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## Tribological Study of TiO<sub>2</sub> Biodegradable Nanolubricant for Cooling in Minimum Quantity Lubrication (MQL)

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**Abstract:** *Minimum Quantity Lubrication (MQL) stands as an inventive machining method designed to reduce coolant usage by delivering a little amount of lubricant (in mist form) directly to the cutting area, aiming to diminish friction and heat during metalworking. Nanolubricant, which integrate nano additives, consists of minuscule particles blended into base lubricants, with the intention of substantially managing friction and wear on components. The integration of nanolubrication into MQL systems has emerged as an optimal approach to machining, providing superior lubrication precisely where it is most required. This advancement not only boosts operational efficiency but also aligns with environmentally conscious practices, thus representing a promising stride in the manufacturing area. This study probes into the thermophysical and tribological characteristics of TiO<sub>2</sub> nanoparticles combined with biodegradable base oil for MQL, evaluating the stability of biodegradable nanolubricant under Triton X-114 surfactant of 0.5% volume. Various volume concentrations, ranging from 0.001%, 0.002%, and 0.003% of TiO<sub>2</sub> biodegradable nanolubricant, underwent examination, assessing stability through visual sedimentation observation. Despite instances of sedimentation, visual observations suggest nanolubricant stability even after 30 days, with the utmost stability noted after 2 hours of sonication. Furthermore, thermophysical properties unveiled the highest dynamic viscosity at a concentration of 0.003% TiO<sub>2</sub> biodegradable nanolubricant, while tribological properties indicated a decrease in coefficient of friction (COF) and wear scar diameter (WSD) at a concentration of 0.003% volume of TiO<sub>2</sub> biodegradable nanolubricant. In summary, TiO<sub>2</sub> in biodegradable base oil emerges as a sustainable selection for advanced cooling agents in MQL.*

**Keywords:** Nanolubricant; TiO<sub>2</sub>; Biodegradable oil; Viscosity; Tribology

### 1. INTRODUCTION

In the course of mechanical motion, the movement of parts and interaction between surfaces generate heat. Prolonged abrasion of the surface had the potential to inflict lasting damage on the component [1]. To proactively address this issue, lubricants were incorporated into the mechanical system to mitigate both friction and heat. The application of lubricants in a mechanical system served the dual purpose of conserving energy and extending the lifespan of the component [2]. Consequently, the utilization of lubricants proved crucial for various functions, encompassing cooling, sealing, and lubrication. To handle demanding conditions, lubricants often contain extreme pressure and anti-wear additives. These minimized friction and wear under harsh circumstances, ultimately enhancing a machine's tribological performance [3].

The current challenges with traditional lubricants in machining performance arose from environmental problems, health dangers, and long-term viability challenges related to flood cooling systems. These methods involved the use of a substantial amount of coolant or lubricant, resulting in an environmental impact. The coolant might also contain toxic additives that, if not controlled effectively, might affect the environment. Therefore, a new cooling approach was devised in which only a small amount of coolant was sprayed at the precise cutting zone. Minimum quantity lubrication (MQL) marks a step forward from conventional machining lubrication practices, such as flood coolant usage [4]. MQL employs a biodegradable fluid in minimal amounts, which provides superior lubrication compared to

excessive flood coolants. Flood coolants present various drawbacks including shop floor messiness, spillage, wet chips, detrimental effects on tools, complex lubrication setups, skin irritations, and disposal challenges. L. N. Lopez et al. [5] delved into the impact of spray-cutting fluids during high-speed milling. They observed that Minimum Quantity Lubrication (MQL) flow serves three crucial functions in the cutting zone: cooling the tool and workpiece, providing lubrication, and efficiently removing chips. MQL significantly influences cutting temperatures across various speeds, resulting in lower tool wear rates compared to completely dry machine [6]. However, even with MQL systems, the use of cutting fluids possessing low thermal conductivity falls short of meeting the requirements for environmentally friendly machining. An alternative, more promising approach involves employing MQL to spray high thermal conductivity by cutting fluid. Studies by E. A. Rahim et al. [7] demonstrated that MQL machining outperformed dry machining in terms of cutting force and cutting temperature. The authors attributed this superiority to the lubricating and cooling effects of the mist, which effectively reduced friction and heat generation at the cutting zone. This MQL machining also yielded better surface finish and reduced tool wear compared to dry machining.

In the wake of the 1970s oil crisis, there arose a pressing demand for more efficient, low-powered machinery. This urgency prompted a surge of interest in high-efficiency appliances. Manufacturers, researchers, and industry leaders tirelessly sought ways to align with societal aspirations for environmentally friendly equipment. However, the pursuit of high efficiency often came at a

cost both in terms of expense and time. Enter nanoparticle dispersion technology: a clever solution that promised to enhance system performance without necessitating the replacement of existing components [8]. These nanoparticles, typically ranging in size from 1 to 100 nanometers, could be composed of metallic, ceramic, or organic materials. Studies have consistently demonstrated that incorporating nanoparticles into lubricants yields remarkable improvements in tribological properties. These enhancements encompass reduced friction, minimized wear, and increased load-carrying capacity. Nanoparticles have attracted significant interest due to their ability to perform multiple functions, interact with various targets, and deliver therapeutic or diagnostic agents and have demonstrated practical applications in a wide range of industries [9, 10]. Consequently, researchers became increasingly curious about how these nano lubricants fared under various lubrication conditions, including Minimum Quantity Lubrication (MQL). Thus, the management of adhesion, stiction, friction, and wear at micro- and nanoscales was coined as ‘nanolubrication’ [11]. The study by S. S. Sanukrishna et al. [12] explored the effects of TiO<sub>2</sub> nanoparticles on the thermal conductivity and viscosity of a polyethylene glycol (PAG)-based lubricant. Their findings revealed that as the volume concentration of nanoparticles and temperature increased, so did the observed values. The authors attributed this phenomenon to the enhanced heat transfer occurring between the nanoparticles and the base fluid. Specifically, this interaction led to an increase in the effective thermal conductivity of the nanolubricant. The maximum enhancement (12.5%) was achieved with a 0.8% volume concentration of nanoparticles at 90°C.

In recent studies, researchers have focused on enhancing Minimum Quantity Lubrication (MQL) performance by incorporating nanofluids. The primary goal is to improve both thermal conductivity and tribological properties within the lubricant mixture [13]. Beyond enhancing tribological conditions, nanofluids facilitate the formation of a thin lubricant film at machining interfaces, effectively reducing friction. C. K. Yildirim et al. [14] conducted experimental investigations while machining Inconel 625 using nano-MQL, where hBN nanoparticles were integrated. This approach was compared to pure MQL and dry machining. The results highlighted the capacity of hBN-nanofluid to enhance surface quality and prolong tool longevity by retaining lubricant within the machining interface for an extended duration, ultimately leading to improved lubrication. Another study by M. Danish et al. [15] explored the influence of graphene-reinforced sunflower oil applied under MQL during Inconel 718 machining. Their findings emphasized the efficacy of nanofluid-MQL compared to traditional dry and flood cooling methods. The key advantage lies in the nanoparticles’ ability to generate a protective tribofilm between machining interfaces, thereby reducing friction and resulting in superior surface quality and overall machining performance. In a recent investigation, M. N. Babu et al. [16] examined Hastelloy C 276 machining under different settings: dry machining, MQL, and nanofluid-MQL. It was found that the nanofluid-MQL demonstrated a remarkable average reduction of 66% in surface roughness compared to dry machining. This reduction suggests that nanofluids

enhance machining performance due to their superior dispersion, high thermal conductivity, effective heat transfer, improved surface wettability, and favorable tribological properties.

Analysis by A. H. Hamisa et al. [17] investigated the behavior of nanolubricant in relation to the concentration of nanoparticles. They found that the coefficient of friction (COF) and wear scar diameter (WSD) initially decreased as the nanoparticle concentration increased, reaching an optimal point. Beyond this point, however, both COF and WSD began to rise. The authors attributed this trend to the formation of a protective nanoparticle layer on the contact surfaces, which reduced surface roughness and minimized metal-to-metal contact. Yet, excessive nanoparticles beyond the optimal concentration led to agglomeration and increased nanolubricant viscosity, subsequently elevating the COF. For achieving the lowest WSD, the optimal volume concentration was determined to be 0.01% for TiO<sub>2</sub> dispersed in polyalphaolefin (POE). The authors explained this behavior by emphasizing the role of nanoparticles in filling surface asperities, thereby reducing contact area and shear stress. However, exceeding the optimal concentration resulted in abrasion and increased WSD. V. Cortes et al. [18] where also demonstrated similar behavior in nanolubricant. As nanoparticle concentration increased, COF and WSD decreased to a certain point, after which they began to rise. Their investigations revealed that the optimal volume concentration for achieving the lowest COF was 0.01% when using TiO<sub>2</sub> dispersed in sunflower oil.

This research evaluates the viability of utilizing TiO<sub>2</sub> dispersed in a biodegradable nanolubricant as an advanced lubricating agent for Minimum Quantity Lubrication (MQL). The study encompasses the formulation and stability assessment of mono TiO<sub>2</sub> dispersion in biodegradable oil. Subsequently, the thermophysical properties of the stable TiO<sub>2</sub> nanolubricant were examined, with a particular focus on dynamic viscosity. Furthermore, the tribological characteristics of mono TiO<sub>2</sub> biodegradable nanolubricant were evaluated across three distinct volume concentrations (0.001%, 0.002%, and 0.003%). This analysis involved assessing the coefficient of friction (COF) and wear scar diameter (WSD) using a Koehler four-ball tester in tribology testing.

## 2. METHODS AND MATERIAL

### 2.1 Lubrication preparation

In this experimental study, TiO<sub>2</sub> nanoparticles were employed, figuring an average size of 50 nm and a high purity level of 99.9%. The TiO<sub>2</sub> nanoparticles were sourced from HWNANO, a supplier of nanomaterials. The diameter of TiO<sub>2</sub> nanoparticles was typically 50 nm. Table 1 displayed the parameters of TiO<sub>2</sub> nanoparticles. The bio-degradable base oil employed in this investigation was Coolube 2210 XP, which had the specifications shown in Table2. Coolube 2210 XP was a lubricant that could be ascribed to a natural lubricant designed for machining ferrous metals, such as steel and iron. It was derived from vegetable oils and contained no volatile organic compounds (VOCs) [18].

Table 1. Properties of TiO<sub>2</sub> [19]

| Properties | TiO <sub>2</sub> |
|------------|------------------|
|------------|------------------|

|                                 |       |
|---------------------------------|-------|
| Density (kg/m <sup>3</sup> )    | 4230  |
| Molecular Mass (g/mol)          | 79.87 |
| Average Particle Diameter (nm)  | 50    |
| Specific Heat (J/kg.K)          | 692   |
| Thermal Conductivity, k (W/m.K) | 8.4   |

Table 2. Properties of biodegradable oil [19]

| Properties                                 | Biodegradable oil |
|--------------------------------------------|-------------------|
| Viscosity @ 40 °C mm <sup>2</sup> /s (cSt) | 10                |
| Pour Point (°C)                            | -12 to -20        |
| Density @ 20 °C (kg/m <sup>3</sup> )       | 890               |
| Flash Point (°C)                           | >200              |
| Thermal Conductivity, k (W/m.K)            | 0.2               |

The base oil and mono nanoparticle were the two major elements required for the formation of mono nanolubricant. The preparation of the nanolubricant involved a two-step process, where nanoparticles (titanium dioxide) were dispersed into this bio-degradable base oil. To achieve a homogenous dispersion of nanoparticles, the liquid was assorted for around 45 minutes using a magnetic stirrer, where high-frequency vibrations struck nanoparticles [20]. It was measured by adding the bio-degradable base oil into four different masses of titanium dioxide nanoparticles to obtain three different volume concentrations of nanolubricant. The surfactant of Triton X-114 with a volume percent of 0.5% was added to each different volume concentration of nanolubricant to reduce the surface tension of a liquid and help disperse solid particles in it. This surfactant was added to nanolubricant to improve their stability and avoid the nanoparticles from settling or agglomeration [21]. Finally, the composite samples were homogenized using ultrasonic sonication. To obtain the necessary nanolubricant, the resulting solution was sonicated for 2 hours each using an ultrasonic sonicator. This step was performed to improve the distribution of nanoparticles. This led to a uniform and consistent dispersion of nanoparticles. The sonication time had a noteworthy outcome on the stability of the nanolubricant prepared [22]. The volume concentration of the nanolubricant was calculated using Eq. (1) [23].

$$\phi_{NL} = \frac{\frac{m_p}{\rho_p}}{\left(\frac{m_p}{\rho_p} + V_{oil}\right)} \times 100 \quad (1)$$

Where  $\phi_{NL}$  is the volume concentration of nanolubricant,  $m_p$  is the mass of nanoparticle in kg,  $V_{oil}$  is the volume of biodegradable base oil in m<sup>3</sup>, and  $\rho_p$  is the density of nanoparticles in kg/m<sup>3</sup>.

## 2.2 Stability of TiO<sub>2</sub> biodegradable nanolubricant

Nanoparticles in lubricants could collide and clump together, causing them to settle down and block the tiny channels. To measure how well the nanoparticles were dispersed and how stable they were, different methods have been used in the past [22, 24]. In this work, the sedimentation method was a simple way to see how the nanoparticles or the process changed over time by observing them for 30 days. It was a useful way to visually check the dispersion stability of nanolubricant. As suggested by A. Ghadimi et al. [25], the commonly

used approach for evaluating the stability of nanolubricant involves comparing and observing sedimentation over time using photographic documentation. This technique serves to confirm that the nanolubricant maintain stability with minimal sediment settling.

## 2.3 Thermophysical properties measurement

In this study, the investigated dynamic viscosity was a crucial property in nanolubricant samples. The employment of the Digital Rheometer Electronic MCR301, generously provided by Anton-Paar was on assessing the viscosity of the nanolubricant. The samples underwent testing across a wide range of rotational speeds, spanning from 0.01 to 1000 revolutions per minute (1/min). Additionally, each sample was evaluated at specific temperatures, varying between 28°C and 90°C. To ensure robust results, the dynamic viscosity measurement was repeated for each sample three times. This approach is aimed at maintaining measurement consistency, enhancing repeatability, and ensuring reliable data.

## 2.4 Experimental apparatus

A Koehler four-ball tribo tester was used to test the nanolubricant tribological performance. This device measured the friction and wear of TiO<sub>2</sub> biodegradable nanolubricant. The test used steel balls that were 12.7 mm in diameter and had a hardness of 65 ± 1 HRC (chromium steel, grade G25) [17]. The test started with pure lubricant and then added TiO<sub>2</sub> bio-degradable nanolubricant with different concentrations from 0.001 % to 0.005%. The test followed the procedures in ASTM D4172-94. For this experiment, the working temperature was at 75°C, and a maximum test duration of 60 minutes was specified. 40.0 kg of weight was placed on the lever arm, and 1200 rpm was set as the rotational speed. The average wear scar diameter was determined by repeating each test three times. In the current experiment, the balls and equipment were cleaned before and after test using a solvent (hexane). Following each test, an optical microscope (light compound microscopy) was used to assess the wear scar diameter (WSD) of the worn materials. Table 3 shows the wear conditions and the ASTM D4172-94 standard.

Table 3. The test conditions for ASTM D4172-94 standard

| Test conditions    |                                                      |
|--------------------|------------------------------------------------------|
| ASTM standard      | D4172-94                                             |
| Test method        | Wear preventive characteristics of lubricating fluid |
| Speed (rpm)        | 1200±60                                              |
| Load (kg)          | 40.0±0.2                                             |
| Duration (Minutes) | 60±1                                                 |
| Temperature (°C)   | 75±2                                                 |
| Remarks            | Wrench on ball pot nut in between 22 to 50 ft. lb    |

## 3. RESULTS AND DISCUSSION

### 3.1 Stability evaluation

#### 3.1.1 Sedimentation observation

The experimental setup, depicted in Figure 1 maintained the nanolubricant samples in a static state for a continuous period of 30 days at room temperature. This extended duration allowed to closely monitor any changes over time. The images of TiO<sub>2</sub> biodegradable nanolubricant for a volume concentration of 0.003% with

different sonication times (0, 0.5, 1, 1.5, and 2 hours) are shown in Figure 1. These images were captured after preparation and continued daily for at least 30 days (or until any significant changes occurred). Encouragingly, Figure 1(a), revealed that no sedimentation of particles occurred immediately after nanolubricant preparation. This stability is crucial for ensuring consistent performance over extended periods. The nanolubricant samples appeared normal, and no clumping of particles was observed; however, the condition of the nanolubrication was murky. The particle sedimentation started on the first day for the 0-hour sample in minimal conditions. After 30 days, the nanolubricant samples had only a small amount of sedimentation, which could be due to gravity. Through visual observation, it was shown that the nanolubricant samples with a 2-hour sonication duration were the most stable.

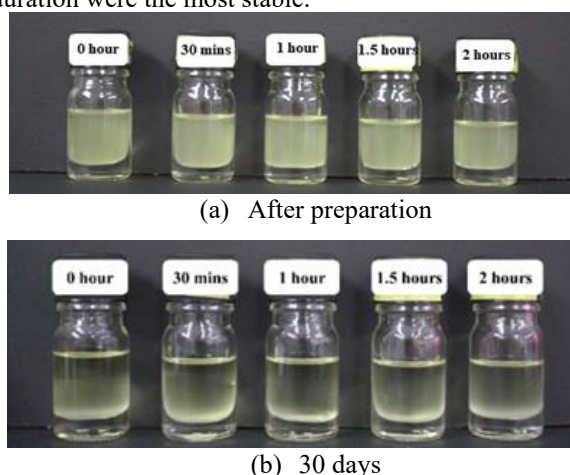


Fig. 1. Sedimentation observation of  $\text{TiO}_2$  biodegradable nanolubricant with different sonication time.

### 3.2 Thermophysical properties

#### 3.2.1 Dynamic viscosity

The study was delved into the viscosity characteristics of a  $\text{TiO}_2$  based biodegradable nanolubricant across a temperature range from  $28^\circ\text{C}$  to  $90^\circ\text{C}$ . Figure 2 illustrated the dynamic viscosity values for  $\text{TiO}_2$  biodegradable nanolubricant with three distinct volume concentrations. This figure depicted the impact of incorporating  $\text{TiO}_2$  nanoparticles into a biodegradable base oil as increased the concentration of  $\text{TiO}_2$  nanoparticles in the biodegradable base oil, the measured viscosity also increased. The maximum viscosity was recorded at a concentration of 0.003%  $\text{TiO}_2$  biodegradable nanolubricant. At the temperature of  $60^\circ\text{C}$ , the highest increment was shown by 0.003% volume concentration of  $\text{TiO}_2$  biodegradable nanolubricant with 14.27% from biodegradable base oil. It was then followed by 0.002% volume concentration of nanolubricant with an increment of 13.35%, and 0.001% volume concentration of nanolubricant (13.23%). Conversely, the lowest behavior of viscosity was observed in bio-degradable base oil. V. Cortes et al. [18] reported similar findings in their investigation of rheological behavior. They studied ( $\text{TiO}_2$ ) nanoparticles dispersed in engine lubricant oil. It is well-established that nanoparticle size and concentration play a pivotal role in shaping the rheological properties of colloidal suspensions, including oil-nanoparticle systems.

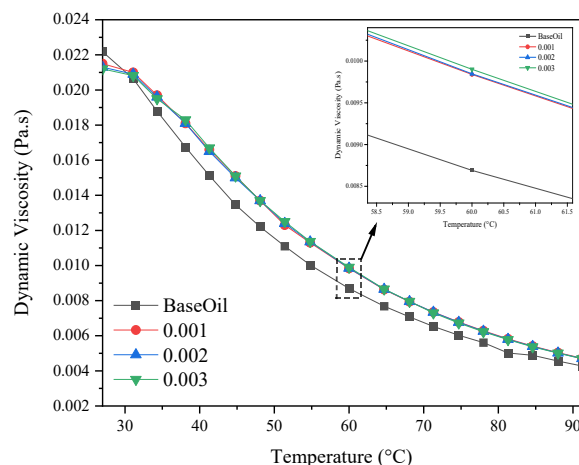


Fig. 2. Dynamic viscosity of nanolubricant.

### 3.3 Tribological characteristics

This section presented assessments of the tribological properties of nanolubricant, encompassing parameters such as coefficient of friction, wear scar diameter, and microscopic observations.

#### 3.3.1 Coefficient of friction evaluation

In Figure 3, the trends of the coefficient of friction over time during tribological tests are depicted. According to these trends, a biodegradable base oil showed a higher COF followed by 0.001% volume concentration of nanolubricant which gave the lowest average decrement with 0.104 standard deviation. The highest average decrement occurred at the 0.003% volume concentration, achieving up to a 16.62% reduction compared to the base oil. The samples remained stable on COF trends from 1000 seconds until 3600 seconds. In this scenario, within the volume concentration range studied, the maximum allowable concentration of nanoparticles was 0.003%, which reduces COF by improving lubrication on the contact surface.

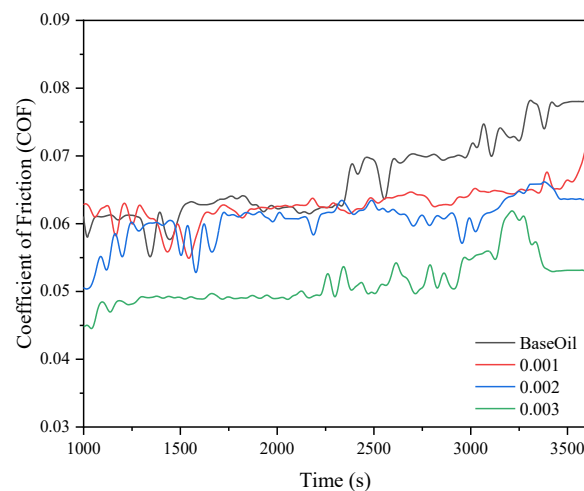


Fig. 3. Coefficient of friction of  $\text{TiO}_2$  biodegradable nanolubricant.

#### 3.3.2 Wear scar diameter evaluation

It was meticulously evaluated the wear properties of a  $\text{TiO}_2$  biodegradable nanolubricant using four-ball wear tests. Figure 4 visually presented the wear scar images. As the nanoparticle concentration increased, the WSD consistently decreased. This behavior can be attributed to the enhanced load-carrying capacity and improved anti-wear performance provided by the nanoparticles [19]. The WSD trend closely aligns with the pattern observed



for COF as the base oil indicated the least effective lubrication as compared to nanolubricant. A volume concentration of 0.003% exhibited the smallest wear scar diameter when compared to biodegradable base oil with a standard deviation of 0.0112 with the improvement of WSD reduction up to 12.42%. Examining worn surfaces of lubricated steel balls (Figure 4), it was found that a volume concentration of 0.001% resulted in a large and rough wear scar (Figure 4(b)) among nanolubricant and this concentration yielded the lowest improvement in WSD (9.09%).

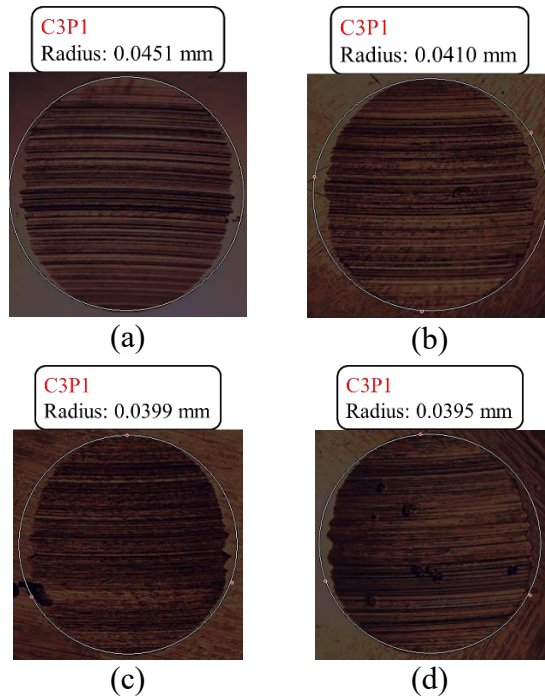


Fig. 4. Optical micrograph of wear scar for (a) Biodegradable base oil, (b) 0.001%  $\text{TiO}_2$  biodegradable nanolubricant, (c) 0.002%  $\text{TiO}_2$  biodegradable nanolubricant, and (d) 0.003%  $\text{TiO}_2$  biodegradable nanolubricant.

#### 4. CONCLUSION

In conclusion, this study effectively examined the stability, thermophysical, and tribological properties of  $\text{TiO}_2$  biodegradable nanolubricant.  $\text{TiO}_2$  was effectively dispersed into biodegradable base oil using Triton-X114 surfactant. Despite some notable sedimentation occurrences in the mixing process, visual observations indicated that nanolubricant with 2 hours of sonication time remained the most stable even after 30 days. Within a temperature range spanning from 27 °C to 90 °C, it was found that the dynamic viscosity reached its maximum at 0.003% volume concentration. Comparing tribological characteristics such as the coefficient of friction (COF) and wear scar diameter (WSD), between the nanolubricant and the biodegradable base oil, it was observed to be significant improvements where the nanolubricant exhibited a lower COF than the base oil, with the most substantial reduction occurring at a volume concentration of 0.003%, reaching an impressive 15%. Similarly, the WSD reduction was highest at the same concentration, decreasing by 12.42%. The groundbreaking finding was that incorporating  $\text{TiO}_2$  nanoparticles into the biodegradable base oil led to superior tribological performance, surpassing other

conventional lubricants. In addition, this study discovered the best volume concentration for enhancing machining performance was achieved at 0.003% volume concentration of  $\text{TiO}_2$  biodegradable nanolubricant. In a nutshell  $\text{TiO}_2$  biodegradable nanolubricant can be employed as an improvise coolant in Minimum Quantity Lubrication (MQL).

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