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RESEARCH ARTICLE

The Tribology Evaluation on a Four-Ball Tribometer Lubricated by Al_2O_3/PAG Nanolubricants

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ABSTRACT - Nanolubricants can improve the tribological properties for application in automotive systems. By reducing the friction rate of the internal components with nanolubricants, the service life of a compressor used in automotive air conditioning (AAC) can be extended. The investigation aims to determine the optimal volume concentration of nanolubricants for achieving the highest performance in tribological properties. Al_2O_3 nanoparticles dispersed in a polyalkylene glycol (PAG ND12) base at volume concentrations of 0.01%, 0.03%, and 0.05% were investigated to improve the lubrication system in the AAC compressor. The stability investigations were carried out by comparing absorbance conditions using a UV-Vis Spectrophotometer at each volume concentration for 210 days. Koehler's four-ball tribometer was used to measure coefficient of friction (COF) and friction torque at a load of 40.0 kg and a speed of 1200 rpm. The stability study of nanolubricant yielded average absorbance values of 0.752, 0.755, and 0.684, respectively. The average COF values of the nanolubricants of 0.01%, 0.03%, and 0.05% were 0.104, 0.078, and 0.117, while the pure lubricant was 0.095. Further investigation on friction torque resulted in a decrease in the pure lubricant of 0.064%, and for nanolubricant Al_2O_3/PAG ND12, a decrease of 0.087%, 0.057%, and 0.092%, respectively. The results indicated that a concentration of 0.03% produced the greatest reduction in COF and torque, namely 0.0078% and 0.0578%, correspondingly. Therefore, it is recommended to use Al_2O_3/PAG ND12 nanolubricant at a volume concentration of 0.03% because it is the most optimal in terms of stability and has the highest COF and frictional torque reduction.

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1.0 INTRODUCTION

Lubricants play a crucial role as working fluids in providing appropriate lubrication for compressors, thereby mitigating excessive friction in the bearings and facilitating temperature reduction of refrigerants throughout the compression process. Pure lubricants are used mainly in automotive compressors to reduce wear on shear parts and maintain stability during operation [1]. The compressors are in sliding contact, causing surface wear at the shear limit during their operation [2]. In addition, the lubrication system also contributes to increased energy savings and the service life of the compressors by slowing down the friction between two friction surfaces to avoid rust and corrosion [3]. Therefore, improper lubricant selection can cause an increase in wear rate and decrease the coefficient of performance (COP). Conversely, an increase in COP and a low coefficient of friction (COF) are signs of a good lubrication system [4-6]. Compressor efficiency can be enhanced by utilizing appropriate lubricants, optimizing performance and minimizing power consumption [7]. The efficiency of compressors can be enhanced through the addition of nanoparticles into the refrigeration system. The performance of the lubrication system is improved by dispersing nanoparticles into the lubricant, leading to better friction reduction and anti-wear properties. This method effectively enhances compressor performance, where nanolubricant is a lubricant with nanoparticles as an addictive substance, which performs better than pure lubricant, as proven by previous research reviews [8-12]. Nevertheless, the advancement and utilisation of nanolubricants could face obstacles due to various aspects, including the compatibility between nanoparticles and base lubricants, as well as the long-term stability of nanoparticle dispersion [13].

Nanolubricants possess significant potential for enhancing automobile cooling capacities and COP performance by reducing the friction associated with compressor operation. Redhwan, et al. [14] revealed the COP, when using Al_2O_3 in polyalkylene glycol (PAG) lubricant, had a significant enhancement of 31% when subjected to a volume concentration of 0.010%. Furthermore, Yilmaz [15] has studied using nanoparticles dispersed as a base material for polyester (POE) lubricants to increase COP in compressor air conditioning systems. They found that Cu/Ag and CuO alloy nanolubricants showed an increase in COP of 20.88% and 14.55%, respectively. Besides increasing COP, nanoparticles have also been proven to reduce COF values and wear rates [16]. Therefore, COF is an essential component in tribological testing in identifying the performance of lubricants with nanoparticles as additives. In a study conducted by Ali, et al. [17] it was observed that the incorporation of Al_2O_3 and TiO_2 nanoparticles at a weight concentration of 0.25% resulted in a significant reduction of the COF by around 40 to 50% and the wear rate by approximately 20 to 30%. Using nanoparticles

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as an additive creates new challenges because the newly formulated nanolubricants have different properties. Due to these issues, a further investigation needs to be carried out to study and compare tribological behavior on friction between the contact of two steel surfaces.

Based on the literature review, nanoparticles are an additive in increasing energy efficiency [18]. At present, efficiency improvements using nanolubricants have been tested by researchers [19]. Nanoparticles dispersed in mineral oil reduce wear and increase load-carrying capacity [20]. Furthermore, Aminullah, et al. [6] have reported that experimental tribology in automotive air conditioning (AAC) systems affects compressor performance, where Al_2O_3 nanoparticles are dispersed into PAG lubricant base. The tribological properties of the Al_2O_3/PAG nanolubricants were improved, and the power savings due to friction losses reached 7.59%. In addition, nanolubricants research continued to be experimentally developed, and a hybrid nanolubricant was introduced. The Al_2O_3-SiO_2/PAG hybrid nanolubricants were evaluated in the AAC system from a tribological perspective, and the results showed that the effect of the nanolubricants resulted in a reduction in COF and a decrease in the wear rate of the nanolubricants up to 4.49% and 12.99%, respectively [21]. Several studies confirm that using nanoparticles in lubricants improves performance [16, 17, 22], such as forming a protective film and the friction effect between surfaces [3]. Thus, this behavior helps extend the mechanical equipment's service life and improve the compressor's performance in the refrigeration system.

However, while there is existing literature on the tribological performance of nanolubricants, no research has been conducted on nanolubricants with PAG ND12 specifically for the AAC system paired with R1234yf. This study mainly evaluated the COF and friction torque properties of the Al_2O_3 nanoparticles dispersed in the PAG ND12. The PAG ND12 is a lubricant for automotive air conditioning pairs with R1234yf refrigerant. As an initial stage, Al_2O_3 nanoparticles were dispersed into PAG ND12 using the two-step method. This method has become familiar to researchers [23-25]. In addition, by applying this method, the test results on nanolubricants samples recorded better stabilisation. Stability evaluation in this study used a UV-Vis spectrophotometer to ensure the stability of the nanolubricants [26, 27]. A four-ball Koehler tribometer is used to measure tribology at a load of 40.0 kg and a speed of 1200 rpm. The COF and friction torque were studied at 0.01%, 0.03% and 0.05% volume concentration. Furthermore, the tribological behavior of steel/steel surfaces lubricated with Al_2O_3/PAG ND12 at different concentrations was compared with pure lubricants.

2.0 METHODOLOGY

2.1 Materials

Nanoparticles in the form of dry powder with metal oxide, namely Al_2O_3, are used to prepare Al_2O_3/PAG ND12 nanolubricants. The Al_2O_3 is spherical, with an average size of 13 nm and a purity level of 99.8%, which was procured from Sigma, St. Louis, MO, USA. The properties of Al_2O_3 nanoparticles are shown in Table 1, and the lubricating properties of PAG ND12 at atmospheric pressure are provided in Table 2. The basic lubricant utilized in this experiment consisted of PAG ND12 lubricants obtained from DENSO. The characterizations of Al_2O_3 nanoparticles are acquired through the field emission scanning electron microscopy (FESEM) technique. The FESEM analysis is conducted with the JEOL JSM 7800F Prime instrument. The presence of Al_2O_3 nanoparticles in the base lubricants was confirmed through observation, wherein the shape, average diameter size, and dispersion of the nanoparticles were examined. The average diameter of Al_2O_3 nanoparticles is shown in Figure 1, as determined from the acquired FESEM images, which was 13 nm. These nanoparticles exhibited a spherical morphology.

Table 1. Properties Al_2O_3 nanoparticles [28, 29]

Properties	Al_2O_3
Molecular mass (g/mol)	101.96
Average particle diameter (nm)	13
Density (kg/m^3)	4000
Specific heat (J/ kg.K)	773
Thermal conductivity (W/m.K)	40

Table 2. Properties of PAG ND12 [30]

Properties	PAG ND12
ISO viscosity grade	46
Density @ 15 °C (kg/m^3)	988
Viscosity index	216
Dynamic viscosity (mPa·s) @ 40 °C	40.13
Dynamic viscosity (mPa·s) @ 100 ℃	8.25
Flash point (°C)	−45

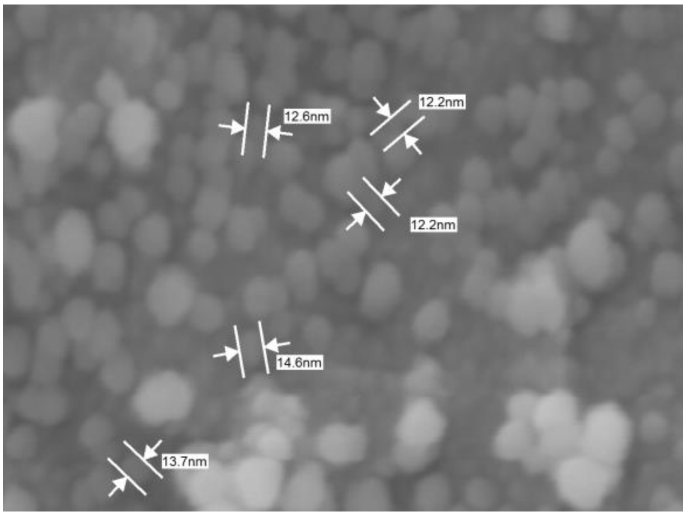

Figure 1. FESEM imaging for Al_2O_3/PAG ND12 nanolubricants

2.2 Nanolubricants preparation

Nanolubricants were made using a two-step method. The two-step method starts with the dispersion of nanoparticles into base lubricants, which is achieved through a magnetic stirrer. Subsequently, the solution undergoes treatment employing diverse homogenization techniques to mitigate the occurrence of suspension aggregation [31, 32]. The Al_2O_3 nanoparticles were dispersed in the PAG ND12 lubricant base at volume concentrations of 0.01%, 0.03%, and 0.05%. At the preparation stage, nanolubricants are made separately at each volume concentration. In the next step, the volume concentration of nanolubricants is calculated using Equation 1, where ϕ is the volume concentration, $m_{n\rho}$ is the mass of the nanoparticle, $\rho_{n\rho}$ is the density of the nanoparticle, m_{bl} is the mass of base lubricant, and ρ_{bl} is the density of base lubricant [29, 33-35].

$$\phi = \left(\frac{m_{np}}{\rho_{np}}\right) / \left(\frac{m_{np}}{\rho_{np}} + \frac{m_{bl}}{\rho_{bl}}\right) x\ 100 \tag{1}$$

In the next step, the nanolubricants were mixed using a magnetic stirrer for 30 minutes with volume concentrations of 0.01%, 0.03%, and 0.05%. Furthermore, a sonication process was done using a Fisherbrand ultrasonic bath vibrator (model: FB15051) to reduce the size of agglomerates and stabilize the nanolubricants. The application of ultrasonic frequency to the nanolubricants facilitates the acceleration of the dissolution process of the nanolubricant mixture by disrupting the intermolecular connections. This process will ensure the nanoparticles can spread homogeneously on the base fluid [36]. The sonification time of 120 minutes was determined using UV-Vis spectrometer analysis to ensure the colloidal stability of the nanolubricants dispersion was well mixed and had less sedimentation potential [14]. Shear surface damage may occur if this step is not carried out [3].

2.3 Nanolubricants stability

The assessment of dispersion and stability characteristics of nanolubricants holds significant importance prior to conducting tests on their tribological properties and performance. Therefore, the careful selection of an appropriate methodology for observing dispersion and stability is a crucial aspect in the process of nanolubricants preparation. Stability testing is a necessary procedure to ascertain the durability of nanolubricant samples over an extended period during the investigation. The stability of nanolubricants was first assessed by the utilization of sedimentation photography techniques. Subsequently, a series of quantitative tests were conducted to assess the stability analysis in greater detail. These experiments involved the utilization of UV-Vis Spectrophotometer measurement and zeta potential analysis [37]. The sedimentation images of the nanolubricants were recorded at regular intervals starting from the initial day of production and extending up to a duration of 6 months. The sedimentation photography technique is employed as an initial approach for observing the behavior of nanolubricant samples. This method is carried out by recording photographs for a particular length of time. The stability of the Al_2O_3/PAG ND12 nanolubricant was then verified by the utilization of UV-Vis and zeta potential analysis. The measurement of the absorbance value of the nanolubricants was conducted using the Genesys 10 UV-Vis Spectrophotometer. The colloidal stability of the nanolubricant dispersion was assessed by measuring the absorbance at the peak wavelength while altering the volume concentrations of the sample. The determination of the zeta potential value of the nanolubricant is conducted using the Anton-Paar Litesizer 500 instrument. Subsequently, the measurement of the absolute value of the zeta potential is compared with the stability classification.

2.4 Test rig description

In the early stages of the tribological measurement process, pure lubricants are used as initial data. The evaluation of the tribological characteristics of the Al_2O_3/PAG ND12 nanolubricants was carried out by employing a Koehler Four-ball Tribo Tester in compliance with the ASTM D4172-94 standard. The experiment was conducted to examine the performance of the lubricant in withstanding high-pressure conditions. Figure 2 shows the Koehler four-ball tribo tester used to evaluate the COF of the nanolubricants. The test equipment had a test ball specification with a diameter of 12.7 mm, Rockwell C hardness (HRC) 60±1, made of chromium grade G20 (ISO 3290) steel. The tribology measurement commenced by utilizing a pure PAG ND12 lubricant, subsequently followed by conducting additional testing using Al_2O_3/PAG ND12 nanolubricant. The testing of nanolubricants Al_2O_3/PAG ND12 with volume concentrations of 0.01%, 0.03%, and 0.05% was carried out in stages according to standard operating procedures ASTM D4172. The ASTM D4172 standard wear characteristics and test conditions are presented in Table 3.

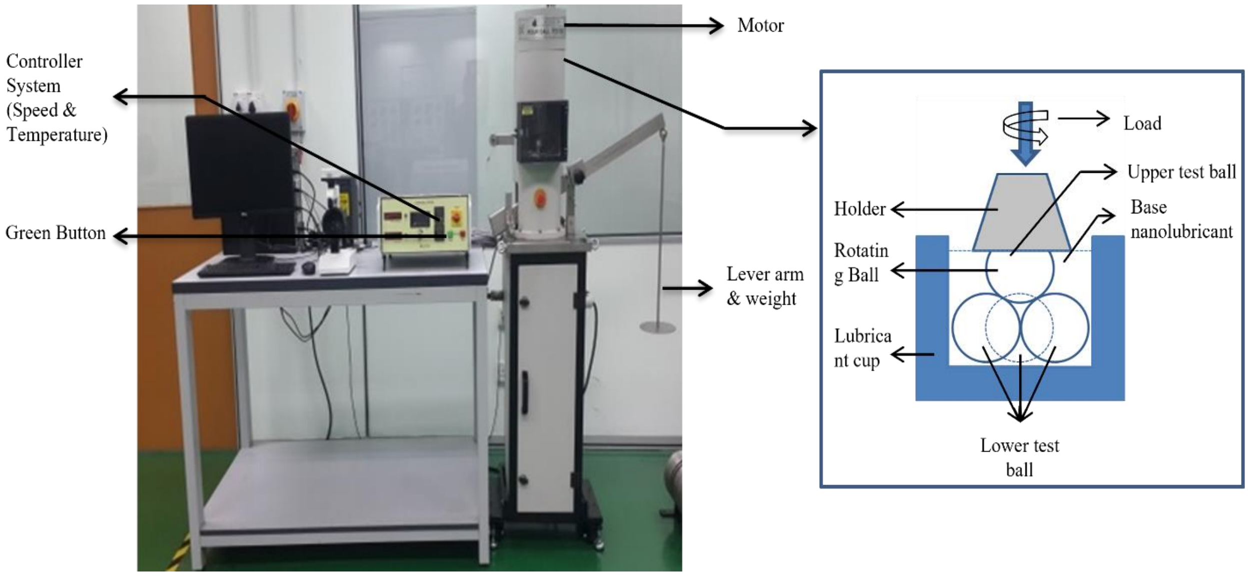

Figure 2. Tribology test using the Koehler four-ball tribo tester

Table 3. Wear characteristics and test conditions for the ASTM D4172 standard [38]

Test method	Test conditions			
	Speed (rpm)	Load (kg)	Duration (min)	Temperature (ºC)
Wear Characteristics	1200 ± 60	40.0 ± 0.2	60 ± 1	75 ± 2

The four metal balls, ball pot, ball locking ring, bearing plate, and collet were initially subjected to a cleaning process utilizing a hexane solvent, followed by air drying. The ball is inserted into the ball collet using an inserter/ejector mechanism located on the test rig base plate, and subsequently inserted into the spindle. Testing using the Koehler four-ball tribo tester was carried out with an operating temperature of 75 °C for 1 hour. The temperature is maintained with the help of an automatic temperature controller installed on the test equipment with the help of the control system module. The control system module's speed is set at 1200 rpm with a loading of 40.0 kg on the lever arm. The friction torque was observed on pure base lubricant and base nanolubricants. The friction torque and constant loading were calculated for all conditions, with COF as the test result. In this case, Equation 2 is used to determine the COF value, where μ is the coefficient of friction, τ (kg·cm) is the frictional torque, and F_N (kg) is the normal load;

$$\mu = \frac{2.23004\,\tau}{F_N} \tag{2}$$

Measurements were made with an optical microscope at an accuracy of 0.01 mm. The wear scar diameters (WSD) were measured three times on average to ensure the results were obtained under constant conditions. In addition, after each test, hexane solvent is always used to clean the testing equipment to prevent contamination. This cleaning aims to get each test relatively close to the perfect measurement value.

3.0 RESULTS AND DISCUSSION

The stability of a system is significantly influenced by the phenomenon of nanoparticle agglomeration, leading to a notable disparity between theoretical predictions and experimental observations [39]. The dispersion stability of the nanolubricants is assessed across a range of volume concentrations. The samples were visually monitored for a maximum duration of 6 months following their preparation. The samples were stored in test tubes and left undisturbed for the whole duration of the observation period. The samples were frequently examined for any observable alterations. Figure 3 displays the samples of Al_2O_3/PAG ND12 nanolubricants at various volume concentrations. All specimens of the nanolubricants Al_2O_3/PAG ND12 exhibited stability, with no notable instances of sedimentation observed. It is also important to acknowledge that all samples were made without the inclusion of any surfactant.

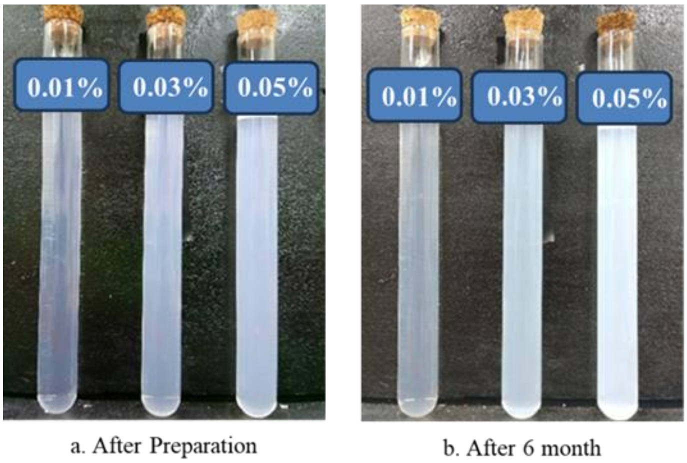

Figure 3. The visual photograph evaluation of Al_2O_3/PAG ND12 nanolubricants

Figure 4 shows the comparative evaluation of sedimentation Al_2O_3/PAG ND12 nanolubricants for different concentrations against sedimentation time for three-volume concentrations of 0.01%, 0.03%, and 0.05% up to 210 days. UV-Vis spectrophotometer was used to obtain the results shown in Figure 6. The test results showed that the Al_2O_3/PAG ND12 nanolubricants decreased gradually until the 210th day of investigation. The stability study of nanolubricants yielded average absorbance values of 0.752, 0.755, and 0.684, respectively. The highest concentration ratio is known to be found in the most stable nanolubricants [35]. The observed trend indicates a decrease in the absorbance ratio as sedimentation time increases, with a notable correlation to the sonication duration. The evaluation results indicate that all volume concentrations exhibit stability and fall within a range of 40% absorbance percentages over the specified time. Notably, the volume concentration of 0.03% demonstrated the highest average stability of above 53% absorbance percentages compared to the other volume concentrations. This trend occurred because the volume concentration of 0.03% shows relatively stable sedimentation, low agglomeration factor and Al_2O_3 nanoparticles as an additive, which can be absorbed well by PAG ND12 as the sedimentation time increases. This phenomenon is similar to the research of Sharif, et al. [26] and Zheng, et al. [40]. Accordingly, it can be concluded that the Al_2O_3/PAG ND12 nanolubricants for all concentrations throughout this investigation remained stable during 210 days of sedimentation time.

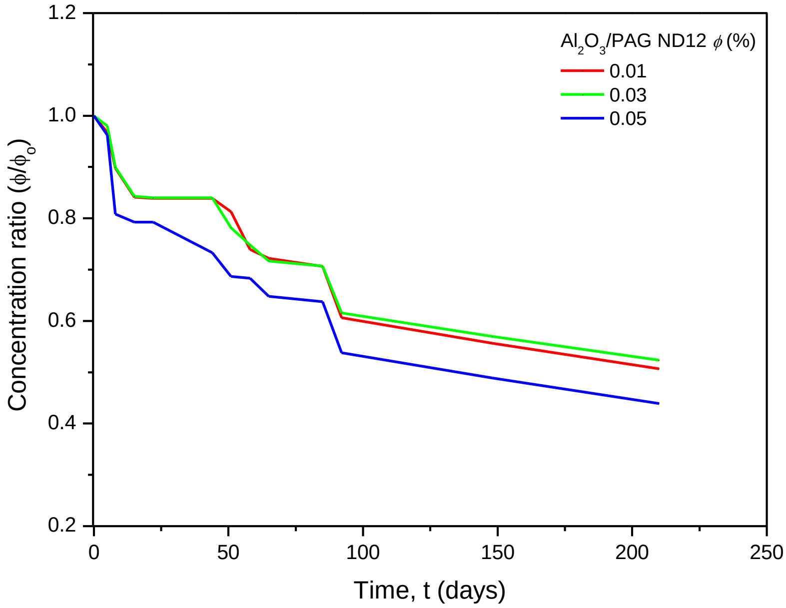

Figure 4. The concentration ratio of nanolubricants with sedimentation time

Figure 5 shows the zeta potential measurement of Al_2O_3/PAG ND12 nanolubricants with a volume concentration of 0.03%. The evaluation results show that the Al_2O_3/PAG ND12 sample has high stability, where the resulting value is 80.6 mV. It is widely accepted that suspensions exhibiting a zeta potential beyond 30 mV are considered to possess stability. Moreover, suspensions characterized by a zeta potential ranging from 40 to 60 mV are deemed to exhibit favorable stability. Lastly, suspensions with a zeta potential surpassing 60 mV are acknowledged to possess exceptional stability. The zeta potential of Al_2O_3/PAG ND12 nanolubricants was found to exceed the excellent stable criterion during the observation. The degree of particle dispersion into the base lubricant is positively correlated with the absolute value, indicating that higher absolute values correspond to improved dispersion and thus enhanced stability [41]. The stability of the nanolubricants is contingent upon the nanoparticles maintaining a state of dispersion without aggregation, facilitating precise zeta potential assessments and, ultimately, enhancing the efficacy of lubrication. Hence, it was determined using visual photograph evaluation, UV-Vis spectrophotometer observation, and zeta potential analysis that Al_2O_3/PAG ND12 nanolubricants exhibited stability across all concentrations.

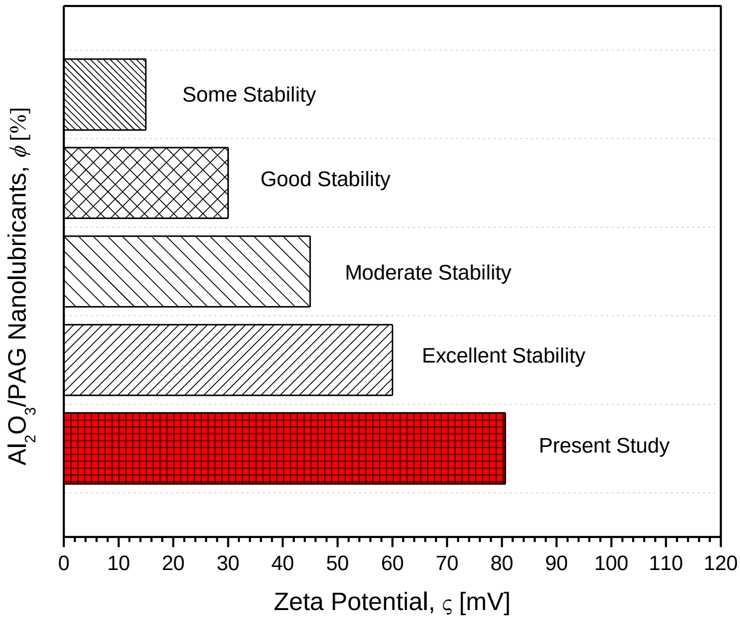

Figure 5. The zeta potential evaluation of Al_2O_3/PAG nanolubricants

Figure 6 shows the results of friction torque testing on pure lubricants and nanolubricants Al_2O_3/PAG ND12 with volume concentrations of 0.01%, 0.03%, and 0.05%. Each sample was tested according to standard operating procedures ASTM D4172 and tested for 1800 seconds. Investigation of pure lubricants resulted in the highest frictional torque of 1.886 and the lowest of 1.537. Furthermore, testing of the Al_2O_3/PAG ND12 nanolubricants with volume concentrations up to 0.05% resulted in the highest frictional torque of 2.158, 1.778, and 2.378, respectively, while the lowest friction torque was 1.601, 1.427, and 1.699, respectively. The frictional torque and frictional coefficient are decreased due to the thin film of lubricant that forms between the balls [42]. Compared to other concentrations, 0.03% Al_2O_3/PAG ND12 nanolubricants are the most effective and have the lowest frictional torque. Therefore, using nanolubricants Al_2O_3/PAG ND12 with a volume concentration of 0.03% is recommended.

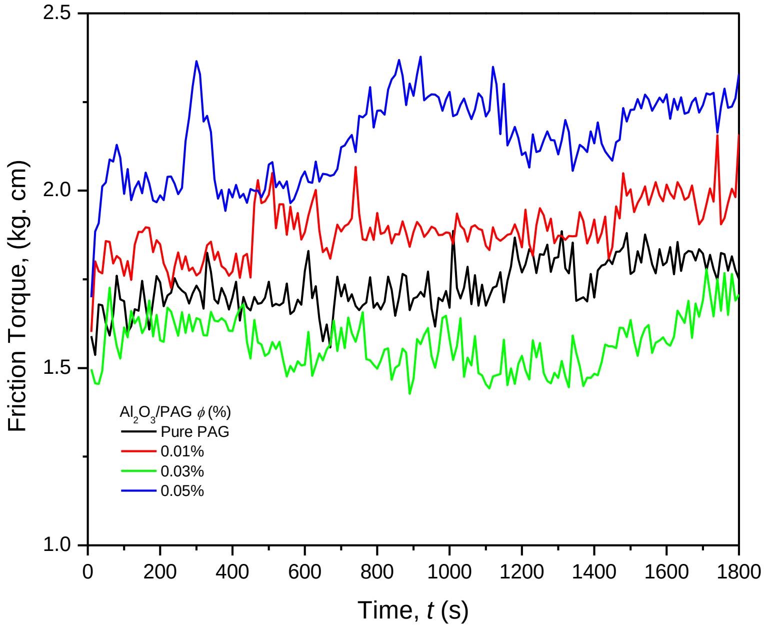

Figure 6. Friction torque evaluation of nanolubricants with time

Figure 7 shows the results of the COF nanolubricants Al_2O_3 with volume concentrations of 0.01%, 0.03%, and 0.05% concerning time. The COF value in the Al_2O_3/PAG ND12 nanolubricants with a volume concentration of 0.01% resulted in an average COF value of 0.104. At 0.03% and 0.5% concentrations, the average COF values were 0.078 and 0.117, respectively. Pure lubricants are superior to volume concentrations of 0.01% and 0.05%, while the COF value of the

Al_2O_3/PAG ND12 nanolubricants with a volume concentration of 0.03% is superior to pure lubricants. The optimum tribological performance of Al_2O_3 nanolubricants at 0.03% volume concentration is the primary reason for COF's trend and behavior with nanolubricants. The average COF values of pure lubricants and nanolubricants with volume concentrations of 0.01%, 0.03%, and 0.05% were compared, and the results showed that the 0.03% volume concentration had the highest COF value. This result is brought on by developing protective films and the rolling effects of friction surfaces [3, 17]. Therefore, it can be concluded that the Al_2O_3/PAG ND12 nanolubricants at an optimal volume concentration of 0.03% are recommended because it has the highest optimal COF compared to pure lubricants.

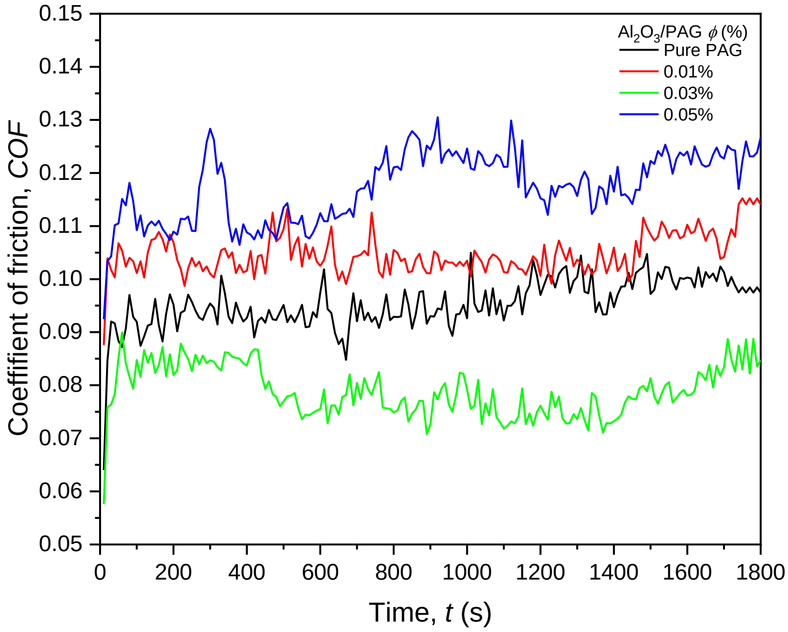

Figure 7. COF evaluation of nanolubricants with time

4.0 CONCLUSIONS

The tribological behavior of steel/steel surfaces lubricated with Al_2O_3/PAG ND12 was investigated with a load of 40.0 kg and a speed of 1200 rpm. The Al_2O_3/PAG ND12 nanolubricants stabilization was evaluated using visual photograph evaluation, UV-Vis spectrophotometer observation and zeta potential analysis at different concentrations, namely 0.01%, 0.03%, and 0.05%. The Al_2O_3/PAG ND12 nanolubricants demonstrated stability at all concentrations, as seen by the absence of sedimentation and a zeta potential value of 80.6 mV, indicating a significant degree of stability. The volume concentration of 0.03% Al_2O_3/PAG ND12 nanolubricants demonstrated superior stability, resulting in the most stable nanolubricants with an absorbance value of 0.75 and absorbance percentages exceeding 52% throughout the sedimentation period. Additionally, the 0.03% volume concentration of Al_2O_3/PAG ND12 nanolubricants experienced the highest average reduction of friction torque of 0.057% and a COF reduction value of 0.078%. The current nanolubricants were designed to be tested in polyalkylene glycol-lubricated R1234yf-coupled automotive air conditioning (AAC-R1234yf) systems. Therefore, employing Al_2O_3/PAG ND12 nanolubricants with volume concentrations of 0.03% is suggested for application in AAC-R1234yf systems as it contributes to higher COF and friction torque reductions, which are crucial in tribological performance. Additional investigation into the tribological performance of mono-metal-oxide nanolubricants is required to build upon the current studies.

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