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Tribology Performance of Polyol-Ester Based TiO_2, SiO_2, and Their Hybrid Nanolubricants

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Abstract: The tribology properties of TiO_2/POE, SiO_2/POE and TiO_2-SiO_2/POE nanolubricants were investigated for an automotive air-conditioning system with an electrically-driven compressor (EDC). A two-step preparation method was used in dispersing TiO_2 and SiO_2 nanoparticles into Polyol-ester (POE)-based lubricant at different volume concentrations of 0.01 to 0.1%. The coefficient of friction (COF) and wear scar diameter (WSD) were investigated using a Koehler four-ball tribo tester and microscopes. For the TiO_2/POE, SiO_2/POE and TiO_2-SiO_2/POE nanolubricants, respectively, the lowest COFs with maximum reduction were attained at 37.5%, 33.5% and 31.6% each at volume concentrations of 0.05%, 0.01% and 0.03%. The highest WSD reduction for the TiO_2/POE and SiO_2/POE mono nanolubricants were attained at 12.5% and 26.4%, respectively, at the same volume concentration of 0.01%. Meanwhile, the maximum reduction of WSD for the TiO_2-SiO_2/POE hybrid nanolubricant was reached at 12.4% at 0.03% volume concentration. As a conclusion, mono and hybrid nanolubricants with volume concentrations of less than 0.05% are suggested for use in air-conditioning systems with EDC because of their outstanding tribology performances. Further performance investigation of nanolubricants in the air-conditioning system is required to extend the present work.

Keywords: tribology; nanolubricants; polyol-ester; four ball tribo tester; coefficient of friction; wear scar diameter

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1. Introduction

In most mechanical systems, lubricants are often used for cooling, sealing and lubrication purposes to improve the system's life cycle, as well as for energy saving. The lubricants usually bind to moving surfaces, forming fluid films that separate moving components' surfaces, while also removing heat and wear particles from the system [1]. Lubricants are sold commercially and made up from a wide range of basic oils and additives. Mineral, synthetic and biological oils are the common three types of basic oils available in the market [2]. Currently, researchers are working hard to identify the optimum formula for developing new innovations in these lubricants in order to cope with the growth of the automotive and manufacturing industries throughout the world [3,4]. The main causes of energy loss and mechanical failure are friction and wear [5,6]. Friction consumes over one third of the world's primary energy, and nearly half of the power of transportation equipment is also consumed in friction [7]. While worn-out parts account for nearly 80% of mechanical failures, friction is identified as a major contributor to surface corrosion and pollution in the environment. Therefore, decreasing friction and wear in a particular mechanical system is critical in extending the service life of mechanical equipment, as well as saving energy and reducing emissions. Lubrication is one of the most effective methods for reducing friction and wear, which is crucial for energy conservation, emission reduction and environmental preservation [8].

Recently, nanoparticles have been identified as the promising candidates for fluid additives by improving the base fluid's properties for specific engineering applications [9]. The nanofluids outperformed the standard base liquid, which is water, in terms of effective thermal conductivity and effective viscosity [10]. Metal, metal oxide, carbon, sulphide, rare earth compounds, nano-composite [11] and others are among nanoparticle additives that are grouped based on their chemical elements characteristics [12]. The addition of nanolubricants in basic fluids has significantly altered the thermo-physical and tribological characteristics of the modified lubricants [13–17]. As mentioned by Falvo and Superfine [18] and Gulzar et al. [19], the relative orientation of the contacting surfaces has an impact on the frictional behaviour for lubrication systems at the nanometer-scale. As a result, a variable performance is likely to be shown under various test conditions. This suggests that the tribological effectiveness of nanolubricants is likely to be system specific. Several experimental experiments were conducted utilising various geometric configurations, including four-ball, ball-on-flat, pin-on-disk, cylinder-on-flat, piston ring on cylinder liner and block-on-ring, to evaluate the tribological performance of nanoparticles as additives in lubricating oils. Rolling, sliding and exfoliation are other lubricating mechanisms of "exfoliation" that solid nanoparticles can engage [20]. Two main categories can be made from these mechanisms. The first is the direct effect of nanoparticles, which comprises protection and the ball bearing action. The second effect improves the surface by polishing and smoothing, as well as fixing and repairing.

According to Lee et al. [21], nanoparticles can perform as friction modifiers through four separate methods when used as a lubricant additive. The ball bearing effect [16,22], mending effect [23], tribo-film generation [24,25] and polishing effect [16,26] are some of the methods by which the dispersed nanoparticles increase the tribological properties of the base lubricants. According to Tao et al. [16], diamond nanoparticles deposited in paraffin oil penetrate rubbing surfaces and operate as a ball bearing, converting sliding action into rolling motion. Lee et al. [21] observed 24% reduction in COF for mineral oil using graphite nanoparticles at 0.5% volume concentration. While in another paper, Chang et al. [27] reported up to 60% reduction in COF for lithium grease with a 2% volume concentration of graphite nanoparticles. The tribological behaviour of nickel nanoparticle dispersion in synthetic oil was studied by Chou et al. [28]. They discovered that mixing nickel nanoparticles into synthetic oil has reduced friction and wear by up to 30% and 45%, respectively. Luo and Wei [8] discovered that employing Al_2O_3 nanoparticles has improved lubrication behaviour. The average COF and wear were reduced by 23.92% and 41.75%, respectively. Tao et al. [16] reported the ball bearing effect for diamond nanoparticles, while Marko et al. [29] revealed the polishing effect for diamond nanoparticles. Later, Raina and Anand [30] discovered the reduction of COF and wear up to 8.34% and 24.5%, respectively, by utilizing diamond nanolubricant at 0.2% mass concentration. According to the tribotest results conducted by A. Elagouz et al. [31], adding ZnO nano-additives reduced the coefficient of friction and specific wear rate of the ring by 20–23% and 43–88%, respectively, when compared to the reference oil. Through the use of four-ball tribology testing, M.F. Ismail [32] investigated the tribological properties of SiO_2 and TiO_2 with PVE nanolubricants for rolling piston rotary systems. These nanolubricants was used in residential air conditioning. With a significant COF reduction of around 15%, the findings were obtained using nanolubricants with concentrations of 0.005 vol% for SiO_2/PVE and 0.015 vol% for TiO_2/PVE. The reduction in friction was caused by the nanoparticle's morphology working on the surfaces of two mechanical components that were in contact to reduce the contact area. It also proved that the wear scar radius on the ball's surface demonstrates the effectiveness of nanoparticles in reducing friction.

The use of nanoparticles in refrigeration systems has lately sparked interest among researchers. Saidur et al. [4] and Alawi and Sidik [33] investigated the potential of nanorefrigerant and nanolubricant for performance improvement in refrigeration systems. Bi et al. [34] used TiO_2 nanoparticles as a lubricant additive in mineral oil (MO) to replace polyol-ester (POE) lubricant for residential refrigerators. They observed that the TiO_2/MO nanolubricant reduced the compressor input power of refrigerators by 26% in comparison to POE lubricant. In another study, Jwo et al. [3] discovered that utilising TiO_2/MO nanolubricant at 0.1% mass concentration in domestic refrigerators decreased the compressor input power by 2.4%. Furthermore, the refrigerator performance increased by 4.4% because of the lower energy usage by the compressor. Based on the reported literature, further experimental investigation of the tribology performance of nanolubricants is crucial to improve the refrigeration system performance, especially in reducing the compressor's work. In the previous study, the tribological behaviour of Al_2O_3/PAG nanolubricants was studied by Aminullah et al. [35] for conventional automotive air-conditioning (AAC) systems. They confirmed that the additional of Al_2O_3 nanoparticles improved the tribology properties of the nanolubricants and affected the AAC compressor piston liner wall. The COF and wear rate dropped by 7.59% and 33.3%, respectively. From the reported literature, the benefits of nanoparticle-enhanced lubricants in automotive applications are significant [36].

The use of nanolubricants for automotive application is not limited to engine lubrication, but also can be employed for other components, such as the compressor of an AAC system. An AAC compressor's performance plays an important role in the overall system performance. In general, the overall AAC system performance can be improved by reducing the compressor's work, which is translated by increasing the coefficient of performance (COP). Continuous operation of the AAC compressor without appropriate lubrication can result in an increase of COF and wear rate, subsequently increasing the compressor's work, which results in a reduction of the COP value. As a solution, the compressor's work can be minimized by improving the tribology properties of the base lubricant in terms of COF and wear rate by introducing nanolubricants.

The air-conditioning system in conventional vehicles is powered by an engine through a belting-driven compressor (BDC). Whereas the HEV used an electrically driven compressor (EDC) to operate the air-conditioning system and is powered by direct current from the vehicle's battery, the power source to operate the AAC-EDC system is one of the major concerns in HEV. The exclusively available energy for HEV propulsion is the electricity stored in the battery pack; thus, the additional power consumed by an AAC-EDC system will affect the overall performance of the HEV. With the compact design of the AAC system, the need for energy will consume size, weight, space and also the cost of the battery. The performance and workload of the AAC system can be improved through vapour compression technologies [37,38]. In the long run, it would give monetary benefits, such as reducing the material used in the refrigeration system. The lighter weight of an AAC-EDC system means the transport runs with better fuel and electrical energy efficiency.

In this study, polyol-ester (POE)-based nanolubricants were formulated for AAC systems with an electrically driven compressor (EDC) specifically for hybrid electrical vehicles (HEV). Previous studies on tribological behaviours of mono and hybrid POE-based nanolubricants are very limited, and no similar investigation is available for application in an AAC system with EDC. Therefore, the present study is to evaluate the tribology characteristics of TiO_2/POE and SiO_2/POE mono nanolubricants, as well as TiO_2-SiO_2/POE hybrid nanolubricants, in comparison to the POE-based lubricant. The investigations were undertaken by formulating an excellent stability of POE nanolubricants and analysing the friction torque, COF and wear scar diameter (WSD) using a four-ball tester tribology machine.

2. Methodology

2.1. Preparation of Nanolubricants

In the present study, metal oxide nanoparticles, namely, SiO_2 and TiO_2, are considered in the preparation of nanolubricants. SiO_2 (amorphous) nanoparticles with 99.9% purity, 30 nm average size and spherical shape are used in this work. The SiO_2 nanoparticles were obtained from DKNANO (Beijing Deke Daojin Science and Technology Co., Ltd., Beijing, China). On the contrary, TiO_2 nanoparticles with 99.9% purity and 30 to 50 nm in size were procured from HWNANO (Hongwu International Group Ltd., Guangzhou, China). The transmission electron microscopy (TEM) was used to characterise these nanoparticles. Table 1 depicted the properties of both SiO_2 and TiO_2 nanoparticles. All safety precautions and adequate personal protective equipment were utilized during the preparation of nanolubricants.

Table 1. Properties of TiO_2 and SiO_2 nanoparticles [39,40].

Property	TiO_2	SiO_2
Thermal Conductivity (W/m·K)	8.4	1.4
Specific heat (J/kg·K)	692	745
Density (kg/m^3) @ 20 °C	4230	2220
Molecular mass (g/mol)	79.86	60.08
Average particle diameter (nm)	50	30

The electrically driven compressor (EDC) in an AAC system for HEVs is powered by an internal electric motor and operated at a high voltage. The electric motor's coil in the compressor comes into touch with the compressor's oil for lubrication. The lubricant in the EDC must be resistant to electrical short circuits at a certain degree. Therefore, POE lubricant is recommended for the EDC specifically in AAC systems for HEVs, as opposed to the conventional AAC system, which uses polyalkylene glycol (PAG) lubricant. POE lubricant has been utilized in electric compressors for more than 20 years, and its high dielectric qualities make it a preferred choice for electric compressors in hybrid automobiles and other vehicles with electric compressors. In this study, POE RL68H, with its properties shown in Table 2, was used in the preparation of nanolubricants. The RL68H is categorized under ISO VG 68 synthetic polyol-ester (POE) lubricant and is specifically designed for application in hydrofluorocarbons (HFC) refrigerant refrigeration and air-conditioning systems. This lubricant is ideal for both initial and service fill to provide effective wear protection for steel and aluminum surfaces, resulting in greater system life and efficiency. POE RL68H lubricant may be used for a wide range of operating temperatures because it has low temperature properties with outstanding chemical and thermal stabilities. The lubricant also offers excellent miscibility with HFCs and other refrigerants, high inherent lubricity over a wide temperature range and good chemical stability with system components.

Table 2. Properties of base fluid.

Properties	POE RL68H
Viscosity @ 40 °C (cSt)	66.6
Viscosity @ 100 °C (cSt)	9.4
Pour Point (°C)	−39
Density @ 20 °C (g/mL)	0.977
Flash Point (°C)	270

A two-step approach was used to prepare the nanolubricant by dispersing TiO_2 and SiO_2 nanoparticles into the POE-based lubricant, as reported by [41]. TiO_2/POE, SiO_2/POE and TiO_2-SiO_2/POE nanolubricants were prepared for tribology investigation in the present study with TiO_2-SiO_2/POE hybrid nanolubricant formulated at a 50:50 ratio of nanoparticles. In previous studies by Redhwan et al. [42] and Sharif et al. [43], the optimum volume concentration for Al_2O_3/PAG and SiO_2/PAG nanolubricants was established at 0.01 and 0.05%, respectively. Therefore, in this study, the nanolubricants were prepared at less than 0.1% volume concentration. In the preparation process, the filtered nanoparticles were disseminated into the POE lubricants and swirled for half an hour using a magnetic stirrer before proceeding to the sonication step. In the present study, the sonication process used an ultrasonic bath with ultrasonic pulses of 100 W and 36 (±3) kHz to improve the stability of the suspended TiO_2 and SiO_2 nanoparticles in the POE-based lubricants, as well as to reduce the agglomeration size. This step is essential to optimize the tribology characteristics of the nanolubricants. Figure 1 shows the two-step process flow of the mono and hybrid nanolubricants preparation utilized for the present work. The volume concentration of the nanolubricant was calculated by using Equation (1) [44].

$$\varphi_{NL} = \frac{\left(\frac{m_p}{\rho_p}\right)}{\left(\frac{m_p}{\rho_p} + \frac{m_L}{\rho_L}\right)} \times 100\% \quad (1)$$

where φ_{NL} is the volume concentration of nanolubricant, m_p is the mass of nanoparticles in kg, m_L is the mass of POE lubricant in kg, ρ_p is the density of nanoparticles in kg/m^3 and ρ_L is the density of POE lubricant in kg/m^3.

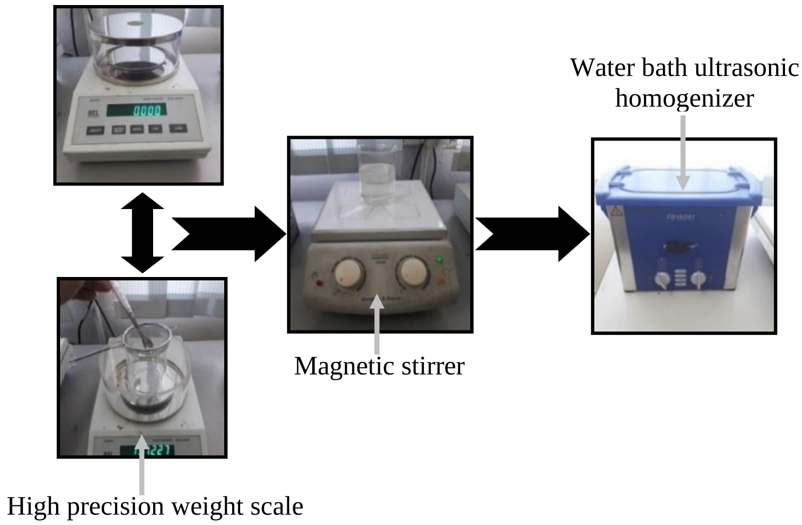


Figure 1. Two-step process in preparation of the nanolubricants.

2.2. Stability of Mono and Hybrid Nanolubricants

The nanolubricant's stability is influenced by the base lubricant and suspended nanoparticle morphology, such as form, shape and structure. In order to assess the stability condition of the nanolubricants, both qualitative and quantitative measurement methods are used. The qualitative method is done by visual sedimentation observation of the sonicated mono and hybrid nanolubricants placed in test tubes at different volume concentrations over a certain period of time. The images of sedimentation for each set of nanolubricants were observed, captured and compared from the first day until 30 days after preparation. According to Ghadimi et al. [45], the most common method for evaluating stability of nanolubricants is done through photo capturing comparison and observation of the sedimentation settling over time. The aim of this technique is to ensure the nanolubricants are in stable condition with minimal sedimentation [46].

The potential difference between the dispersion medium and the stationary layer of fluid connected to the particle is known as the zeta potential. In dispersion, the zeta potential represents the degree of repulsion between adjoining, similarly charged particles. Colloids with a high zeta potential (negative or positive) are electrically stable, whereas those with a low zeta potential are coagulate or flocculate. Zeta potential analysis was undertaken for the quantitative assessment to validate the stability condition of the prepared nanolubricants by examining its electrophoretic behaviours [47]. In this work, the zeta potential for mono and hybrid nanolubricants were measured using Anton-Paar Litesizer 500. A large absolute value indicates good particle dispersion into the base lubricant; hence, it can be translated as high stability [42]. Further characterization of SiO_2 and TiO_2 nanoparticles was accomplished from Transmission Electron Microscopy (TEM) imaging. TEM was also used to confirm the nanoparticle size in suspended form, while simultaneously observing the state of nanolubricants' aggregation and dispersion. The average size of the nanoparticle is determined using the number-based size of the nanoparticles from the TEM images.

2.3. Measurement of Tribology Properties

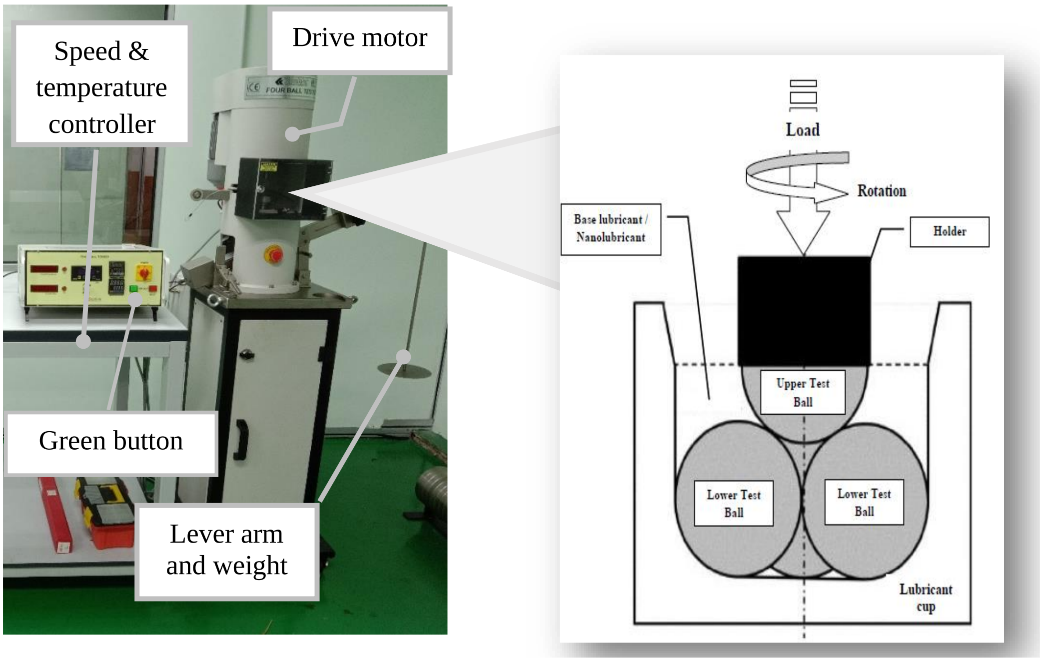
The tribological performance of TiO_2/POE, SiO_2/POE and TiO_2-SiO_2/POE nanolubricants was performed using a Koehler four-ball tribo tester according to ASTM D4172-18 standard, as shown in Figure 2a. Test balls with diameter of 12.7 mm, hardness Rockwell C (HRC) of 60 ± 1 and made of chromium steel grade G20 under ISO 3290 were used in this study [45–47]. The tribology measurement was started with pure POE lubricant, which was then followed by TiO_2/POE, SiO_2/POE and TiO_2-SiO_2/POE nanolubricants at different volume concentrations of 0.01%, 0.03%, 0.05%, 0.07% and 0.10%. The test condition for the ASTM D4172-18 standard is presented in Table 3. For this experiment, the operating temperature was set at 75 °C in a test duration of up to 60 min. An automatic temperature controller was used to regulate the heater to maintain the lubricants' constant temperature. A load of 40.0 kg was applied on the lever arm, and the rotary speed was set at 1200 rpm. The friction torque of pure POE lubricant and nanolubricants was recorded. Coefficients of friction (COF) were calculated from Equation (2) for all experimental conditions, using the measured friction torque and constant load.

$$\mu = 2.23004 \frac{\tau}{F_N} \quad (2)$$

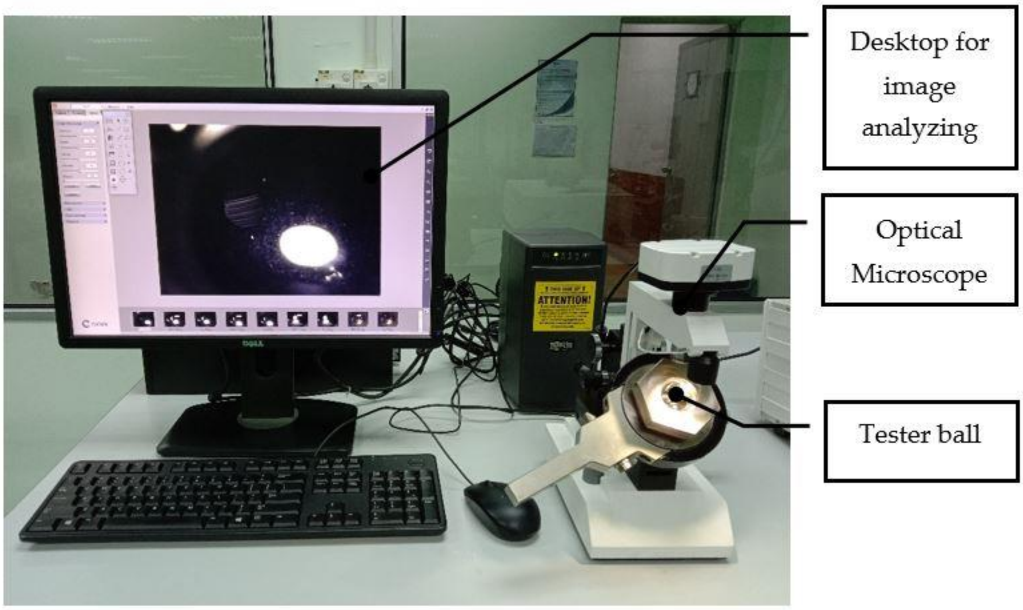
where τ is the friction torque, kg·cm; F_N is the normal load, kg; and μ is the coefficient of friction. Later, the wear scar diameters (WSD) of the three lower balls were measured by an optical microscope at an accuracy of 0.01 mm, as shown in Figure 2b. Each test was performed three times, and the average wear scar diameter was calculated. Solvent, such as hexane, was used in the present study to clean the tools and balls before and after each test.

Table 3. The test conditions for ASTM D4172-18 standard.

ASTM Standard	Test Method	Test Conditions				Remarks
		Speed (rpm)	**Load (kg)**	**Duration (Minutes)**	**Temperature (°C)**	
D4172-18	Wear Preventive Characteristics of Lubricating Fluid	1200 ± 60	40.0 ± 0.2	60 ± 1	75 ± 2	Ball pot to be torqued down between 25 and 50 ft·lb



(**a**) Four-Ball tribology tester and its schematic configuration



(**b**) Wear scar diameter microscope

Figure 2. Four ball tribology tester and optical microscope.

3. Results and Discussion

3.1. Stability of Nanolubricants

The stability results for both mono and hybrids of TiO_2/POE, SiO_2/POE and TiO_2-SiO_2/POE nanolubricants using both qualitative and quantitative methods are presented in this section.

3.1.1. Sedimentation Observation

The samples of the nanolubricants at different volume concentrations are shown in Figure 3. TiO_2/POE, SiO_2/POE and TiO_2-SiO_2/POE nanolubricants were observed to be visually stable in Figure 3a–c, respectively, with only minimum sedimentation up to 30 days after preparation. For TiO_2/POE nanolubricant, a small amount of sedimentation occurred for samples at 0.07% and 0.1% volume concentrations, which is similar to TiO_2-SiO_2/POE hybrid nanolubricant, as well. For both nanolubricants, minimum sedimentation only occurred for samples at volume concentrations of 0.01% and 0.03%. However, in general, the visual stability of TiO_2-SiO_2/POE hybrid nanolubricant was found to be better than TiO_2/POE nanolubricant. Meanwhile, all SiO_2/POE nanolubricant samples were found to be stable, without any significant sedimentation. Overall, observation confirmed no significant separation between the POE-based lubricant and nanoparticles for up to 30 days after preparation. It should be noted that no surfactant was utilized in the preparation of all nanolubricants. This is important to avoid any drawback to the refrigeration system during operation and application. In summary, SiO_2/POE nanolubricant exhibited the best stability condition, according to visual observation, which was then followed by TiO_2-SiO_2/POE and TiO_2/POE nanolubricants. The presence of an SiO_2 nanoparticle with TiO_2 nanoparticles in hybrid form improved the stability condition of the TiO_2-SiO_2/POE nanolubricants. Further quantitative evaluation by zeta potential is conducted in the next section to confirm these findings.

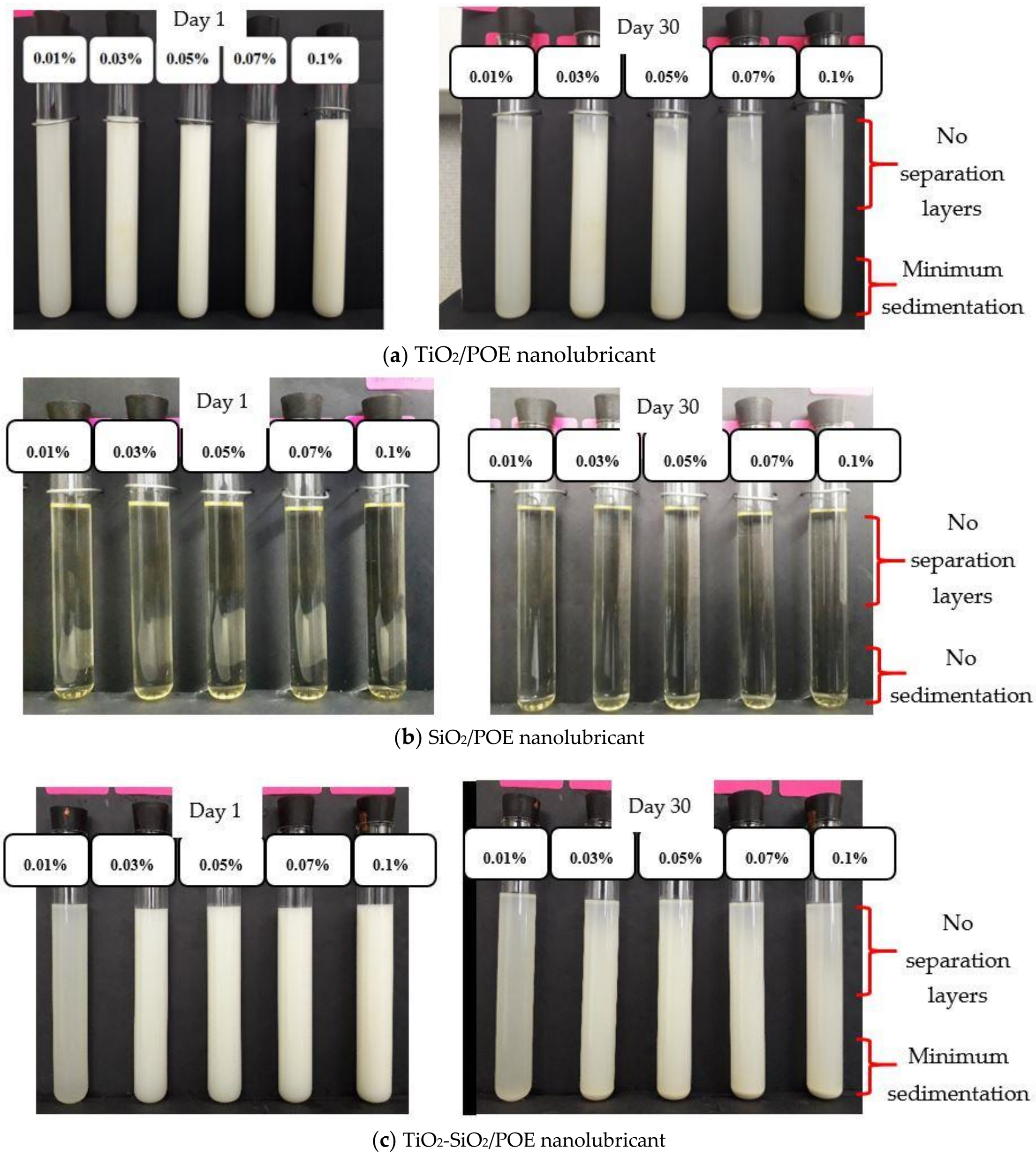


Figure 3. Nanolubricants sedimentation observation for up to 30 days after preparation.

3.1.2. Zeta Potential Evaluation

Figure 4 shows the zeta potential measurement for both mono and hybrid nanolubricants. The zeta potential for TiO_2/POE and SiO_2/POE mono nanolubricants was attained at the value of up to 81.1 and 102.5 mV, respectively. Meanwhile, the zeta potential value for TiO_2-SiO_2/POE hybrid nanolubricant was achieved at 276.8 mV, which is higher than the TiO_2/POE and SiO_2/POE mono nanolubricants. These results were compared with the classification of the stability condition for the different range of the zeta potential by Ghadimi et al. [45]. The absolute value of zeta potential of above 60 mV is desirable for excellent stability conditions of the nanolubricants. The mono and hybrid nanolubricants of this study exhibited zeta potential values of more than 60 mV, which is proven to be beyond the stability limit. The TiO_2-SiO_2/POE hybrid nanolubricant was recorded with the highest zeta potential value among the other nanolubricants; hence, it confirmed the findings made from the visual observation. Its electrokinetic properties have a significant impact on the stability of TiO_2-SiO_2/POE nanolubricants. The strong repulsive forces produced by the increased surface charge density are what cause the decrease in coagulation likelihood [48]. According to stabilisation theory, a large absolute value of zeta potential indicates that the electrostatic repulsive forces between nanoparticles are very strong and, hence, reflect a stable suspension [45]. Therefore, all nanolubricants of the present study are concluded to have excellent stability conditions, which can be used for tribology behaviour evaluation.

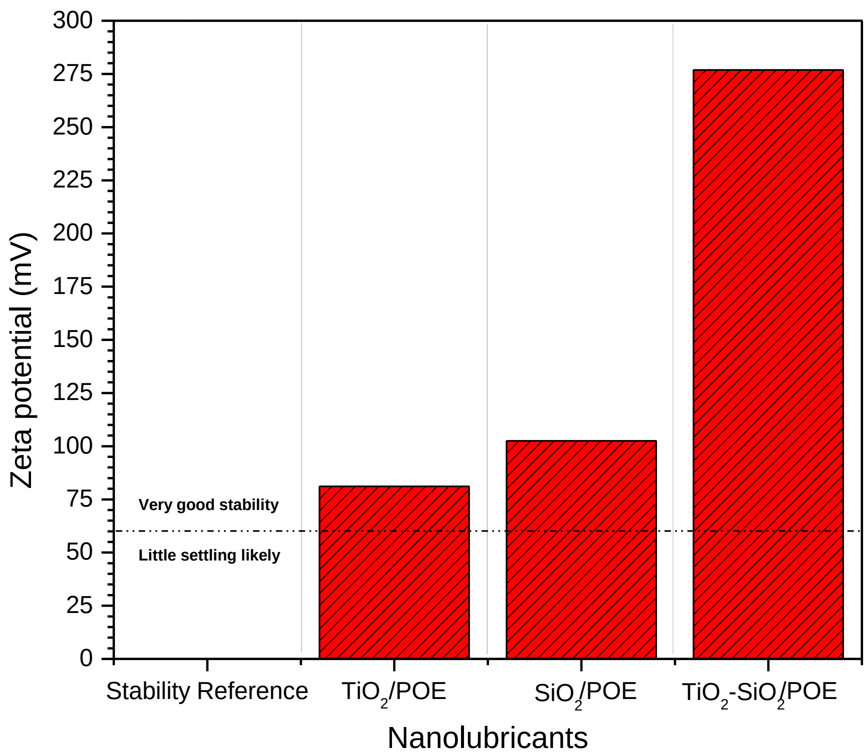
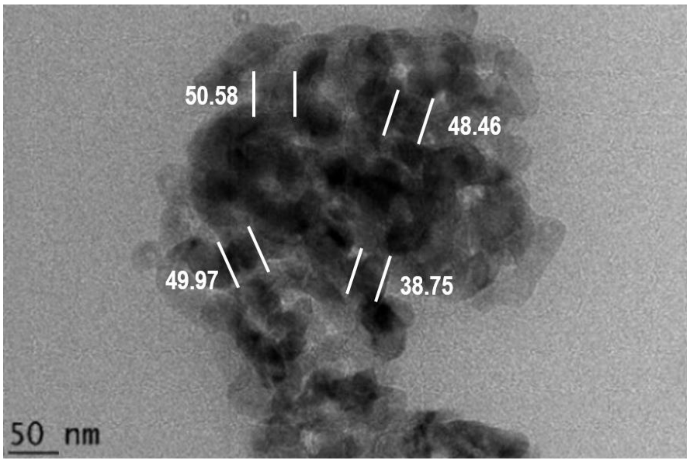


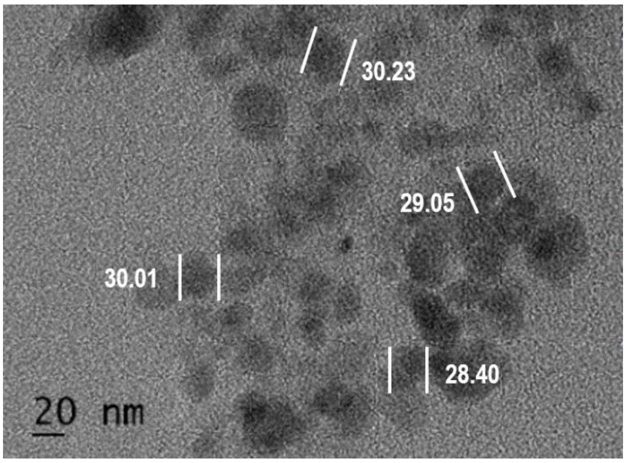
Figure 4. Zeta potential of TiO_2/POE, SiO_2/POE and TiO_2-SiO_2/POE nanolubricants.

3.1.3. Micrograph Observation

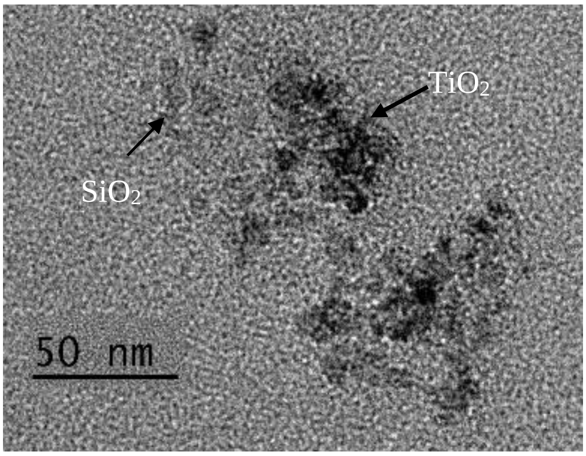
The TEM images of TiO_2 and SiO_2 nanoparticles suspended in POE RL68H lubricant at the amplification of ×88,000 and ×180,000 are shown in Figure 5a,b, respectively. The size of TiO_2 was confirmed to be 30 to 50 nm, while SiO_2 nanoparticles are 30 nm in size, with both nanoparticles observed to be spherical in shape. For both mono nanolubricants, minimal clustering and agglomeration of nanoparticles were observed, with well dissemination of nanoparticles in the POE RL68H-based lubricant. Meanwhile, Figure 5c shows the TEM image for the TiO_2-SiO_2 hybrid nanolubricant, where the particles distribution of TiO_2 and SiO_2 nanoparticles is clearly indicated. The clustering and agglomeration of these nanoparticles is found to be uniform and irregular in shape. From these TEM observations, it can be concluded that the nanoparticles demonstrated good dispersion and less agglomeration while suspended in POE-based lubricant.



(**a**) TiO_2/POE nanolubricant (×88,000)



(**b**) SiO_2/POE nanolubricant (×180,000)



(**c**) TiO_2-SiO_2/POE nanolubricant (×88,000)

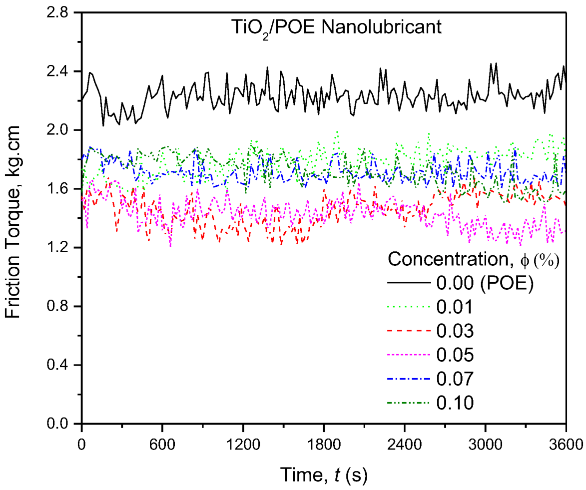
Figure 5. TEM images for mono and hybrid nanolubricants.

3.2. *Tribology Properties*

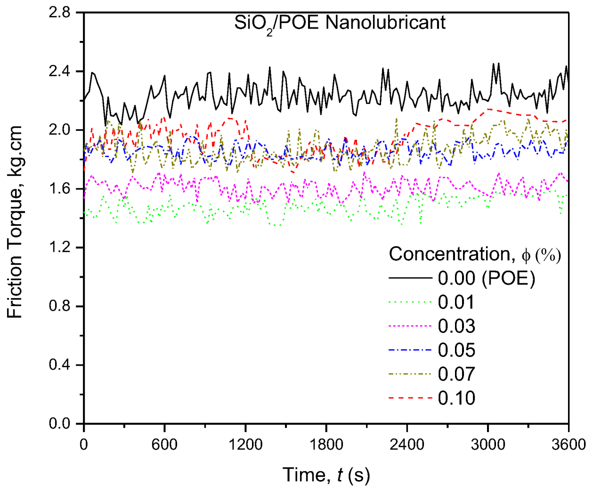
The evaluations of the tribology properties for both mono and hybrid nanolubrications, including friction torque, coefficient of friction, wear scar diameter, as well as microscopic observation, are presented in this section.

3.2.1. Friction Torque Evaluation

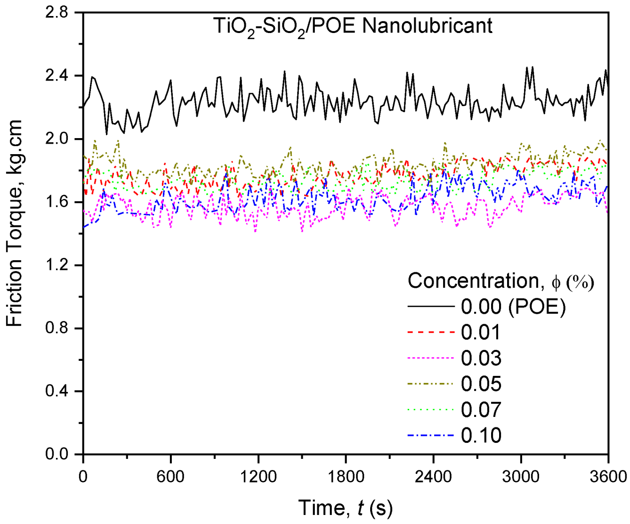
Figure 6 presents the friction torque for mono and hybrid nanolubricants. It was observed that the friction torque for both mono nanolubricants of TiO_2/POE (Figure 6a) and SiO_2/POE (Figure 6b) performed at lower values than the pure POE lubricant at all volume concentrations. The highest reduction of 36% occurred at 0.05% volume concentration for TiO_2/POE nanolubricant, which was then followed by 0.03%, 0.07% and 0.1% volume concentrations with friction torque reduction of 34.9%, 23.4% and 23.4%, respectively, as shown in Figure 6a. Meanwhile, the lowest reduction of 20.2% happened at 0.01% volume concentration. However, the highest friction torque reduction for SiO_2/POE nanolubricants in Figure 6b was obtained at 0.01% volume concentration. The reduction percentage was gradually decreased for volume concentration higher than 0.01%, as depicted in Figure 6b. The highest reduction was obtained at 33.4% at 0.01% volume concentration and decreases to 28%, 17%, 16.1% and 14% at volume concentrations of 0.03%, 0.05%, 0.07% and 0.1%, respectively. Figure 6c shows the friction torque reduction for TiO_2-SiO_2/POE hybrid nanolubricant. The hybrid nanolubricant performed with significant reduction of friction torque at all volume concentrations and at lower value than pure POE lubricant. The highest friction torque reduction for hybrid nanolubricant was obtained at 29.8% at 0.01% volume concentration. Generally, the lowest friction torque with the highest reduction for TiO_2/POE, SiO_2/POE and TiO_2-SiO_2/POE nanolubricants was observed at 36%, 33.7% and 29.8% at volume concentrations of 0.05%, 0.01% and 0.03%, respectively. The summary of friction torque reductions is presented in Table 4.



(**a**) TiO_2/POE nanolubricant



(**b**) SiO_2/POE nanolubricant



(**c**) TiO_2-SiO_2/POE nanolubricant

Figure 6. Friction torque for mono and hybrid nanolubricants.

Table 4. The performance of friction torque, COF and WSD for mono and hybrid nanolubricants.

Nanolubricants	Volume Concentration (%)	Reduction of Friction Torque (%)	Reduction of COF (%)	Reduction of WSD (%)
TiO_2/POE	0.01	20.2	22.1	12.5
	0.03	34.9	36.1	11.6
	0.05	36.0	37.5	2.0
	0.07	23.4	24.1	1.2
	0.1	23.4	24.9	0.7

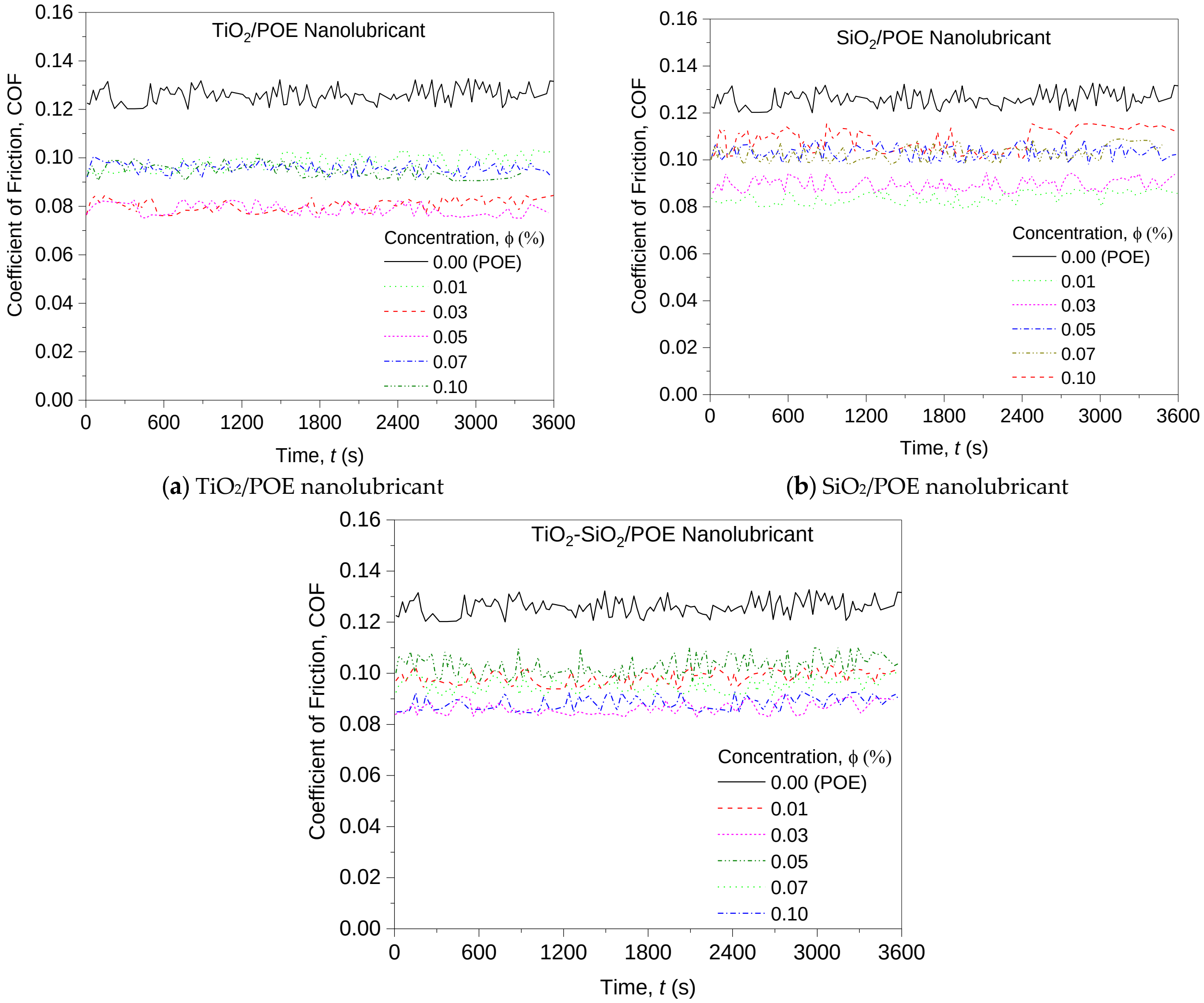
Table 4. *Cont.*

Nanolubricants	Volume Concentration (%)	Reduction of Friction Torque (%)	Reduction of COF (%)	Reduction of WSD (%)
SiO_2/POE	0.01	33.7	33.5	26.4
	0.03	28.0	28.9	8.6
	0.05	17.0	18.1	8.2
	0.07	16.1	18.5	8.6
	0.1	14.0	14.4	9.1
TiO_2-SiO_2/POE	0.01	21.0	21.9	10.8
	0.03	29.8	31.6	12.4
	0.05	17.9	18.5	10.6
	0.07	22.1	24.4	11.4
	0.1	26.7	29.9	8.9

3.2.2. Coefficient of Friction Evaluation

Figure 7 illustrates the coefficient of friction (COF) for mono and hybrid nanolubricants at different volume concentrations for up to 3600 s or 1 hour experimental evaluation. A similar trend was observed for COF, as compared to the trend for friction torque, with a significant difference in percent improvement. The COF performance of nanolubricants was mostly influenced by the volume concentration of nanolubricants and in agreement with the previous finding [49]. Figure 7a shows COF values for mono TiO_2/POE nanolubricant, as compared to the pure POE lubricant. The COF was attained at lower values than the POE lubricant at all volume concentrations. The highest average decrement was found to be at 0.05% volume concentration and reached up to 37.5% less than the POE-based lubricant. Meanwhile, the lowest average decrement occurred at 0.01% volume concentration with 22.1% improvement. Figure 7b presents COF values for mono SiO_2/POE nanolubricant in comparison to the pure POE lubricant. Similarly, all COF values at all volume concentrations generally decreased and performed at lower values than the pure POE lubricant. The highest average reduction reached up to 33.4% at 0.01% volume concentration, which was then followed by 28.9%, 18.5% and 18.1% for 0.03%, 0.07% and 0.05% volume concentrations, respectively. The lowest average reduction was attained at 0.1% volume concentration with 14.2% increment.

The COF values for TiO_2-SiO_2/POE hybrid nanolubricant are shown in Figure 7c. All samples of the hybrid nanolubricant at all volume concentrations performed with lower COF values than the pure POE lubricant. The highest average decrement of 31.6% was observed at 0.03% volume concentration, while the lowest average decrement of 18.5% occurred at 0.05% volume concentration. Correspondingly, the COF values for hybrid nanolubricant at 0.07% and 0.1% volume concentrations were reduced with decrements of 24.4% and 29.9%, respectively. Interestingly, the COF value decrements were recorded at all volume concentrations of both mono and hybrid nanolubricants. It was expected that the mono and hybrid nanolubricant would behave with high anti-friction performances, as compared to the POE-based lubricant. Therefore, the addition of nanoparticles into the base lubricant can improve the tribology performance of the original lubricant. From the overall findings, it can be inferred that the anti-friction performance of the base lubricant can be improved significantly using different types of nanoparticles at particular concentrations [50]. For instance, the present study suggested the optimum volume concentration of 0.01%, 0.03% and 0.05% for SiO_2/POE, TiO_2-SiO_2/POE hybrid and TiO_2/POE nanolubricants, respectively, for air-conditioning system application specifically with EDC. Furthermore, the primary cause of the reduction of friction torque and COF of the present nanolubricants is due to the amount of effective nanoparticles that can convert from pure sliding friction into rolling friction (nanoparticle mechanism) as a result of lower interfacial frictional surface action [25]. It is thought that the spherical and quasispherical nanoparticles behave as tiny ball bearings that roll onto the contact region. Such shaped nanoparticles are thought to change sliding friction into a combination of sliding and rolling friction. This lubricating process is related to the tribo-pair system, which maintains the nanoparticles' stiffness and form by creating stable low-load conditions between the shearing surfaces. For better understanding, COF values reduction is summarized in Table 4.



(**a**) TiO_2/POE nanolubricant (**b**) SiO_2/POE nanolubricant

(**c**) TiO_2-SiO_2/POE nanolubricant

Figure 7. Coefficient of friction for mono and hybrid nanolubricants.

Figure 8 shows the COF reduction of present nanolubricants in comparison to the existing studies in the literature. Luo et al. [51] used Al_2O_3/TiO_2 nanocomposites dispersed in lubricating oil. The Al_2O_3/TiO_2 nanolubricant attained COF reduction of up to 13.2%. In comparison to the mono TiO_2/POE nanolubricant, the present nanolubricant shows better performance than Al_2O_3/TiO_2 nanolubricant, with COF reduction improvement up to 16.7%. In another paper, Jiao et al. [52] used mineral oil as a base lubricant, with Al_2O_3/SiO_2 nanoparticles dispersion as the lubricant additive. According to the findings, the COF was reduced up to 25%, which is considerably lower than the COF of the present study for mono SiO_2/POE nanolubricant with 3.9% difference. On the contrary, another study reported by Gulzar et al. [53] employed TiO_2-SiO_2 hybrid nanoparticles dispersed in palm oil-based trimethylolpropane (TMP) esters. The reduction for COF was reported up to 16.1%. This finding is substantially lower than the present study for TiO_2-SiO_2/POE hybrid nanolubricant, with 13.8% enhancement of COF reduction. In general, although the study on the POE-based nanolubricant is not extensive, it demonstrates better COF performance as compared to other base types of nanolubricants.

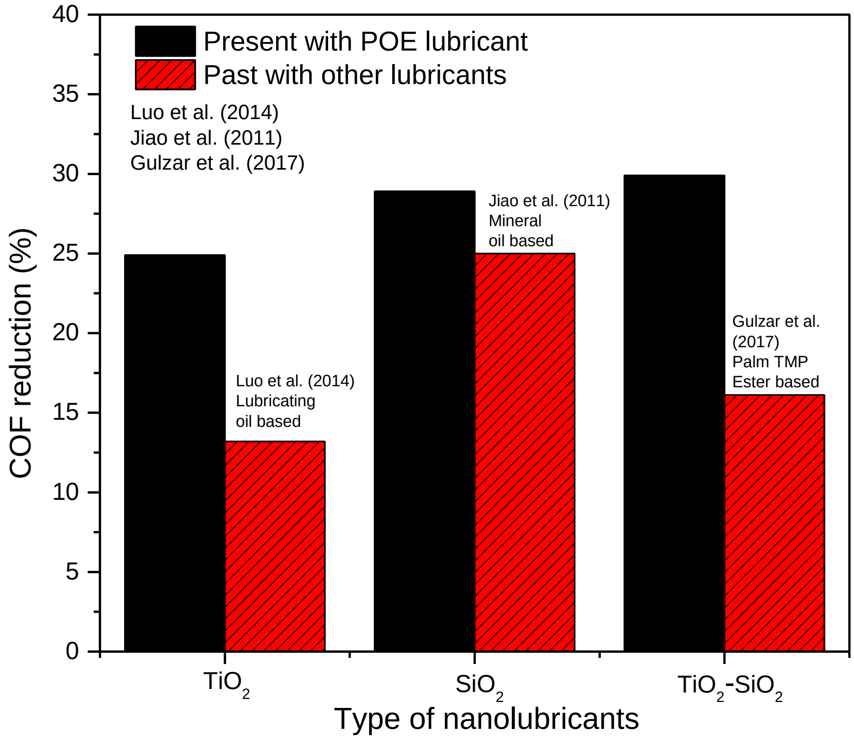
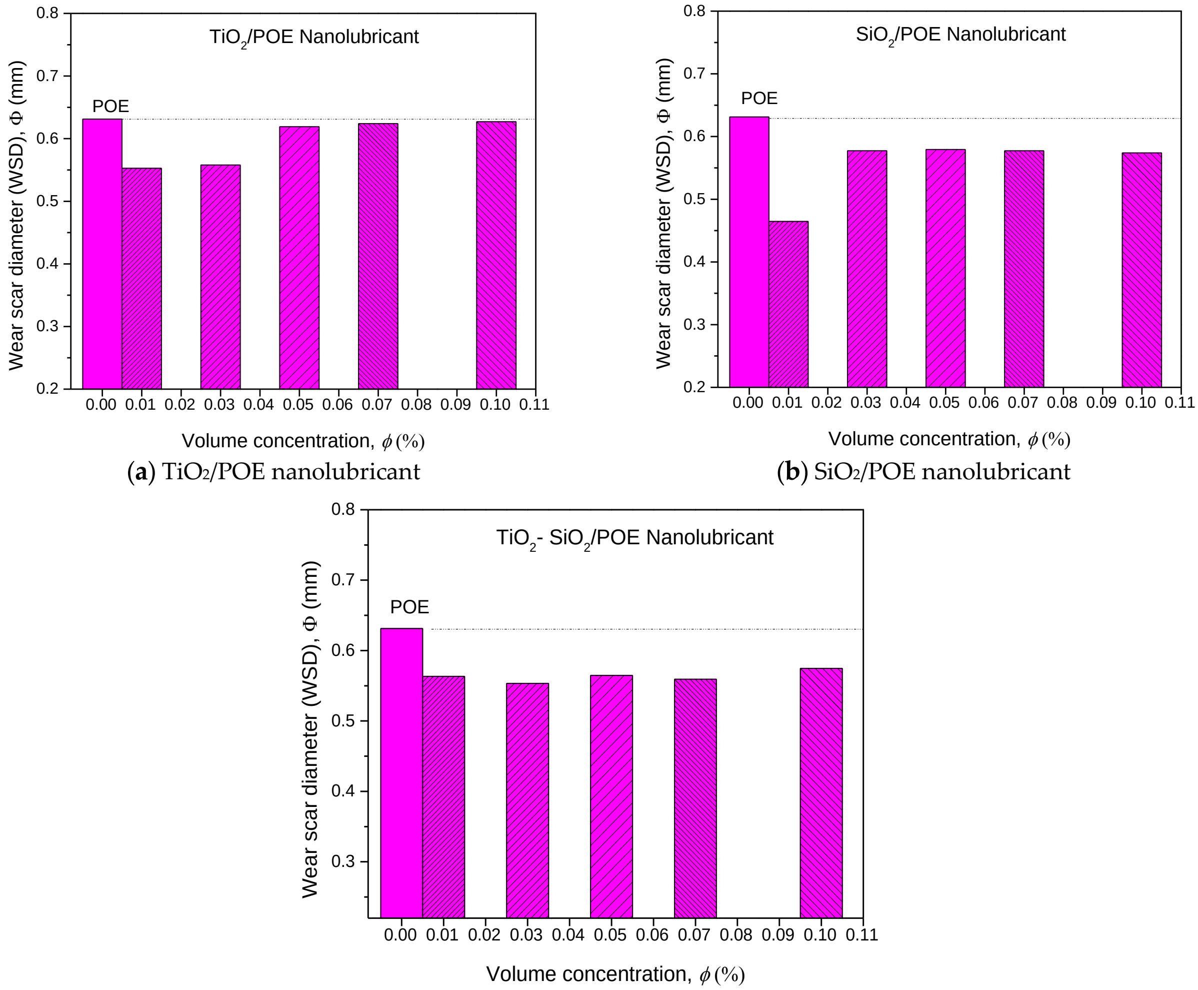


Figure 8. Comparison of coefficient of friction reduction with previous studies.

3.2.3. Wear Scar Diameter Evaluation

According to the ASTM D4172-18 standard, the compensation scar radius is defined as the average radius in millimetres of the wear scar on the stationary lower balls, and it occurred after the tribology test evaluation. The revolving top ball was pressed against the stationary balls at the bottom by an axial force in the presence of lubricant, thus creating identical wear scar diameter (WSD) without causing any scuffing or welding to the balls. Therefore, surface wear performance is another crucial factor that needs to be considered during selection of an effective lubricant for the refrigeration system and specifically for AAC systems with EDC. Figure 9 shows the WSD of mono and hybrid nanolubricants in comparison to the pure POE lubricant. As shown in Figure 9a, the TiO_2/POE nanolubricant attained lower WSD than the pure POE lubricant at 0.01% and 0.03% volume concentrations with high average reductions of 12.5% and 11.6%, respectively. In contrast, the WSD for TiO_2/POE nanolubricant was decreased significantly at volume concentrations of 0.05%, 0.07% and 0.1% with small average increments of 2.0%, 1.2% and 0.7%, respectively. These findings indicate that the compensation scar radius for TiO_2/POE nanolubricant outperformed the POE lubricant at a volume concentration of less than 0.05% with optimum concentration of 0.01%.

Figure 9b indicates the WSD for mono SiO_2/POE nanolubricant. In general, the WSD for SiO_2/POE nanolubricant is better than TiO_2/POE nanolubricant, with a substantial decrement trend for all volume concentrations, as compared to the pure POE lubricant. The highest WSD reduction was observed at 26.4% at 0.01% volume concentration, which was then followed by 9.1%, 8.61%, 8.6% and 8.4% for 0.1%, 0.07%, 0.03% and 0.05% volume concentrations, respectively. The reduction trend for WSD of SiO_2/POE nanolubricant is almost consistent at 0.03% to 0.1% volume concentrations. Interestingly, the optimum volume concentration for SiO_2/POE nanolubricant also occurred at 0.01% volume concentration, which followed a similar trend of optimum COF reduction. This could happen due to the lack of nanoparticles at low volume concentrations in the nanolubricants. An optimum amount of nanoparticles are necessary to generate rolling friction on the frictional surface, yet a large number of nanoparticles results in a high wear rate due to the agglomeration of excess nanoparticles [52]. Figure 9c presents the WSD variation of TiO_2-SiO_2/POE hybrid nanolubricant at different volume concentrations. Similar to the mono nanolubricants, the WSD for hybrid nanolubricant at all volume concentrations is always lower than the pure POE lubricant. The maximum reduction of 12.4% was recorded at 0.03% volume concentration, which was then followed by 0.07%, 0.01%, 0.05% and 0.1% volume concentrations with 11.4%, 10.8%, 10.6% and 8.9%, respectively. The reduction occurred because, when the roughness of the lubricating surface is decreased by nanoparticle-assisted abrasion, the polishing effect, also known as the smoothing effect, is thought to be observed. The spaces between the rough asperities, which can serve as reservoirs of solid lubricants (nanoparticles) within the contact, may be filled with nanoparticles in tribological contacts [54]. Table 4 summarizes the WSD reduction for mono and hybrid nanolubricants.



(**a**) TiO_2/POE nanolubricant (**b**) SiO_2/POE nanolubricant

(**c**) TiO_2-SiO_2/POE nanolubricant

Figure 9. Wear scar diameter for mono and hybrid nanolubricants.

Figure 10 displays a WSD reduction comparison between the current study and the prior findings. The previous studies also experienced the reduction of WSD for nanolubricant at much better values than their base lubricants. Luo et al. [51] studied Al_2O_3 with TiO_2 nanoparticles dispersed in lubricating oil, which showed 11.8% WSD reduction. The present study for TiO_2/POE nanolubricant is 0.7% better than their finding. Meanwhile in another study by Gulzar et al. [53], the improvement of WSD reduction up to 10.4% for TiO_2-SiO_2 hybrid nanolubricant using palm oil-based TMP Ester was observed. In comparison to the TiO_2-SiO_2/POE nanolubricant, the current study provides higher WSD reduction with 2% improvement. Therefore, it can summarised that the use of nanoparticles in POE lubricant will improve the tribology performance better than other types of lubricants.

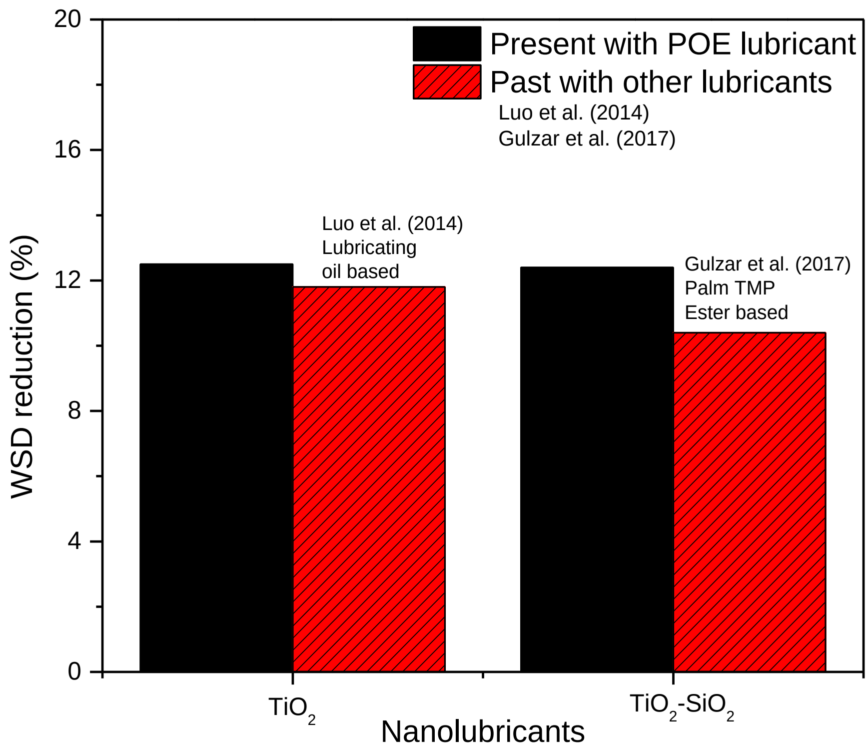
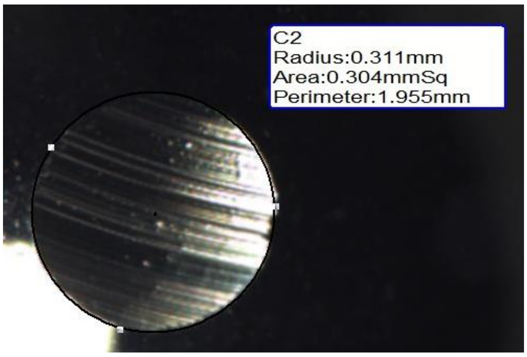


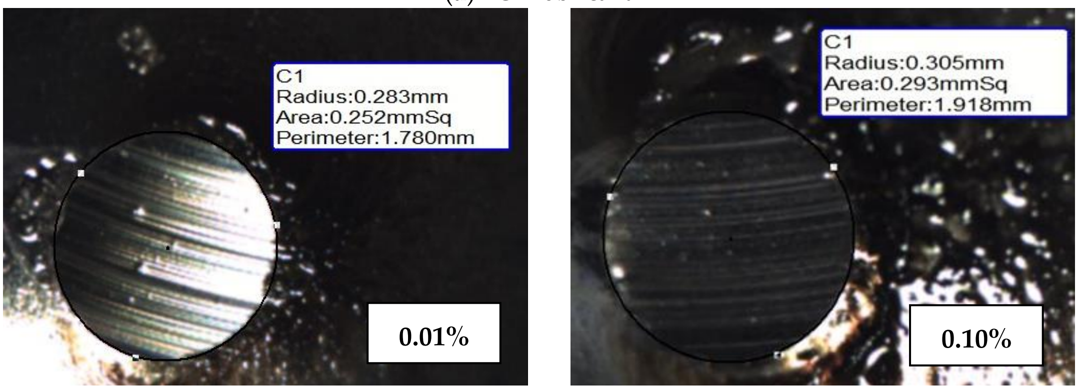
Figure 10. Wear scar diameter reduction comparison with previous studies.

3.2.4. Microscopic Observation

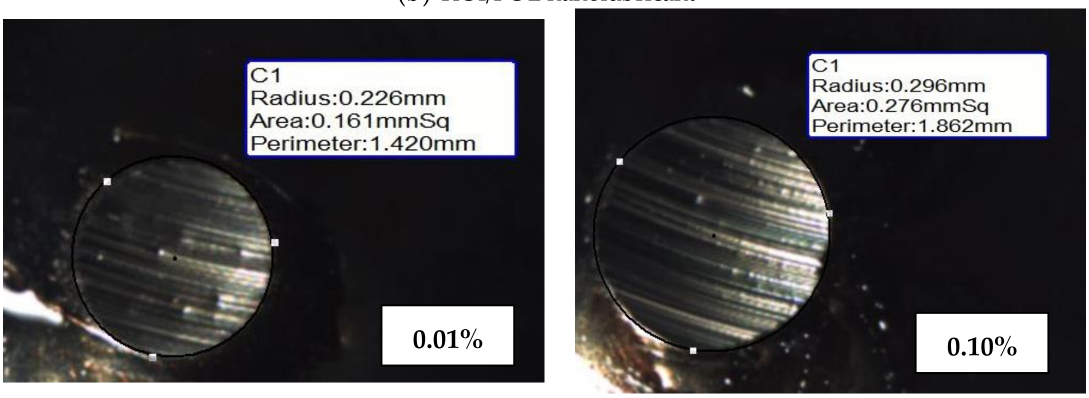
Figure 11 depicts microscopic observation of wear scar for mono and hybrid nanolubricants. The wear scar appearance on the ball surface with the implementation of POE-based lubricant is presented in Figure 11a as the reference data. The wear radius for POE lubricant is 0.311 mm. The wear scar images for nanolubricants are presented in Figure 11b–d with only images for 0.01% and 0.1% volume concentrations considered to represent the minimum and maximum amount of nanoparticles in the lubricants. Figure 11b shows the wear scar images by the application of TiO_2/POE nanolubricant. The wear radii of 0.283 and 0.305 mm were generated by the nanolubricant at volume concentrations of 0.01% and 0.1%, respectively. Meanwhile, the wear scar radii of 0.226 and 0.296 mm are shown in Figure 11c for SiO_2/POE nanolubricant at volume concentrations of 0.01% and 0.1%, respectively. Lastly, the scar radii for TiO_2-SiO_2/POE hybrid nanolubricant is displayed in Figure 11d. The wear scar radii at 0.01% and 0.1% volume concentrations for hybrid nanolubricant is consistent and almost similar by 0.284 and 0.281 mm, respectively. The wear scar radii for all samples of nanolubricants are lower than POE lubricant, hence confirming the behaviour of wear resistance with the use of nanoparticles as additive in lubricant for up to 0.1% volume concentration. The lowest scar radius was produced by SiO_2/POE nanolubricant at 0.01% volume concentration and in agreement with the previous COF findings.



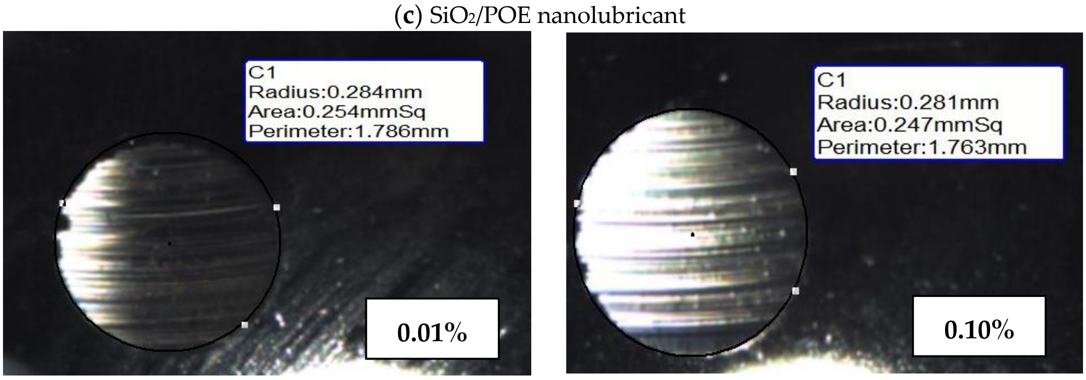
(**a**) POE lubricant



(**b**) TiO_2/POE nanolubricant



(**c**) SiO_2/POE nanolubricant



(**d**) TiO_2-SiO_2/POE nanolubricant

Figure 11. Microscopic observations for mono and hybrid nanolubricants.

4. Conclusions

The tribology characteristics in terms of the friction torque, COF and WSD of mono and hybrid nanolubricants in comparison to the POE-based lubricant were undertaken in the present study. All nanolubricants were found to be in excellent stability conditions, with zeta potential values of more than 60 mV. The friction torque and COF of mono and hybrid nanolubricants are generally lower than the POE lubricant for all volume concentrations. The lowest COF with the highest average reduction for TiO_2/POE, SiO_2/POE and TiO_2-SiO_2/POE nanolubricants was attained at volume concentrations of 0.05%, 0.01% and 0.03% and reached up to 37.5%, 33.5% and 31.6%, respectively. Similarly, the WSD for mono and hybrid nanolubricants at all volume concentrations appeared lower than the POE-based lubricant. The TiO_2/POE and SiO_2/POE mono nanolubricants attained the lowest WSD at 0.01% volume concentration, with the maximum reduction at 12.5% and 26.4%, respectively. In contrast, the TiO_2-SiO_2/POE hybrid nanolubricant achieved the highest reduction of WSD at 0.03% volume concentration and dropped down to 12.4%. The lowest scar radius was produced by SiO_2/POE nanolubricant at 0.01% volume concentration. The use of TiO_2 and SiO_2 nanoparticles in POE-based lubricant improved the tribology performances significantly and was found to be better than other types of lubricants. The present study suggested the optimum volume concentrations of 0.01%, 0.03% and 0.05% for SiO_2/POE, TiO_2-SiO_2/POE hybrid and TiO_2/POE nanolubricants, respectively. Consequently, mono and hybrid nanolubricants with volume concentrations of less than 0.05% are recommended for application in air-conditioning systems specifically with EDC.

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