Enhanced Lubrication and Photocatalytic Degradation of Liquid Paraffin by Hollow MoS$_2$ Microspheres

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ABSTRACT: Nowadays, with the rapid development of environmental protection awareness, the demand for the emergence of a green counterpart of lubricant additive plays a more and more important role in reducing friction and wear as the times require. In this paper, full-hollow and semihollow molybdenum disulfide (MoS$_2$) microspheres were prepared via a hydrothermal method and were characterized and confirmed by X-ray diffraction (XRD), scanning electron microscopy (SEM), and transmission electron microscopy (TEM). According to our results, both fully hollow and semihollow MoS$_2$ microspheres possessed excellent lubrication-enhancing effects for liquid paraffin (LP), while full-hollow samples after friction provided better photocatalytic degradation properties than semihollow samples after friction. Related analysis indicated that curved layer opened structures with more rim and edge sites, bigger surface area, and narrower band gap made full-hollow MoS$_2$ samples achieve a better photocatalytic level. Thus, it was a sustainable solution for both lubrication-enhancing and photocatalytic degradation functions during different stages of the usage of lubricating oils, which suggests a potential strategy for achieving environmentally friendly developments.

1. INTRODUCTION

Friction is a common physical phenomenon which consumes a great deal of energy, and wear caused by friction is one major reason for mechanical failures. It is estimated that about 1/3 of the energy in the world is consumed by friction in various forms, and about 80% of the machine component failures are generated from friction in different ways. Improving lubrication, controlling friction and reducing wear are important strategies to save energy and achieve high reliability. For example, great efforts have been made, and many additives have been introduced to lubricating oils to improve their lubrication performance.1−4 Nevertheless, they may also cause subsequent pollution, and developing green lubrication systems with both better lubricating performances and degradation properties is of great practical significance.

Due to the weak van der Waals force between layered structures, MoS$_2$ has been used as a solid lubricant and additive for lubricating oil and/or solid materials as lubricants for many years.5−11 Related lubricating mechanisms, such as rolling, deformation, exfoliation, and chemical stability, have been illustrated.12−14 Especially, nanosized or microsized MoS$_2$ with high surface areas also show excellent photocatalytic activity in the photoinduced degradation of organic pollutants.15−17 Compared to those layer-closed structures (such as fullerene-like and hollow sphere-like structures), MoS$_2$ with layer-opened structures can possess better photocatalytic performances due to more active atoms located at rim and edge sites.18−21 The microscopic morphology of MoS$_2$ plays a crucial role in its macroscopic properties, which can be controlled or modulated by the proper method.22−28 In this work, MoS$_2$ with special microscopic structures was prepared, which helps to achieve a sustainable solution combined lubrication-enhancing and photocatalytic degradation promotion for lubricating oil at different stages.

2. RESULTS AND DISCUSSION

2.1. Characterizations of Samples. XRD patterns of full-hollow MoS$_2$ and semihollow MoS$_2$ samples are presented in Figure 1a, and both of them contain four diffractive peaks at 13.7°, 33.1°, 39.5°, and 58.9°, corresponding to (002), (100), (103), and (110) crystal planes of MoS$_2$, respectively. No other diffractive peaks corresponding to impurity phases are detected. TEM and SEM images of the two samples (Figure 1b to Figure 1e) show that full-hollow and semihollow microspheres with uniform size have been successfully prepared, while the full-hollow sample possesses a slightly thicker shell.

The hydrothermal formation process of full-hollow structure is illustrated in Figure 2a. Bu$_4$N$^+$ ionized from Bu$_4$NBBr can form small aggregates, and then vesicles are formed by increasing aggregate size. Plenty of MoO$_4^{2−}$ in solution can be absorbed to...
Figure 1. (a) X-ray diffraction patterns of two as-prepared structures. (b) TEM of full-hollow MoS$_2$. (c) SEM of full-hollow MoS$_2$. (d) TEM of semihollow MoS$_2$. (e) SEM of semihollow MoS$_2$.

Figure 2. Schematic illustration of the formation process of (a) hollow MoS$_2$ microspheres and (b) core–shell MoS$_2$ microspheres.

Figure 3. Variations of the average friction coefficient and AWSDs with the increase of (a) full-hollow and (b) semihollow MoS$_2$ microsphere concentration (from 0 to 0.70 wt %). Insert at top right corner: the changing tendency of the real-time friction coefficient at different concentrations of relative MoS$_2$ microspheres.
the vesicles by electrostatic interaction. With temperature increasing, H$_2$S generates from the decomposition of KSCN, which can react with MoO$_4^{2-}$ and form Mo$_2$S$_3$. For semihollow structure (Figure 2b), thiomolybdate precursor (Mo$_3$(S$_2$)$_6$S)$_2^{2-}$ can be formed by the reaction between Na$_2$MoO$_4$ and KSCN. Then Bu$_4$N$^+$ can be absorbed to (Mo$_3$(S$_2$)$_6$S)$_2^{2-}$ also by electrostatic interaction, and MoO$_4^{2-}$ can be absorbed to Bu$_4$N$^+$ similarly. Finally, H$_2$S reacts with MoO$_4^{2-}$ to form MoS$_2$, and (Mo$_3$(S$_2$)$_6$S)$_2^{2-}$ gradually decomposed into MoS$_2$.

### 2.2. Tribological Properties

Figure 3a and Figure 3b show that the average friction coefficient and the average wear scar diameter (AWSD) decrease rapidly when MoS$_2$ concentration is quite lower, which results from MoS$_2$ microspheres entering into the surface of friction pairs along with LP, due to its spherical structures including rolling and deformation. When the concentrations of full-hollow and semihollow MoS$_2$ are higher than 0.20% and 0.30%, respectively, the friction coefficients for the two systems increase in different ways. There are sufficient additives between the surfaces of friction pairs, and varying degrees of exfoliations are produced, as shown in Figure 4a to Figure 4c. Obviously, the resistance to friction pairs is stronger when microspheres are exfoliated directly, so the average friction coefficient rises as in Figure 3a and Figure 3b. In addition, AWSD can reach the smallest value because direct exfoliation can lead to smaller contacting areas between friction pairs. When the concentrations of full-hollow and semihollow MoS$_2$ are higher than 0.30% and 0.50%, respectively, fewer microspheres can enter the surface of friction pairs owing to agglomeration, which results in lower average friction coefficient under the same lubrication mechanism. As illustrated in the inset pictures at the top right corner of Figure 3a and Figure 3b, the friction coefficient of LP containing semihollow samples is relatively stable. TEM and SEM images of samples after friction (Figure 4d–Figure 4g) indicate that they are destroyed in different degrees by friction, and layer-closed structures have turned into layer-opened structures. Plenty of curved layer-opened fragments appear, thus friction makes samples obtain bigger surface area and more rim and edge sites. For semihollow samples, only small amounts of them are destroyed (Figure 4f and Figure 4g), due to the good mechanical property of its core–shell structure.

### 2.3. Photocatalytic Degradation

Figure 5 gives the photocatalytic degradation behaviors generated by full-hollow MoS$_2$ before friction (FB), full-hollow MoS$_2$ after friction (FA), semihollow MoS$_2$ before friction (SB), and semihollow MoS$_2$ after friction (SA) in the photocatalytic tests. Figure 5a indicates that $c/c_0$ (where $c_0$ is initial concentration of LP, $c$: real-time concentration of LP in photocatalytic degradation) has a positive correlation with MoS$_2$ concentration. The optimum concentration for FB and FA is 0.40 wt %, while the optimum concentration for FB and FA is 0.40 wt %.
concentration for SB and SA is 0.60 wt %. In photocatalytic tests, the full-hollow sample can obtain same photocatalytic effects (corresponding to the same contact areas) by rather smaller amounts than semihollow sample or other solid materials. Supposing that the shell size of semihollow and full-hollow samples is the same, the contact area between semihollow morphology and LP is \( S_a \) and the contact area between full-hollow morphology and LP is \( S_f \):

\[
S_f = S_t = 4\pi a^2
\]

The average mass of a single full-hollow sphere and semihollow sphere \((m_f, m_s)\) can be expressed as

\[
m_f = V_f \rho = \left(\frac{4}{3} \pi a^3 - \frac{4}{3} \pi b^3\right) \rho
\]

\[
m_s = V_s \rho = m_f + \frac{4}{3} \pi a^3 \rho
\]

where \(V_f\) or \(V_s\) are the average volume of a full-hollow sphere or a semihollow sphere, and \(\rho\) is the average density of samples.

Figure 5a also indicates that the incensement of MoS \(_2\) concentration leads to the abatement of light transmittance of the LP mixture, so that \(c/c_0\) gradually decreases with increasing MoS \(_2\) concentration. FA has better photocatalytic properties than SA since there are more curved fragments in the former one. As shown in Figure 4c to Figure 4f, hollow structures are destroyed more seriously by friction, and many curved nanoplatelets or nanosheets form sequentially. In this way, there are larger contact areas between MoS \(_2\) and LP, and more active rim-edge atoms with dangling bonds in nanoplatelets and nanosheets can result in higher catalytic activity.

Although FA possesses a higher catalytic activity, its photoinduced degradation rate for LP is only about 15.0%, which can be improved by other means. It is shown in Figure 6 that the photocatalytic degradation rate induced by FA can be greatly improved in acid solution (reaches \(\sim 50\%\) at \( pH = 3 \)). On the contrary, alkaline environment can lead to a decrease in degradation rate. The hydroxyl radical (\(\bullet OH\)) plays an important role in degradation of organic compound due to its strong oxidizability, which is partly determined by \( pH \) value.

At \( pH = 3 \), the oxidation potential of \(\bullet OH\) ranges from 2.65 to 2.80 V, and its oxidizing property is next only to fluorine in nature; the value is only 1.9 V when \( pH \) value reaches 7. \(^{30,31}\) Obviously, the oxidizability of \(\bullet OH\) is much stronger in acid environment than that in neutral environment. Proton can reduce the concentration of \(\bullet OH\) when \( pH \) values are smaller than 3, as parts of \(\bullet OH\) have been converted into HO\(^-\), resulting in a decrease of LP degradation. \(^{32}\) On the other hand, when the \( pH \) value is higher than 7, the hydroxyl radical is rapidly converted into \(\bullet O^–\):

\[
\bullet OH + OH^- \rightarrow \bullet O^- + H_2O
\]

\(\bullet O^-\) works more slowly than \(\bullet OH\), so that the photocatalytic efficiency is greatly reduced. \(^{33}\) By adjusting \( pH \) values, the degradation efficiency can be greatly enhanced from 15.0% to 45.0%.

In order to confirm the effect of \( pH \) value on the concentration of \(\bullet OH\), a kinetic modeling of \(\bullet OH\) is suggested and discussed. Considering the photocatalytic reaction process, \(\bullet OH\) is produced by O\(_2\) and H\(_2\)O:

\[
O_2 + H_2O \rightarrow \bullet OH\ (reaction\ rate: \ k_1)
\]

LP degradation by hydroxyl radicals is expressed as

\[
LP + \bullet OH \rightarrow oxidized\ products\ (reaction\ rate: \ k_2)
\]

Thus, the degradation rate of LP can be written as

\[
-\frac{d[LP]}{dt} = k_2[LP][\bullet OH]
\]

However, there are some hydroxyl radical scavengers \((X)\) in the reaction system, and the effect of \(X\) can be written as

\[
X + \bullet OH \rightarrow products\ (reaction\ rate: \ k_3)
\]

Then, according to eqs 5, 6, and 8, the changing rate of hydroxyl radical concentration can be expressed as

\[
-\frac{d[\bullet OH]}{dt} = k_1[O_2][H_2O] - k_2[LP][\bullet OH] - k_3[X][\bullet OH]
\]

Generally, pseudo steady state is assumed with regard to reactants, so that the concentration of hydroxyl radicals can be considered as constant at a certain time, thus:

\[
-\frac{d[\bullet OH]}{dt} = 0
\]

Combining eqs 9 and 10:

\[
[\bullet OH] = \frac{k_1[O_2][H_2O]}{k_2[LP] - k_3[X]}
\]

Substituting eqs 11 into 7:

\[
-\frac{d[LP]}{dt} = -\frac{k_1[LP][O_2][H_2O]}{k_2[LP] + k_3[X]}
\]

In the experiments, the amount of LP is relatively small, and it can be regarded as effective degradation, thus:

\[
k_2[LP] + k_3[X] \approx k_3[X]
\]

Combining eqs 12 and 13, eq 12 can be changed as

\[
-\frac{d[LP]}{dt} = -\frac{k_1k_2[O_2][H_2O]}{k_3[X]}[LP]
\]
or
\[
\ln \left( \frac{[LP]_0}{[LP]} \right) = k_{ap}t
\]

where \( k_{ap} = \frac{k_f([O_2]([H_2O]))}{[O_2]} \), and \( k_{ap} \) is the pseudo-first-order rate constant and depends on the concentration of \( \bullet OH \).

\( k_{ap} \) can be calculated by the slope of kinetic fit (Figure 7), and it also represents the reaction rate affected by \( \bullet OH \). pH value has great influence on \( k_{ap} \), which indicates that it also has a great influence on the concentration of \( \bullet OH \). When pH value is 3, \( k_{ap} \) reaches the maximum value, which is the best parameter for photocatalytic degradation.

On the other hand, dye absorbed on the surface of MoS\(_2\) is stimulated to the excited state (dye\(^+\)) by absorbing visible light. The excited photoelectrons transfer to the conduction band of MoS\(_2\) from dye\(^+\) and resulted in the conversion of dye to the radical cation \( \bullet dye^+\). \( O_2 \) absorbed on the surface of MoS\(_2\) can be reduced to superoxide radicals \( \bullet O_2^- \), which have the ability of degrading an organic compound (LP and \( \bullet dye^+\)) to small molecules by photoelectrons on the conduction band. In our experiment, only 0.9 wt \% of methyl orange (MO: a kind of dye) is added into the LP mixture (pH = 3). Figure 8 indicates that MO can significantly improve the efficiency of photocatalytic degradation for LP (to about 68.0%). What is particularly worth mentioning is that MO can be almost completely degraded after 8 h. Therefore, there is no new pollutant appearing at the end of the photocatalytic degradation reaction.

The catalytic activity maintenance of FA after several usage cycles is also important. Figure 9 gives circular tests of photocatalytic degradation of LP catalyzed by FA. Results indicate that the value of \( c/c_0 \) does not increase obviously after four cycles (lasting 48 h, pH = 3, without MO), indicating its excellent recyclable property and stability in photocatalytic degradation. However, the degradation rate is only 48.6% after four cycles when MO is involved (lasting 48 h, pH = 3), probably because some degradation products of MO are adsorbed on the surface of MoS\(_2\).

The light absorption properties of FB, FA, SB, and SA have been investigated by UV–vis diffuse reflectance spectra (DRS) (Figure 10), and the reflection pathway of light on the surface of these MoS\(_2\) microspheres with different morphologies is schematically illustrated in the inset at the top right corner of Figure 10. Semihollow MoS\(_2\) microspheres exhibit stronger photoabsorption behavior almost in the whole detected wavelength range because semihollow structure allows more multiple reflections of light within the interior cavity, corresponding to more efficient use of the light source.

However, FA has the best photocatalytic property because of so many curved nanoplatelets and nanosheets with active rim-edge atoms, which indicates active rim-edge atoms are more important than light absorption properties. Better light absorption performance can improve the charge separation. The estimated band gap values of MoS\(_2\) before and after friction are 2.02 and 2.33 eV, respectively, as shown in the inset at the top left corner of Figure 10. The narrower band gap of friction-induced samples indicates that more photogenerated charges are separated, which can enhance the photocatalytic performance.

Investigations on other important factors for photocatalytic properties (Figure 11) indicate that water and light are determining factors in photocatalysis, which can be confirmed by contrasting experimental results of photocatalysis by adding light or deionized water. In addition, triethanolamine (TEOA), tert-butanol (t-Bu), and \( N_2 \) as photohole (h\(^+\)) scavenger, radical scavenger, and \( O_2 \) scavenger, respectively, are also involved in examining the effects of these scavengers on photocatalysis (pH = 3). As shown in Figure 11, TEOA can reduce the photocatalytic activity to a certain value, which means photoholes (h\(^+\)) play a major role in photocatalysis. Photoholes (h\(^+\)) with positive charge have a strong oxidizability, and water (H\(_2\)O) and hydroxyl (OH\(^-\)) can be oxidized and generate plenty of hydroxyl radicals (\( \bullet OH \)). On the other hand, some other organic materials absorbed on the surface of the photocatalyst can also be oxidized by photoholes (h\(^+\)) effortlessly. In addition, \( N_2 \) and t-Bu also have some negative influence on photocatalysis.

### 3.4. Suggested Mechanism for Photocatalytic Degradation

A possible mechanism is proposed to explain the high photocatalytic degradation efficiency of FA under visible-light
irradiation. As shown in Figure 12a, full-hollow MoS2 microspheres will be destroyed by friction. Plenty of curved nanoplatelets and nanosheets are generated subsequently, and curved structure allows multiple reflections of light, resulting in more efficient use of the light source. In addition, a layer-opened structure has more rim and edge sites produced by friction (Figure 12b), which are more active than basal surfaces. Bigger surface area and narrower bandgap of FA also lead to better photocatalytic properties. In Figure 12c, when FA is irradiated by a high-pressure mercury lamp, photoelectrons ($e^-$) jump from the valence band to conduction band, and therefore photoholes ($h^+$) are formed in the valence band. Moreover, the dye absorbed on the surface of MoS2 is stimulated to the excited state (dye*) by absorbing visible light, and then excited photoelectrons transfer to the conduction band of MoS2 from dye*. As photoelectrons ($e^-$) have high reductive ability and can be easily captured by O2, they eventually generate plenty of •OH. Water or other organic materials absorbed on the surface of the photocatalyst can be oxidized effortlessly by oxidizing photoholes ($h^+$), and •OH can also be generated by the reactions of $h^+$ and H2O or OH·.29 All the radicals can degrade most organic compounds to CO2 and H2O or other molecules.

3. CONCLUSIONS

The tribological performances and photocatalytic degradation effect of full-hollow and semihollow MoS2 microspheres on paraffin have been investigated. Both of them can exhibit good tribological behaviors, while full-hollow samples possess better photocatalytic efficiency. Full-hollow structure destroyed seriously by friction can result in many curved layer-opened fragments, and more rims and edges can perform more efficient photocatalytic reactions. Furthermore, our results also showed that acid solution can enhance photocatalytic efficiency greatly, which can be illustrated by a kinetic model. In a word, a green and sustainable strategy for the design of combining both lubricating additive and photocatalyst at different stages of LP mixtures has been suggested, which is just a preliminary attempt to move along the great idea of full-life-cycle green design of a lubricating oil system.
4. EXPERIMENTAL SECTION

4.1. Materials. Potassium thiocyanate (KSCN) and TEOA were purchased from Sinopharm Chemical Reagent Co., Ltd., and sodium molybdate (Na₂MoO₄) was provided by Shanghai Macklin Biochemical Co., Ltd. Tetrabutyl ammonium bromide (Bu₄NBr) and t-Bu were bought from Shanghai Lingfeng chemical reagent Co., Ltd., and MO, LP, absolute alcohol, and other common chemicals were commercially available and used without further purification.

4.2. Techniques. XRD was carried out by a Rigaku K/Max-γA X-ray diffractometer. SEM images were obtained on a FEI Inspect F50 instrument, and TEM images were obtained by a JEM-2100 instrument. UV−vis absorption spectra and UV−vis DR S were carried out on a UV-3600 spectrometer and a UV-2600 spectrometer, respectively. The tribological properties were explored by a MRS-10A four-ball testing machine under the condition of a constant load of 280 ± 4 N and a rotational speed of 1450 rpm at 75 ± 2 °C. Photocatalytic degradation tests were carried out on a photochemical reaction instrument (JT-GHX-AC, Hangzhou, China).

4.3. Preparation of MoS₂ Samples. Full-Hollow MoS₂ Microspheres. Amounts of 0.67 g of KSCN and 0.56 g of Na₂MoO₄ were dissolved in 65 mL of deionized water by magnetic stirring, and 0.23 g of Bu₄NBr was added into the obtained solution. Then the pH value of the above solution was adjusted to ∼1. The solution was transferred into a 100 mL Teflon-lined stainless autoclave and heated at 240 °C for 24 h. The obtained black precipitates were washed by deionized water and absolute ethanol and dried in vacuum at 60 °C for 20 h.

Semihollow MoS₂ Microspheres. The preparation was the same as that for full-hollow MoS₂, except that the pH value was controlled around 5.

4.4. Tribological Tests. Full-hollow and semihollow MoS₂ microspheres were used as lubricating additives for LP. The steel balls used in MRS-10A four-ball testing machine were made from GCr15 materials with hardness of 61−64 HRC and the roughness of Ra = 0.02 μm. The wear rate was determined by the AWSD (±0.01 mm) of the three bottom balls.

4.5. Photocatalytic Degradation Test. Certain amounts of reclaimed MoS₂ powders after friction tests were added in a mixture of 1.0 g of LP and 1.5 g of deionized water by ultrasonic dispersion for 40 min in the dark. Each system was put into a photochemical reaction instrument under continuous stirring and irradiated by a high-pressure mercury lamp. After photocatalytic reaction, MoS₂ was removed by centrifugation, and 10.0 g of dichloromethane was added in each solution. The mixtures were separated by a separating funnel. The concentration of LP was determined by a UV−vis spectrometer and repeated three times.

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Notes
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