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# ASSESSING THE LUBRICATION PERFORMANCE OF SUNFLOWER OIL MODIFIED WITH MONTMORILLONITE CLAY (MMT) NANOPARTICLES FOR INDUSTRIAL APPLICATIONS

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## ABSTRACT

*Because of the environmental impact and price volatility, there has been a growing concern about using petroleum-based lubricants. This issue has encouraged research into the development of biodegradable lubricants like vegetable oils. This study assessed the tribological behavior of sunflower oil modified with Montmorillonite nanoclay (MMT) as lubricant additives. A block-on-ring tribometer was used to assess the wear and friction characteristics of the nano-lubricants. A custom-made tapping torque tester was used to evaluate the nano-lubricants in a real-world application. It was found that the volumetric wear, Coefficient of Friction, and torque of the system decreased when MMT nanoparticles were added. The experimental results indicated that sunflower oil modified with 2.0 wt.% MMT exhibited the best tribological behavior, reducing COF and wear volume loss by 88% and 63%, respectively. Based on the results, the newly developed sunflower oil-based nano-lubricants, with the addition of MMT nanoclay, could be a promising environmentally friendly solution to compete with petroleum-based lubricants.*
Keywords: Montmorillonite clay, sunflower oil, lubrication performance

## 1. INTRODUCTION

Concerns over environmental pollution and health have sparked an interest in sustainable development, with researchers exploring viable alternatives to mineral oil, the most commonly used lubricant. Around 38 million metric tons of lubricants are used annually, and mineral oil is preferred for its performance and low cost [1]. However, reducing fossil fuels and fluctuating petroleum prices have led researchers to investigate vegetable oils as a non-toxic alternative with good lubrication properties, including high lubricity, low volatility, and high viscosity index [2,3]. Vegetable oils were extensively used for lubrication until petroleum-based oils were discovered. However, increasing regulations have pushed for sustainable production, making vegetable oils a viable alternative [4]. With over 350 crops that can be used to extract vegetable oil, common oils used in industry include canola, coconut, and sunflower oil [5,6]. Vegetable oils have polar and non-polar regions and are liquid at room temperature, making them suitable for lubrication [7]. Research is focused on improving the thermo-oxidation properties of vegetable oils, and they must meet wear and friction requirements while also meeting demand [2].

Sunflower oil, extracted from sunflower seeds, is a readily available and renewable non-volatile vegetable oil that is biodegradable and eco-friendly. Its composition varies based on the manufacturing process and selective breeding, but most sunflower oil contains linoleic acid, oleic acid, polyunsaturated fat, and monounsaturated fat. The high percentage of oleic acid (>30%) gives sunflower oil high thermo-oxidative properties compared to other vegetable oils [8,9]. Sunflower oil also has good tribological characteristics, although its composition of less stable monounsaturated and polyunsaturated fatty acids makes it susceptible to degradation by heat and air. However, adding lubricant additives can improve its lubrication properties [8].

Additives have been utilized to enhance lubricant oil properties, including extreme pressure, antiwear, film-forming, viscosity control, and deposit control additives [10]. Recent research indicates that nanoparticles have the potential to reduce friction and wear as lubricant additives. Due to their size, nanoparticles can penetrate between two contact areas, potentially reducing friction and wear through mechanisms such as the mending effect, rolling effect, protective film formation, and polishing effect [6]. Nanoparticles (NPs) have been employed as lubricant additives in vegetable oils, leading to improvements in reducing wear and friction, improving surface roughness, and enhancing tribological behavior. Several studies have investigated the effects of different types of nanoparticles as lubricant additives, including CuO, $SiO_2$, $TiO_2$, molybdenum disulfide ($MoS_2$), and Halloysite clay nanotubes (HNTs) [3, 8 11-15]. The studies utilized various tribological testing methods, such as pin-on-disc tribometer, block-on-ring tribometer, and ball-on-disk experiments. The research showed that NPs added to vegetable oils as lubricant additives reduced wear volume loss and coefficient of friction (COF), improved the formation of smoother and more compact tribofilms, and exhibited friction reduction capability comparable to conventional lubricants. The optimal concentration of NPs in the vegetable oils was also identified to enhance tribological behavior. However, a challenge in using NPs as lubricant additives is their tendency to sediment over time, leading to a non-uniform composition of the lubricant. Nevertheless, the research showed that nanoparticles have significant potential as effective lubricant additives in vegetable oils, improving their tribological behavior and extending their service life. Further research is needed to explore using nanoparticles as lubricant additives in vegetable oils under various operating conditions and address the sedimentation issue.

The addition of clay-based nano-additives to lubricants, including water-based, oil-based, synthetic, and polymeric lubricants, has been shown to enhance tribological properties, particularly friction, and wear, according to studies [16]. Montmorillonite, also known as MMT, is a naturally occurring phyllosilicate material with a layered structure that has been widely used as a reinforcement for composite materials [17]. This clay-based nanoparticle has been suggested as a lubricant additive to enhance tribological performance. MMT is environmentally friendly and occurs naturally. In a prior investigation by our group, the authors evaluated the lubrication performance of corn and peanut oils enhanced with Halloysite clay nanotubes (HNTs) and Montmorillonite nanoclay (MMT) as green lubricant additives [18]. Wear tests and surface analysis were conducted, revealing significant reductions in coefficient of friction (COF) and wear volume loss with nanoparticle additions. Tapping torque tests in an industrial application further supported the improved performance.

The main aim of this study is to assess the lubrication performance of sunflower oil modified by adding MMT (Montmorillonite) nanoparticles at various concentrations, which have not been previously explored to the best of our knowledge. We propose the use of sunflower oil as base lubricant due to the higher concentration of mono-unsaturated and saturated fatty acids, compared to corn and peanut oils previously studied by our group. These fatty acids form a fatty acid monolayer that reduces contact and friction between moving surfaces according to the literature, therefore, would provide a higher tribological performance [19]. The morphology of the MMT nanoparticles was analyzed by scanning electron microscopy (SEM). The tribological performance of the sunflower oil was evaluated experimentally through block-on-ring experiments, and the worn surfaces were analyzed by SEM and profilometry. In addition, tapping torque tests were performed to assess the nano-lubricant's performance in a real-world application.

## 2. MATERIALS AND METHODS

### 2.1 Materials

For this study, MMT nanoparticles, $(Na, Ca)_{0.33}$ $(Al\ Mg)_2$ $(Si_4O_{10})$ $(OH)_2$. $nH_2O$, were obtained from Sigma-Aldrich, and their morphology was analyzed using a field-emission scanning electron microscope (FE-SEM) ZEISS SIGMA VP. SEM micrographs of the MMT nanoparticles are presented in Figure 1. It can be observed that the MMT nanoparticles were spherical and formed large and small aggregates [20]. The surface morphology of the MMT nanoparticles displayed a layered surface with some large flakes and pores. Figure 1b shows that the mastoid was comprised of several fragments resembling a flower cluster [21]. An image processing program was employed to analyze over fifty data points in order to determine the size of the nanoparticles. The average diameter of the MMT nanoparticles was determined to be 7.57 μm, with the majority of the nanoparticles ranging from 0-5 μm. In contrast, some were significantly larger in diameter than others, which could be the reason for the large average diameter.

Additionally, commercially available sunflower oil was used as base oil, presenting the following characteristics: density (40 °C): 0.90 g/cm$^3$, viscosity (40 °C): 35 mPas, iodine value: 120-145 gl/100 g, and acid number: 0.2-0.5 KOH/1 g. The fatty acid content was 4% palmitic acid, 65% oleic acid, 26% linoleic acid, and 5% stearic acid.

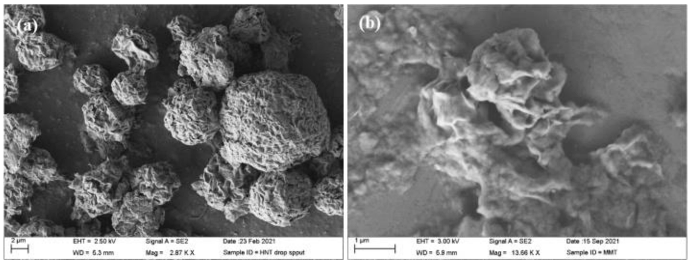

**FIGURE 1:** Morphology of the MMT nanoparticles at (a) 2.87 KX, and (b) 13.66 KX magnifications.

### 2.2 Formulation of Nanolubricants

To formulate the nanolubricants, MMT nanoparticles were dispersed at varying concentrations in commercially available sunflower oil. The nanolubricants were prepared separately using different concentrations of MMT nanoparticles (ranging from 0.50 to 3.0 wt.%) and were ultrasonically dispersed for five minutes using a 120-Watt sonic dismembrator at a frequency of 20 kHz to achieve a uniform dispersion and good suspension stability.

### 2.3. Tribological Characterization

The optimal concentration of MMT nanoparticles in sunflower oil was determined by conducting tribological tests under different conditions. A custom-made block-on-ring tribometer was used to evaluate the lubrication performance of sunflower oil with and without MMT nanoparticles under sliding conditions, in accordance with ASTM G-077-05 standards [22]. The test involved loading an AISI 304 stainless-steel block (dimensions: 14 × 6.35 × 6.35 mm, and hardness: 128 HRB) against a rotating AISI 52100 steel ring (d = 40 mm, hardness: 60 HRC), with the lubricants placed in an oil bath container to maintain constant lubrication during the test. A load of 266 N was used, and the test was performed at a temperature of 23°C, at 173 rpm, and at a contact pressure of 218 MPa for a total time of about 1200 seconds. Each test was repeated three times to ensure repeatability. Wear mass loss was determined gravimetrically using an electronic balance, with a specific density of 8 g/cm$^3$ for the stainless-steel block to convert the wear mass loss into wear volumetric loss. The friction force and temperature were also continuously measured and recorded during each test. The tribological experiments were conducted three times to ensure reliability. After the experiments, the wear areas on the worn specimens were analyzed for their morphology using a field emission scanning electron microscope (FE-SEM) ZEISS SIGMA VP equipped with an energy dispersive x-ray spectrometer (EDS) analyzer. The surface roughness on the worn areas was analyzed using a MahrSurf M300C surface profilometer.

### 2.4. Tapping Torque Tests

A custom-made tapping torque tester was used to perform tapping torque tests on newly developed nanolubricants, following the ASTM D5619 (2011) standard procedure [23,24]. Aluminum 6061 cylindrical specimens were used for the testing, with dimensions of 25.4 mm in diameter and 38.1 mm in length, and high-speed steel taps of size M6 x1.00 mm. The tests were conducted at a machining speed of 90 rpm, with a hole depth of 33.02 mm and hole diameter of 5 mm. To lubricate the tools during the tapping operation, 0.5 mL of the lubricant sample was poured into the specimens. The tapping procedure was repeated five times to arrive at an average value for the thrust force and torque. The tests were performed using a milling machine that was equipped with the custom-made tapping torque tester.

## 3. RESULTS AND DISCUSSION

### 3.1. Tribological Results

The present study evaluated the tribological performance of sunflower oil with and without MMT nanoparticles at concentrations ranging from 0.5 to 3.0 wt.%. Data was collected from block-on-ring tribometer experiments under consistent test conditions. Figure 2 shows representative plots of the frictional force acting on the block specimen with and without MMT nanoparticle additives in sunflower oil at different concentrations. Results indicate that MMT nanoparticles effectively reduced the initial frictional force for all concentrations tested. The decrease in friction force over time can be attributed to nanoparticles initially forming a more effective boundary layer between the surfaces, resulting in reduced friction. As time progressed, factors such as shearing, wear, or other phenomena may have altered the nanoparticle-surface interactions, leading to changes in the frictional force. MMT nanoparticles can undergo degradation or chemical transformations when exposed to certain conditions, such as high temperatures or chemical reactions with the lubricant. These degradation processes may affect the nanoparticles' lubricating properties over time, causing changes in the frictional force. Furthermore, contacting surfaces also become smoother over time which reduces friction significantly.

It can be observed that the lowest frictional force was obtained with a 2.0 wt.% MMT concentration. Additionally, nanolubricants showed no increase in frictional force above the base sunflower oil friction force, with the lowest frictional force again produced by the 2.0 wt.% MMT concentration. The addition of MMT nanoclays to vegetable oils can decrease the contact area and friction between the surfaces of the tribo-pair due to the exfoliation of the weakly-bonded layers of MMT nanoclays. This reduction in friction and wear is consistent with the findings of Peña-Parás et al., who investigated the optimization of machining parameters and MMT clay NPs in a cutting fluid and obtained comparable results [16]. In a prior investigation, the authors examined the lubrication performance of corn and peanut oils with added Halloysite clay nanotubes (HNTs) and Montmorillonite nanoclay (MMT) as eco-friendly lubricant additives [18]. The study revealed that the greatest reduction in friction force occurred when MMT nanoparticles were incorporated into the corn and peanut oils at concentrations of 1.5 and 2.0 wt.% concentrations, respectively.

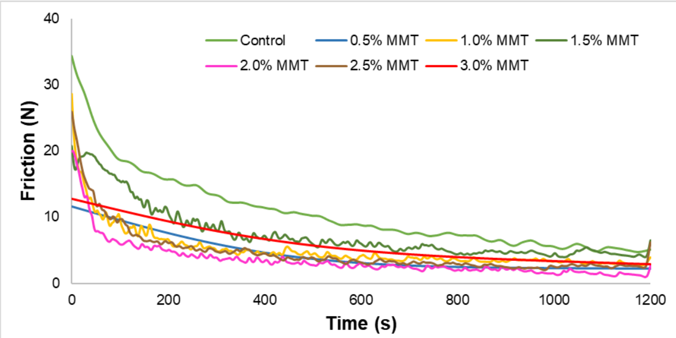


**FIGURE 2:** Friction force vs time graph for sunflower oil-based nano-lubricants.

The coefficient of friction or COF is a measure of the resistance encountered when two surfaces come into contact and slide against each other. It quantifies the frictional forces between the surfaces and is expressed as the ratio of the force required to initiate or sustain the sliding motion to the normal force pressing the surfaces together. Here, COF was calculated by dividing the measured frictional forces, shown in Figure 2, by the applied normal force of the test (266 N). A higher COF indicates greater resistance to sliding and more significant frictional forces, while a lower COF signifies smoother sliding and reduced friction. The average values of coefficient of friction reported from the block specimens lubricated with sunflower oil nanolubricants at various concentrations is shown in Figure 3. The error bars in the plot indicate the standard deviation of the tests. It can be observed that the base sunflower oil had a COF of 0.0398, and the addition of MMT nanoparticles significantly reduced the coefficient of friction. At first, the COF decreased suddenly at 0.50 wt.% MMT concentration, then increased gradually up to 1.5 wt.% and the COF at 1.5 wt.% was 0.0164, which was the highest COF value. This can be attributed to the increased contact area between the MMT particles and the surfaces in contact, leading to higher frictional forces. After that, suddenly, the COF decreased again at 2.0 wt.% to 0.0049 and further increased after 2.0 wt.%. This can be explained by the formation of a lubricating layer or a boundary film between the surfaces due to the presence of MMT particles. This film acts as a barrier, reducing the direct contact between the surfaces and resulting in a lower coefficient of friction. Beyond the 2% concentration, the COF increases again. This could be due to the excessive presence of MMT particles, which can lead to agglomeration or clustering. These clusters may cause roughness or irregularities on the surfaces, leading to an increase in frictional forces and hence an increase in the coefficient of friction. Therefore, for sunflower oil, the optimal concentration was found to be at 2.0 wt.%, and it reduced the COF of the base sunflower by 88%. Earlier research indicates that the addition of NPs to coconut and sunflower oils significantly reduced the COF. For example, coconut oil containing $SiO_2$ and CuO NPs decreased COF by 94% and 93%, respectively, compared to coconut oil without NPs [12]. Sunflower oil containing $TiO_2$ and $SiO_2$ NPs exhibited a reduction in COF of 94% and 78%, respectively, in comparison to sunflower oil without NP additives [8]. Similarly, sunflower oil fortified with HNTs showed a 29% reduction in friction coefficient [15]. In a similar study, Ortega et al. investigated the lubrication efficacy of corn and peanut oils reinforced with Halloysite clay nanotubes (HNTs) and Montmorillonite nanoclay (MMT) as environmentally friendly lubricant additives [18]. They discovered a substantial decrease in the coefficient of friction (COF) of up to 77% and 81% for the corn and peanut oil specimens, respectively, when reinforced with MMT nanoparticles compared to the oils without any additives.

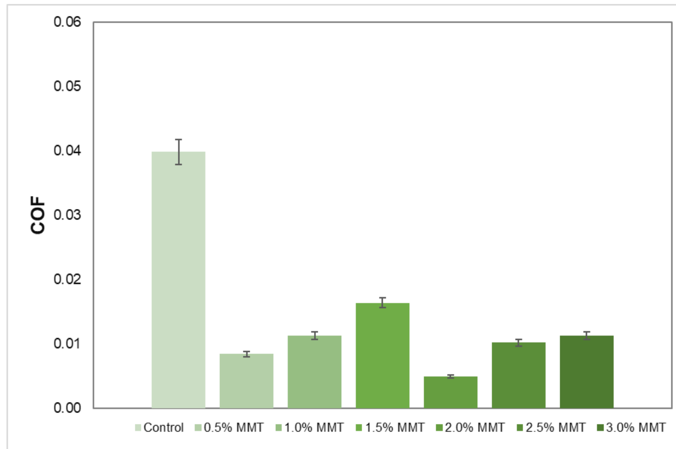


**FIGURE 3:** Coefficient of friction (COF) results for sunflower oil modified with MMT nanoparticles at different concentrations.

Figure 4 illustrates the average mean volumetric wear of AISI 304 blocks lubricated with sunflower oil and sunflower oil modified with MMT nanoparticles. The error bars in the plot indicate the standard deviation of the tests. The results of the tribological tests showed that the addition of MMT nanoparticles to sunflower oil reduced the volumetric wear loss compared to the base sunflower oil. The lowest volumetric wear was observed at 2.0 wt.% concentration, reducing wear by 63%. This can be attributed to the MMT nature. MMT are flake-like structures that are weakly bonded with van der Waals bonds, and they tend to exfoliate and may deposit onto moving surfaces, protecting them against friction and wear. In an earlier investigation, the authors examined the lubrication effectiveness of corn and peanut oils with the addition of Halloysite clay nanotubes (HNTs) and Montmorillonite nanoclay (MMT) as eco-friendly lubricant additives [18]. They observed a decrease in volumetric wear of up to 75% and 62% for the corn and peanut oil samples, respectively, when reinforced with MMT nanoparticles, in comparison to the oils without any lubricant additives.

At lower concentrations than 2.0 wt.% nanoparticles were not able to form a sufficient lubricating layer or boundary film, resulting in less effective friction reduction. Furthermore, the volumetric wear increased again as the concentration increased above 2.0 wt.%, likely due to the agglomeration of the nanoparticles. These clusters may cause surface roughness and irregularities, resulting in increased frictional forces and elevated coefficients of friction. Excessive nanoparticles can also lead to overcrowding, hindering the smooth sliding motion between surfaces.

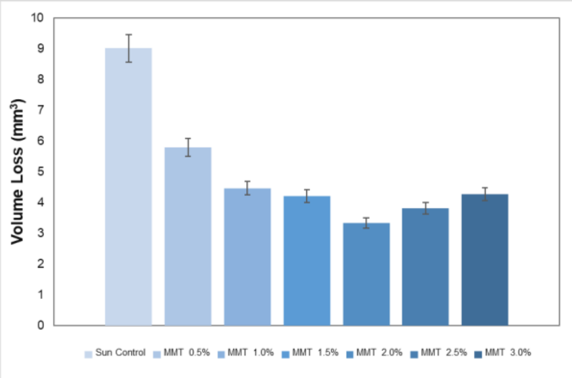


**FIGURE 4:** Mean volumetric wear of AISI 304 specimens lubricated with sunflower oil modified with MMT nanoparticles.

### 3.2. Tapping Torque Results

The tapping torque results conducted the ASTM D5619 standard procedure is shown in Figure 5. Experiments were performed on sunflower oil and the sunflower nanolubricant with the best tribological performance according to the block-on-ring tests, being the 2.0 wt.% MMT.

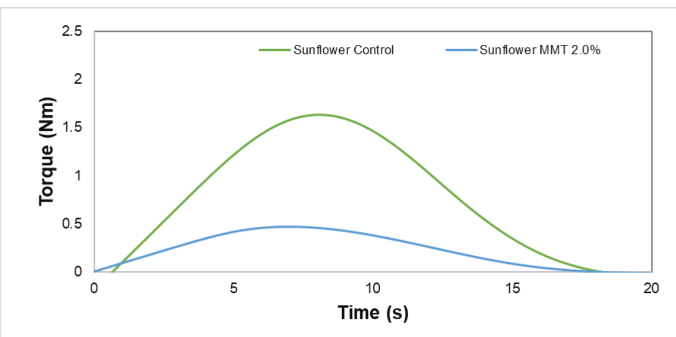


**FIGURE 5:** Tapping torque results for base sunflower oil and the best concentration sunflower nanolubricant.

The tapping torque test results showed that the maximum tapping torque for base sunflower oil was 1.64 Nm. For the 2.0 wt.% sunflower nanolubricant a maximum tapping torque of 0.471 Nm was obtained, with a 65% reduction from the base sunflower oil. The thin lubricating film formed by the MMT nanoparticles in the sunflower oil allowed for a transition from sliding to rolling friction, leading to a reduction in force and torque required for tapping. The MMT nanoparticles were able to exfoliate and deposit onto surfaces, which created a thin lubricating film in the oils. This led to a decrease in the force and torque needed for tapping. These results are similar to those obtained by Biswas et al. [15] when evaluating sunflower oil reinforced with HNT NPs and Talib et al. [25] when evaluating jatropha modified with hBN NPs. In a prior investigation, Ortega et al. conducted a study on the lubrication effectiveness of corn and peanut oils fortified with Halloysite clay nanotubes (HNTs) and Montmorillonite nanoclay (MMT) as environmentally friendly lubricant additives [18]. They observed a decrease in tapping performance of up to 59% and 55% for the corn and peanut oil samples, respectively, when reinforced with MMT nanoparticles, in comparison to the oils without any lubricant additives.

### 3.3. Wear Scar Analysis

The SEM images of the worn surfaces of the blocks after the wear tribological test are shown in Figure 6. From Figure 6a, it can be observed that in case of base sunflower oil, there were large number of burrs and furrows which resulted in increase in friction and wear. In the absence of a tribofilm, the wear track for the base lubricant exhibited numerous grooves and furrows caused by adhesive wear on the surface of the lubricants. This burrs and furrows increased the friction between sliding surfaces which resulted in a higher mass loss. The SEM image of wear scar of the block lubricated with 2.0 wt.% MMT modified sunflower oil is shown in Figure 6b. After incorporating MMT nanoparticles, furrow-depth and metal burr have both decreased significantly. The wear scar has limited number of burrs and furrows which helped to reduce the frictional force as well as low volumetric wear. Moreover, it can also be observed that a smaller number of grooves were present compared to the base sunflower oil and the exfoliation occurred on the surface of the block of the best modified nano lubricant which reduced the overall friction during the lubrication.

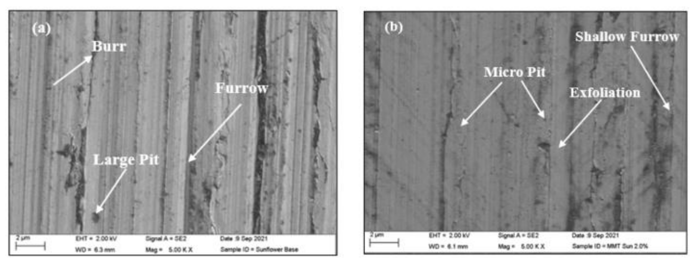


**FIGURE 6:** SEM micrographs of worn surfaces on blocks lubricated with: (a) sunflower oil, (b) sunflower oil modified with MMT nanoparticles at 2.0 wt.%.

### 3.4. Surface Roughness Analysis

The average surface roughness of the AISI 304 Blocks (before and after testing) lubricated with base sunflower oil and sunflower oil modified with MMT nanoparticles is shown in Figure 7. The Ra values of the specimen before testing, lubricated with base sunflower oil, sunflower oil with 2.0 wt.% MMT were 0.167 µm, 0.339 µm and 0.092 µm respectively. When compared to the controlled untested sample, it can be observed that base sunflower oil significantly increased the roughness of the block by 102.99% due to the higher concentrations of palmitic and oleic acids. For 2.0 wt.% MMT-modified sunflower oil, the roughness was decreased by 45% compared with the initial roughness of the block.

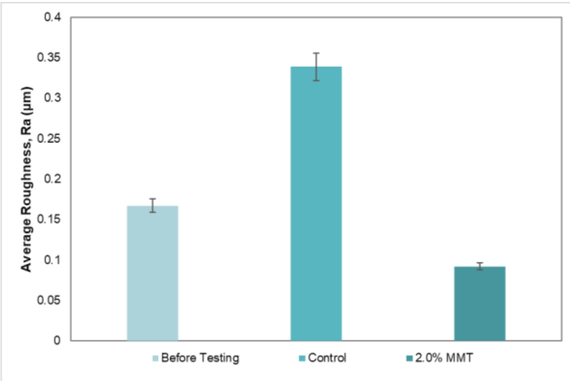


**FIGURE 7:** Average surface roughness of AISI 304 blocks before testing, lubricated with sunflower oil, and sunflower MMT 2.0%.

## 4. CONCLUSIONS

The present study investigated the effects of MMT nanoparticles on the lubrication performance of sunflower oil. The conclusions drawn from the results are summarized as follows:

- The sunflower nano-lubricant's tribological characteristics greatly depend on the concentration of MMT nanoparticles. Sunflower oil modified with 2.0 wt.% MMT exhibited the best tribological behavior, reducing COF and wear volume loss by 88% and 63%, respectively.
- Adding the MMT nanoparticles to the sunflower oil decreased the maximum torque of the system by 65.3%. This can be attributed to the exfoliation of the MMT nanoparticles and depositing them onto surfaces, creating a thin lubricating film.
- MMT nanoparticles reduced the surface roughness compared to the initial roughness of the specimens. The roughness of the specimen was reduced by 45% with sunflower oil modified with 2.0 wt.% MMT.
- SEM and profilometry investigations demonstrated the surface enhancement of worn surfaces caused by the protective film effect of the MMT nanoparticle additions.

It can be concluded from the above results that sunflower oil modified with MMT nanoparticles show great potential to develop new lower-cost and environmentally friendly nanomaterial-based nanolubricants as a replacement for petroleum, mineral, and synthetic oil. Future work will focus on studying and improving the dispersion and stability of MMT nanoparticles to employ them in industrial applications.


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