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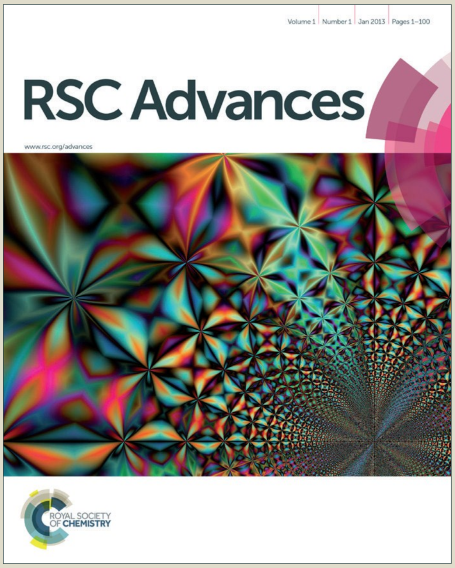

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Preparation and Tribological Properties of Organically Modified Graphite Oxide in Liquid Paraffin at Ultra-low Concentration

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Abstract

In recent years, two-dimensional (2-D) materials have attracted more and more attentions due to their 2-D anisotropy and special physicochemical properties. In this work, hexadecyltrimethoxysilane (HDTMS) and octyltrimethoxysilane (OTMS) were employed to modify graphite oxide (GO) nanosheets for the potential application as lubricating oil additives. The organically modified GO nanosheets were characterized by FTIR, XRD, TGA, UV-vis spectrum and SEM, which indicated that HDTMS and OTMS had successfully entered GO layers. Furthermore, the tribological properties of liquid paraffin containing HDTMS and OTMS modified GO were investigated by a four-ball friction testing machine. The results show that ultra-low concentration (0.008 wt%, the optimal value) of HDTMS or OTMS modified GO in liquid paraffin can reduce the friction coefficients of the mixtures by 34.0% and 15.5% respectively. HDTMS who possesses longer alkyl chain is better for improving the organic compatibilities and helps GO nanosheets to exfoliate more significantly and disperse in paraffin more homogeneously, thus corresponding to better effectiveness in decrease the friction coefficient.

Keywords: graphite oxide; organic silane; four-ball test; friction coefficient

1. Introduction

Two dimensional (2-D) nanomaterials have attracted more and more attentions due to their anisotropy and special physicochemical properties, which differ from those of their 3-D (bulk) counterparts [1-11]. As the most attractive 2-D nanomaterial discovered by Geim [12], graphene and related 2-D nanosheets possess remarkable electrical, mechanical, optical and thermal characteristics along with very high specific surface areas, which promise their potential applications in frontier research field such as nano-electronics, composite materials, sensors, catalysis, solar cells, hydrogen storage, drug delivery, batteries.

In the recent years, 2-D nanomaterials such as graphene and graphite oxide sheets are expected to play more important roles in the research field of friction and wear, which are the major causes for energy dissipation and material loss in mechanical processes and mechanical movements. From a microscopic point of view, there exist ridges, valleys, asperities and depressions at microscale or nanosacle on the surface of a pair of friction pairs, which will result in friction and wear. Generally, lubrication is a principal ways to decrease the friction and wear, thus improve energy efficiency and mechanical durability. Accordingly, more effective controls or reductions of the friction in moving mechanical systems are important for a sustainable consideration. As one kind of attractive lubricant materials or additives for lubricant oils, graphene and graphene related 2-D nanomaterials have been shown to be impermeable to liquids and gases, and they possess extreme mechanical strength which can suppress wearing. These properties make them attractive for tribological applications to achieve low friction and low wear regimes. On the other hand, if they are used as additives for lubricating oils, graphene and related 2-D nanomaterials should be more compatible with organic phase. In order to increase the organic compatibilities of these 2-D nanomaterials and achieve better dispersion and better effectiveness in improving the fiction properties, physical and/or chemical modification had been carried out: Zhang dispersed the oleic-acid modified graphene in PAO9 lubricating oil, and they found that the friction coefficient and wear scar diameter of the oil with addition of functionalized graphene were reduced by 17% and 14% respectively [13]. Lin modified graphene platelets by using of stearic acid and oleic acid to improve the stability in lube oil, and the results indicated that modified graphene possessed high load-carrying capacity, friction reduction and anti-wear ability [14]. Yang modified graphene by using 3-aminopropyltriethoxylsilane (APTS) and found that APTS grafted graphene in silica monoliths showed improved compressive strength [15]. Unfortunately, the friction improvements for lubricating oils containing organic silane modified graphene or related 2-D nanomaterials haven't been discussed yet to the best of our knowledge. In this work, hexadecyltrimethoxysilane (HDTMS) and octyltrimethoxysilane (OTMS) were employed to modify graphite oxide (GO), and the tribological properties of liquid paraffin containing OTMS and HDTMS modified GO were investigated.

2. Experimental

2.1 Materials

Natural flake grahite was purchased from Qingdao Jin Ri Lai Graphite Co., Ltd.

Concentrated sulfuric acid, potassium permanganate, 30% H_2O_2 and concentrated hydrochloric acid were obtained from shanghai reagent Co., Ltd., N,N-Dimethylformamide (DMF), triethylene-amine (TEA), anhydrous methanol, petroleum ether, liquid paraffin, octyltrimethoxysilane and hexadecyltrimethoxysilane were commercial available and they were of analytical grade. Ultra-pure water (resistivity 18.25 MΩ.cm) was used for the preparation and rinsing.

2.2 Preparations

In our work, GO was prepared by using Hummers method [16-17]. Typically, 3g graphite was gradually added into a beaker with 70 mL concentrated sulfuric acid which was pre-cooled. Afterwards, 9g $KMnO_4$ was gradually added into the mixture under an ice bath. The rate of addition must be controlled in order to keep the temperature lower than 20 ^oC. Successively, the suspensions were transferred into 40^oC water bath for 35min. Then 150 mL water was added and the solution was agitating for 15min at 95 ^oC. Additionally, 500 ml water and 15 mL H_2O_2 were added successively. The mixture was centrifuged and washed with 250 mL HCl aqueous solution. Finally, the products were centrifuged, washed and dried at 55 ^oC.

Octyltrimethoxysilane midified GO (OTMS-GO) and hexadecyltrimethoxysilane midified GO (HDTMS-GO) were prepared according to Ma’s method [13]. Typically, 200 mg GO was ultrasonically dispersed in 100 mL DMF. Afterwards, 0.3 mL TEA and 2.7 g HDTMS (or 1.8 g OTMS) were added into the mixture and stirred for 0.5 h. Then they were heated to 110 ^oC under continuous agitation for 24 h in Nitrogen atmosphere. In the end, the mixture was diluted with methanol to dissolve the unreacted HDTMS (or OTMS), followed by being filtrated using 0.45μm PTFE ultrafiltration membrane. The filter cake was washed with methanol and water sequentially at least four times since then. The resultant was dried in oven at 60 °C.

The liquid paraffin mixtures with 0.004 wt%, 0.008 wt%, 0.01 wt%, 0.015 wt%, 0.02 wt% of OTMS-GO (or HDTMS-GO) were prepared and their tribological properties were tested by a four-ball friction testing machine.

2.3 Techniques

XRD patterns were obtained by using a Rigaku K/max-γA X-ray diffractometer with a Cu Ka (λ=1.5415 Å) at the scanning rate of 0.02°/s; Infrared spectra were obtained by using a MAGNA-IR 750 spectrometer. Thermal gravimetric analysis (TGA) was performed on a Netzsch STA-409c Thermal Analyzer under a 50×103 mm^3/min nitrogen or air flow with the heating rate of 10 °C/min; Scanning electron microscopy (SEM) was carried out on the FEI Inspect F50 instrument.

The tribological properties of the liquid paraffin mixtures were tested by a MRS-10A four-ball testing machine (load: 300 N ± 4 N, rotating speed: 1450rpm, oil temperature: 75±2 °C). The steel balls were made from GCr15 materials with hardness of 61-64 HRC with the roughness of Ra=0.02. Before each test, the steel balls were cleaned by petroleum ether several times. Each test was performed three times under the same condition and the friction coefficient value was calculated via averaging of the real-time friction coefficient over the entire testing duration, as far as each time. The final friction coefficient value together with error bars was obtained by calculating average value of FC of different times. The acquirement of wear scar diameter (WSD) was by calculating the average of the three bottom balls at bottom.

The dispersion stability of OTMS-GO and HDTMS-GO in paraffin was evaluated by an Ultraviolet-visible spectrometer. OTMS-GO and HDTMS-GO were dispersed in liquid paraffin by ultra-sonication (150W, 1h), and the mixture's absorbance was measured immediately after ultra-sonication. Successively, the mixture was centrifuged at 1000rpm and the supernatant was decanted at 20-min intervals to evaluate absorbency by an ultraviolet-visible spectrometer.

3. Results and discussion

3.1 Synthesis and characterizations of OTMS-GO and HDTMS-GO

FTIR spectra of GO, OTMS-GO and HDTMS-GO are showed in **Fig.1**. For GO, the wide adsorbing peak at 3400 cm^{-1} is attributed to the stretching vibration of hydroxyl group (O-H) from GO and water. Other adsorbing peaks corresponding to the characteristic functional groups of GO are listed as followings: C-O-C (1057cm^{-1}), C-O (1223 cm^{-1}), C=C (1620cm^{-1}), C=O (1735cm^{-1}). After being modified by organic OTMS and HDTMS, new adsorbing peaks at 2919cm^{-1} and 2856cm^{-1} related to C-H vibration from alkyl chain of OTMS or HDTMS can be found in the spectra of OTMS-GO and HDTMS-GO, while the adsorbing peak corresponding to O-H vibration disappears. Comparing with GO, OTMS-GO as well as HDTMS-GO shows sharp adsorbing peaks at 1000~1100 cm^{-1} due to Si-O-C/Si-O-Si groups. In addition, the adsorbing peak corresponding to carbonyl group (C=O) is weakened, which illustrates that partial reduction happens simultaneously during modifications. These changes can give evidence for OTMS and HDTMS entering GO layers.

Fig. 2 shows X-ray diffraction patterns of GO, OTMS-GO and HDTMS-GO. For GO, (001) diffraction peak can be found at 12.2°, corresponding to the interlayer space of 0.722 nm. At the same time, there are no other diffraction peaks, indicating that graphite has been oxidized. For HDTMS-GO, the diffraction peak shifts to 5.7° and the corresponding interlayer space is 1.55nm, which indicates that HDTMS has entered GO sheets and exfoliated structure has been formed. In addition, a sharp peak at 21.3° is very close to that of graphene indicating a recovery of graphitic structure [18]. For OTMS-GO, the peak at 21° was broadening and lower than HDTMS-GO which indicates that OTMS-GO possesses smaller interlayer space.

Thermal gravimetric analysis (TGA) results of GO, OTMS-GO, and HDTMS-GO are showed in **Fig.3**. The curve for GO shows three degradation steps: the first step of weight loss below 100 °C attributes to the loss of water in the GO; the second step between 160 °C and 230 °C corresponds to the condensation between carboxyl and hydroxyl groups; the mass loss occurred beyond 230 °C is related to the decomposition of other stable functional groups. For the TGA curve for HDTMS-GO, there is little weight loss from 160 °C to 230 °C, manifesting the removal or converting of the main oxygen-containing moieties after modification by HDTMS. The slight weight loss from 230 °C to 420 °C is owing to the loss of unreacted oxygen-containing species on the organic sheets. Then there is a sharp mass drop (~60.4%) from 420 °C to 550 °C which is caused by the thermal decomposition of HDTMS chains bonded on GO surface [19]. As for the curve for OTMS-GO, slight mass loss from 212 °C to 250 °C can be observed which is due to the pyrolysis of oxygen-containing groups. Additionally, a sharp mass loss from 250 °C to 550 °C is observed in OTMS-GO curve. Comparing TGA curves for HDTMS-GO and OTMS-GO, the residue for HDTMS-GO is less than that for OTMS-GO, which is accounted for the difference in the length of alkyl chain in HDTMS and OTMS.

The morphology of GO, OTMS-GO and HDTMS-GO are characterized by SEM, as showed in **Fig.4**. In these SEM images of OTMS-GO and HDTMS-GO, wrinkled or folded platelets can be observed, which due to the functional groups or organic chains introduced to the GO surface. OTMS-GO and HDTMS-GO samples exhibit fluffy accumulation because of the entrance of long alkyl chains into grapheme layers.

3.2 Tribological properties of OTMS-GO and HDTMS-GO in liquid paraffin

Fig.5 shows the changing tendencies of the friction coefficients of pure paraffin and its mixtures versus the concentration of HDTMS-GO. It can be found that the friction coefficient decreases with HDTMS-GO concentration increasing when it is lower than 0.008 wt%. On the contrary, the friction coefficient increases with HDTMS-GO concentration increasing continuously when its concentration is bigger than 0.008 wt%. Based on our experiments, the optimal concentration for the lowest friction coefficient is 0.008 wt%, and the corresponding value of friction coefficient is 0.0626, which is decreased by 34.0% in comparison with that obtained in pure paraffin. Similarly, **Fig.6** gives the variations of friction coefficient versus the concentration of OTMS-GO. The optimal concentration for the lowest friction coefficient is also 0.008 wt%, and the corresponding value of friction coefficient is 0.826, which is decreased by 15.5% in comparison with that obtained in pure liquid paraffin.

Evidently, ultra-low concentration (0.008 wt%) of HDTMS-GO or OTMS-GO in liquid paraffin can reduce the friction coefficient effectively. As we known, HDTMS-GO and OTMS-GO contain long organic chains in their interlayer structures, which can improve their organic compatibilities with paraffin and make organically modified nanosheets to exfoliate and disperse in the base oils more homogeneously. It is good for reducing the friction coefficient of paraffin mixtures.

3.3 Mechanisms for the improvements in friction properties

As additives, HDTMS-GO works better than OTMS-GO in reducing the friction coefficient of pure paraffin, although the optimal additions of them are both at 0.008 wt%. At the same concentration, the only difference between HDTMS-GO-paraffin system and OTMS-GO-paraffin system is the length of alkyl chains, which plays an important role in the modified additives. Obviously, longer alkyl chains are useful for organic compatibilities which can help GO sheets to exfoliate more significantly and disperse more homogeneously. The detailed mechanisms for the improvements in friction can be suggested as followings:

On one hand, organic silanes (HDTMS and OTMS) work as a friendly media between liquid paraffin and GO nanosheets. HDTMS and OTMS who contain organic chains can improve the organic compatibilities of GO nanosheets with liquid paraffin and thus result in better dispersion stability and uniformity, which is better for reducing the friction coefficient. In our experiment, the dispersion stability of HDTMS-GO-paraffin system is better than that of OTMS-GO-paraffin system, which have been checked and testified by UV-vis spectrophotometry. **Fig.7** shows the dispersion stability curves of paraffin containing 0.03 wt% of HDTMS-GO and OTMS-GO respectively. The relative concentration can be measured by normalizing the UV absorbance of paraffin mixtures, calculating the ratio of UV absorbency of the supernatant fluid at each measurement period which is divided by the initial intensity of the suspension. According to Lambert-Beer law, UV-vis absorbance is proportional to concentration. The value of the relative concentration lower than 1.0 means particle sedimentation and the slope is regarded as the rate of precipitation. Here centrifugation between two consecutive UV-vis absorbance measurements is to speed up the particle sedimentation and amplify the changing tendency of dispersion stability. It can be found that both HDTMS-GO and OTMS-GO show rapid sedimentation at the first stage, followed by relatively mild decreasing with the centrifugation time going. However, the suspension of HDTMS-GO showed better dispersion stability at each stage. When organically modified GO are dispersed in oil, the alkyl chains can generate steric hindrance against accumulations, helping to prevent modified GO sheets from each other. As for HDTMS-GO suspensions, longer alkyl chains generate more effective steric hindrance effect than OTMS-GO, thus resulted in better dispersion stability.

On the other hand, organic silane (HDTMS and OTMS) works as an effective assistant for GO nanosheets in improving the friction properties for paraffin. **Fig.8** gives the real-time changing tendency of friction coefficients for paraffin, HDTMS-GO-paraffin system, HDTMS-paraffin system, OTMS-GO-paraffin system and OTMS-paraffin system respectively. At the very beginning, the real-time friction coefficient of each system starts from a similar level. With time passing, the friction coefficient of OTMS-GO-paraffin system increases gradually, while that of HDTMS-GO-paraffin system still keeps at a low level almost constantly. Comparatively speaking, the friction coefficient changing tendencies for HDTMS-paraffin system and OTMS-paraffin system are similar with that of pure paraffin. Obviously, HDTMS and OTMS themselves cannot improve the friction properties of paraffin, at least at this concentration. As we known, HDTMS and OTMS are two kinds of organic molecules with middle molecular weights, and their viscosities are smaller than liquid paraffin. So the viscosity of the mixture will be decreased slightly, thus friction coefficient are slightly improved from 0.0999 (paraffin) to 0.1006 (HDTMS-paraffin system) and 0.1015 (OTMS-paraffin system) respectively, as showed in the inserted picture of **Fig.8**.

The process for HDTMS-GO and OTMS-GO improving friction properties of paraffin mixtures can be suggested and illustrated as **Fig.9a**: HDTMS-GO nansheets or OTMS-GO nansheets enter the gap between the contact surfaces of the upper and lower steel balls with the flow of paraffin. Shedding or curling of GO nanosheets will occur under compressive stress and shear stress in four ball friction test. The shedding GO nanosheets will fill in the sunken areas of surface of the friction pair, thus decrease the friction coefficient. Since alkyl chains of HDTMS and OTMS can help GO nanosheets to exfoliate, the interaction force between organically modified GO nanosheets are smaller than that without organic modification. That is to say, HDTMS-GO nanosheets and OTMS-GO nanosheets are easily to slide and shed, which is better for antifriction. **Fig.9b** gives the wear scar photos of the steel balls after friction tests for 0.008 wt% HDTMS-GO-paraffin system and 0.008 wt% OTMS-GO-paraffin system respectively. The wear scar diameters obatined from HDTMS-GO-paraffin system are smaller than that from OTMS-GO-paraffin system. Obviously, longer alkyl chains can help GO sheets to exfoliate more significantly and the force between the layers is smaller. In the same amount of addition of modified GO nanosheets, the additive modified by HDTMS can achieve better effectiveness in antifriction.

4. Conclusions

In summary, HDTMS and OTMS-GO modified GO nanosheets were prepared and used as additives for liquid paraffin to improve its tribological properties. The results showed that ultra-low concentration (0.008 wt%) of HDTMS-GO and OTMS-GO nanosheets in liquid paraffin can significantly reduce the friction coefficient of base oil by 34.0 % and 15.5% respectively. HDTMS who possesses longer alkyl chain is better for GO nanosheets to achieve better organic compatibilities in liquid paraffin and help them to exfoliate more significantly and disperse more homogeneously, thus corresponding to better effectiveness in reducing the friction coefficient of pure paraffin.

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Figure captions

Figure 1: FTIR spectrum of (a) graphite oxide (GO), (b) octyltrimethoxysilane modified graphite oxide (OTMS-GO) and (c) hexadecyltrimethoxysilane modified graphite oxide (HDTMS-GO)

Figure 2: XRD patterns of (a) hexadecyltrimethoxysilane modified graphite oxide (HDTMS-GO), (b) octyltrimethoxysilane modified graphite oxide (OTMS-GO) and (a) graphite oxide (GO)

Figure 3: Thermal gravimetric analysis (TGA) curves of (a) graphite oxide (GO), (b) octyltrimethoxysilane modified graphite oxide (OTMS-GO) and (c) hexadecyltrimethoxysilane modified graphite oxide (HDTMS-GO)

Figure 4: Scanning electron microscopy (SEM) images of (a) graphite oxide (GO), (b)

octyltrimethoxysilane modified graphite oxide (OTMS-GO) and (c) hexadecyltrimethoxysilane modified graphite oxide (HDTMS-GO)

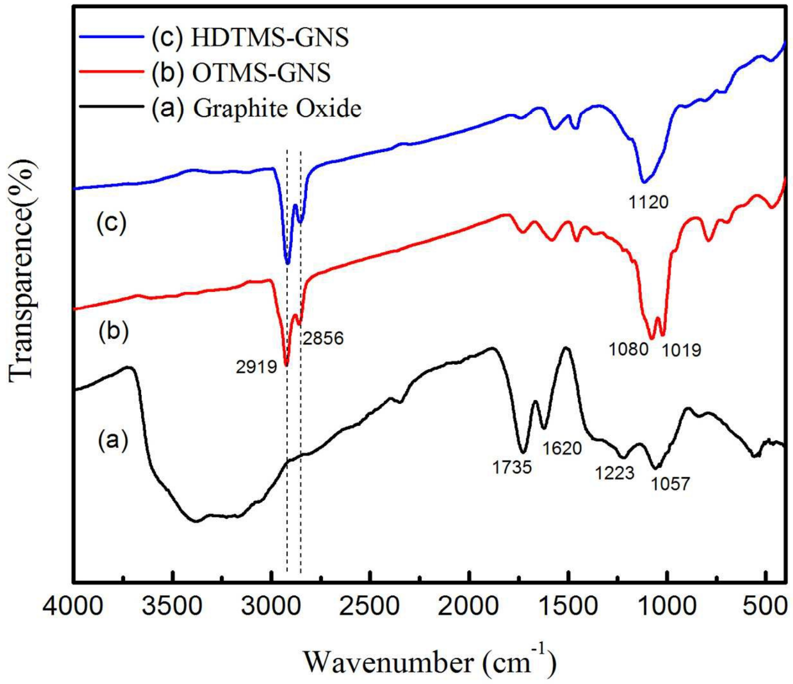
Figure 5: Friction coefficient variation versus the concentration of hexadecyltrimethoxysilane modified graphite oxide (HDTMS-GO).

Figure 6: Friction coefficient variation versus the concentration of octyltrimethoxysilane modified graphite oxide (OTMS-GO).

Figure 7: Dispersible and stable ability of HDTMS-GO and OTMS-GO as additives in lube oil determined by ultraviolet-visible spectrometer.

Figure 8: Real-time friction coefficient variation for (a) paraffin, (b) 0.008 wt% HDTMS-GO-paraffin system, (c) 0.008 wt% HDTMS-paraffin system, (d) 0.008 wt% OTMS-GO-paraffin system and (e) 0.008 wt% OTMS-paraffin system respectively. The inserted picture is the average friction coefficient for (a), (b), (c), (d), (e).

Figure 9: (a) The illustrated process for HDTMS-GO and OTMS-GO improving friction properties; (b) the wear scar photos of the steel balls after friction test for 0.008 wt % HDTMS-GO-paraffin system (H-1, H-2, H-3) and 0.008 wt % OTMS-GO-paraffin system (O-1, O-2, O-3), and the scale bar in each photo is 0.2 mm



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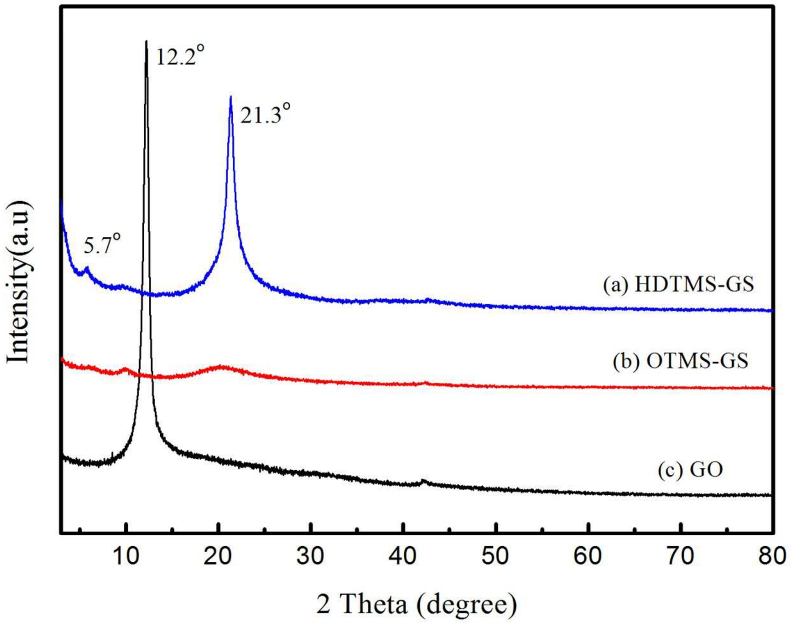

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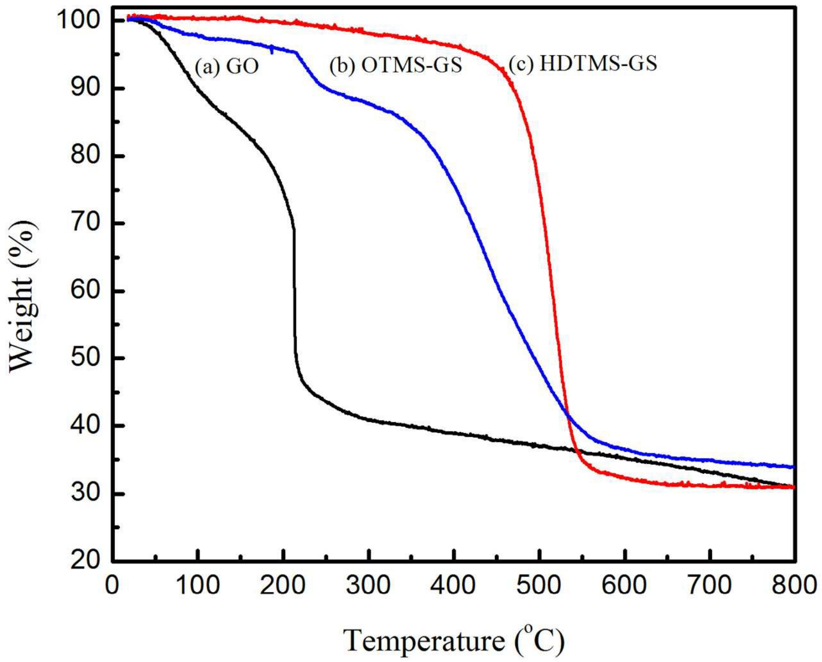

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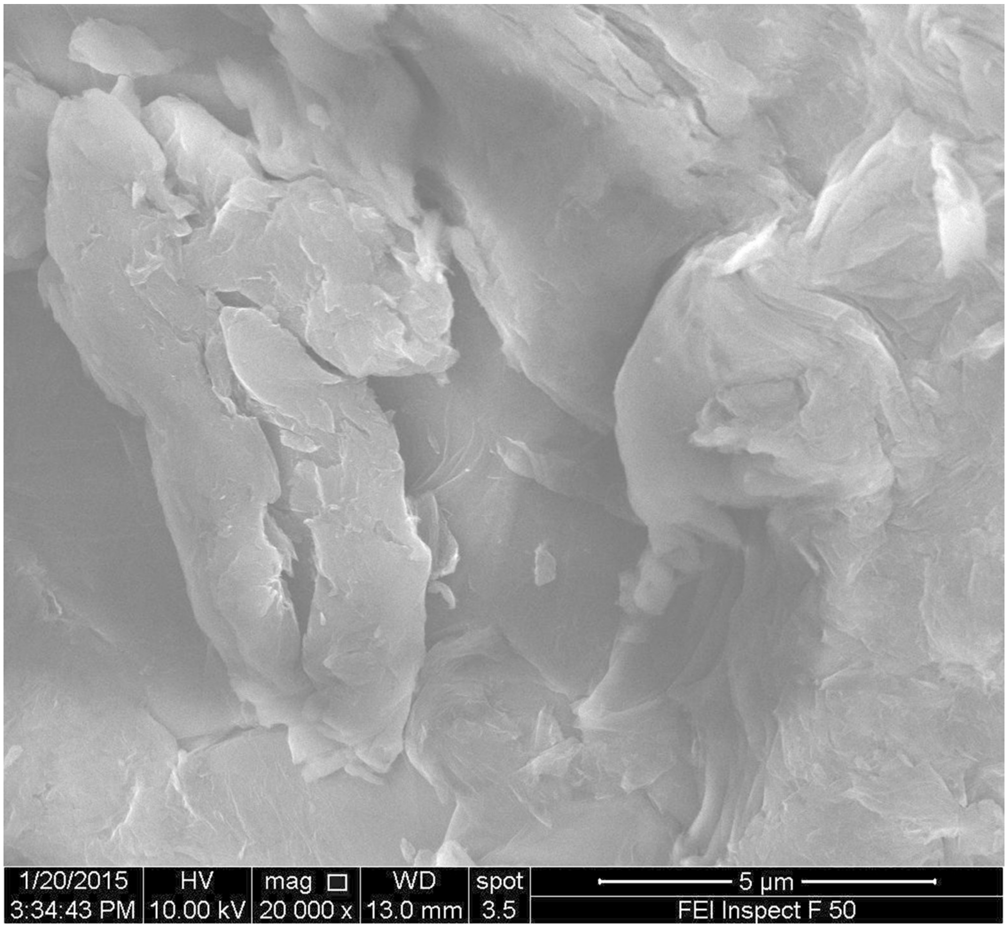

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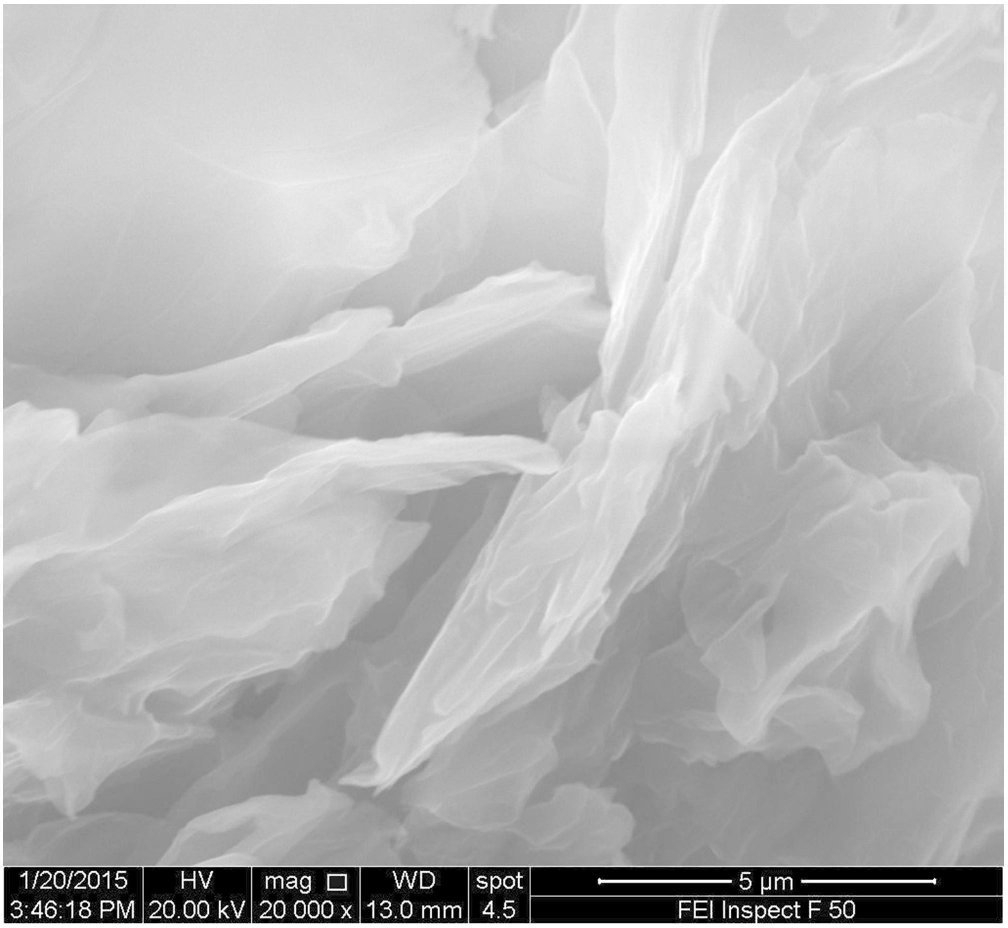

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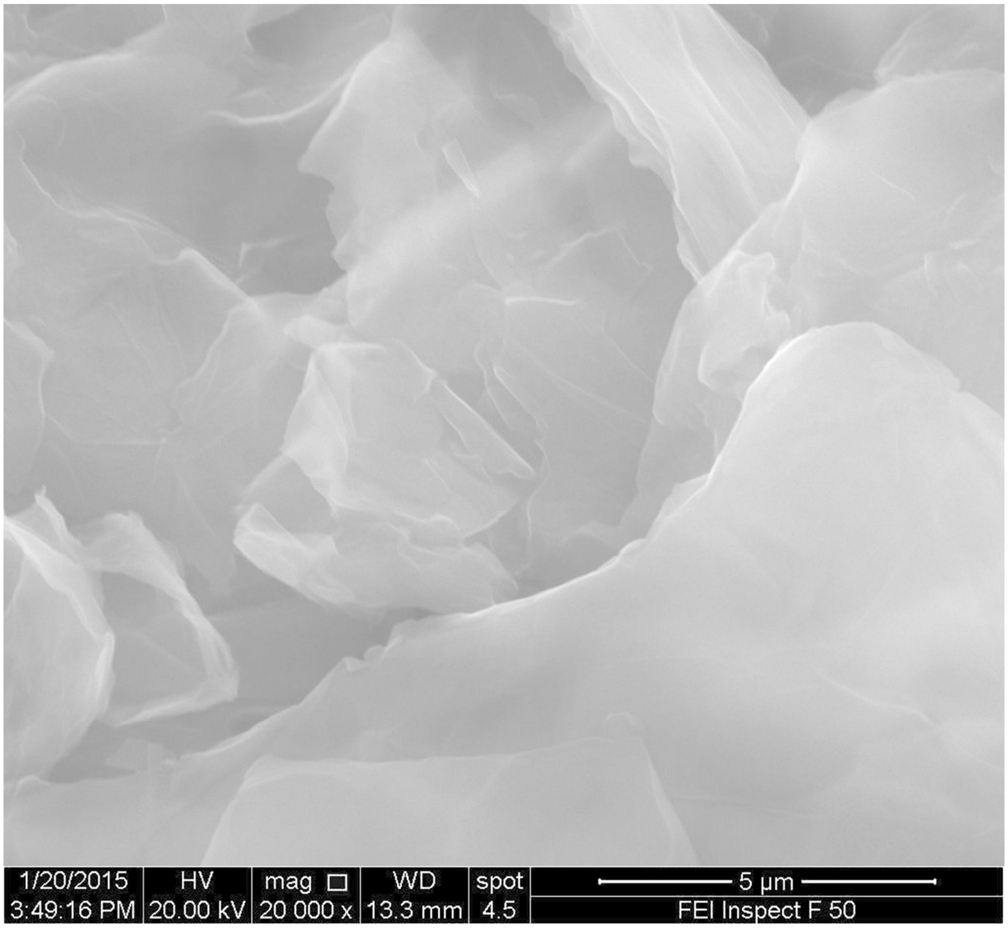

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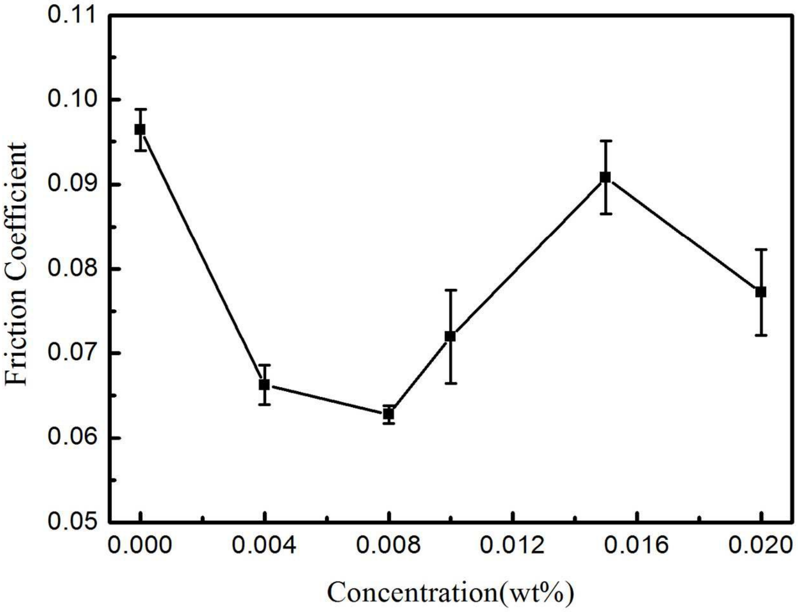

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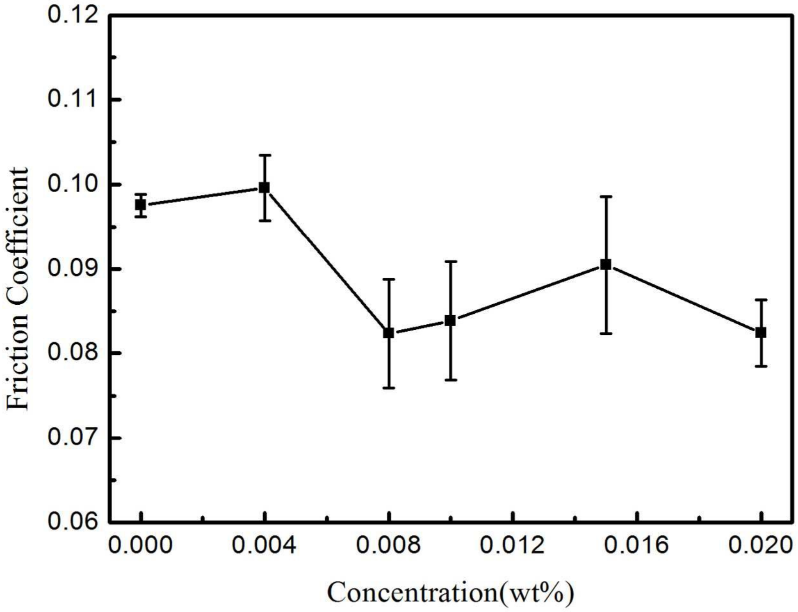

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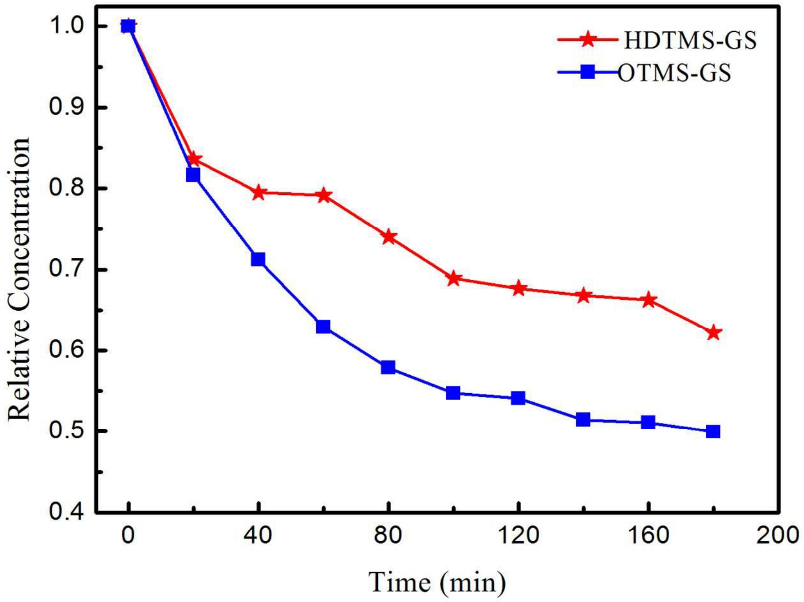

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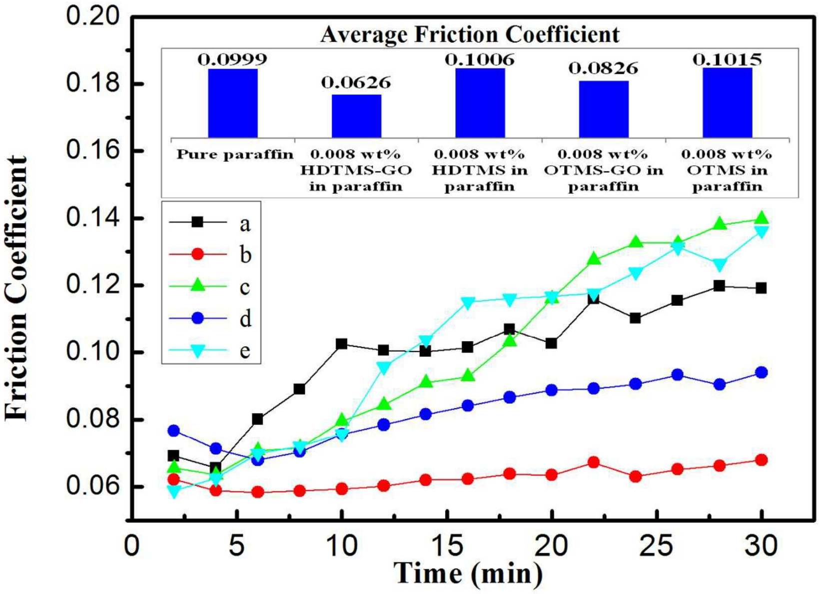

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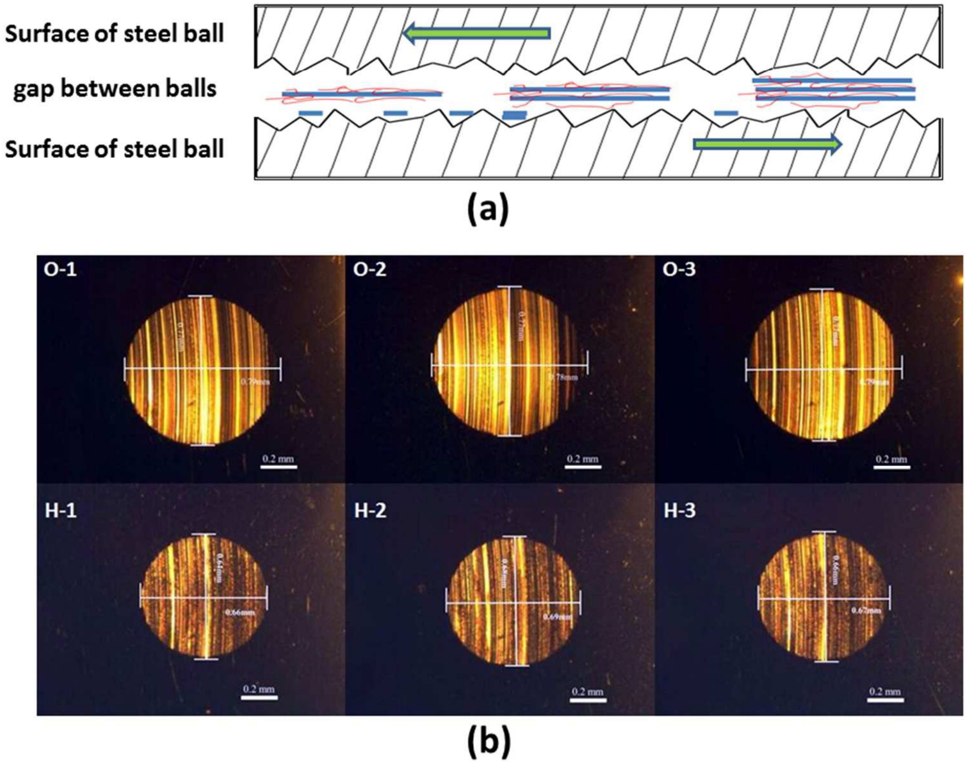
Surface of steel ball
gap between balls
Surface of steel ball
(a)
O-1
0.77mm
0.79mm
0.2 mm
O-2
0.77mm
0.78mm
0.2 mm
O-3
0.79mm
0.2 mm
H-1
0.64mm
0.66mm
0.2 mm
H-2
0.69mm
0.2 mm
H-3
0.66mm
0.67mm
0.2 mm
(b)