Tribological behaviors and self-healing performance of surface modification nanoscale palygorskite as lubricant additive for the steel pair

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Abstract
Palygorskite is an environmentally friendly, inexpensive, and promising silicate mineral. In this research, nanoscale palygorskite additive powders (NPA) were prepared by high energy ball milling with the modifier γ-aminopropyltriethoxysilane (KH550). The NPA was sustainably dispersed in base oil 150 N, exhibiting no particle sedimentation for one week. The self-healing behavior and tribological performance of NPA as lubricant additive were investigated by a ring-on-disc tribometer under different concentrations for 50 h. The results indicate that NPA possesses excellent friction-reducing and anti-wear performance. The 3.0 wt.% and 4.0 wt.% concentrations of NPA displayed the best friction-reducing and anti-wear effects, respectively. The average friction coefficient and wear mass loss decreased by 31.93% and 26.92%, in contrast to that of base oil 150 N. It was also found that different NPA concentrations possessed different friction states. A tribo-film with bilayer structure was formed during the friction process, the first one of which contained compound made up of multiple elements, and the second one of which contained NPA nanoparticles deposited on the friction interface. Moreover, the friction coefficient increased dramatically during the formation of the tribo-film.

1. Introduction

Oil lubricants are widely used in the moving components of a machine to reduce friction and wear, as well as to diminish noise and dissipate heat. Accordingly, they have a significant influence on service-life extension, efficiency enhancement, and energy conservation for the apparatus. The additive can enhance the capacity of base oil lubricants. Traditional additives containing sulfur and phosphorus elements may cause serious environmental pollution [1, 2]. Therefore, novel and environmentally friendly additives are needed in applied science and industrial field. Since last decade, with the development of nanotechnology, nanoparticle additive attracts great attention due to their minuscule size, enabling them to optimize the rubbing surface [3–5]. Nanoparticles of hard materials, such as SiO2 [6, 7] and Al2O3 [8], used as lubricant additives have a promising future because of their ability to improve properties of various base fluids. Among the several types of hard materials nano-additives, phyllosilicate mineral powders such as serpentine are also proven to be effective in enhancing the tribological ability of lubricating oil [9–11]. The investigation into serpentine powders as a lubricant additive is comprehensive and insightful. Although palygorskite has a similar chemical constituent and crystal structure as that of serpentine, there are few reports about the use of palygorskite as a lubricating oil additive. It can be hypothesized that nanoscale palygorskite is able to reduce friction and wear as lubricant additives. Furthermore, this nanoscale powder is suitable due to its low cost and simple preparation procedure.

Palygorskite is a natural one-dimensional nanofiber material with the typical molecular formula of Si₉O₃₀Mg₆(Al)(OH)₂(H₂O)₄·4H₂O [12], also known as attapulgite, which has been widely investigated in some research fields such as environmental protection and energy materials [13, 14]. Some studies suggest that
palygorskite is feasible for application in the field of tribology. The friction and wear behaviors of Polytetrafluoroethylene (PTFE) after the addition of nano-attapulgite with acid and thermally treatment are researched. The results demonstrate that although the friction coefficient changes negligibly, the wear resistance of composite PTFE was greatly improved [15, 16]. Meanwhile, surface-modified attapulgite is reported to be a thickener to synthesize environmentally-friendly lubricating grease with superior tribological properties [17–19]. Due to the high thermal stability and salt resistance, organo-palygorskite is used as the additive to modify the rheology and tribological characteristics of oil-based [20, 21] and water-based [22, 23] drilling fluids. With the addition of palygorskite powders into lubricating oils, the tribological behavior of the steel tribo-pair can be significantly improved [24, 25]. This is attributed to the formation of multiple elements tribo-film. Moreover, nanocomposites based on attapulgite used as lubricating oil additives were investigated [26, 27]. Even so, the lubrication and self-healing mechanisms of palygorskite are still obscure, and the experiment period of previous researches is relatively short, usually less than 5 h. The tribological performance during tribo-film formation and the self-healing ability of palygorskite in different friction periods are rarely investigated. However, those are crucial for understanding and improving the self-healing and tribological capabilities of palygorskite.

Based on the above description, in the present work, nanoscale palygorskite additives powders (NPA) were fabricated for base oil 150 N. The self-healing and tribological performances of different concentrations of NPA were explored using ring-on-disk friction and wear tester. The influence of NPA on the worn surface in different experiment periods was investigated to reveal the tribo-film formation process and self-healing behavior. The dispersion property, friction-reducing, anti-wear, and self-healing characteristics of NPA were further explored to lay a foundation for its industrial application.

2. Experiment details

2.1. Material processing and sample preparation

The raw material with a diameter of 0.15 mm was fabricated by artificially purifying and crushing the natural palygorskite mineral, whose chemical composition is shown in table 1. They were placed in a jar and dry ground by a high energy ball mill at 150 r min⁻¹ for 30 h, and then wet ground with the addition of 12 ml absolute ethanol (99.5%, Nanjing Reagent Co., Ltd, China) for 30 h to attain nanoscale palygorskite powder (NP). Nanoscale palygorskite additives (NPA) were obtained through a similar method as that of NP except for additional addition of 5.0 wt.% of γ-aminopropyltriethoxysilane (KH550, 98%, Nanjing Reagent Co., Ltd, China) during the wet grinding process. Both NP and NPA were dried to constant weight in a vacuum oven before use. NPA with weight percentage of 0.0, 1.0, 2.0, 3.0, 4.0, and 5.0 were respectively mixed in oil 150 N. These mixtures were mechanically stirred at a speed of 900 r min⁻¹ for 30 min. And then they are further treated with ultrasonic dispersion for 30 min. Finally, oil samples with different concentrations of NPA were obtained, which are shown in table 2. The base lubricant was mineral lubricating oil 150 N (S-oil Corp., Korea). The representative physicochemical properties of base oil 150 N and NPA-5 oil sample are mentioned in table 3. There are not much significant changes except in the kinematic viscosity. The results show that the kinematic viscosity of NPA-5 has increased. And a similar phenomenon is also observed by other research [21].

| Table 1. Chemical composition of palygorskite powders. |
|---------------------------------|
| Component | S₂O₂ | Al₂O₃ | Fe₂O₃ | MgO | CaO | K₂O | Na₂O | H₂O⁺ | H₂O | Others |
| Content (wt.%) | 56.10 | 12.50 | 1.68 | 8.72 | 0.85 | 0.12 | 0.12 | 14.80 | 4.76 | 0.35 |

| Table 2. Component of oil samples fabricated in the present work. |
|---------------------------------|
| Code name | Component |
| NPA-0 | 150 N |
| NPA-1 | 150 N + 1.0 wt.% NPA |
| NPA-2 | 150 N + 2.0 wt.% NPA |
| NPA-3 | 150 N + 3.0 wt.% NPA |
| NPA-4 | 150 N + 4.0 wt.% NPA |
| NPA-5 | 150 N + 5.0 wt.% NPA |
2.2. Tribological test

The friction-reducing and anti-wear performance of different oil samples was investigated using a tribometer with a ring-on-disc configuration as shown in figure 1. The ring and disc were made of thermal refining AISI 1045 steel, and the hardness was 56–59 HRC. The surface roughness of the friction pair was 0.8 μm. All the experiments were explored at ambient temperature with a 200 N set load. The upper ring rotated clockwise at a speed of 400 r min⁻¹ (0.561 m s⁻¹), whose inner and outer diameters were Φ 20.0 mm and Φ 25.6 mm, respectively. The lower disc was fixed in the oil bath, whose diameter and thickness was respectively Φ 41.0 mm and 6.0 mm. During the test, the friction interface was immersed in oil samples. The average friction coefficient was calculated based on the statistics obtained from the tribometer. The tribological test lasted for 50 h. In order to reveal the mechanism of tribofilm and self-healing behavior, the rubbing surface, friction coefficient, and wear mass loss were observed every 5 h when the lubricants were NPA-4 oil samples. Before observing the worn surface, the friction pair should be cleaned in toluene (99.5%, Nanjing Reagent Co., Ltd, China) by ultrasound for 10 min and air-dried.

2.3. Evaluation methods and characterization technology

The dispersion property of NPA was evaluated by the sedimentation method, transmission electron microscope (TEM) and ultraviolet-visible (UV–vis) spectrophotometry absorbance measurements. Lubricating oil dispersions (NPA-0 and NPA-5) were prepared according to the method of 2.1 and table 3. Then the mixtures were slowly poured into a sample bottle and let them rest for 2 weeks. The sedimentation height was recorded once a day. The shape and size of NPA powders were measured by TEM (JEM-2000FX, JEOL Ltd, Japan; accelerating voltage of 180 kV). For UV–vis (AUV-1900, Macy Instrument, China) spectral analysis, the test was performed immediately after the ultrasonic dispersion of NPA in the base oil. Oil samples were placed in glass cuvettes with a capacity of 3.5 ml. The changes in the absorbance level of light were recorded every day.

The tribological experiment was explored immediately after preparation of oil samples. The friction surface of the lower disc was preliminary detected by a metallographic microscope (XJL-03, Nanjing Jiangnan Novel Optics Co., Ltd, China). The wear mass loss was measured by electronic balance with an accuracy of 0.1 mg (AR224CN, Ohaus, America). The morphologies and elemental content of the rubbing surface were detected by electro probe-X-ray microanalysis (EXMO, EPMA-1600, Shimadzu Corp. Japan.) provided with energy dispersive spectroscopy (EDS).

3. Results and discussion

3.1. Dispersion property of NPA

The dispersion stability measurement results of the oil samples are presented in figure 2. After being stored for 1 day, it can be seen from figure 2(a) that the settling height of NP lubricant dispersion (Sample 1) increases dramatically, while the settling height of NPA-5 (Sample 2) is zero. And this phenomenon lasts for a week.
However, after a week, Sample 2 began to have slower settling rates, which means the tribological test can be investigated within 7 days. Figure 2(b) shows the UV–vis spectra of the oil sample NPA-5. It is clear from the results that as the time prolonged, absorbance values of NPA-5 have the same trend and fluctuate within a certain range. There are no significant changes within two weeks. The stable absorbance rate reveals that NPA exhibits good dispersion stability and less agglomeration in the base oil. In fact, when further prolonging the static duration, only a short time of stir is needed before use to get a good dispersion for NPA contained oil samples. Figure 3 displays the TEM image of as-prepared NPA. According to this figure, the maximum diameter, minimum diameter, and mean diameter of NPA particles were 262 nm, 48 nm, and 126 nm, respectively. The nanoscale of as-prepared NPA was suitable for dispersion in base oil 150 N.

The role of KH550 is to disperse, couple, and stabilize the nanoparticles in the process of modifying NP. The sketch map of the surface modification process is shown in figure 4. In the first step, KH550 is hydrolyzed to form silanol with a large number of hydroxyl groups. In the second step, dehydration reactions occur between the silanol component and the hydroxyl groups on the surface of Np. As a result, the organic group is grafted on the Np, and the core–shell structure is generated [28], where the core is Np and the shell is KH550 group. The shell can weaken the agglomeration phenomenon by increasing the steric hindrance of nanoscale powder [29], thus improving the stability of NPA in base oil 150 N by improving the solution solubility. Consequently, NPA can be suspended in base oil 150 N without precipitation.
3.2. Tribological performance

Figure 5 shows the evolutionary relationship of NPA concentrations with the average friction coefficient (a) and wear mass loss (b) of the steel tribo-pairs. Without the addition of NPA (NPA-0), the average friction coefficient is relatively high (0.055), and the wear mass loss is the maximum (2.7 mg), indicating the severe wear of the friction pairs. It is proven that the base oil plays a certain role in friction, though its lubrication performance is poor. An obvious distinction is displayed after introducing different concentrations of NPA into oil 150 N. Both the average friction coefficient and the wear mass loss decrease gradually first and increase progressively later given the different amounts of NPA, although the two dividing points are different. When the content of NPA increases to 3.0 wt.%, the average friction coefficient is the lowest. Compared with NPA-0, the average friction coefficient and wear mass loss are reduced by 31.93% and 15.38%. When the oil sample is NPA-4, the wear mass loss is the minimum. Compared with oil 150 N, the wear mass loss is reduced by 26.92%. However, the average friction coefficient is higher than that of 150 N. When the oil sample is NPA-5, the average friction coefficient and wear mass loss displayed an increase of a different magnitude. From the above discussion, we can see that when the concentration is lower or higher than the dividing point, both the friction-reducing or anti-wear performances are weakened.

The SEM photos of the rubbing surface under the lubrication of 150 N with and without NPA are presented in figure 6. For the base oil, a typical plastic deformation zone with numbers of wide and deep furrows and some exfoliated pits are evident on the contact surface (figure 6(a)), indicating serious adhesive wear and abrasive wear. For NPA-2, the plastic deformation zone and exfoliated pits are almost non-existent. Moreover, the furrows are shallow and narrow (figure 6(b)), implying slight abrasive wear. For NPA-3, the friction surface is relatively flat because it is covered by a tribo-film, and the furrows disappear and are replaced by minor scratches with edge blur (figure 6(c)). The results indicate that rolling-sliding friction is displayed in the experiment. This condition is advantageous in improving friction-reducing performance. The friction interface for NPA-4 exhibits no obvious signs of wear. Instead, a flat, dense, continuous, and steady tribo-film is formed (figure 6(d)).
indicating sliding friction between films. This state is beneficial for enhancing abrasive resistance. As the NPA amount increases to 5.0 wt.%, plenty of thin and shallow scratches are observed on the rubbing surface (figure 6(e)). This result shows that the friction state transforms into abrasive wear. Compared with figure 6(d), the tribo-film with speckles is generated in figure 6(f), illustrating that uninterrupted friction is conducive to forming uniform tribo-film.

Figure 7 exhibits the EDS spectrums and elemental contents of the contact surface of figure 6. It is observed that Fe, Mn, and Si exist on the frictional interface under the lubrication of 150 N (figure 7(a)). With the addition of NPA into 150 N, the characteristic elements of palygorskite, such as Al and Mg, can be detected on the frictional interface. Furthermore, in contrast to the base oil, there is a decrease content of Fe and an increased content of Si and O. It is well known that Si is also one of the characteristic elements of palygorskite. Compared with NPA-0 (figure 7(a)), the content of elements in NPA-3 (figure 7(c)) changes significantly. In particular, the content of Fe reduces from 98.08% to 82.51%, the content of Si grows from 1.08% to 3.62%, the content of O, Al, and Mg rises respectively to 9.39%, 1.56%, and 1.91%. It is suggested that a tribo-film enriched Si, Al, Mg, O, and Fe is generated. For NPA-4 (figure 7(d)), the content of Si, Al, Mg, and O reaches the highest value of 10.30%, 3.26%, 3.23%, and 30.06% in sequence. Inversely, the content of Fe is found to be the least (52.64%), indicating that a tribo-film with a higher content of the characteristic elements of palygorskite is formed on NPA-4 treated specimen. As seen in figure 7(e), the content of each element on the friction surface is similar to figure 7(c) as the amount of NPA increased to 5.0 wt.%. This result implies that the tribo-film is slightly damaged by abrasive wear. Although the uniformity of tribo-film is different (figures 6(d) and (f)), the content of each
Element is close to each other, as shown in figures 7(d) and (f). This result suggests that the influence of palygorskite concentration on tribo-film is greater than the continuity of tribological experiments.

3.3. Self-healing ability
The excellent self-repairing ability of NPA is a unique phenomenon in the tribological test. Figure 8 exhibits changes in metallographic images of the friction surface every 5 h under the lubrication of NPA-4. In the beginning, only signs of friction are shown on the mating surface, and the wear becomes severer when prolonging the testing time. After 20 h, dark areas start to appear on the rubbing surface. Notably, an increasing trend of dark areas and a smoothing trend of the worn surface seems to happen. When the experiment lasts for 30 h, the friction interface is covered by a dark tribo-film and the steel matrix is invisible. In combination with the EDS results, it can be concluded that NPA possesses the self-healing ability to generate the tribo-film with the help of friction behavior. More importantly, the generation rate of tribo-film is higher than the wear rate. When the experiment time is longer than 30 h, the formation and wear of the tribo-film reach a dynamic exchange.
state. That is to say, at this stage, the friction between the steel substrate transforms into the friction between the tribo-film. Therefore, the anti-wear performance of the steel matrix is greatly improved (shown in figure 5(b)). It should be noted that an uneven tribo-film is generated (30 h~50 h in figure 8) due to discontinuous cleaning, which is consistent with SEM observation in figure 6(f).

Figure 9 presents the friction coefficient curves of tribo-pair lubricated with NPA-0, NPA-3, and NPA-4 as a function of rotation time. The friction coefficient of base oil is higher than the oil samples with NPA in the first 10 h. A relatively stable stage of the friction coefficient of base oil is exhibited when the testing varies from 10 to 40 h. After that, the friction coefficient of the base oil grows significantly. This indicates that the friction coefficient of base oil experiences initial worn stage, normally worn stage, and acutely worn stage during the entire test. For NPA-3, the friction coefficient fluctuates slightly with time, and the value is always much lower than that of base oil. This result reveals that a better friction reduction ability is displayed when lubricated with NPA-3, as seen in figure 5(a). The evolution of the friction coefficient for NPA-4 is more attractive. Initially, the friction coefficient of the steel tribo-pair is less than that of base oil. But the friction coefficient rises dramatically in the test period of 20 to 30 h due to the formation of hard tribo-film, which can be obviously observed in figure 8. This implies that the extension and abrasion of the tribo-film along the steel matrix contribute to a rapid
rise of the friction coefficient. After that, the tribo-film is formed, and the friction coefficient has entered the period of the run-in and stable wear. The formation of tribo-film improves wear resistance capability but at the cost of friction coefficient increase, as shown in figure 5. It is worth mentioning whether improving the anti-wear performance or enhancing friction-reducing property can both extend the service life of the steel matrix.

3.4. Discussion

From the experiment results, it can be indicated that the friction-reducing and anti-wear performances of NPA depend on the friction behavior and the formation state of tribo-film under different concentrations. For NPA-0, both adhesive wear and abrasion wear exist at the frictional interface. The abrasive particles are made up of rough peaks on the steel matrix surface and the particles falling off due to adhesion and fatigue. In this state, the values of friction coefficient and wear mass loss are both very high. For NPA-2, the friction process is dominated by abrasive wear. NPA powders, which are very hard, produce a nano-ball effect and cause the abrasive particles to become round and thin. The sliding friction is replaced by rolling-sliding friction [30], which reduces the friction coefficient and the wear mass loss. For NPA-3, a thin tribo-film enriched with multiple elements is generated [24]. Because of a relatively flat surface and rolling-sliding friction, the friction coefficient is the lowest and the wear mass loss is less. When lubricated with NPA-4, the steel substrate is covered by a relatively thicker tribo-film with a bilayer structure (shown in figure 11), which possesses the flattest contact surface (shown in figure 6(d)). Thus, the rolling-sliding friction becomes into sliding friction of the tribo-film, indicating a rise in friction coefficient. Also, the deterioration in anti-friction property with excessive NPA volume fraction may be due to an increase in viscous friction [7]. Conversely, the anti-wear performance of the steel matrix is improved by the formation of thick tribo-film. This behavior is consistent with figure 5. A similar phenomenon is also observed by other investigations [31, 32]. When the concentration of NPA was too high, such as 5.0 wt.%, the high surface energy makes NPA particles agglomerate together and forms clusters, which weakens the friction-reducing and anti-wear capabilities of the steel substrate.

As a three-layer phyllosilicate, palygorskite is composed of the Si-O tetrahedral layer and [Mg, Al]-OH octahedron layer, as shown in figure 10 [33]. The layers are arranged in a 1:2 ratio by hydrogen bonds and van der Waals force. This crystal structure brings many physicochemical characteristics to palygorskite, such as high adsorption and large surface energy. When the content of NPA is lower (below 3.0 wt.%), the nanoscale powders get together at the friction interface due to the flow of the oil sample and its own high adsorption. This phenomenon brings two effects. The first is the polishing and ball effect, which changes the sliding friction into rolling-sliding friction [30]. The other is to increase the viscosity of the oil samples and even transforms the oil into a solid-like film with a certain thixotropic strength [34]. The above two effects work together to improve the friction-reducing and anti-wear performance of the steel matrix. As the NPA concentration is 4.0 wt.%, because of the effect of friction heat, and shearing force, the crystal structure of palygorskite is destroyed, releasing active oxygen and secondary particles [35]. At the same time, new iron atoms are exposed on the surface of the steel matrix. Due to stronger absorption and activity, the smaller nanoscale powders preferentially gather in the friction area to form a tribo-film. According to the XPS results of the published researches [25–27], it is concluded that the tribo film mainly composed of iron oxides, silicon oxides, and organic compounds. Thereafter, the larger nanoscale powders are ceaselessly deposited on the rubbing surface [25]. Finally, a dense, flat, thick, and steady tribo-film with bilayer structure is formed, as shown in figure 11. More importantly, the

![Figure 10. Schematic crystal structure of palygorskite.](image-url)
tribo-film has excellent plasticity and ductility like metal, which also possesses higher hardness. In the future, the effect of work conditions such as normal loads, rotation rate, and temperature will be investigated to further explore the tribological and self-healing mechanism of NPA powders.

4. Conclusions

The NPA powders were successfully prepared by high energy ball milling method. The self-healing behavior and tribological performance of NPA on the steel matrix were investigated through a ring-on-disc tribometer under different concentrations at room temperature. The friction coefficient, wear mass loss, elemental analysis, and microstructure of the rubbing surface were analyzed. The main conclusions are listed as follows:

1. NPA powders with an average diameter of 168 nm were fabricated using KH550 as the modifier. The prepared NPA showed good stability in base oil 150 N, and no particle sedimentation was observed for one week.

2. The concentration of NPA in the base oil plays a vital role to control the wear mass loss and friction coefficient. Compared with base oil 150 N, the 3.0 wt.% concentration of NPA presented the best friction-reducing effect, and the mean friction coefficient decreased by 31.93%. The 4.0 wt.% content of NPA exhibited an optimal anti-wear performance, and the maximum wear mass loss reduction is 26.92% compared with that of 150 N. A dark tribo-film was clearly observed on the frictional interface.

3. The excellent self-healing behavior of NPA should be attributed to the tribo-film with bilayer structure, the first one of which contained compound made up of multiple elements, and the second one of which contained NPA nanoparticles deposited on the friction interface. In addition, the friction coefficient increased rapidly during the tribo-film formation.

4. The friction-reducing and anti-wear performance of NPA depended on the friction behavior and the tribo-film formation state under different concentrations. Different NPA concentrations possessed different friction states.

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