



# Preparation of graphene-MoS<sub>2</sub> hybrid aerogels as multifunctional sorbents for water remediation

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**ABSTRACT** The increasing demand of clean water and effective way to recycle industrial wastewater has offered a new application for carbon-based three-dimensional (3D) porous networks as sorbents due to their superior sorption abilities. Through the surface modification and hybridization with functional materials, the physical and chemical properties of the 3D carbon-based materials can be engineered. In this work, graphene-MoS<sub>2</sub> aerogels (GMAs) with bulky shape are synthesized *via* a one-pot hydrothermal method. The obtained GMAs show quick sorption rate and high sorption capacity towards a wide variety of contaminants. The sorption covers not only organic solvents or organic dyes, but also toxic heavy metals ions such as Hg<sup>2+</sup> and Pb<sup>2+</sup>. More importantly, the sorption capacity towards metal ions can be optimized by simply changing the loading amount of MoS<sub>2</sub>.

**Keywords:** Graphene, MoS<sub>2</sub>, aerogels, multifunctional sorbents, water remediation

## INTRODUCTION

Carbon-based three-dimensional (3D) architectures have attracted considerable attention in the past two decades due to their unique properties, such as huge surface area, interconnected porous structures, and macroscopic bulky shape, enabling them to be one of the most promising materials for water treatment [1–6]. However, the carbon-based 3D porous networks have limited compatibility and most of them only show affinity to certain category of contaminants [7–11]. This limitation hinders

the real industrial wastewater treatment since it normally contains diverse types of pollutants (e.g., heavy metal ions, dyes, oils, and other organic compounds) [12–15]. Therefore, it is desired to develop new hybrid carbon-based sorbents, i.e., porous carbon networks incorporated with other functional materials to enhance their sorption capabilities and simultaneously absorb multiple types of pollutants from wastewater [16–18].

Recent studies have demonstrated that the graphene-like two-dimensional (2D) transition metal dichalcogenides (TMDs) are promising sorbents for removal of contaminants due to their unique layered structures and large surface area [19–21]. For example, Chao *et al.* [22] used ultra-thin MoS<sub>2</sub> nanosheets to remove toxic organics. Our previous work also indicated that organic dyes could be absorbed onto the surface of MoS<sub>2</sub> nanosheets to facilitate the photocatalytic degradation of dyes by TiO<sub>2</sub> nanobelts [23]. However, MoS<sub>2</sub> nanosheets cannot be completely recycled from treated water, which might lead to the secondary environmental pollution. In contrast, the carbon-based 3D materials in macroscopically bulky shape are easy to be collected and recycled. Therefore, it is reasonable to combine MoS<sub>2</sub> with 3D carbon networks to prepare hybrid materials, which could have both excellent sorption performance and good recyclability.

Here, a series of graphene-MoS<sub>2</sub> aerogels (GMAs) with porous architectures and bulky shapes are prepared *via* a facile hydrothermal method using graphene oxide (GO)

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as carbon source. The prepared GMAs are then used as multifunctional sorbents for water remediation, which exhibit outstanding sorption performance for organic compounds in wastewater. In addition, GMAs also show good sorption ability towards both heavy metal ions and dyes.

## EXPERIMENTAL SECTION

### Preparation of GO

GO was produced according to the Hummers method [24]. Typically, 4 g of graphite powders, 2 g of sodium nitrate and 92 mL of sulfuric acid (98 wt.%) were added into a 500-mL flask and mixed together. To prevent the temperature exceeding 20°C, the flask was immersed into an ice bath and then 12 g of potassium permanganate was slowly added into it. After that, the flask temperature was increased to 35°C and kept for one day, followed by addition of 184 mL deionized (DI) water. The obtained suspension was heated to 98°C for 15–30 min to increase the oxidation degree of the GO product. After mixing with 560 mL of DI water, the brown suspension was further treated with 12 mL of hydrogen peroxide (30 wt. %). Finally, the mixture was separated by filtration or centrifugation and washed several times using diluted hydrochloric acid (5 vol%) and DI water followed by drying at 40°C in the vacuum oven for two days.

### Preparation of aerogels

After 10 mL of GO solution (2.5 mg mL<sup>-1</sup>) was mixed with 15 mg of thioacetamide (C<sub>2</sub>H<sub>5</sub>NS) and 30 mg of sodium molybdate (Na<sub>2</sub>MoO<sub>4</sub>). The suspension was sonicated for 10 min and placed into a polytetrafluoroethylene (PTFE)-lined autoclave. Then the autoclave was heated to 200°C for one day. After naturally cooled down to room temperature, the black product was washed with DI water several times and subsequently freeze-dried for two days to form porous aerogel structure, referred as GMA-1. As for the GMA-2, GMA-3 and GMA-4, all experimental procedures were similar with those for preparation of GMA-1, except for the amounts of C<sub>2</sub>H<sub>5</sub>NS and Na<sub>2</sub>MoO<sub>4</sub>. 60 mg of C<sub>2</sub>H<sub>5</sub>NS and 30 mg of Na<sub>2</sub>MoO<sub>4</sub> were used for preparation of GMA-2. 90 mg of C<sub>2</sub>H<sub>5</sub>NS and 45 mg of Na<sub>2</sub>MoO<sub>4</sub> were used for preparation of GMA-3. 120 mg of C<sub>2</sub>H<sub>5</sub>NS and 60 mg of Na<sub>2</sub>MoO<sub>4</sub> were used for preparation of GMA-4. As for the pure reduced graphene oxide aerogel (PGA), the experimental procedures were similar with those for preparation of GMAs, but without adding any C<sub>2</sub>H<sub>5</sub>NS or Na<sub>2</sub>MoO<sub>4</sub>. In order to measure the loading amount of

MoS<sub>2</sub> in GMAs, GMAs were immersed into Aqua Regia overnight, and the corresponding solutions were diluted and filtrated before the inductively coupled plasma-optical emission spectrometry (ICP-OES) measurements.

### Characterization of materials

Scanning electron microscopy (SEM) images were collected using a JSM-7600F field-emission SEM (FESEM) operated at 5 kV. The transmission electron microscopy (TEM) samples were prepared by dropping about 5 μL sample solutions on copper grid (200 mesh) with lacey carbon-coating and then drying at ambient condition. High-resolution TEM (HRTEM) and high-angle annular dark-field scanning TEM (HAADF-STEM) images were collected using a JEM 2100F transmission electron microscope operated at an accelerating voltage of 200 kV. The X-ray photoelectron spectroscopy (XPS) data were collected using a theta probe electron spectrometer (ESCA-Lab-200i-XL, Thermo Scientific). The XPS samples were prepared by drying the droplets of sample solutions on Si/SiO<sub>x</sub> substrates under ambient condition. Ion concentrations were measured using an ICP-OES (Perkin Elmer). UV-vis spectra were collected using a Shimadzu UV-2550 spectrometer.

### Sorption capacity measurements for oils and organics

The obtained GMAs were immersed into the oils and organic solvents for 2 min before weight measurements. Note that these measurements should be done in a short time to avoid the evaporation of absorbed organics. The weights of GMAs before and after sorption tests were recorded to calculate the weight gain (g g<sup>-1</sup>), which was defined as the mass of contaminants absorbed per unit mass of sorbent. The recyclability test was performed with the distillation method. For example, after hexane (boiling point 68.5°C) was fully absorbed into GMA, the GMA was heated to 80°C to evaporate the absorbed hexane. The residual mass was then weighed. This cycle was repeated 10 times.

### Sorption capacity measurements for organic dyes and heavy metal ions

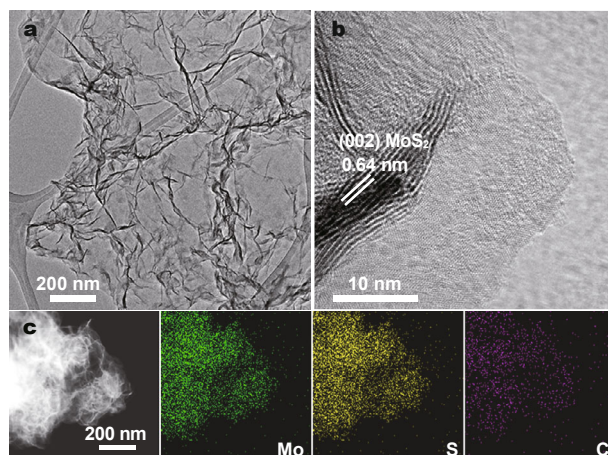
Various amounts of dyes (methyl orange (MO) and methylene blue (MB)) and salts of heavy metals (HgCl<sub>2</sub> and PbCl<sub>2</sub>) were dissolved in DI water with a specific concentration. A piece of GMA was immersed into the solution for one to four days. The dye concentrations in the solution were then measured with a UV-vis spectrophotometer, and the concentrations of heavy metal ions in solutions were measured with ICP-OES. The weight

gains ( $\text{mg g}^{-1}$ ) were calculated.

## RESULTS AND DISCUSSION

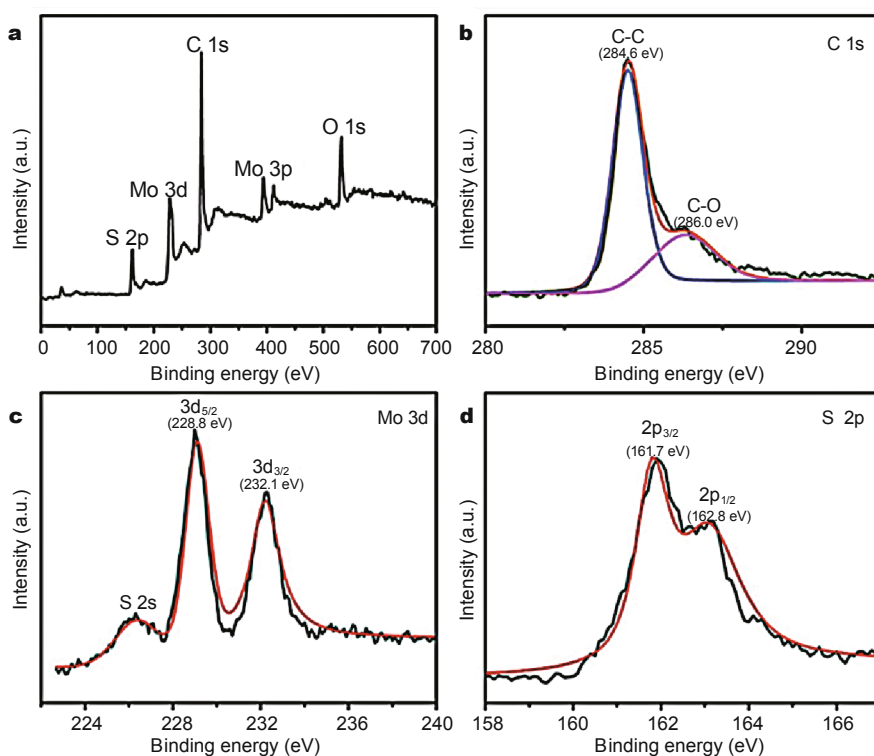
GMA with black spongy texture were prepared *via* a simple one-pot hydrothermal method (see the EXPERIMENTAL SECTION for details). Briefly, after the  $\text{C}_2\text{H}_5\text{NS}$  (used as sulphur source) and  $\text{Na}_2\text{MoO}_4$  (used as molybdenum source) were added into a GO solution, the mixed solution was transferred into a PTFE-lined autoclave and then heated at  $200^\circ\text{C}$  for one day. The final products were washed and subsequently freeze-dried for two days to form porous GMA, referred as GMA-1. A typical TEM image of GMA-1 confirms the ultrathin nature of obtained hybrid sheets (Fig. 1a). These nanosheets are highly wrinkled, which are different from the reduced GO (rGO) nanosheets (Fig. S1, Supplementary information). As shown in the HRTEM image of the obtained hybrid nanosheets (Fig. 1b), the lattice spacing of 0.64 nm corresponds to the (002) planes of  $\text{MoS}_2$  [25]. The HAADF-STEM image and the corresponding energy dispersive spectroscopy (EDS) mappings of GMA-1 (Fig. 1c) reveal the uniform distribution of Mo and S on rGO nanosheets.

The electron binding energy of GMA-1 was analyzed by



**Figure 1** (a) Low-magnification TEM image, (b) HRTEM image and (c) STEM image of GMA-1 and its corresponding EDS elemental mapping of Mo, S, and C.

XPS (Fig. 2). The XPS survey spectrum contains several sharp peaks (Fig. 2a), suggesting the presence of Mo, S, and C elements. The electron at C 1s level can be observed from two peaks. As shown in Fig. 2b, the main peak at around 284.6 eV matches the C–C bonds of rGO nanosheets, while the lower peak at 286.0 eV corresponds

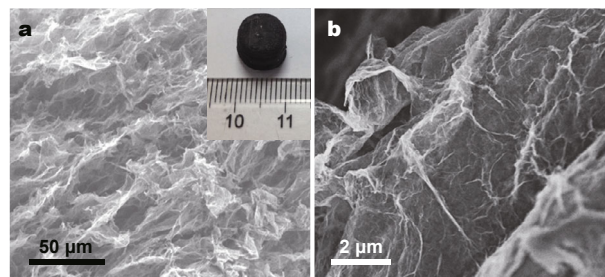


**Figure 2** XPS spectra of GMA-1 showing its electronic binding energy. (a) XPS survey spectrum of GMA-1. (b–d) XPS spectra of C 1s (b), Mo 3d (c), and S 2p (d).

to C–O bond [26]. In addition, the chemical states of Mo and S in GMA-1 were also investigated (Fig. 2c, d). The peaks of Mo 3d<sub>3/2</sub> and Mo 3d<sub>5/2</sub> were located at 232.1 and 228.8 eV, respectively, revealing the characteristic of Mo<sup>4+</sup> state in MoS<sub>2</sub> (Fig. 2c) [27]. The peaks at 161.7 and 162.8 eV in S 2p spectrum separated by a spin-orbit splitting of 1.1 eV were attributed to the S<sup>2-</sup> of MoS<sub>2</sub> (Fig. 2d) [28]. All the aforementioned results confirm the formation of MoS<sub>2</sub> on rGO nanosheets.

The morphology of GMA-1 was further investigated by SEM (Fig. 3). Microscopic pores with interconnected networks were clearly observed in the prepared GMA-1. Importantly, a mirror reflection was observed when a piece of GMA-1 was immersed into water (Fig. S2a), indicating its high hydrophobicity [29], which was further confirmed by the water contact angle of 130°±3° measured on the GMA-1 surface (Fig. S2b). Interestingly, the GMA-1 can be shaped into a cylinder in centimeter scale (inset of Fig. 3a), making it easy to be collected and recycled after it is used for water treatment which is shown below [30].

Given its 3D porous structure and surface hydrophobicity, the prepared GMA-1 is an ideal candidate for the removal of contaminants, such as oils and organic solvents, from wastewater. The sorption capacities of GMA-1 were evaluated by sorption of a series of organic liquids, such as chloroform, benzyl alcohol, heptane, and acetone. The GMA-1 shows high sorption capacities towards them. Especially, about 65 times its weight of chloroform can be absorbed (Fig. S3). As known, surface area, porosity, and surface functionality are among the most important factors which affect the sorption capacity of sorbents [31]. There are two main sorption mechanisms for GMAs during the liquid sorption. One is the surface adsorption, i.e., the organic liquids are adsorbed onto the surface of sorbents. The other one is absorption. It is believed that the latter gives greater contribution to a high sorption capacity in aerogels [31]. Note that when a piece of GMA-1 (15 mg) was in contact with cyclohexane (200 mg, dyed with Sudan red 5B), it quickly absorbed the cyclohexane within 9 s (Fig. S4a–d). Moreover, the obtained GMA-1 can also selectively absorb heavy organics under water. As shown in Fig. S4e–h, chloroform (300 mg, dyed with Sudan red 5B) at the bottom of water was entirely absorbed into a piece of GMA-1 (11 mg) within 12 s. In addition to its high sorption capacity and fast sorption rate, GMA-1 also showed good recyclability towards organic solvents. No obvious change in the sorption ability of GMA-1 was observed even after 10 cycles of tests using the distillation method. After each



**Figure 3** (a, b) SEM images of GMA-1 under different magnifications. Inset in (a): photograph of GMA-1.

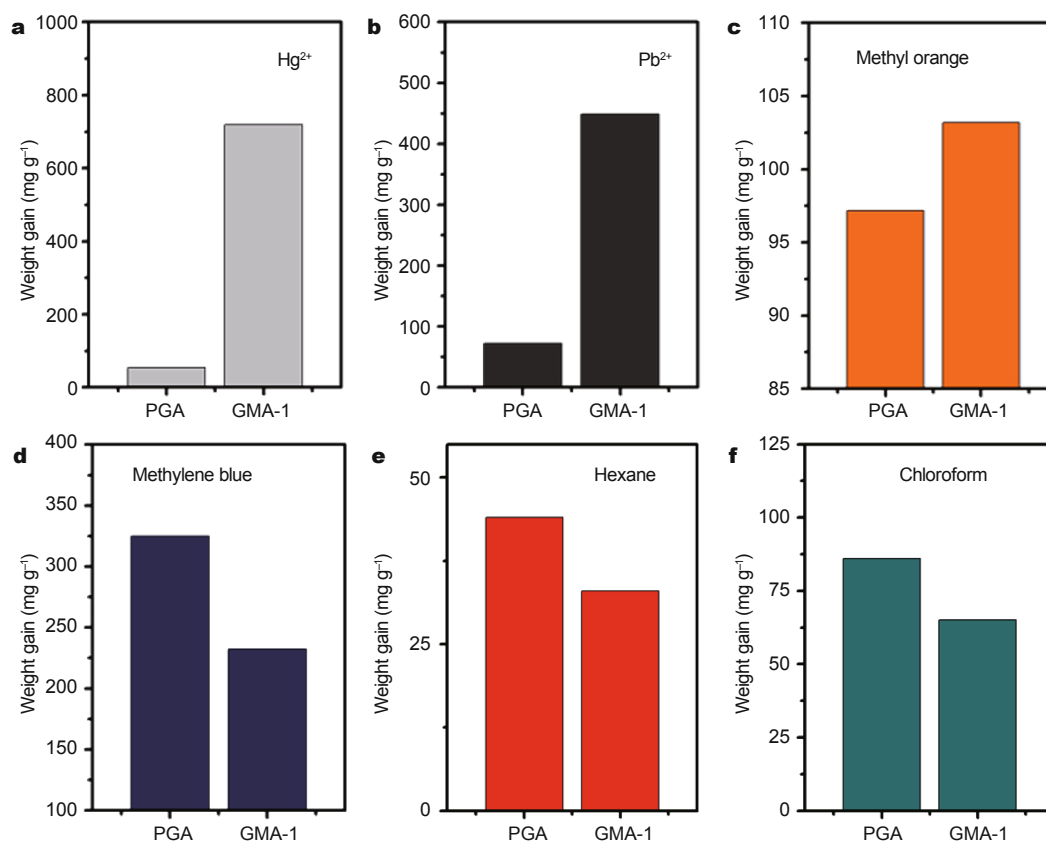
cycle, the weight of residual hexane in the GMA-1 is less than 5% of the absorbed hexane, indicating the highly stable recycling performance (Fig. S5).

As known, industrial wastewater usually contains various kinds of pollutants. As a proof-of-concept application, the obtained GMA-1 was used to absorb several pollutants from wastewater, not only oils and organic solvents, but also dyes and heavy metal ions. The sorption capacities of GMA-1, compared with those of PGA, are quite different (Fig. 4). The GMA-1 shows superior sorption capacities towards both Hg<sup>2+</sup> (719 mg g<sup>-1</sup>) and Pb<sup>2+</sup> (449 mg g<sup>-1</sup>), while the sorption capacities of PGA are only 55 and 72 mg g<sup>-1</sup> for Hg<sup>2+</sup> and Pb<sup>2+</sup>, respectively (Fig. 4a, b). Furthermore, in sorption of anionic methyl orange, GMA-1 still shows better performance than PGA (Fig. 4c), although for the sorption of cationic methylene blue, GMA is not as good as PGA (Fig. 4d). Moreover, in the sorption of organic solvents (e.g., hexane and chloroform), GMA-1 could not compete with PGA (Fig. 4e, f).

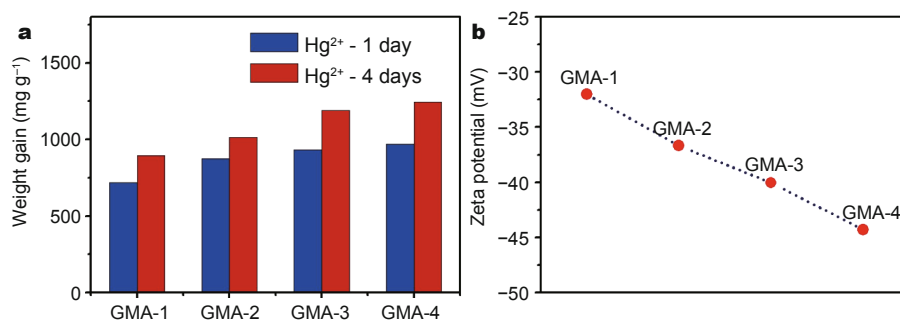
To investigate the effect of the loading amount of MoS<sub>2</sub> in GMAs on the sorption capacity, a series of GMAs, referred as GMA-1, GMA-2, GMA-3, and GMA-4 with different wt.% of MoS<sub>2</sub>, were prepared. The corresponding morphologies of GMAs with increased loading amount of MoS<sub>2</sub>, i.e., GMA-1 (22.9 wt.%), GMA-2 (25.8 wt.%), GMA-3 (28.0 wt.%) and GMA-4 (34.8 wt.%), were shown in Fig. S6. The one-day sorption capacities for Hg<sup>2+</sup> are 719, 876, 930 and 970 mg g<sup>-1</sup> for GMA-1, GMA-2, GMA-3, and GMA-4, respectively, and the corresponding four-day sorption capacities for Hg<sup>2+</sup> are 897, 1015, 1188, and 1245 mg g<sup>-1</sup>, respectively (Fig. 5a). The sorption capacity of GMA-4 is much higher than or comparable with many previously reported materials, such as GO nanosheets [32] (Table S1, Supplementary information).

Generally, the sorption capacity and sorption rate of water-soluble ions and dyes are influenced by the specific





**Figure 4** The comparison of performances between PGA and GMA-1 in removal of heavy metal ions (a, b), organic dyes (c, d), and organic solvents (e, f).



**Figure 5** (a) The sorption capabilities of GMAs towards Hg<sup>2+</sup> ions. (b) Zeta potentials of the respective GMAs before sorption.

surface area of sorbents and the interaction between sorbents and sorbates [33]. To deduce the possible sorption mechanism, the zeta potentials and surface areas of GMAs were measured. It is noted that the surfaces of the GMAs are negatively charged which become more negative as the wt.% of MoS<sub>2</sub> increased from GMA-1 to GMA-4 (Fig. 5b). Meanwhile, the surface area of GMAs decreased as the wt.% of MoS<sub>2</sub> increased (Table S2). These results suggest that the electrostatic interaction between

the surfaces of GMAs and the sorbates is the dominant sorption mechanism for water-soluble ions.

## CONCLUSIONS

In summary, we report that 3D GMAs with high porosity can be simply synthesized by hydrothermal method followed by a typical freeze-drying process. The structural modification by MoS<sub>2</sub> equips the GMAs with good sorption ability towards several organic contaminants,

such as organic solvents and dyes. Impressively, the prepared GMAs can absorb  $\text{Hg}^{2+}$  ions up to  $1245 \text{ mg g}^{-1}$ . It is believed that GMAs are highly promising as multi-functional sorbents for real-life industry wastewater remediation.

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**Author contributions** All authors contributed to the discussion and

preparation of the manuscript. The final version of the manuscript was approved by all authors.

**Conflict of interest** The authors declare that they have no conflict of interest.

**Supplementary information** Supporting data are available in the online version of the paper.



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## 基于石墨烯-二硫化钼复合气凝胶的多功能吸附材料的制备及其在水污染处理方面的应用

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**摘要** 三维碳基多孔材料因其独特的结构和超高的吸附性能, 已成为最有水污染处理应用前景的吸附材料之一. 本文通过有效的调控, 合成了多种具有不同孔道结构和成分组成的石墨烯-二硫化钼复合气凝胶材料. 这种材料在吸附重金属离子, 有机染料, 油及有机溶剂方面都有很多优异的表现. 通过调控二硫化钼的比例, 可以有效改善材料的吸附性能. 得益于此, 其吸附重金属汞离子的效率可以达到  $1245 \text{ mg g}^{-1}$ .