The Hybrid TiO2:SiO2 in Biodegradable oil: Effect of the Mixture Ratio

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https://doi.org/10.5109/7395615

出版情報：Proceedings of International Exchange and Innovation Conference on Engineering & Sciences (IEICES). 11, pp.874-880, 2025-10-30. International Exchange and Innovation Conference on Engineering & Sciences
バージョン：

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Abstract: *In machining industries, Minimum Quantity Lubricant (MQL) is employed in machining operations. It is preferred over conventional flood methods due to its advantages. Traditional flood machining requires large amounts of lubricant, increasing costs and harming health and environmental risks. However, MQL faces challenges in reducing heat generation, particularly hard-to-cut materials like low alloy steel AISI 4340, due to long cutting hours. Therefore, the implementation of nanolubricant minimum quantity lubrication (NMQL) is studied due to its potential to enhance thermal conductivity and viscosity characteristics. This paper reviews the stability and thermos-physical properties of the nanolubricants in the green base fluid using volume concentrations of 0.1% of TiO2:SiO2 at a mixture ratio of 30:70 and 50:50. Stability evaluations are visual inspection, UV-Vis spectrophotometry, and Zeta potential analysis. These findings underscore the potential of NMQL as an eco-friendly lubricant, prolonging the life of cutting tools and efficient energy consumption in the machining process.*

Keywords: *Green Nanolubricant; TiO_2:SiO_2; NMQL; Stability; Thermo-physical*

1. INTRODUCTION

Implementing environmentally friendly machines is a crucial step toward accomplishing sustainable development. It is a known fact for the industry to always utilize machinery to the maximum as they prioritize productivity above all. A critical component of this process is the use of Metal Working Fluid (MWF), which serves multiple functions, including removing heat, lubricating the machinery, preventing corrosion, and washing off metal chips from the workspace. While effective in its purpose, conventional mineral oil-based MWF poses significant environmental and health challenges. Improper disposal of these fluids leads to water and soil pollution, contributing to long-term environmental degradation [1]. Moreover, extended exposure to these fluids presents health risks to workers, especially machine operators, who are in constant contact with conventional lubricants during long working hours [2]. Instead of using mineral MWF, there has been a corresponding rise in the demand for more environmentally friendly cutting fluids. Vegetable oil is considered an eco-friendly alternative due to its biodegradability and sustainability, making it a preferred choice for cutting fluids from an environmental protection standpoint [3]. A study used vegetable cooking oil as a lubricant when machining AISI 4340 Alloy steel. The results showed better surface finish and lower cutting forces compared to dry cutting. [4]. Vegetable oil-based cutting fluids commonly include oils derived from plant-based, such as sesame, coconut, soybean, palm, and castor [5].

One primary goal in machining is to reduce cutting fluid consumption while maintaining or improving performance. The Minimum Quantity Lubrication (MQL) method offers a solution by ensuring that the MWF can also reach the critical tool-workpiece interface to effectively dissipate heat even though in the form of fine mist as there is a study on MQL technique shown to have better tribological qualities, safe, and environmentally friendly [6]. Moreover, MQL has been shown to be more effective in significantly reducing grinding forces, wear, and surface roughness of workpiece materials [7]. A study by M.K. Gupta et al. using MQL with vegetable oils containing MoS_2 and graphite-based nanofluids showed improved cooling and lubrication by reducing thrust force and torque [8]. Although MQL has gained attention for its ability to reduce lubricant consumption and environmental impact, it has its limitations, particularly in maintaining performance during high-speed machining operations [9]. Furthermore, mineral-based flood cutting is commonly employed in industrial settings due to its effectiveness in dissipating the excessive heat generated at the cutting zone when machining high-strength materials [10], such as low alloy steel AISI 4340 and Inconel. In contrast, the MQL method is unable to produce a high-quality product as the prolonged machining duration can lead to material properties deteriorating, which will influence the tool wear [11]. To address these challenges, the introduction of nanoparticles as additives in MQL referred to as Nanoparticle-Enhanced Minimum Quantity Lubrication (NMQL) has gained attention. NMQL reduces cutting fluid consumption, minimizes waste generation, lowers overall costs, including disposal costs, and significantly reduces harm to both the environment and workers. Traditional cutting fluids often contain chemical additives that inhibit bacterial growth, extend the fluid's lifespan, and improve performance. However, these additives frequently include harmful substances such as sulphur compounds, which are known to cause serious health issues, including respiratory problems, skin irritation, and even cancer [12]. Additionally, the disposal of these non-biodegradable fluids contributes to environmental pollution, contaminating water sources and damaging ecosystems [13]. The technique of NMQL with vegetable cutting fluid can increase the surface quality of a product while reducing power consumption and approaching sustainable manufacturing [14]. Studies by T. He et al., the cooling and lubricating properties of the base fluid can be enhanced by adding nanoparticles to the cutting fluid or by replacing the cutting fluid with nanofluid directly [15].

An advanced lubricant where dispersion of two types of nanoparticles in base fluids, known as a hybrid nanolubricant offers a better alternative to both mono nanolubricant and conventional lubricant as it enhances heat transfer efficiency [16]. Studies by M. Sandeep Kumar et al. [17] showed that the thermo-physical characteristics of mono are further improved with hybrid nanolubricant. Experimental investigations involving a few mixture ratios of Cu-Zn hybrid nanoparticles (50:50, 25:75, and 75:25) revealed notable improvements in thermophysical characteristics, as the 50:50 hybrid nanofluid exhibited the greatest enhancements compared to the mono composite with a reduction in surface roughness of 13.72%. In addition, another study on the Al_2O_3/CNT dispersed in Extra Virgin Olive (EVO) oil enhances the thermal conductivity, dynamic viscosity also heat capacity [18]. Not only does NMQL have better thermal conductivity where the heat of the tool-workpiece can transfer better [19], but the additive of nanoparticles of MQL is also able to enhance other thermophysical properties such as its dynamic viscosity.

Thermal conductivity is crucial in determining the performance of a system that involves heat transfer from one region to another. Thermal conductivity influences the heat transfer rate within the application. Meanwhile, viscosity influences the flow behaviour of the liquid. Both properties can significantly influence energy consumption [20]. The primary factors affecting thermal conductivity include particle shape, nanofluid preparation, interfacial layers, brownian motion, and particle clustering and aggregation. On the other hand, dynamic viscosity is primarily influenced by the physical behaviour of the particles, nanofluid preparation, and particle clustering and aggregation. The nanolubricants in machining operations have resulted in positive effects, enhanced lubrication efficiency, decreased friction and wear, and also effectively control heat dissipation in various machining operations [21]. The thermophysical properties of nanolubricants are key to improving the heat dissipation ability during machining. An increase in thermal conductivity ensures efficient removal of heat from the cutting zone, which is vital for prolonging tool life and maintaining workpiece quality. The viscosity of the nanolubricant, another important property, affects its flow behaviour and the formation of a lubricating film under operational conditions. Furthermore, a study by Das et al. on high-strength and durability metal on AISI 4340 steel under the turning machining process using nanolubricant Al_2O_3, Fe_2O_3, and CuO dispersed in rice bran oil has shown improvement in terms of surface tension, contact angle, thermal conductivity, and viscosity as compared to traditional cutting fluid [22]. This research aims to evaluate the performance of green nanolubricants formulated with 0.1% titanium dioxide and silicon dioxide (TiO_2:SiO_2) nanoparticles dispersed in Coolube 2210 XP, specifically at mixture ratios of 30:70 and 50:50. The investigation begins with the preparation of a stable hybrid nanolubricant using TiO_2:SiO_2 nanoparticles. Subsequently, the thermo-physical properties of the nanolubricants are analysed to assess their suitability for sustainable lubrication in machining applications.

2. MATERIALS AND METHODS

2.1 Nanolubricant Preparation Process

The experimentation used hybrid nanoparticles TiO_2 and SiO_2 with a ratio of (30:70 and 50:50) which have an average dimension of 50nm and 30nm were used in the study. The used titanium dioxide of 99.9% high purity level was sourced from HWANO meanwhile silicon dioxide of 99% was obtained from Xuzhou Jiechuang New Material Technology (Guangzhou Jiechuang Trading Co., Ltd). The physical properties of TiO_2 and SiO_2 can be seen in Table 1. As for the MQL biodegradable base oil, Coolube 2210 XP is used. The base fluid is manufactured by Unist Incorporation. Table 2. shows the properties of the base, which is free of volatile organic compounds (VOC), it is environmentally friendly.

Table 1. Properties of TiO_2 and SiO_2 [23]

Properties	TiO_2	SiO_2
Density(kg/m)	4230	2220
Molecular mass(g/mol)	79.87	60.08
Average particle diameter(mm)	50	30
Specific heat (J/kgK)	692	745
Thermal conductivity (W/mK)	8.4	1.4

Table 2. Properties of Coolube 2210 XP [24]

Properties	SiO_2
Viscosity @ 40 ^oC mm^2/s (cSt)	10
Pour Point (^oC)	-12 to -20
Density @ 20 ^oC (kg/m^3)	890
Flash Point (^oC)	>200
Thermal conductivity (W/mK)	0.2

The combination of two mono nanoparticles created hybrid nanoparticles. Hence, the experimentation used the method called the two-step as demonstrated in previous studies by [25], to prepare the hybrid nanolubricants. TiO_2 and SiO_2 nanoparticles have been utilised in this experiment. The weight of the nanoparticles for a given volume concentration of nanolubricants was based on Equation 1. A similar equation has been used in other studies to produce nanolubricants [26].

$$\phi = \frac{\frac{m_n}{\rho_n}}{\frac{m_n}{\rho_n} + \frac{m_{base}}{\rho_{base}}} \times 100\% \quad (1)$$

Volume concentration of nanolubricant, ϕ. m_n (kg) is mass of the nanoparticle, ρ_n (kg/ m^3) is density of nanoparticles, m_{base} is mass of base oil and ρ_{base} is density of base oil.

In this study, nanolubricants were prepared in volume concentrations of 0.1%. The process begins with the precise weighing of nanoparticle masses using a high-precision analytical balance. After the nanoparticles were weighed, a specific volume of base oil was added to the particles, along with 0.5% Triton X-114 surfactant for each sample. The surfactant was used to reduce the liquid's surface tension and enhance the dispersion of solid particles within the fluid medium [27]. Surfactant played a critical role in ensuring uniformity by preventing nanoparticle clumping and improving stability. Next, each sample underwent an initial mixing phase using a magnetic stirrer for 30 minutes. This step facilitated the even dispersion of nanoparticles and surfactant in the base oil. After stirring, the samples were subjected to sonicate for 2 hours. The ultrasonic waves generated during sonication ensured a more effective dispersion of nanoparticles throughout the fluid [28], preventing them from clustering. This process significantly enhanced the stability of the nanolubricant, allowing the nanoparticles to remain evenly distributed and suspended for longer periods [29].

2.2 Stability of TiO_2:SiO_2/ Coolube 2210 XP

The stability of nanolubricants was tested by using 3 methods[6] to prevent nanoparticles from settling at the bottom of the test tube and clumping, which would indicate instability. The first method employed was a qualitative technique known as visual sedimentation. This test evaluated the stability of the nanolubricant based on visible sedimentation patterns. After preparing the nanolubricant samples, it was left undisturbed in a fixed location to prevent external disturbances that might interfere with the results. Sample sonicate for 0 hours, 0.5 hours, 1 hour, 1.5 hours, and 2 hours. Daily observations and photographs of the samples were recorded over a month, monitoring changes in sedimentation. This method provided valuable qualitative data on the dispersion of nanoparticles over time and helped identify trends in stability based on visual indicators.

The second stability evaluation utilized Ultraviolet-Visible (UV-Vis) spectrophotometry to measure the absorbance of light in the nanolubricant samples. The machine used for this test was the Jasco/V 670-EX UV-Vis spectrophotometer. This method measured the absorption and transmission of ultraviolet light as it passed through a cuvette containing the nanolubricant [30]. By analysing how much light was absorbed and how much passed through the sample, the dispersion and uniformity of the nanoparticles in the base fluid were assessed. Observations were conducted for a month to identify changes in the stability and uniformity of the nanolubricant.

The final method for stability evaluation was zeta potential analysis. This test was carried out with the Malvern Zetasizer, measuring the electrostatic stability of the nanoparticle dispersion. A high zeta potential value, whether positive or negative, indicated strong repulsive forces between particles, reducing the likelihood of aggregation. This single-point test served to confirm the stability results obtained from UV-Vis measurements and visual sedimentation tests.

2.3 Thermophysical Characteristics

After confirming the stability of the nanolubricant, the next step involves measuring its thermophysical properties, specifically the thermal conductivity. This was achieved using the TCi Thermal Conductivity Analyzer by C-Therm, which provides data on thermal conductivity within a measurement range of 0 to 500 W/mK and a temperature range of 30 to 90°C. In this process, the samples are placed inside an oven-like space equipped with a thermal sensor. This sensor, directly connected to the analyser, records the thermal effusivity of the sample. The measurement follows the standards outlined in ASTM D7984, employing the Modified Transient Plane Source (MTPS) method, which ensures accurate and reliable results. The test allows the nanolubricant's potential effectiveness in heat transfer applications under varying temperature conditions.

The Digital Rheometer Electronic 301 was used to measure the rheological properties of the samples within a temperature range of 28˚C to 90˚C. This device operates on the Searle principle, which involves a static measuring cup and a rotating measuring bob. The sample is held within a double-gap stationary lower cup and a concentric cylinder measuring system. The stationary lower cup ensures the sample remains securely positioned, while the concentric cylinder is directly connected to the rheometer's motor. This allows the rotating measuring bob, which serves as the moving part, to come into direct contact with the sample during the test. This configuration ensures precise measurements of the sample's viscosity under a wide range of temperatures.

3. RESULTS AND DISCUSSION

3.1 Analysis on Stability

The stability of the nanolubricant was monitored from the day of preparation until the 30th day, or until visible sedimentation occurred, with evaluations conducted every ten days. The study used a 0.1% TiO_2:SiO_2 vol% concentration. In Fig.1., the nanolubricant was analysed with a single mixture ratio (30:70) subjected to varying sonication durations of 0 hours (immediately after stirring), 30 minutes, 1 hour, 1.5 hours, and 2 hours. It was observed that, on the first day, all samples appeared uniformly muddy yellowish free of clumps and visually indistinguishable. However, by the 15th to 30th day, slight sedimentation became visible at the bottoms of the test tubes. Sedimentation became evident at the bottom of the test tubes. A similar observation was reported by Kamal Kamarulzaman et al. [31], who investigated the stability of CNC-CuO nanoparticles in engine oil over 30 days. Their results showed that the nanolubricant remained uniformly dispersed without noticeable agglomeration at the bottom during the first week. However, a distinct separation layer gradually formed as time progressed. Among these samples, the one subjected to 2 hours of sonication was the murkiest, indicating better dispersion and reduced agglomeration of nanoparticles, while the 0 hours sample was the clearest, suggesting poor stability and higher particle aggregation.

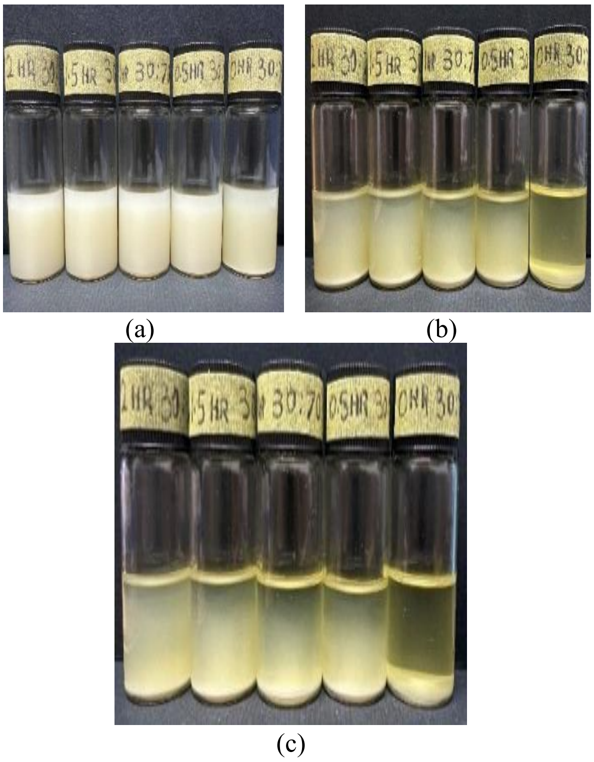

Fig. 1. Sedimentation observation of different sonication hours (a) Day 0, (b) Day 15 and (c) Day 30.

The colloidal stability of the samples was assessed using a consistent wavelength of approximately 386 nm. According to Fig. 2., a declining trend in absorbance was observed from the first day of sample preparation through the thirtieth day. This trend also correlated with the sonication duration, where samples subjected to longer sonication periods exhibited higher initial absorbance and a gradual decline over time. The absorbance volume concentration used for the UV-Vis stability test was 0.001%, differing from the intended 0.1%. This adjustment was necessary because the machine used for measuring UV-Vis absorbance could not read values exceeding seven. Additionally, at the intended concentration, the nanolubricant appeared excessively murky, making accurate measurements challenging. As a result, the sample was diluted to a new volume concentration suitable for the UV-Vis test. On the first day, the nanolubricant had the murkiest appearance, corresponding to the highest absorbance values, as less light passed through the sample. By the thirtieth day, some sedimentation had occurred, allowing light to pass through when exposed to UV light, which explains the reduction in absorbance. This indicates a decrease in colloidal stability over time. Additionally, the analysis suggests that two hours is the optimum sonication period. Samples subjected to this duration were well-mixed, as evidenced by higher absorbance values, meaning less light could pass through. The result indicates that a sonication duration of 2 hours yields optimal stability, consistent with the findings reported by Zawawi et al., [32] who identified the same duration as the most effective for achieving superior dispersion stability.

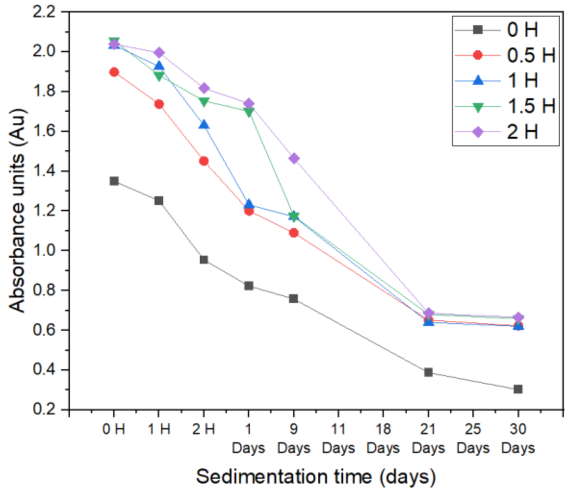

Fig. 2. Absorbance units against sedimentation time.

The zeta potential presented in Fig. 3. shows a value of 97.5 mV for the 0.001% (30:70) nanolubricant. Reading exceeding 60 mV signifies exceptional stability [33]. This indicates a highly stable dispersion, where the particles are strongly repelled from one another, significantly reducing the likelihood of aggregation and ensuring optimal performance of the lubricant.

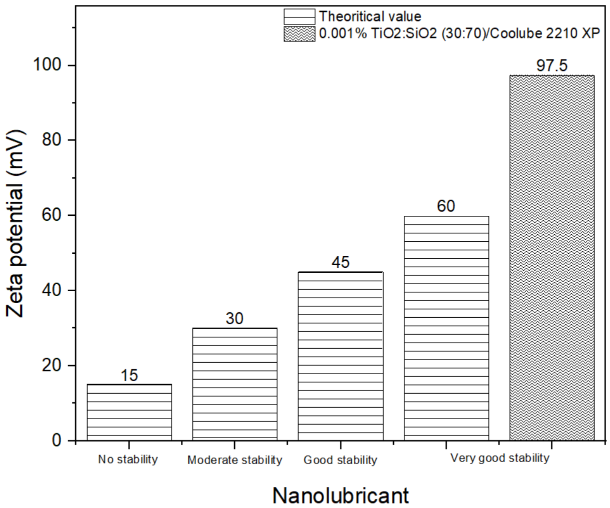

Fig. 3. Zeta potential measurement.

3.2 Measurement on Thermophysical Properties

Fig. 4. shows the measurement of thermal conductivity at 3 different temperatures. It was observed that, at every temperature, the nanolubricant dispersed in the base had better results than the base fluid. The data also shows that between the two mixtures ratios, 50:50 has a higher value than 30:70 for every temperature. The thermal conductivity at a temperature of 70 °C for a mixture ratio 50:50 is 0.146W/mK, and the improvement value is 0.006, which is a 4.3% enhancement from the base lubricant which is 0.14W/mK. It has been noted that the presence of nano additive in lubricant enhanced the Coolube 2210 XP 's heat conductivity. A previous study was done [34], where the thermal conductivity is enhanced with additional nanoparticles in comparison with the base as the transmission of heat improves. Moreover, the thermal conductivity enhancement as nanoparticles are added is due to the tribology motion effect, where the aimless motion of particles of the fluid hit among other active particles and base molecules which helps for better disperse of heat. The improvement

in thermal conductivity observed in the nanolubricants can be attributed to the intensified Brow motion of the dispersed brownnanoparticles. This random motion facilitates enhanced thermal energy distribution by promoting more efficient heat exchange between the nanoparticles and the surrounding molecules, thereby increasing overall thermal transport within the fluid [35].

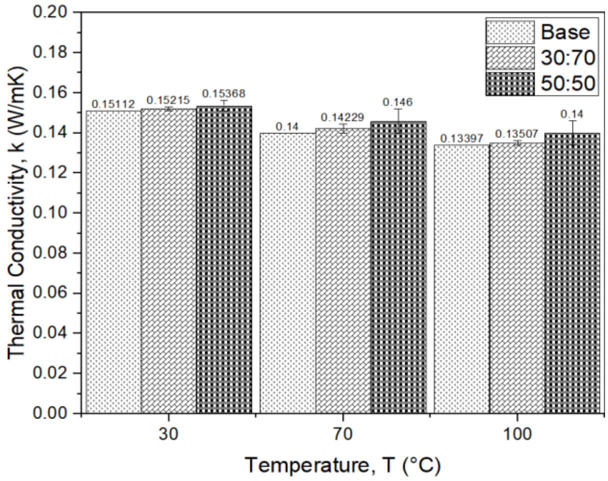

Fig. 4. Thermal conductivity against temperature.

Fig. 5. shows the relationship between dynamic viscosity and temperature. As the temperature increases from 30°C to 90°C, the dynamic viscosity steadily decreases. This reduction is attributed to the increased kinetic energy of molecules at elevated temperatures, which disrupts intermolecular interactions and consequently leads to a decrease in viscosity [36]. Among the tested samples, the mixture ratio 50:50 shows better results than 30:70 and the base oil exhibits the lowest dynamic viscosity throughout the temperature range. In Fig. 6., a specific temperature of 90°C is highlighted. At this temperature, the highest reading of dynamic viscosity is observed for the 50:50 mixture ratio, which also represents the highest concentration of TiO_2: SiO_2 additives. This ratio achieves a 13.3% improvement in dynamic viscosity compared to the base oil. In contrast, the base oil shows the lowest dynamic viscosity at this temperature, reinforcing the significant impact of the additives in enhancing the viscosity of the biodegradable oil. The 50:50 mixture ratio not only improves viscosity performance but also maintains stability at higher temperatures, making it the optimal concentration for enhancing the rheological properties of biodegradable lubricants.

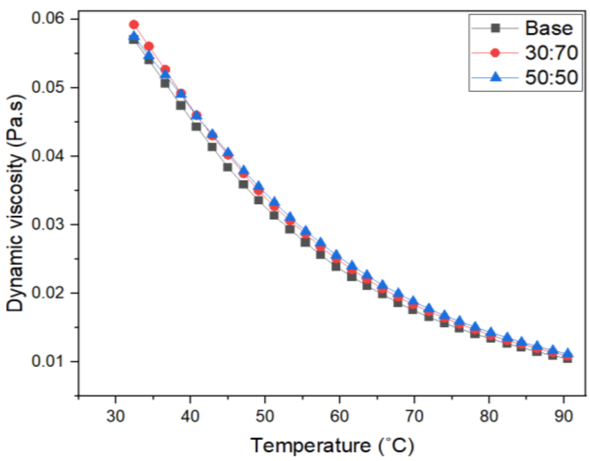

Fig. 5. Dynamic viscosity against temperature variation.

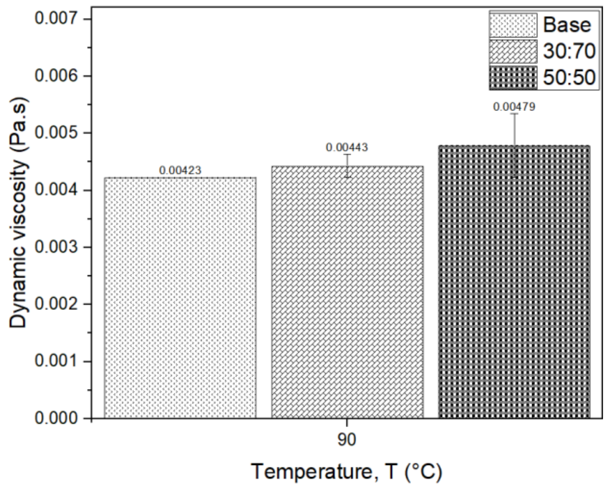

Fig. 6. Dynamic viscosity against a temperature of 90°C.

4. CONCLUSION

In the current study, the stability of the preparation and thermo-physical properties measurement was influenced by the mixture ratio of the hybrid nanolubricant and the base fluid. The stability of the samples prepared was confirmed based on visual inspection, UV vis spectral measurement, and zeta potential measurement. It was confirmed that the samples prepared were in an excellent stability region, and the preferred sonication duration is 2 hours to ensure maximum stability. Meanwhile, the thermo-physical nanolubricants characteristics show that the dynamic viscosity and thermal conductivity of the TiO_2:SiO_2 mixture ratio (30:70 and 50:50)/Coolube 2210 XP nanolubricants are improved than base. However, 50:50 shows higher enhancement compared to 30:70. The mixture ratio 50:50 shows that the thermal conductivity at a temperature of 100°C was improved by 4.5%, on the other hand, dynamic viscosity at 90°C was enhanced by 13.3% respectively as compared to the base lubricant. These improvements are attributed to the optimal nanoparticle mixture ratio, which facilitated the study of thermophysical properties in NMQL for the industrial machining process. However, further experimental work in actual application should be performed to validate such findings.

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