase the water-based lubricant viscosity to enhance the performance of the lubricated applied machine. On the other hand, if the nanofluid is unstable, particle flocculation or lumps will be an interference that increases the COF of the system (Omrani et al., 2019). Many researchers use surfactants to overcome this matter. Surfactant is a dispersing agent that works by absorbing onto the surface of nanoparticles and forming a protective layer around them. This prevents or reduces the agglomeration of nanoparticles, promoting their uniform dispersion within the fluid. Abdelrazika et al. have reported that water based  $Ti_3C_2$  MXene nanofluid had good stability when incorporated with surfactant (Cetrimonium Bromide or CTAB) (Abdelrazik et al., 2020). However, the MXene concentration is relatively low and does not have high viscosity.

Sodium carboxymethyl cellulose (CMC), also known as cellulose gum, is one of the lubricant additives as viscosity modifiers, which is biodegradable, non-toxic, and soluble in water. The tribological properties of CMC-water suspension have been previously studied and had a very low friction coefficient (COF) (Rahmadiawan, Fuadi, et al., 2022). CMC could also act as a surfactant due to its nature that it can form noncovalent  $\pi$ - $\pi$  interaction and have a negative charge (Yu et al., 2017). On the other hand, MXene also has a strong negative charge.

As far as the author is aware, no prior research discusses the preparation and characterization of MXene water-based nanofluid incorporated with CMC. This scarcity of research can be attributed to the limited number of studies conducted on MXene thus far. In this work, we present the first-ever preparation of MXene water-based nanofluid using CMC as the surfactant. Our hypothesis is that the interaction between CMC and MXene nanofluid will yield improved stability through electrostatic repulsion. Furthermore, the high viscosity of CMC is expected to enhance the tribological performance. Therefore, our work focuses on preparing and characterizing an MXene water-based nanofluid with high MXene concentration and high viscosity by incorporating CMC as an additive. Subsequently, we investigate the stability, viscosity, thermal conductivity, and tribological properties of the  $Ti_3C_2$  MXene/CMC nanofluid.

## 2.0 MATERIALS AND METHODS

### 2.1 Materials

Table 1 provides the main properties of MXene, Carboxymethyl Cellulose, and CTAB. Titanium Carbide ( $Ti_3C_2$ ) MXene was purchased from Wuhan Golden Wing Industry & Trade, Wuhan, China. CMC was purchased from Changsu Wealthy Science and Technology, China. CTAB was purchased from HIMEDIA, India. Deionized water used for the base fluid was purchased locally.

Table 1. The main properties of MXene, Carboxymethyl Cellulose, and CTAB.

Specification	MXene ( $Ti_3C_2$ )	CMC	CTAB
Color	Black	White	White
Purity (%)	99%	98%	99%
Molecular Weight (g/mol)	-	-	364.45
Degree of Substitution	-	0.9	-

### 2.2 Lubricant Preparation

MX0.35 and MX0.7: MXene powder (0.35. wt% for MX0.35; 0.7. wt% for MX0.7) was dispersed to 300 g of deionized water and stirred at 350 rpm in 40 °C for 30 minutes. Then, 0.4. wt% of CTAB was added. To fully dispersed the MXene and water, the solution was then sonicated by a sonicator (SJIA-1200W, Ningbo Yinzhou Sjia Lab Equipment, China) with 60% power and 15 minutes duration.

MXCMC0.35 and MXCMC0.7: First, 300 g of deionized water was mixed with 0.4. wt% CMC in a beaker glass, followed by stirring at 350 rpm at 100 °C to fully disperse the CMC powder. Then, MXene powder was added to the mixture (0.35. wt% for MXCMC0.35; 0.7. wt% for MXCMC0.7). Then, it was sonicated with 60% power and 15 minutes duration. The composition of all nanofluids is presented in Table 2.

Table 2. The composition of nanofluid lubricants.

Sample	Composition in mass fraction (wt%)			
	Water	CMC	MXene	CTAB
MX0.35	98.65	0	0.35	0.4
MX0.7	98.3	0	0.7	0.4
MXCMC0.35	99.25	0.4	0.35	0
MXCMC0.7	98.9	0.4	0.7	0

### 2.3 Characterization of MXene Nanoparticles

The particle size of MXene nanoparticles was measured by using Particulate Systems Nano-Plus Zeta/Nano Particle Analyser. The measurement was repeated three times. The morphology and chemical compositions of the particles was examined using a Field Emission Scanning Electron Microscopes (JIB-4610F) and energy-dispersive spectroscopy (EDS). The thickness of the MXene sheet was investigated using ImageJ software.

### 2.4 Nanofluid Lubricant Characterization

The stability, viscosity, and thermal conductivity of all samples were investigated. The stability was characterized by visual observation and a zeta potential analyzer (HORIBA SZ-100). The zeta potential measurements were repeated three times. After preparation, four hours, and eight hours. The viscosity or rheology of the samples was measured using a rheometer (RheolabQC, Anton-Paar, Germany). The viscosity was measured in room temperature conditions, and each sample was tested three times. Thermal conductivity was investigated using a thermal conductivity analyzer (TEMPOS, Germany). A circulating thermostatic bath was used to control the temperature and measure the high-temperature thermal conductivity value. Figure 1 shows the TEMPOS thermal analyzer and circulating thermostatic bath. Since temperature consistency is vital for the TEMPOS, the sample and sensor must reach temperature equilibrium for 30 minutes before any measurements may be taken. To ensure the accuracy of thermal conductivity readings and lower the error points the device indicates, the equipment is rested for 10 minutes after each measurement before conducting the following test. The test is repeated three times to ensure its accuracy.



(a)

(b)

Figure 1: Equipment for thermal conductivity measurement. (a) Thermostatic bath; (b) Thermal conductivity analyzer.

## 2.5 Tribological Properties Investigation

A pin-on-disk reciprocating tribometer is operated to assess the lubricant samples' tribological performance. A grey cast iron (FC25), 30 mm in diameter and 5 mm thickness was used as a disk and contacted with a pin (4 mm in diameter and 20 mm in length) made of alloy steel (AISI52100). Before the test, the disk was polished with emery paper (grid #320) for 10 minutes at 200 RPM. The tribometer was loaded with a normal force of 10 N. The test duration for each nanofluid lubricant sample was four hours. Figure 2 shows the reciprocating pin-on-disk tribometer setup.

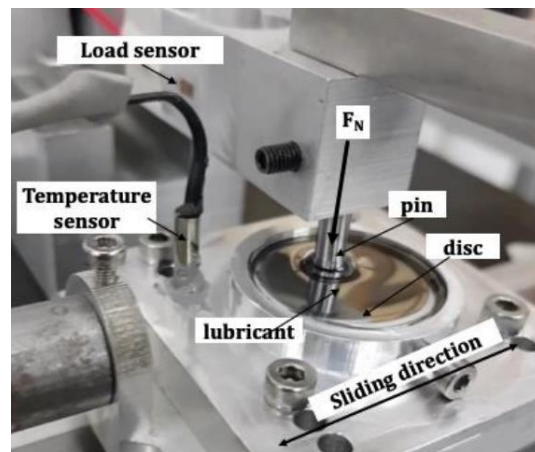


Figure 2: Reciprocating pin-on-disk tribometer setup (Fuadi et al., 2023).

## 3.0 RESULTS AND DISCUSSION

### 3.1 MXene Nanoparticles Morphology and Stability

Figure 3(a) illustrates the FESEM image of MXene nanoparticles with 5000 times magnification, while Figure 3(b) is in 50,000 times magnification, and Figure 3(c) presents the chemical composition of the particles. The  $Ti_3C_2$  MXene is obtained by exfoliating the Al element from its precursor ( $Ti_3AlC_2$ ) (Asfattahi et al., 2020). This process generally involves etching the  $Ti_3AlC_2$  with high-concentration hydrofluoric acid (HF). From Figure 3(a,b), it can be confirmed that the purchased product is a  $Ti_3C_2$  MXene. From EDS, the particle has a high amount of Ti and small amounts of Al, indicating that the etching process of the MXene preparation is successful (Naguib et al., 2012). In Figure 3(b), it also can be seen that the MXene used in this work is a layered structure, exhibiting a sheet-like morphology with a layer thickness ranging from 15 to 50 nm. This is consistent with previous research (Asfattahi et al., 2020; Feng et al., 2017; Nguyen & Chung, 2020). Figure 3(c) presents the particle size distribution of the MXene nanofluid, revealing an average particle size of 408.4 nm. This falls within the nano range, as supported by previous literature findings (Pauzi et al., 2020). This is because the sonication process breaks the flakes into smaller sizes. Sonication has been proven and guaranteed to reduce the particle size of nanoparticles in suspension form (Guo et al., 2016; Mahdavi & Talesh, 2017; Zakani et al., 2022). Particle size is an important factor in preventing agglomeration. This is because small particles have a higher surface area-to-volume ratio, which promotes stronger repulsive forces between particles (Krishna et al., 2020).

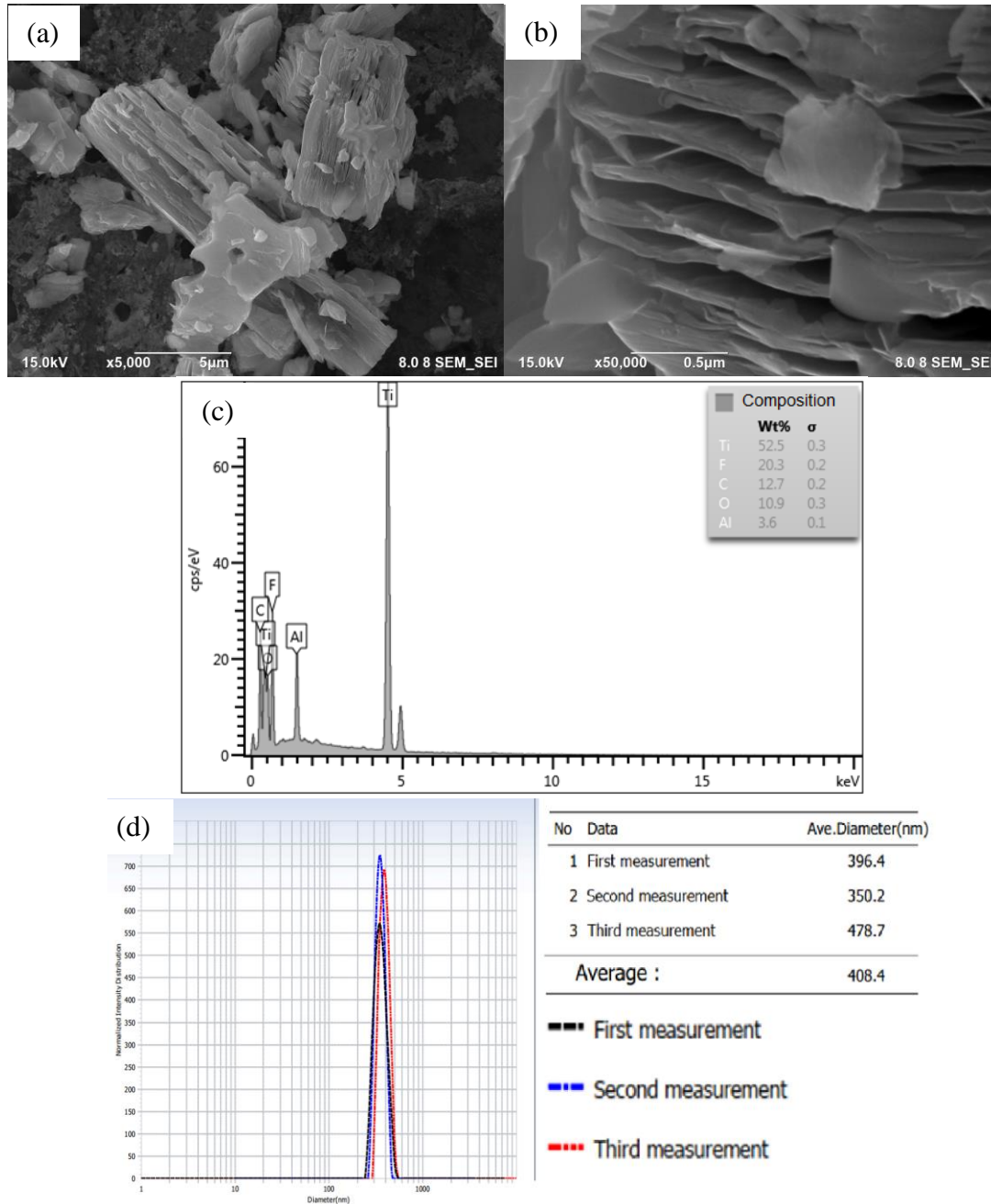


Figure 3: (a) FESEM image of MXene particles in 5000x magnification (b) 50,000x magnification; (c) EDS pattern; (d) particle size distribution.

Figure 4 presents the stability of all nanofluid lubricant samples investigated (a) after preparation, (b) at 24 hours, and (c) at 3 days. Table 3 displays the stability of all samples observed by zeta potential analyzer. According to Table 3, the MXCMC0.7 exhibited the highest

stability with a value of 30.5 mV. All samples were fully dispersed after the sonication process. The observation was conducted for three days. When applied as a lubricant, stability is an essential factor. Stable nanofluids help maintain a consistent lubricant film thickness between the moving surfaces, improving lubrication performance. If the nanofluid undergoes agglomeration or settling, it can result in uneven distribution of nanoparticles, leading to localized film thickness variations and potential performance issues (Huang et al., 2021b).

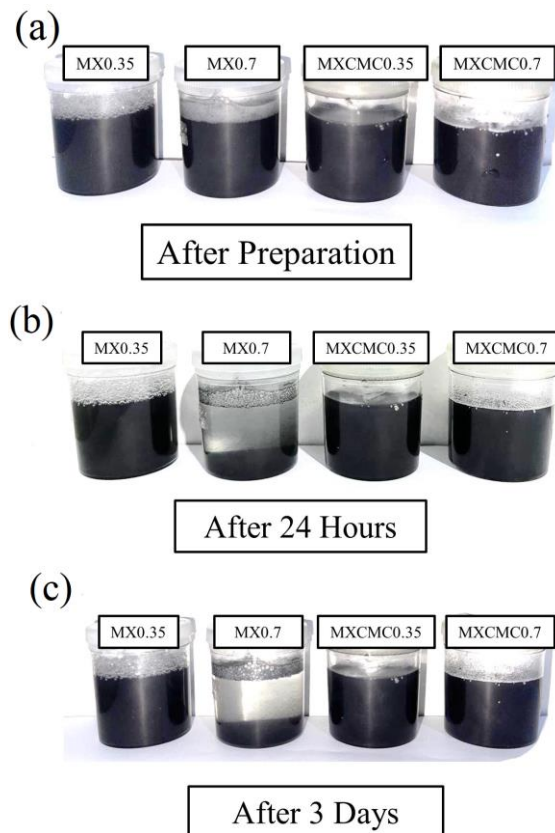


Figure 4: Visual observation of MXene nanofluid lubricants. (a) After preparation; (b) After 24 hours; (c) After 3 days.

Table 3. Average zeta potential value of all MXene nanofluid lubricants.

Sample	Zeta potential value (mV)
MX0.35	20.3
MXCMC0.35	-30.4
MX0.7	12.2
MXCMC0.7	-24.5

The MX0.35 and 0.7 samples were prepared with CTAB surfactant, and a small amount of foam appeared at the top of the samples. CTAB is a cationic surfactant known for its ability to reduce

the surface tension of liquids and facilitate foam formation (Chaturvedi et al., 2021). This foam formation phenomenon aligns with findings reported in the literature (Sarsam et al., 2016). A similar work of water/MXene (0.05. wt%) nanofluid conducted by Abdelrazik et al. reported that the nanofluid samples prepared using the CTAB surfactant are stable (Abdelrazik et al., 2020). According to our results, samples with CTAB and low MXene concentration (MX0.35) also had fair stability with no sedimentation detected, while the samples with high MXene concentration (MX0.7) were sedimented, which increased with time. This is also consistent with the zeta potential value (Table 3), which is the lowest among all. This is probably because the CTAB concentration is insufficient to create a layer or coat all MXene particles. Therefore, adding more surfactant is necessary. However, it is important to consider that the surfactant layer may hinder the efficient transfer of thermal energy, and excessive surfactant amount may lead to reduced thermal conductivity (Khairul et al., 2016).

On the other hand, it is surprising that the samples with CMC had better performance in stabilizing MXene nanofluid. Samples with CMC had higher zeta potential values compared to nanofluid prepared with CTAB. This is because of the high viscosity provided by CMC (Zainith & Mishra, 2021). Literature discloses that nanofluid settling velocity is slower when the viscosity of the base fluid is lower (Rubbi et al., 2021). Furthermore, CMC and  $Ti_3C_2$  MXene both have a negative charge (Kim et al., 2021; Z. Li et al., 2017). This leads to electrostatic repulsion, which repels the negatively charged MXene particles, preventing agglomeration or settling (Chakraborty & Panigrahi, 2020). This is consistent with the zeta potential value that shows a negative number for the MXCMC0.35 and MXCMC0.7 samples.

### 3.2 Viscosity Analysis

Figure 5 presents the viscosity of all samples at room temperature. The viscosity of nanofluids plays a crucial role in determining their lubricating performance and efficiency. It affects the thickness of the lubricant film formed between the moving surfaces. A thicker lubricant film can provide better separation and reduce the direct contact between the surfaces, minimizing friction and wear (Quinchia et al., 2014). However, it must be in a suitable amount. Thus it will be easier to pump and distribute within the lubrication system (Rahmadiawan et al., 2021). As shown in Figure 5, adding CMC increases the base fluid's viscosity significantly. This is consistent with the previously reported work (J. Li et al., 2015; Zainith & Mishra, 2021). Several researchers have investigated the viscosity performance of MXene nanofluid (Jin et al., 2022; Mao et al., 2022). It can be stated that adding MXene increases water viscosity, although it is not significant and not effective if it applies as a lubricant. Adding 0.4. wt% CMC to water increases water viscosity almost four times. The MXCMC0.7 has the best viscosity value of 3.64 mPa.s.



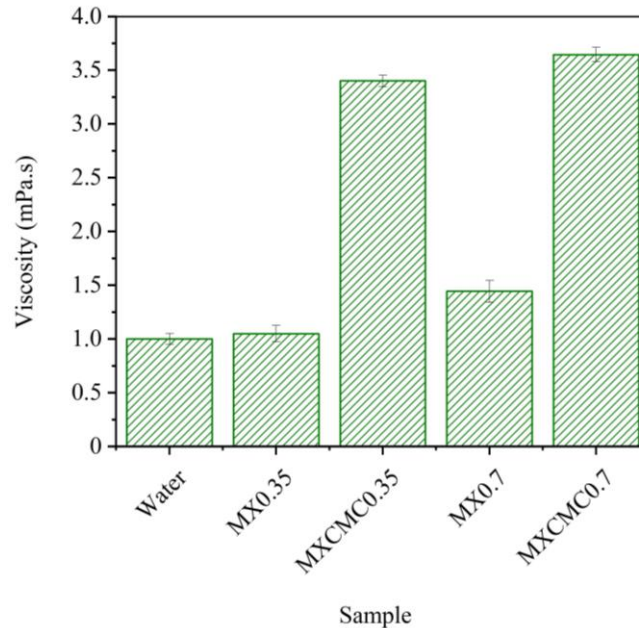


Figure 5: Viscosity of MXene nanofluid lubricants in room temperature.

### 3.3 Thermal Conductivity Analysis

The thermal conductivity and enhancement of thermal conductivity are shown in Figure 6(a) and 6(b), respectively. The error bars displayed along the Y-axis in the graphs presented in Figure 6(a) represent the uncertainties associated with the measured values. The measured thermal conductivity exhibited a maximum deviation of  $\pm 3.81\%$ . It can be noted that all MXene nanofluid has higher thermal conductivity than water. The thermal conductivity of MX0.35, MX0.7, MXCMC0.35, and MXCMC0.7 at maximum testing temperature are 0.78, 0.82, 0.89, and 0.98 W/m.K respectively, with maximum enhancement relative to water is 52% acquired by MXCMC0.7 sample. This indicates that MXene is preeminent in increasing thermal conductivity. One similar previous research worked on water-based MXene nanofluid with 0.5. wt% MXene concentration achieved a 30% increment compared to water (Mao et al., 2022). Note that samples with CMC have better thermal conductivity compared to CTAB.

The unstable MX0.7 sample has a lower thermal conductivity value than MXCMC0.7. This is probably due to the better stability that the MXCMC sample has. Furthermore, unstable nanofluid with higher settling velocity and van der Waals forces can result in reduced surface contact between MXene nanoparticles and water, ultimately resulting in lower thermal conductivity (Rubbi et al., 2021). It was stated that stability can affect the thermal conductivity of a nanofluid (X. Li et al., 2014). Agglomeration can create barriers to heat transfer, limiting the effective interparticle contact area and reducing the overall thermal conductivity (Rubbi et al., 2021).

Literature discloses that efficient heat dissipation is essential to prevent excessive temperature rise, which can lead to lubricant degradation, component damage, and reduced performance (Syahir et al., 2017). A nanofluid with higher thermal conductivity can help dissipate heat more effectively from the lubricated components, preventing overheating (X. Li et al., 2020).

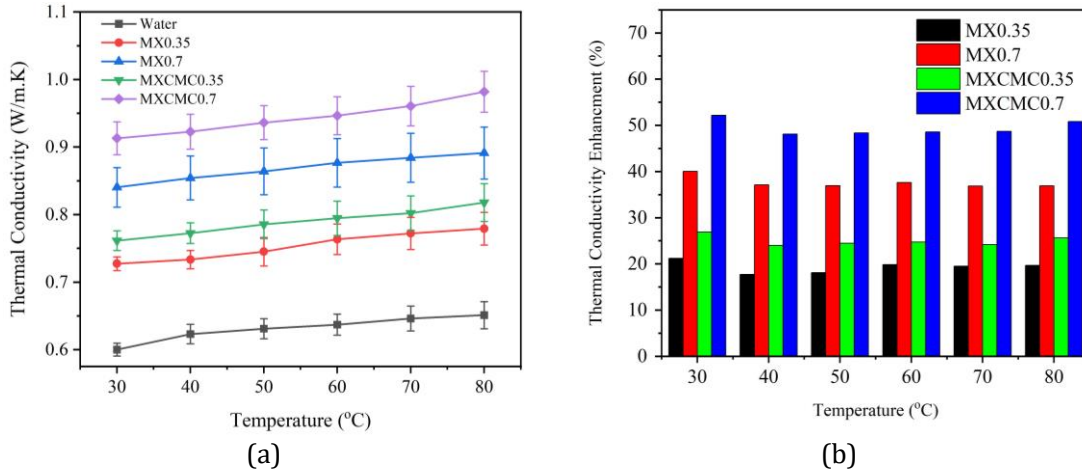


Figure 6: (a) Thermal conductivity; (b) Thermal conductivity enhancement of MXene nanofluid lubricants.

### 3.4 Tribological Properties Analysis

Figure 7 shows the COF value of the pin-on-disk tribological test using nanofluid lubricants as the lubricating fluid. Figure 8 (a), (b), (c), and (d) were the disk after the test condition lubricated with MX0.35, MX0.7, MXCMC0.35, and MXCMC0.7 respectively. It can be seen from Figure 7 that the best COF was achieved by MXCMC0.7, with a minimum COF value of 0.04, and the average was 0.45, a 25% improvement compared to water. This is consistent with good stability and high viscosity value because these will provide a thick and uniform lubricating film that reduces the impact of the rubbing surface (Bonu et al., 2016). Adding more MXene concentration also reduces the COF value, which is also consistent with thermal conductivity test results.

Meanwhile, a stick-slip was observed during the friction test using water and MX0.7. This is due to the high difference between the static and kinetic coefficient of friction value (K. Li et al., 2019). MX0.7 has low stability, and agglomeration may occur during the test, resulting in higher COF due to interference from particle lumps (Omrani et al., 2019). According to Figure 8, it can be observed that the disk lubricated with MX0.35 and MX0.7 are corroded. This is probably because MXene is not fully dispersed with water. Thus, the disk comes into contact with water instead of MXene suspension. The presence of water, combined with other factors such as dissolved oxygen and corrosive ions, can lead to corrosion of the disk surface (Rahmadiawan, Fuadi, et al., 2022).

On the other hand, as shown in Figure 8(c,d), the disks lubricated with MXCMC samples exhibit less corrosion compared to those without CMC (Figure 8(a,b)), which can be stated that MXene has anti-corrosion abilities. This observation is in line with previous studies that reported similar findings (Yan et al., 2019). Our morphology analysis also supports this result, as their layered structure and nanosheet morphology provide a large surface area. This large surface area allows for a higher concentration of active sites, which can interact with corrosive species and form protective films on the metal surface. The formation of these films hinders the diffusion of corrosive agents and reduces the likelihood of corrosion initiation (Yan et al., 2019).

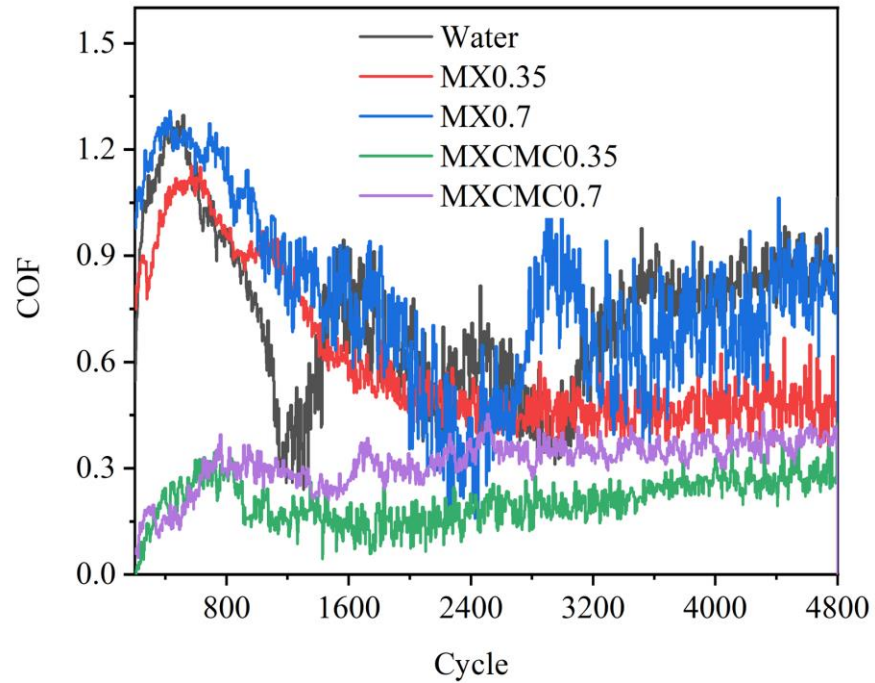


Figure 7: The friction coefficient of tribological properties test.

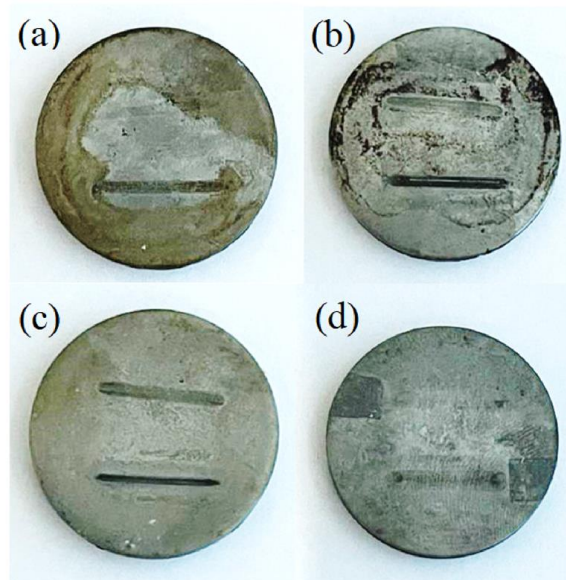


Figure 8: Disk condition after undergoing tribological test lubricated with (a) MX0.35; (b) MX0.7; (c) MXCMC0.35; (d) MXCMC0.7.

## CONCLUSIONS

This study effectively synthesizes a new nanofluid lubricant using water mixed with MXene ( $\text{Ti}_3\text{C}_2$ ) nanoflakes as an additive and CMC as a stabilizer and a viscosity modifier. The FESEM and particle size distribution test confirmed that the MXene had a layered structure with nano-size particles, with average particle size and thickness are 408.4 nm and 38 nm, respectively. The MXene nanofluid lubricant with CMC had better performance than CTAB. This is proven by better stability, thermal conductivity, and COF value. The addition of CMC improved the stability of the MXene nanofluid, resulting in higher thermal conductivity and COF and incorporating 0.7 wt% MXene in 0.4 wt% CMC improved the viscosity, thermal conductivity, and COF remarkably. The MXCMC0.7 sample achieves the best COF value. A 4-hour tribological test provides an average COF value of 0.45, 25% lower than lubricated water. This high viscosity, thermal conductivity, good stability, and low COF value suggested that this CMCMX nanofluid lubricant could be utilized as a high-performance water-based lubricant for machining applications.

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