

Research Paper

Tribology Properties on 5W-30 Synthetic Oil with Surfactant and Nanomaterial Oxide Addition

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Abstract

This study analyzes the tribological properties of 5W-30 synthetic oil with the addition of surfactants and oxide nanomaterials. This research used SAE 5W-30 lubricant base material with the addition of Aluminum Oxide (Al_2O_3), Titanium Dioxide (TiO_2), and Hybrid Aluminum Oxide (Al_2O_3) - Titanium Dioxide (TiO_2) nanomaterials. The nano lubricants were synthesized using a two-step method by adding nanomaterials by 0.05% volume fraction, followed by 50 ml of 5W-30 synthetic oil and polyvinylpyrrolidone (PVP) surfactant by 0.1%. Then, it was stirred using a magnetic stirrer for 20 minutes, followed by an ultrasonic homogenizer process for 30 minutes. Further, the nanolubricant was tested to identify its thermophysical properties, including density, dynamic viscosity, and sedimentation. It also underwent tribological testing, including wear, coefficient of friction, and surface roughness. Further, the nanomaterial was characterized using SEM, XRD, and FTIR. The morphological analysis using SEM suggested an irregular shape of the Al_2O_3 nanomaterial surface, while TiO_2 has a spherical shape. Besides, phase identification with XRD testing showed corundum and anatase phases. Functional group analysis through the FTIR showed the presence of Ti-O and Al-O. The highest density and viscosity results without surfactants were obtained in hybrid nanolubricant 779 kg/mm^3 and 0.0579 Pa.s , while the use of surfactants resulted in 788.89 kg/mm^3 of density and 0.0695 Pa.s viscosity. Tribological gray cast iron FC25 results in the best COF value observed in SAE 5W-30 + PVP- TiO_2 lubrication (0.093). The lowest wear mass without surfactant was obtained in the Al_2O_3 - TiO_2 nanolubricant hybrid (0.02 grams), the lowest surface roughness in a mixture of PVP and TiO_2 surfactants was $0.743 \mu\text{m}$. Meanwhile, the surface morphology of gray cast iron FC25 with hybrid nanolubricant SAE 5W-30 (Al_2O_3 - TiO_2) and Nanolubricant SAE 5W-30+ (PVP- TiO_2) produced the smoothest surface.

Keywords: Oil 5W-30; Al_2O_3 ; TiO_2 ; Surfactan; Nanolubricant; Tribology

1. Introduction

Population growth in Indonesia which is increasing every year has an impact on the need for adequate transportation to support human activities. To meet human needs related to transportation, the automotive industry has also increased production by 10% annually since 2018 [1]. Along with the increasing population of

vehicles, it must be directly proportional to the production of spare parts and lubricants that can support the maintenance of automotive vehicles, more specifically motorcycles. General lubricants are a necessity in the automotive world because they play an important role in reducing circulation between components to support engine performance in maximum condition [2].



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Lubricants in motorized vehicles also function as vibration dampers, coolers, and transport of dirt found in internal combustion engines [3]. Selection of the quality of the lubricant according to the engine specifications can extend the service life of the vehicle and can improve the work performance and durability of the motorcycle engine. Using the wrong lubricant will cause a decrease in engine performance and component wear and tear which can lead to severe damage [4]. To reduce losses that occur in motorcycle engine components due to wear and tear, this can be done by considering the selection of the type of lubricant according to the required specifications.

Lubricants have a viscosity at high temperatures or low temperatures when the engine is operated, so lubricants have their own grade (degree) regulated by the Society of Automotive Engineers (SAE). The SAE 5W-30 number on the lubricant packaging means a viscosity of 5 at cold temperatures (winter), and a viscosity of 30 at 100 degrees Celsius [5]. Nanoparticles have been suggested as a lubricant additive to improve thermophysical performance. The addition of nanomaterials to base fluids changes their thermophysical features such as density, viscosity and thermal conductivity. The dimensions and shape of the nanomaterial, the concentration of the nanomaterial, the viscosity of the base fluid, and the surfactants used for the formulation are the main parameters that affect the performance of the lubricant [6].

The addition of oxide nanomaterials, such as Aluminum Oxide (Al_2O_3) and Titanium dioxide (TiO_2), on basic lubricant reduces friction and wear since Al_2O_3 has excellent heat transfer nature, while TiO_2 can quickly spread, resulting in more excellent surface quality [7]. Thus, the addition of polyvinylpyrrolidone (PVP) surfactant inhibits agglomeration on nanolubricant, enhances the efficiency, as well as decreases friction and wear on gray cast iron FC25 material [8].

Based on the description above, it is hoped that this research can be a solution in overcoming the problem of friction loss and wear caused by friction between the cylinder block and piston ring in vehicle engines. Apart from that, it can be a way to save fuel and extend engine life by paying attention to the type of lubrication used. This research focuses on studying tribological

properties by synthesizing nanolubricants added with Al_2O_3 and TiO_2 nanomaterials and 5W-30 synthetic oil as well as adding polyvinylpyrrolidone (PVP) as a surfactant additive to gray cast iron FC25 specimens.

2. Method

2.1. Additive Materials

In this study, Aluminum Oxide from SIGMA ALDRICH dengan with 99.5% was used, with white color and powder shape at an average size $\leq 10 \mu\text{m}$. Meanwhile, Titanium Dioxide nanoparticle is formed from titanium oxide. Titanium oxide (TiO_2) is a widely accessible dielectric nanomaterial with high thermal conductivity properties. Besides, Titanium Dioxide (TiO_2) can precipitate and improve the properties of base oil, as well as carrying sufficiently low concentration for conquering friction [9]. In this study, Titanium Dioxide brand SIGMA ALDRICH with 99.7% purity, white color, and powder form (average particle size of $\leq 25 \text{ nm}$) was used.

Surfactant is a critical component in the preparation of nanolubricant. The addition of surfactant enhanced the nanoparticle's dispersion stability [10]. It had hydrophobic tails and hydrophilic heads. The polyvinylpyrrolidone (PVP) was used as the surfactant in this study. It is a commercial, industrial polymer with a low cost that has been commonly utilized in numerous fields, such as lithium batteries, water electrolysis, anti-bacterial agents, food additives, and surfactants [11]. Polyvinylpyrrolidone (PVP) is also referred to as a nonionic polymer containing robust hydrophilic molecules and can adhere easily to materials in solution [12]. PVP has also been reported to lower the repulsive force between hydrophobic chains in materials containing two atoms in one molecule [13]. The structure of polyvinylpyrrolidone (PVP) is presented in Figure 1.

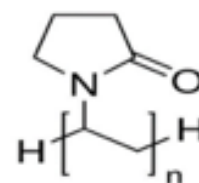


Figure 1. Structure of polyvinylpyrrolidone (PVP)

2.2. Additive Materials Characterization

Material characterization was carried out to identify the properties of the material. This research used three types of characterization, including SEM (Scanning Electron Microscopy), to identify the surface morphology of nanoparticles [14], XRD (Xray-Diffraction), and FTIR (Fourier Transform Infrared). The XRD was used to determine the crystalline phase structure of nanoparticles by investigating the the material [15]. Lastly, the FTIR (Fourier Transform Infrared) was performed to find the functional group using infrared [16].

2.3. Nanolubricant Preparation

SAE 5W-30 nanolubricant was used as the base oil. In this study, seven samples with 0.05% fraction volume were prepared, consisting samples 5W-30 base oil, Al₂O₃ nanolubricant + SAE 5W-30, TiO₂ nanolubricant + SAE 5W-30, hybrid nanolubricant Al₂O₃ - TiO₂+SAE 5W-30, as well as the samples with the same volume fraction added with polyvinylpyrrolidone (PVP) surfactant. After the samples had been prepared, the lubricant and nanoparticle were stirred using a magnetic stirrer at 1250 rpm speed and room temperature for 20 minutes. Then, we conducted homogenization using an ultrasonic homogenizer for 30 minutes to produce a nanolubricant with excellent stability. The prepared nanolubricant with and without Polyvinylpyrrolidone (PVP) surfactant are illustrated in [Figure 2a](#) and [Figure 2b](#).

2.4. Nanolubricant Characterization

2.4.1. Analisis of Thermophysical Properties

Thermophysical properties determine the performance, pressure drop, heat transfer

coefficient, and thermal efficiency of a system [17]. The thermophysical properties parameters for nanolubricant include viscosity and density. Viscosity indicates the amount of force needed to move an object in a fluid. Besides, it can serve as an indicator of fluid viscosity, reflecting the level of friction within the fluid [18]. In this study, Viscometer type NDJ-8S was utilized, with rotor number 1 and different speeds of 6 rpm, 12 rpm, 30 rpm, and 60 rpm, as well as temperature ranging between 30 °C – 100 °C. Further, Eq. (1) was used to estimate the dynamic viscosity.

$$v = \frac{\mu}{\rho} \quad (1)$$

Where, v = Kinematic viscosity (m²/s); μ = Dynamic viscosity (kg/m.s); ρ =density (kg/m³)

Density represents the mass per unit volume that is symbolized by (ρ) with the international unit of kg/m³ and a British unit of slug/ft³ [19]. The density of fluid was calculated using Eq. (2).

$$\rho = \frac{m}{V} \quad (2)$$

Where, ρ = density (kg/m³); m = masss of fluid (kg); V = Fluid volume (m³).

2.4.2. Analysis of Tribology Properties

Tribology is the study of friction, wear, and lubrication [20]. In a more extensive range, tribology is defined as a reciprocal relationship between the surface of a solid and a moving object, as well as the reasons for its emergence. For the tribology analysis, we used the pin-on-disc tribometer. In specific, we used the pin on disc from Stainlees steel, named Ducom Instruments,

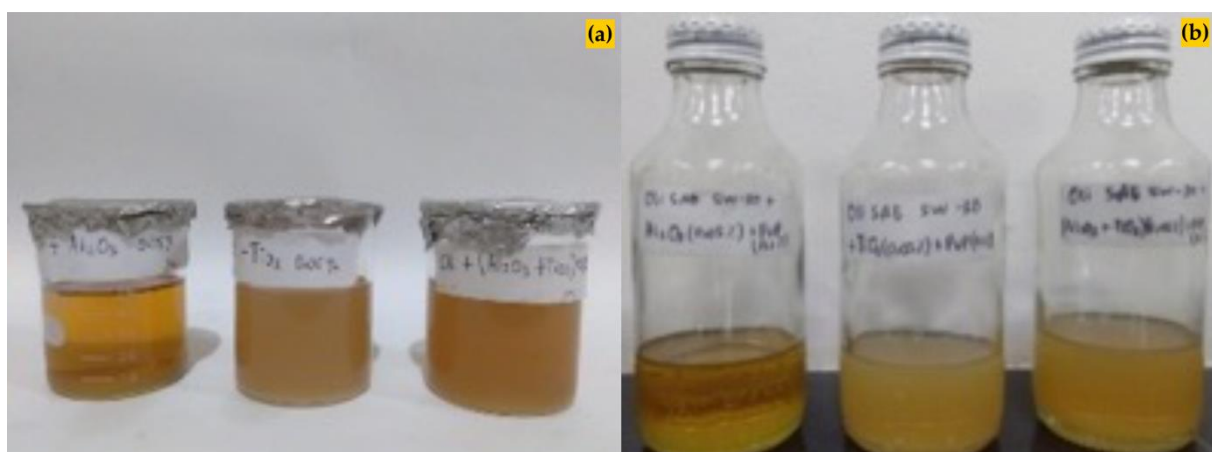


Figure 2. Prepared nanolubricant (a) without surfactant and (b) with polyvinylpyrrolidone (PVP) surfactant

with blue display, digital display type, 3-12 mm pin diameter and 165 x 8 mm disc diameter, and a load capacity of 5 - 200 N. Tribometer pin on the disc contains pins and discs that are commonly used to measure friction and wear. The schematic of the pin-on-disc test equipment is illustrated in [Figure 3a](#), while the rotary movement of the pin-on-disc is shown in [Figure 3b](#).

One of the applicable instruments for this analysis is the pin on the disc, where the disc rotates while the pin is motionless and pressed against the disc. This frictional loading will result in repeated contact between surfaces, which will eventually take some of the material on the surface of the pin on the disc [21]. Friction and wear have a close correlation with tribology testing as friction represents the interaction between two nudging surfaces with relatively constant movement, while wear is the gradual loss of material from two nudging surfaces due to contact [22]. The friction and wear were estimated using Eq. (3) and Eq. (4), respectively.

$$\mu = \frac{F}{P} \quad (3)$$

Where, μ = Friction coefficient; F : Measured frictional force; P : The normal load attached to the pin seat

$$\Delta V = \frac{m_0 - m_1}{\rho} \quad (4)$$

Where, ΔV = Wear volume of a material (m^3); m_0 = Mass of specimen before testing (kg); m_1 : Mass of specimen after testing (kg); ρ = Specimens density (kg/m^3)

2.4.3. Analysis of Surface Roughness

The roughness analysis was completed using Stylush Probe or Surfcom Touch instrument. The roughness test was conducted on the disc surface which directly touched the pin. This surface roughness test was conducted to measure and analyze the roughness of a material or surface [23]. For the Surfcom Touch, we used the ones with code no. 178-395, speed of 1 mm/s, cable length of 1 m and mass of 190 g. The surfcom touch instrument used in this study is illustrated in [Figure 4](#).

3. Result and Discussion

3.1. Scanning Electron Microscopy (SEM) Analysis on Aluminum Oxide (Al_2O_3) and Titanium Dioxide (TiO_2)

Scanning Electron Microscopy (SEM) on Aluminum Oxide (Al_2O_3) and Titanium Dioxide (TiO_2) nanomaterials was performed to identify their morphology [24], [25]. This test was carried out using SEM brand FEI type *Inspect-S50* purchased in Japan. The obtained SEM characterization on Aluminum Oxide and Titanium Dioxide nanoparticles is illustrated in [Figure 5a](#). and [Figure 5b](#).

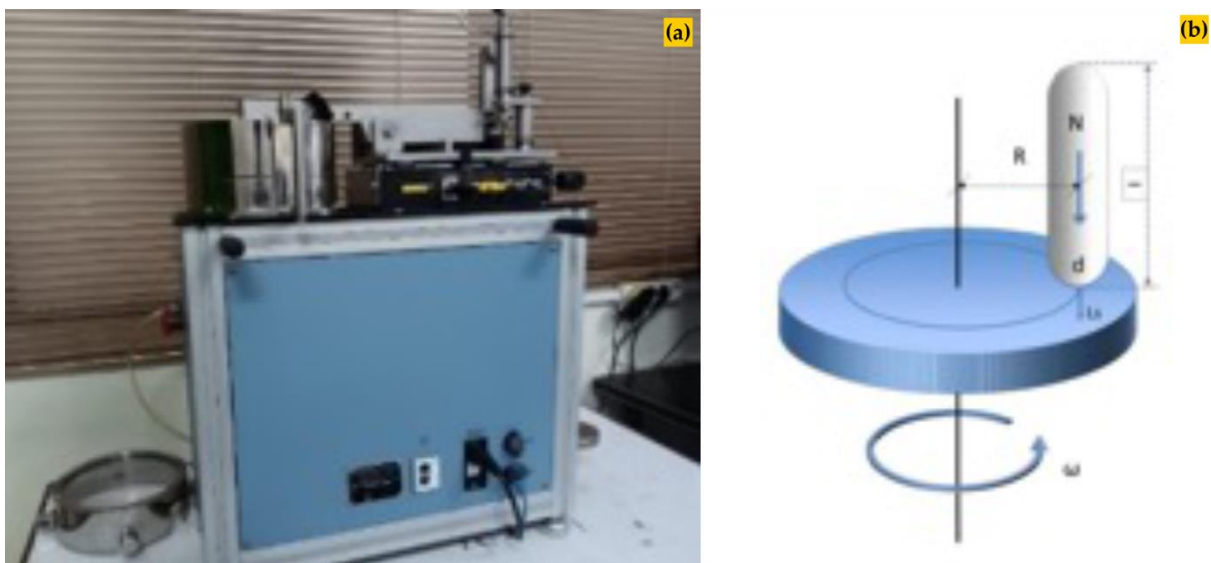


Figure 3. Tribology Test (a) Tribometer pin on disc (b) Rotating scheme of pin on disc tribometer (N= the force exerted on pin (N); R= distance between disc and pin (mm); D= ball/pin diameter (mm); W= rotation (rpm))



Figure 4. Surface roughness Instrument

The SEM analysis results on Aluminum Oxide and Titanium Dioxide at 1000x and 10000x magnification suggested the dominant presence of the Corundum phase (Al_2O_3) with trigonal crystal structure [26]. Meanwhile, the results of the microstructure test showed that the Aluminum Oxide nanomaterial has irregular grain shapes and nanoparticles that do not bind to each other. Further, the SEM analysis results on the titanium dioxide indicated its anatase phase with a tetragonal crystal structure, tendency to agglomerate, and spherical shape similar to a ball [27].

3.2. X-Ray Diffraction (XRD) Analysis on Aluminum Oxide and Titanium Dioxide

Section The X-Ray-Diffraction (XRD) test was carried out to identify the phase structure,

crystallinity, and grain shape of Aluminum Oxide and Titanium Dioxide nanomaterials [28]. The analysis was performed explicitly on the diffraction peaks on the XRD graph. For the instrument, we adopted the Pananalytical brand type Expert Pro equipped with SHP (Software High Score Plus) and PDF2. The results of the XRD test on Aluminum Oxide (Al_2O_3) and Titanium Dioxide (TiO_2) nanomaterials are presented in **Figure 6a** and **Figure 6b**.

As illustrated in **Figure 6a**, Al_2O_3 carries the highest peak intensity at 2θ angles of 25.47, 35.04, 37.67, 43.26, 52.45, 57.41, 66.42, 68.12, 76.80, which correspond to the miller index of (hkl) (012), (104), (110), (113), (204), (116), (214), (300), (119). The crystallite shape of *Aluminum Oxide* (Al_2O_3) was analyzed using *MATCH software*. The results showed a trigonal crystal shape, similar to the findings reported in a study [29] describing the trigonal crystal shape of (Al_2O_3). For calculating its crystallite size, the Scherrer formula (Eq. (2)) was conducted [30], [31].

$$D = \frac{K \cdot \lambda}{\beta \cos \theta} \quad (4)$$

Where, D = Crystal size (nm); K = Form factor of the crystal (0.9); λ = X-ray wavelength (0.15406); β = Value of FWHM (Full Width at Half Maximum) (rad); θ : Diffraction angle ($^\circ$)

The obtained crystallite size at 35.043 positions with an FWHM of 0.1224 was 68.0622 nm.

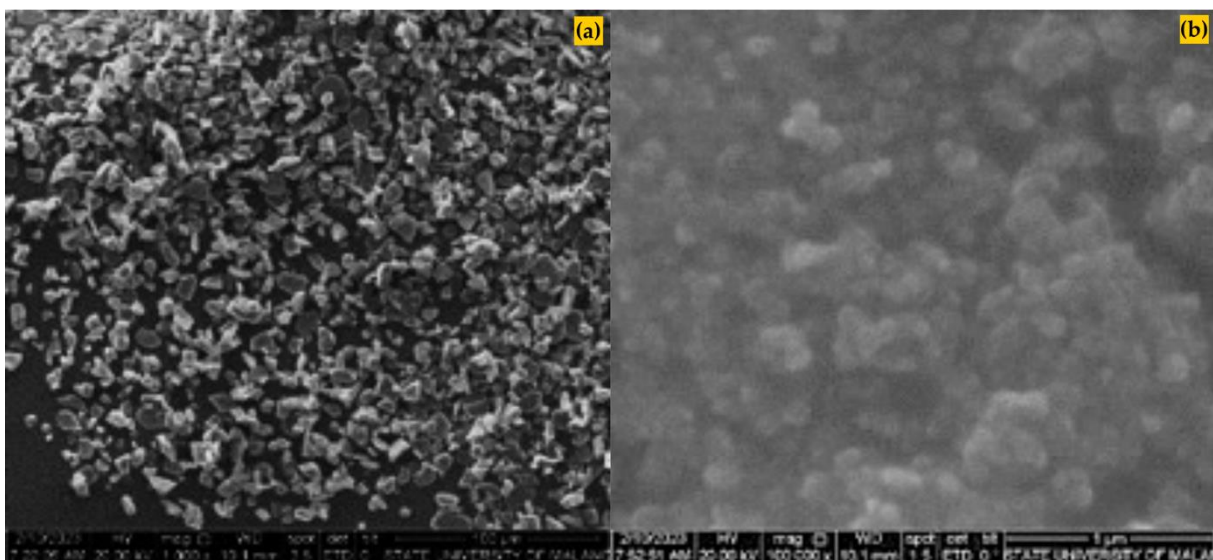


Figure 5. Results of SEM for (a) Aluminum oxide (Al_2O_3) at 1000x magnification and (b) Titanium dioxide (TiO) with 10000x magnification

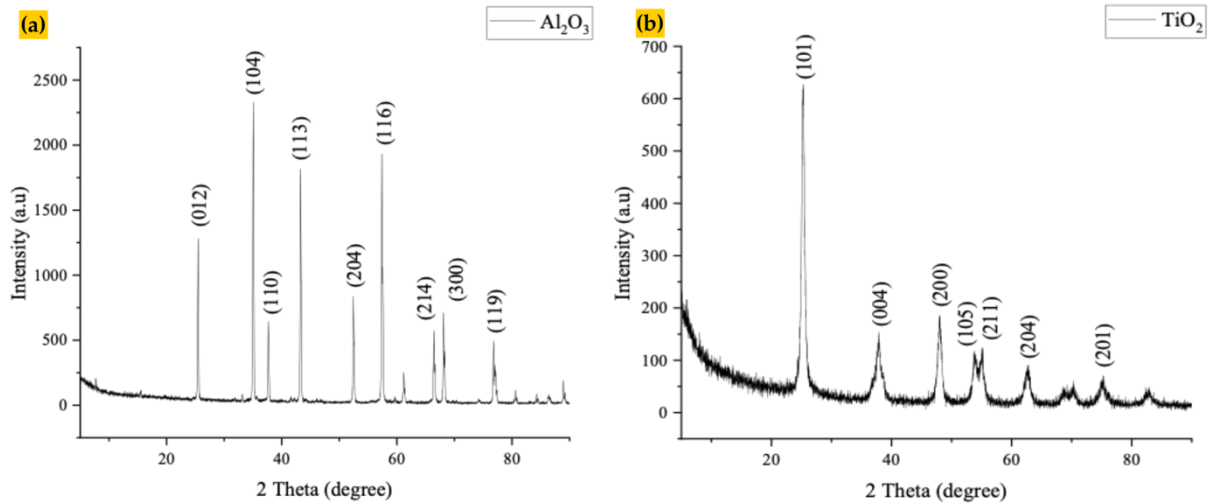


Figure 6. (a) XRD graph for Al₂O₃ nanomaterial; (b) XRD graph for TiO₂ nanomaterial

In addition, TiO₂ presents the highest peak intensity at 2theta angle: 25.35, 37.89, 47.95, 53.84, 55.12, 62.84, 75.13 with miller index value of (hkl) (101), (004), (200), (105), (211), (204), (201), as presented in **Figure 6b**. Besides, according to the Match software analysis results, TiO₂ has tetragonal crystal shape, linear to the results discovered in a study [32]. For its crystallite size, we performed calculations using the Scherrer equation, showing the 20.2806 nm crystallite size at 25.3464 position with FWHM of 0.4015.

3.3. Fourier Transform Infrared (FTIR) Analysis on Aluminum Oxide and Titanium Dioxide

The Fourier Transform Infrared (FTIR) test results indicate the content and functional groups of Al₂O₃ and TiO₂ nanomaterial. In this study, we used FTIR brand Shimadzu type IRprestige 21. The obtained data are in the form of graphics at around 4000-500 wavelength [33] **Figure 7a** and

Figure 7b. depicts the FTIR spectrum for Al₂O₃ and TiO₂ around 4000-500 cm⁻¹ wavelength.

Figure 7a shows that Al₂O₃ has O-H stretching bond at 3524 [34]. Besides, it also presents a C-H vibration bond [35], a symmetric bending Al-O-H vibration bond [36], [37] Al-O stretching bond [38] at 2309, 1058.99, and 400-800 peaks, respectively.

In addition, the Al₂O₃ has O – H stretching vibration at 3300 – 3500 [39], Ti-OH at 2300 and 1630 [40], along with bending Ti – OH and C – O stretching vibration at 1151-1106 and 1054-1041 [41], as well as Ti-O stretching bond and O-Ti-O at 895 and 500-700 peak, respectively [42].

3.4. Sedimentation

The nanolubricant stability test was carried out using the sedimentation method. The sedimentation method can only be observed visually by measuring the height of the mixture in 0, 4, 8, 12, 16, and 20 days [43]. The stability can be

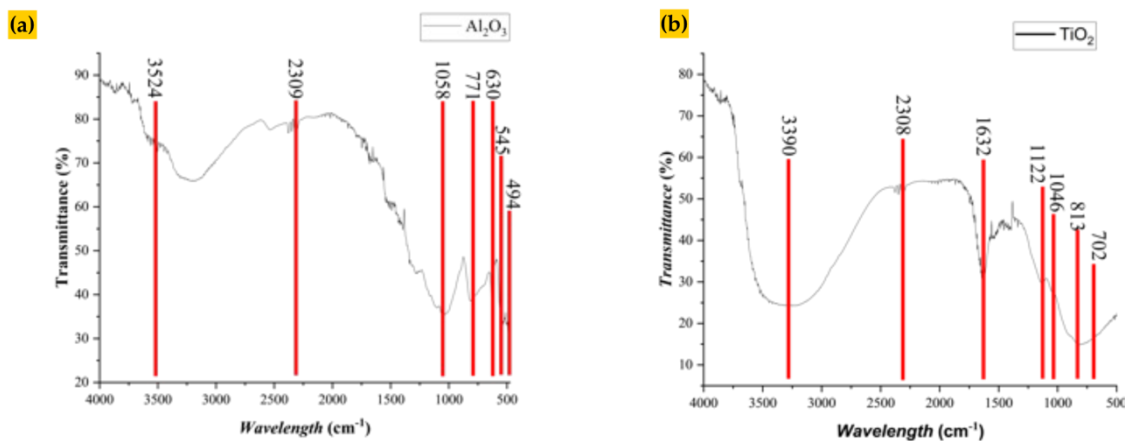


Figure 7. (a) FTIR spectrum for Al₂O₃ nanomaterial; (b) FTIR spectrum for TiO₂ nanomaterial

easily evaluated by taking a photo of the sedimentation of the sample in a beaker or clear bottle. Analysis using the sedimentation method is usually carried out immediately after the nanoparticle dispersion in the lubricant. Consequently, sedimentation time protects against wear and reduces friction [44].

3.5. Density

The density test involved measuring the weight of the fluid and then dividing it by its volume. The density test results for the nanolubricant added with and without PVP surfactant are shown in Figure 8.

Based on Figure 8, the highest density value for the 5W-30 hybrid nanolubricant is 779 kg/m^3 , while the lowest density was observed on the 5W-30 base lubricant. The density value is influenced by several parameters, such as temperature, as well as the addition of chemical composition and additives [45]. In Figure 8, the density of lubricant added with surfactant is represented by the green stick. The highest density observed on TiO_2 added with PVP surfactant is $778,889 \text{ kg/m}^3$. As the nanoparticle density has been reported to affect the density of the prepared nanolubricant [46], [47], initially, the density of TiO_2 and Al_2O_3 is 4,23 and 3,7 g/cm^3 , respectively. The addition of PVP surfactant in nanolubricant enhances its density, thus, the nanolubricant added with surfactant presents greater density [48].

3.6. Viscosity

The viscosity test was performed using the viscometer type NDJ-8S at $1 - 2000000 \text{ mPa}\cdot\text{s}$. The

obtained viscosity for the samples with and without PVP surfactant is presented in Figure 9, with the pink stick representing the results for samples with no surfactant addition, while the green stick shows the viscosity of samples with PVP surfactant. The results suggested that the basic lubricant with and without PVP surfactant presents $0.0442 \text{ Pa}\cdot\text{s}$ dynamic viscosity, while nanolubricant SAE 5W-30 + Al_2O_3 , SAE 5W-30 + TiO_2 , and hybrid nanolubricant + SAE 5W-30, have 0.0468, 0.0563, and $0.0579 \text{ Pa}\cdot\text{s}$ dynamic viscosity, respectively. This result is induced by the increase of internal shear stress at concentration with higher concentrations [49]. Further, the dynamic viscosity on the nanolubricant added with a surfactant is linear with their density since viscosity, as influenced by the nanoparticle's density. Meanwhile, the hybrid $\text{Al}_2\text{O}_3\text{-TiO}_2$ nanolubricant presents the features from both nanoparticles, thus, its dynamic viscosity lies between the two of them [50].

3.7. Tribology

3.7.1. Surface Wear of Gray Cast Iron FC25 with Nanolubricant Variations

Figure 10 shows average wear mass test results for gray cast iron FC 25 without and with surfactant. The bar in green represents the wear from no lubrication, with the highest wear of 0.082 grams. Meanwhile, the yellow bar shows the wear mass of SAE 5W-30 base oil which reaches 0.012 grams. The red bars show the wear mass from the nanolubricant added with PVP surfactant. The use of SAE 5W-30 + Al_2O_3 and SAE 5W-30 + TiO_2 nanolubricant results in 0.003 and 0.004 grams of

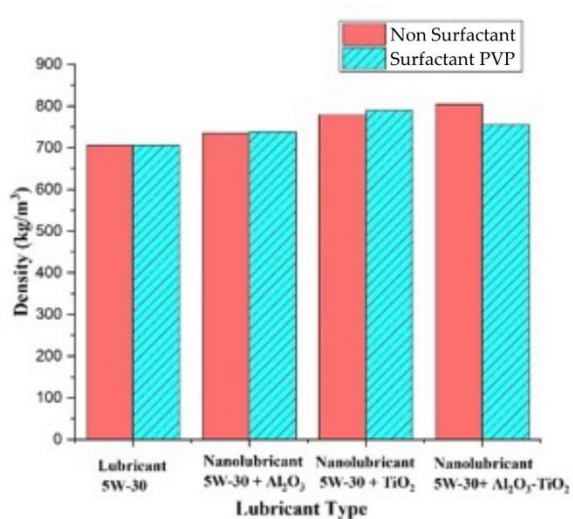


Figure 8. Density test results

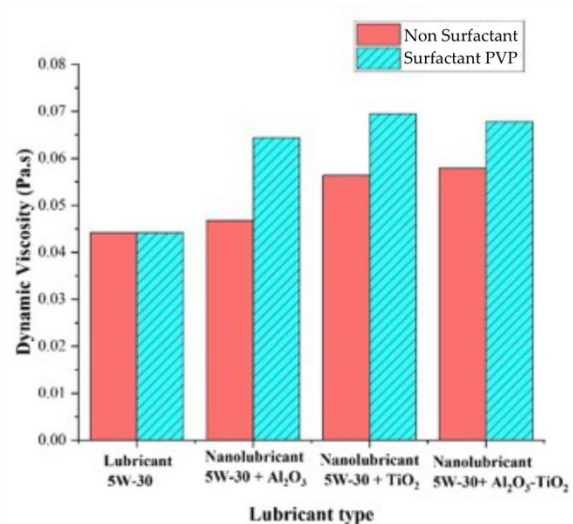


Figure 9. Viscosity of lubricant

wear mass. The lowest average wear for the samples with no surfactant was observed from the hybrid nanolubricant + SAE 5W-30 sample (0.002 gram). The lowest wear mass on the use of hybrid nanolubricant samples with no surfactant is caused by the addition of Al_2O_3 and TiO_2 , which significantly lowers the wear mass, in comparison to the use of base oil or lubricant added with single oxide nanomaterial [51]. Additionally, the hybrid nanoparticle Al_2O_3 and TiO_2 is a modified additive that significantly reduces the friction coefficient, resulting in excellent anti-wear effects than the pure Al_2O_3 and TiO_2 [52]. Lastly, the blue chart in Figure 10 shows the COF test results on samples added with surfactant. The addition of PVP surfactant on lubrication affects the wear of gray cast iron FC25, as illustrated in Figure 10. The lowest wear was observed on the samples added with TiO_2 due to the low friction coefficient of nanolubricant with PVP and TiO_2 . The friction coefficient is directly proportional to the wear mass of gray cast iron FC25 [53]. The excellent stability of nanolubricant presents a positive contribution to the moving element due to the more extended period of nanoparticle agglomeration, which inhibits massive abrasive wear [54].

3.7.2. Surface Friction Coefficient of Gray Cast Iron FC25 with Nanolubricant Variation

Figure 11 presents the COF test results on gray cast iron FC25 with and without surfactant. The

green bar represents the COF results with no lubrication of 0.727. This finding signifies that the higher COF is induced by the unlubricated gray cast iron surface, which results in wear and a greater amount of friction. The yellow bar in Figure 11 shows the COF results for the SAE 5W-30 base oil lubrication, which reaches 0.108. The base oil on the friction surface decreases the severity of abrasion caused by the nanoparticle, which decelerates the COF value. The red bar represents the 0.154 COF results for the SAE 5W-30 + Al_2O_3 nanolubricant with no PVP. Meanwhile, the use of hybrid nanolubricant SAE 5W-30 results in a 0.143 value which does not significantly differ from the results of SAE 5W-30 nanolubricant. Further, the lowest COF result was found on the SAE 5W - 30 + TiO_2 nanolubricant (0.093), similar to the result reported in a study. That study further explained that the lowest COF value mainly occurs due to the reduction of the frictional torque due to the presence of nanoparticles which effectively transforms pure friction into scrolling friction. Besides, the spherical shape of Titanium Dioxide (TiO_2) nanoparticles can serve as the ball bearings that roll into the contact area, enabling the change of sliding friction into combination friction (sliding and rolling). Additionally, the lubrication also correlates with a tribo-pair system, which maintains the nanoparticle's stiffness and shape by establishing low and stable load among the friction surface, which results in better and

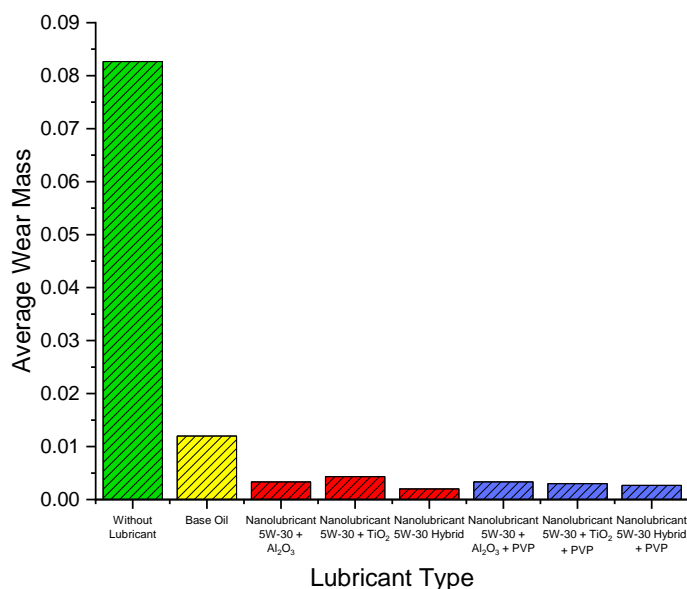


Figure 10. Wear test on gray cast iron FC25 with nanolubricant variation

smaller friction [55], [56]. The blue chart in Figure 11 shows the COF results on the samples with surfactant addition. The highest friction coefficient test results on samples with PVP surfactant were identified on the Al₂O₃ + SAE 5W-30 samples (0.174), while the lowest was in TiO₂ + SAE 5W-30 (0.093). [57] described that the smaller nanoparticle size reduces friction. Meanwhile, the sphere shape nanoparticle presents a rolling effect between the surface, which enhances the wear endurance [58].

3.7.3. Surface Roughness of Gray Cast Iron FC25 with Lubricant Variations

Figure 12 illustrates the surface roughness test on gray cast iron FC25 with and without

surfactant. The red bar represents the roughness in the Ra unit, while the blue bar shows the roughness in the Rq unit. The highest roughness of 1.67 μm Ra 2.12 μm Rq was found from the gray cast iron FC25 without lubrication, while for the gray cast iron FC25 with a base oil lubricant, we obtained 0.96 Ra and 1.17 μm Rq. Meanwhile, for the SAE 5W-30 + Al₂O₃ nanolubricant, .86 μm Ra and 096 μm Rq were obtained. The addition of Al₂O₃ into the basic lubricant enables the Al₂O₃ to bind the surface roughness and conductivity. Besides, the Al₂O₃ coating has been reported as an alternative for regulating surface roughness [59]. For the roughness test on gray cast iron, FC25 with lubrication using TiO₂ nanolubricant and PVP surfactant was identified as the lowest, with a

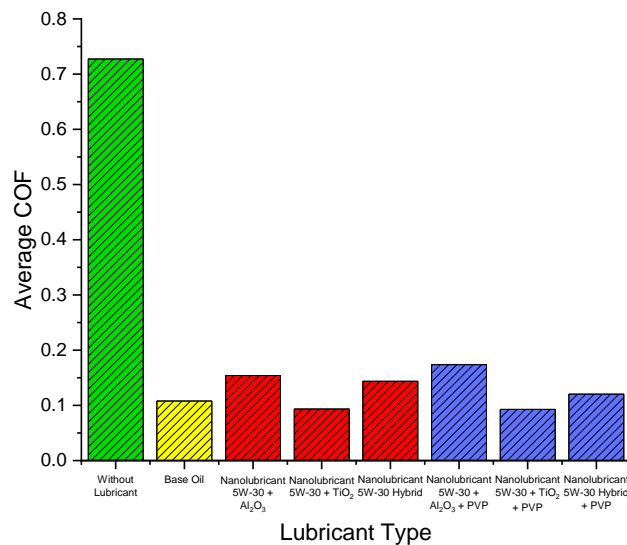


Figure 11. COF results on gray cast iron FC25 with nanolubricant variation

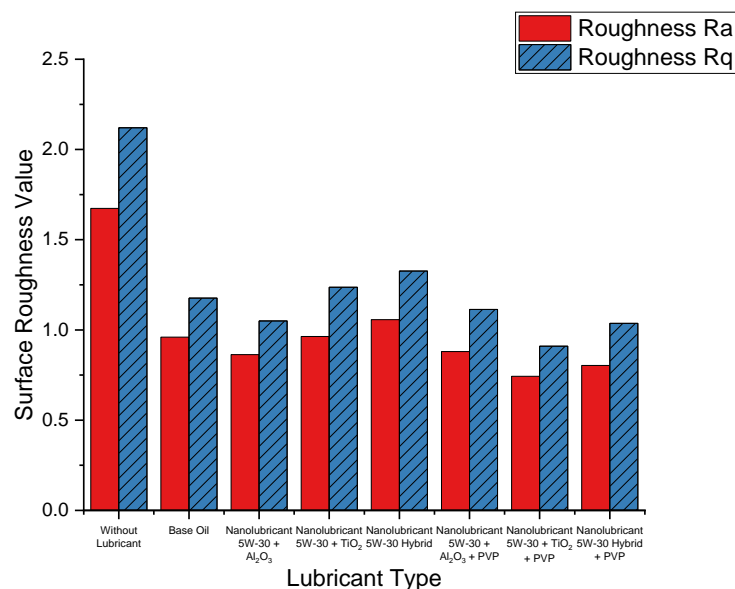


Figure 12. Surface roughness of gray cast iron FC25 with nanolubricant variation

roughness value of $0.743\ \mu\text{m}$. The results from Al_2O_3 nanolubricant + SAE 5W-30 and hybrid nanolubricant $\text{Al}_2\text{O}_3 - \text{TiO}_2$ + SAE 5W-30 were 0.880 and $0.803\ \mu\text{m}$, respectively. The surface roughness is affected by the nanoparticle's abrasive nature [57], contact voltage, adhesion, and surface friction, which results in wear on the wear track and increases the surface roughness [60].

3.7.4. Macroscopic Observations on Gray Cast Iron FC25

The macroscopic observation was performed to analyze and identify the defects or damages on an object. Besides, it also facilitates the identification of cracks and corrosion. Figure 13 shows the macro picture of the gray cast iron FC25.

Figure 13a–Figure 13h presents the result of macro photo observations on the surface of gray cast iron FC25 using $250\times$ magnification. The macro photo observations in this study were divided into two, namely the observations on samples without surfactant (Figure 13a–Figure 13e) and the ones with surfactant (Figure 13f–Figure 13h). As presented in Figure 13a, the gray cast iron with FC25 without lubricant has dominant scratches on its surface with plastic deformation caused by high contact and debris [61]. Figure 13b shows the macro photo test of gray cast iron FC25 using base oil as a lubricant. It shows a dark area on its wear track which indicates the presence of an adhesive layer from debris or an interface layer established during the friction process. A study [62] uncovered that the SAE 5W-30 lubricant generates smaller friction due to its more excellent

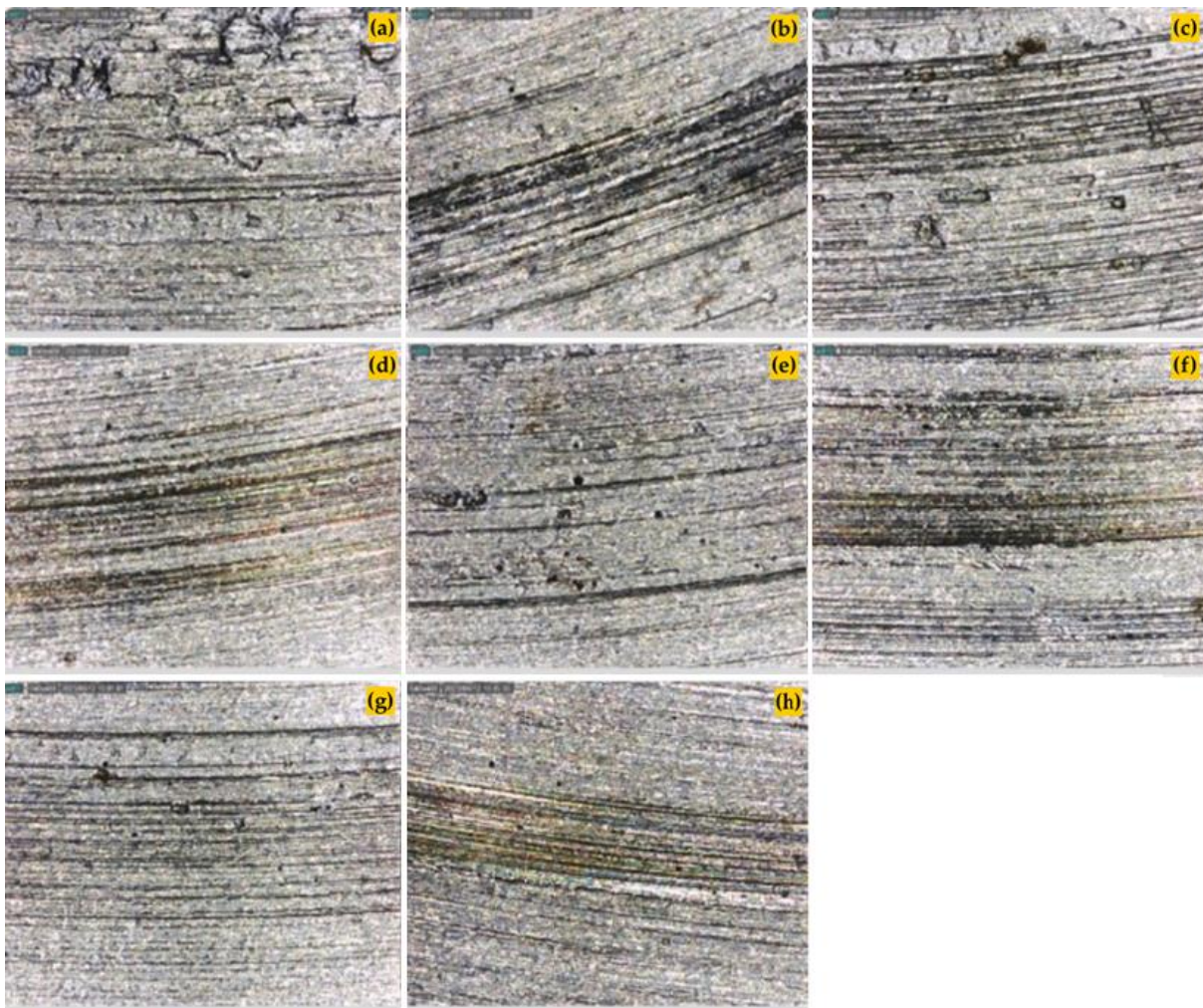


Figure 13. Macro Picture Observation (a) Without lubrication; (b) SAE 5W-30 base oil; (c) 5W-30 + (Al_2O_3) nanolubricant; (d) 5W-30 + (TiO_2) nanolubricant; (e) Hybrid 5W-30 + (Al_2O_3 - TiO_2) nanolubricant; (f) 5W-30 + (Al_2O_3 -PVP) nanolubricant; (g) 5W-30 + (TiO_2 -PVP) nanolubricant; (h) Hybrid 5W-30 + (Al_2O_3 - TiO_2 -PVP) nanolubricant

properties. Further, the same study also describes the correlation between the friction diameter with the lubrication performance, in which the lower friction signifies greater lubrication performance. **Figure 13c** illustrates the test results for SAE 5W-30 + Al₂O₃ nanolubricant, while **Figure 13f** shows the results for SAE 5W-30 + PVP + Al₂O₃ nanolubricant. From those two figures, we observed more significant scratches from the results of samples with No. PVP. This scratch is caused by the addition of PVP, which activates the abrasive nature of Al₂O₃. Further, we also identified a clear dominating mechanical scratch on the wear surface, but it has lower dark areas in comparison to the SAE 5W-30 base oil sample. Linearly, [63] concluded that Al₂O₃ nanoparticle forms minor grooves on the contact surface induced by nanoparticle piracy effects. Meanwhile, **Figure 13d** shows the test results for SAE 5W-30 + TiO₂ nanolubricant, which indicates the specimen surface with a smoother groove than the specimens coated with Al₂O₃ nanolubricant + SAE 5W-30, SAE 5W-30, and without lubricant. The addition of PVP surfactant on TiO₂ forms tribofilm layer on the gray cast iron FC25 surface. Therefore, the gray cast iron FC25 surface added with PVP + TiO₂ appears to be smooth, as presented in 13g. The addition of Titanium Dioxide lowers the friction and wear on the gray cast iron FC25 through the tribological reaction between the modified TiO₂, the nanoparticle's surface, and the gray cast iron FC25 surface during the sliding surface, along with the adsorption of decomposed particles [64]. Additionally, the photo macro analysis on the hybrid nanolubricant + SAE 5W-30 shows the role of Al₂O₃ + TiO₂ as the additive material tends to produce a smooth surface in comparison to the specimens with SAE 5W-30 + TiO₂ nanolubricant. The addition of PVP surfactant on hybrid Al₂O₃-TiO₂ generates a more significant size of scratch than from the specimens coated with nanolubricant with no surfactant because the addition of PVP induces abrasive Al₂O₃ on the surface of gray cast iron FC25. This finding is in accordance with the results of the wear mass test presented in **Figure 13g**. Besides, the addition of Al₂O₃ and TiO₂ hybrid nanoparticles on the basic oil (5W-30) fills the wear and curvature on the wear surface (a physical mechanism), aiding separation, reducing contact between metals,

thereby providing a hydrodynamic effect that minimizes the coefficient of friction and making the surface smoother [65].

3.7.5. Morphology Observation on Wear Track of Gray Cast Iron FC25

Figure 14 and **Figure 15** present the SEM observation results on the surface morphology of gray cast iron FC25 using 30x and 100x magnification. The morphological observations in this study were divided into two, namely without polyvinylpyrrolidone surfactant (**Figure 14c-Figure 14e** and **Figure 15c-Figure 15e**) and with Polyvinylpyrrolidone surfactant (**Figure 14f-Figure 14h** and **Figure 15f-Figure 15h**). The result presented in **Figure 14a** and **Figure 15a** are the morphology of FC25 gray cast iron without the use of lubricants. These two figures indicate greater roughness and wear on the specimen surface in comparison to the specimens with SAE-30 base oil lubricants and other variations of lubricants. On the specimens with no lubricant, we observed signs of extensive plastic deformation with deep grooves that appear on the wear track. This is in line with the statement xx. The emergence of plastic deformation is caused by thermal softening, which is formed due to repeated loading without base oil or lubricants. Besides, it also generates other signs, such as deep grooves and material removal, resulting in plastic deformation, abrasion, and delamination that determine the mechanisms causing the wear and tear. The porosity results after testing on samples without nanolubricant were 61,174%.

Furthermore, **Figure 14b** and **Figure 15b** shows the results of the wear track on the FC25 gray cast iron test with SAE 5W-30 base oil lubricant. The figures indicate the scratches that cause abrasion grooves in the traces where the pin hit the disc [66]. This surface wear signifies the decrease of collected tribofilm, causing friction between the contacts which results in a porosity of 41.2%, slightly lower than the specimens without lubricant. **Figure 14c** and **Figure 15c** illustrate the results of the wear track on FC25 gray cast iron coated with nanolubricant SAE 5W-30 and Al₂O₃, showing a fairly deep wear surface and plastic deformation, which causes wear on the surface. This is due to the continuously increasing number of indentations or grooves and levels of delamination resulting in erosion on the transfer

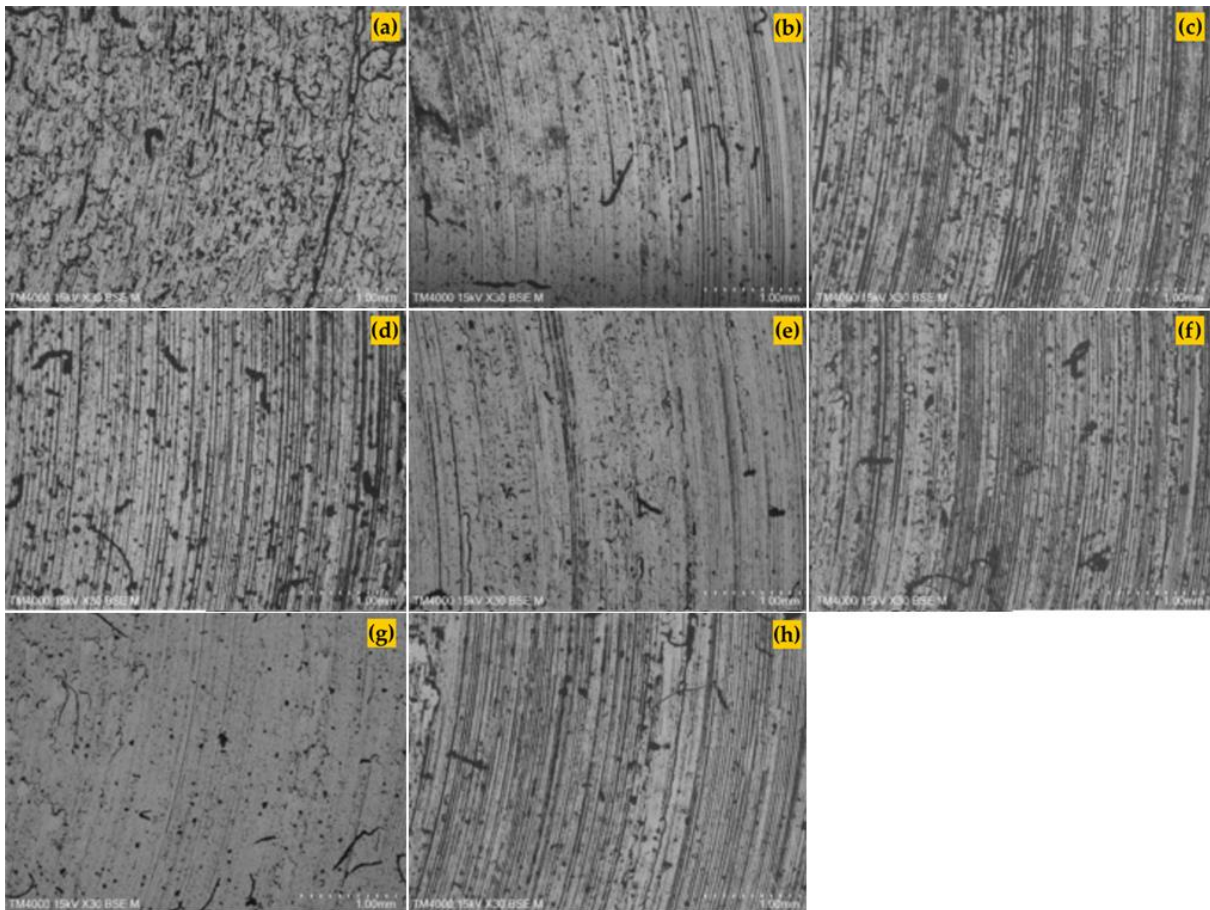


Figure 14. Morphology analysis on gray cast iron FC25 at 30x magnification (a) Without lubrication; (b) With SAE 5W-30 Base Oil; (c) Nanolubricant 5W-30 + (Al_2O_3); (d) Nanolubricant 5W-30 + (TiO_2); (e) Hybrid Nanolubricant 5W-30 + (Al_2O_3 - TiO_2); (f) Nanolubricant 5W-30 + (Al_2O_3 -PVP); (g) Nanolubricant 5W-30 + (TiO_2 -PVP); (h) Hybrid Nanolubricant 5W-30 + (Al_2O_3 - TiO_2 -PVP)

of material and pins to the disc. This erosion is formed from the Al_2O_3 particle fraction, which produces a porosity value of 66.96%. **Figure 14d** and **Figure 15d** show the results of the wear track from the use of SAE 5W-30 and TiO_2 nanolubricant, signifying abrasion grooves accompanied by surface delamination, which is characterized by the continuous appearance of Al_2O_3 deposits on the surface. This is due to the influence of the TiO_2 particle size, which can cause surface damage because of its adhesive wear nature transformed into become abrasive wear [66]. In addition, the addition of TiO_2 nanoparticles into the base oil can reduce the level of abrasive wear, enabling the base oil to carry away the particle fragment efficiently. However, this effect is not observed on the TiO_2 lump.

In **Figure 14e** and **Figure 15e**, we can see the results of the wear track using a hybrid nanolubricant on gray cast iron FC25. The figures show the use of a hybrid nanolubricant SAE 5W-

30 dan Al_2O_3 , nanolubricant SAE 5W-30 and TiO_2 , base oil, and without lubricant. The use of hybrid nanolubricant SAE 5W-30 can produce wider tracks but does not result in deeper scratches. Besides, the contact between hybrid nanoparticles forms tribofilms as an anti-wear protective layer that can improve the tribological nature. The porosity test results for the gray cast iron FC25 sample with the SAE 5W-30 hybrid nanolubricant were 20.718%, suggesting the superiority of hybrid nano lubricant in comparison to other types of lubricants without the addition of surfactants. The results of wear track morphology testing of gray cast iron FC25 using lubricant with the addition of PVP surfactant showed abrasive wear on the surface, as shown in **Figure 14f**, **Figure 14h** and **Figure 15f**, **Figure 15h**. Lubrication using TiO_2 produces the smoothest surface among the use of other lubricants. The rolling effect behavior of spherical nanoparticles can increase wear resistance [58]. The PVP and TiO_2 addition to the

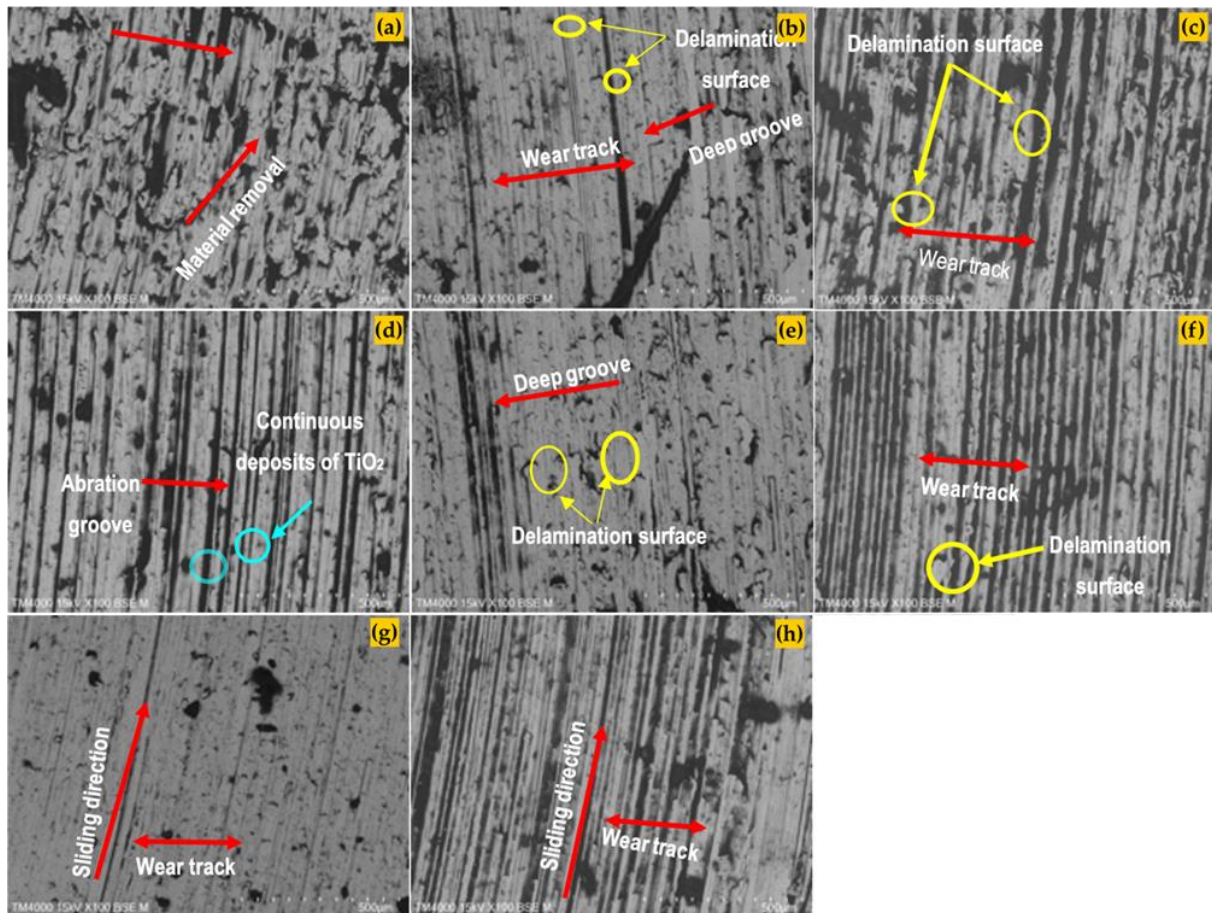


Figure 15. Morphology observation results on gray cast iron FC25 at 100x magnification (a) Without lubrication; (b) With SAE 5W-30 base oil; (c) Nanolubricant 5W-30 + (Al_2O_3); (d) Nanolubricant 5W-30 + (TiO_2); (e) Hybrid nanolubricant 5W-30 + (Al_2O_3 - TiO_2); (f) Nanolubricant 5W-30 + (Al_2O_3 -PVP); (g) Nanolubricant 5W-30 + (TiO_2 -PVP); (h) Hybrid nanolubricant 5W-30 + (Al_2O_3 - TiO_2 -PVP)

lubricant presents low COF value than the lubricant added with PVP and Al_2O_3 or the Hybrid Al_2O_3 and TiO_2 . Consequently, the gray cast iron FC25 surface coated with PVP and TiO_2 has a smoother surface as there is no significant abrasive wear [67].

4. Conclusion

In this paper, the authors have checked and applied a modified application of the multi-pass Based on the analysis results, several conclusions are discussed in the following.

The surface morphology of Aluminum Oxide nanoparticles has an irregular particle shape. The crystallite size of 20.28 nm is observed for the Aluminum Oxide nanoparticles at a peak of $2\theta = 25.34^\circ$. Meanwhile, the Titanium Dioxide nanoparticles have a spherical (spherical) particle shape resembling a ball with a crystallite size of 68.06 nm, observed at a peak of $2\theta = 35.04^\circ$. The functional groups of Aluminum Oxide

nanoparticles are identified at the peak of $3524 - 494 \text{ cm}^{-1}$ and the functional groups of Titanium Dioxide nanoparticles are located at the peak ranging between $3390 - 702 \text{ cm}^{-1}$.

The addition of nanoparticles and surfactants in SAE 5W-30 changes the thermophysical properties of the nanolubricant. The highest density ($788,89 \text{ kg/mm}^3$) and dynamic viscosity values (0.0695 Pa.s) are obtained from the addition of PVP and TiO_2 surfactants. Meanwhile, for the nanolubricant without the addition of surfactants, the highest density (779 kg/m^3) and dynamic viscosity ($0,0579 \text{ Pa.s}$) values are identified in the addition of hybrid Al_2O_3 - TiO_2 . The best stability is obtained by adding a surfactant to each nanolubricant because the addition of PVP surfactant enhances the stability of the nanolubricant.

The tribological properties of gray cast iron FC25 produce the best value of friction coefficient from the coating with SAE 5W-30 + PVP + TiO_2

(0.093), while the lowest wear mass is from the lubrication without hybrid Al₂O₃-TiO₂ surfactant (0.002 gram). The lowest surface roughness is found in a mixture of PVP and TiO₂ surfactants by 0.743 μm. The surface morphology of gray cast iron FC25 coated with SAE 5W-30 + hybrid Al₂O₃-TiO₂ and SAE 5W-30 + PVP + TiO₂ lubrication produces the smoothest surface.

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Author's Declaration

Authors' contributions and responsibilities

The authors made substantial contributions to the conception and design of the study. The authors took responsibility for data analysis, interpretation and discussion of results. The authors read and approved the final manuscript.

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Availability of data and materials

All data are available from the authors.

Competing interests

The authors declare no competing interest.

Additional information

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