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A three-dimensional porous MoS$_2$-PVP aerogel as a highly efficient and recyclable sorbent for oils and organic solvents

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Abstract: Three-dimensional (3D) aerogels are attracting more and more attention in oil-water separation ascribe to its advantages of low density, high porosity, and large specific surface area. However, its application was greatly limited due to its hydrophilic and low adsorption properties. In this work, we report a 3D MoS$_2$-polyvinylpyrrolidone (PVP) aerogels by freeze-drying method, where PVP was used as a skeleton to support aerogels. As a surfactant, PVP could easily attach to the surface of MoS$_2$ nanosheets and facilitates the interconnection between nanosheets. The 3D MoS$_2$-PVP aerogel exhibits low density, high porosity, good hydrophobicity, and excellent adsorption capacity (195–649 times). Moreover, after 30 cycles, the structure of 3D MoS$_2$-PVP aerogel is well kept and the adsorption capacity still retained 93.5% and 92.9% by squeezing and distillation. Therefore, the obtained 3D MoS$_2$-PVP aerogel is a promising adsorption material and has great potential practical application value in oil-water separation.

Keywords: PVP, 3D MoS$_2$-PVP aerogel, hydrophobicity, adsorption capacity, oil/water separation.
Introduction

With the development of society, the problem of water will be more and more serious. Oil leakage, industrial wastewater discharge, and organic solvent leakage are the main causes of water pollution. These problems have caused the serious ecological crisis, which leads to the wide attention of basic and applied research all over the world.\textsuperscript{1-4} Some traditional approaches had been adopted to solve this serious problem. For example, including dispersant, adsorbent material, oil skimmer vessel, and oil containment boom.\textsuperscript{5-13} Among the above methods, the adsorbent material is one of the most promising approaches because of its simple process and excellent adsorption efficiency. The porous materials were widely used as absorbents because they can separate oil and water through a simple and effective absorption process.\textsuperscript{14-17} Generally, the ideal absorbent material should have low density, high absorption capacity, excellent recyclability, and environmental friendliness. Thus, the large number of adsorbent materials have been used to treat sewage, including wool fibers,\textsuperscript{18} activated carbon,\textsuperscript{19} expanded graphite,\textsuperscript{20} BN nanosheets because of their microporosity.\textsuperscript{17, 21-23} Although these adsorbent materials are effective to a certain extent, the practical application is severely limited due to low adsorption capacity and poor recyclability. Therefore, it is highly desired to develop adsorption material with a high adsorption capacity and excellent recyclability.

Recently, it has been greatly attended to the preparation of porous polymer aerogels by a freeze-drying method.\textsuperscript{24-28} This method exhibits great advantages of simple and controllable, which has been widely studied and applied in various fields.\textsuperscript{29-33} It has been reported that GO-PVA aerogel was successfully obtained,\textsuperscript{34} PVA can be used as the skeleton to support GO. The boron nitride-modified PVA aerogel was obtained via a freeze-drying method, and it possesses low density, high porosity, and outstanding adsorption capacity for various solvents.\textsuperscript{35} MoS\textsubscript{2} is a layered transition metal disulfide with a graphene-like structure.\textsuperscript{36} Its unique structure advantages make it has a great prospect in the fields of catalysis, batteries, and sensors.\textsuperscript{37-42} Due to the extensive study of there-dimensional (3D) aerogels, the demand for MoS\textsubscript{2} aerogels is increasing to realize its potential application.

Herein, the 3D MoS\textsubscript{2}-PVP aerogel has been successfully fabricated by the freeze-drying method. As a surfactant, PVP could easily attach to the surface of MoS\textsubscript{2} nanosheets and facilitates the interconnection between nanosheets. The obtain 3D MoS\textsubscript{2}-PVP aerogel exhibits great advantages of high adsorption capacity and strong recyclability. In addition, after 30 cycles, the
structure of 3D MoS₂-PVP aerogel is well kept and the adsorption capacity still retained 93.5% and 92.9% by squeezing and distillation. It can be served as a promising adsorption material for environmental remediation.

**Results and discussion**

Fig. 1 depicts the assembling process of 3D MoS₂-PVP aerogel. Here, amphiphilic PVP can be used as a framework to support aerogel (Fig. S1). The MoS₂ shows a lateral size at ~200-600 nm (Fig. S2a). The specific surface area (SSA) of pure PVA aerogel is 44.5 m² g⁻¹, with the increase of MoS₂, the specific surface area of 3D MoS₂-PVP increased to 75.2, 79.6, 83.5 and 90.4 m² g⁻¹, respectively (Table S1).

Fig. 2a-b shows the XRD of the samples. Two diffraction peaks at 11.3° and 19.7° confirm the amorphous nature of PVP (Fig. 2b). The diffraction peaks of MoS₂-PVP aerogel match well with the MoS₂ (Fig. 2a). Nevertheless, it can be seen the intensity ratio of (002)/(103) of MoS₂-PVP aerogels (7.3) is higher than the MoS₂ (1.6), confirming the PVP promotes the exfoliation of MoS₂ nanosheets.³⁷, ⁴³

The morphologies of these samples are shown in Fig. S2. Fig. S2a exhibit the MoS₂ was 2D sheet-shaped morphology. Especially, when MoS₂ is combined with PVP, the irregular porous structure of MoS₂ nanosheets can be obtained (Fig. S2b). Furthermore, PVP can easily insert into MoS₂ nanosheets to generation thinner nanosheets.⁴³

The FTIR spectra in Fig. 2c-d can further indicate the interaction between MoS₂ and PVP. These peaks of PVP at 3320, 2917 and 1425, 1725, 1086 cm⁻¹ were attributed to O-H, –CH₃, C=O and C–O functional group, respectively. The peaks of MoS₂ at 3349, 1722, 1440 and 1158 cm⁻¹ corresponding to O-H, C=O, –CH₃ and C–O functional group, respectively. However, the characteristic peaks of MoS₂-PVP aerogel blue shift to 3289, 1686, 1420 and 1131 cm⁻¹, indicating the interaction between MoS₂ and PVP.⁴⁴

Fig. S3 exhibits optical photos of 3D MoS₂-PVP aerogel with different concentrations of MoS₂, which are basically the same in macroscopic morphology. Moreover, the PVP aerogel can also be obtained by the freezing method, which indicates its potential as the 3D framework to support aerogel. Importantly, our method is widely applicable to the preparation of other 1D or 2D material-PVP aerogels. Take BN-PVP and CNTs-PVP aerogels as examples, they were also successfully obtained by this method (Fig. S4).
It has been reported that the high specific surface area is beneficial to the improvement of adsorption performance. Therefore, we selected the 3D MoS$_2$-PVP aerogel (20 mg cm$^{-3}$) with a high specific surface area as the research object. The 3D MoS$_2$-PVP aerogel can stand stably on the surface of the flower (Fig. 3a). Fig. S5 shows the 3D MoS$_2$-PVP aerogel can support water droplets on its surface ascribe to its good hydrophobic. The hydrophobicity of adsorption material has great significance for the application of oil-water separation. Fig. 3b-c exhibit the water contact angle (WCA) of 3D MoS$_2$-PVP aerogel. The WCAs of 3D MoS$_2$-PVP aerogel increase from 91° to 113° with the increase of MoS$_2$ (Fig. 3c), indicating the hydrophobicity of 3D MoS$_2$-PVP aerogel. As shown in Fig. 3d, the mirror-reflection is observed ascribe to the formation of a new interface between aerogel and surrounding water, which further confirms the hydrophobicity of the 3D MoS$_2$-PVP aerogel.

Furthermore, the 3D MoS$_2$-PVP aerogel shows excellent mechanical performance. Fig. S6 shows these compressive curves have good hysteresis loops, which are consistent with the typical behavior of porous materials. With the increase of MoS$_2$, the mechanical properties become better. When the concentration of MoS$_2$ is 5 mg cm$^{-3}$, the compress strength is 4 KPa. The compress strength of MoS$_2$-PVP was reinforced to 25, 45, 63 KPa at the concentration of 10, 15 and 20 mg cm$^{-3}$, respectively. The result shows that the MoS$_2$-PVP aerogel had outstanding compress performance, which may be attributed to the great contribution of the ordered structure.

3D MoS$_2$-PVP aerogel is one of the idea adsorption materials for removing various oils and organic solvents due to its good hydrophobicity and excellent mechanical stability. As shown in Fig. 4a and Movie S1, when the 3D MoS$_2$-PVP aerogel is placed on the surface of the oil-water mixture, it can absorb the oil completely and quickly. These results confirm it has a great prospect in the field of oil-water separation.

To explore the adsorption efficiency of the 3D MoS$_2$-PVP aerogel, the weight gain (wt %) was defined as adsorbed the weight of the solvent per unit weight by dry aerogel. Herein, Table S1 shows the SSA of 3D MoS$_2$-PVP aerogel is increased with the increasing MoS$_2$. Moreover, the adsorption capacity of 3D MoS$_2$-PVP aerogel increases with the increasing SSA (Fig. S7).

The adsorption capability of 3D MoS$_2$-PVP aerogel on various oils and organic solvents were measured. The resulting exhibit that the 3D MoS$_2$-PVP aerogel has excellent adsorption capacity and can absorb the solvents 195-649 times of its weight (Fig. 4b).
In addition, the adsorption capacity of 3D MoS\textsubscript{2}-PVP aerogel is higher than that of previously reported adsorption materials (Table 1),\textsuperscript{46, 48-73} such as exfoliated graphite (60-90 times),\textsuperscript{74} carbon nanotube sponges (80-180 times),\textsuperscript{50} graphene sponge (60-160 times),\textsuperscript{54} and reduced graphite oxide foam (5-40 times).\textsuperscript{55} Moreover, the method to prepare 3D MoS\textsubscript{2}-PVP aerogel is the relatively simplest and its precursor is the relatively cheapest. Therefore, the obtained 3D MoS\textsubscript{2}-PVP aerogel is regarded as the most promising adsorbent for environmental remediation.

The key for oil-water separation is the recyclability of contaminants, as most contaminants contain both valuable and harmful materials. Two typical methods had been reported for recover contaminants, including squeezing and distillation. As observed in Fig. 5a, the octadecene absorbed by the 3D MoS\textsubscript{2}-PVP aerogel can be eliminated by squeezing. When the pressure was released, the aerogel can return its original shape (Fig. 5a).

Moreover, the 3D MoS\textsubscript{2}-PVP aerogel was repeated to recycle tests by squeezing and distillation (Fig. 5b-c). Fig. 5b shows the absorption-squeezing cycle process of the 3D MoS\textsubscript{2}-PVP aerogel. The structure of 3D MoS\textsubscript{2}-PVP aerogel was well kept and the adsorption capacity still retained 93.5% after 30 cycles (Fig. S8a and S8b, Supporting Information). As illustrated in Fig. 5c, the adsorption capacity of 3D MoS\textsubscript{2}-PVP aerogel still retained 92.9% after 30 cycles, which suggests its good recyclability. In addition, the structure of 3D MoS\textsubscript{2}-PVP aerogel was well kept after 30 cycles adsorption-distillation process (Fig. S9a and S9b, Supporting Information). Therefore, the obtained 3D MoS\textsubscript{2}-PVP aerogel can be used to remove contaminants by squeezing, distillation, or a combination of two methods. It shows a great potential practical application value in the field of oil-water separation.

Conclusions

In conclusion, 3D MoS\textsubscript{2}-PVP aerogel with low density, good hydrophobicity, high adsorption capacity, and excellent recyclability was fabricated by the freeze-drying method. The adsorption capacity of the 3D MoS\textsubscript{2}-PVP aerogel can be as high as 195-649 times of its weight. Moreover, after 30 cycles, the structure of 3D MoS\textsubscript{2}-PVP aerogel is well kept and the adsorption capacity still retained 93.5% and 92.9% by squeezing and distillation. Therefore, the obtained 3D MoS\textsubscript{2}-PVP aerogel has an extremely potential application for oil-water separation.

Experimental section
Materials

MoS\(_2\) powders were purchased from Nanjing XFNANO Materials Tech Co., Ltd. PVP and various organic solvents were from Shanghai Aladdin Biochemistry Technology Co., Ltd. Gasoline oil was provided by the local gas station. Pump oil, sesame oil, colza, and olive oil were obtained in the local market.

Preparation of 3D MoS\(_2\)-PVP aerogel

The 3D MoS\(_2\)-PVP aerogel was prepared by the freeze-drying method.\(^{45, 75-78}\) Briefly, MoS\(_2\) powder were uniformly dispersed in PVP solutions (1 mg cm\(^{-3}\)) under stirring and the concentration of MoS\(_2\) was designed 5, 10, 15 and 20 mg cm\(^{-3}\), respectively. Then the suspension was poured into the rubber mold placed at the top of a cold steel rod, where was cooled by adding liquid nitrogen and kept at -70 °C for 1h. Then the obtained frozen sample was further freeze-drying by using a Labconco-195 freeze-drier for 72 h. Finally, the 3D MoS\(_2\)-PVP aerogel was obtained.

Characterization

The morphology was observed by SEM (S-4800, Hitachi) at an accelerating voltage of 10 kV. The X-ray diffraction spectrum was collected by XRD-6000 (Shimadzu) with Cu K\(\alpha\) radiation (\(\lambda = 1.54178\) Å). The Fourier Transform Infrared Spectroscopy (FTIR) was characterized from 4000 to 400 cm\(^{-1}\) wavenumber by Nicolet 6700. The contact angle was characterized with water by OCA 15 Pro. The specific surface area was conducted by the ASAP 2020 system at 77K. The compression test was carried out by Instron 5565A at the speed of 0.1 mm/min.

Absorption of oils and organic solvents

During the adsorption test, these 3D MoS\(_2\)-PVP aerogels were required to completely filled with oils or organic solvents. To avoid evaporation of solvents with low boiling points, the weight measurements should be made quickly. The weight of 3D MoS\(_2\)-PVP aerogels before and after adsorption was recorded.

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Fig. 1 Assembling process of 3D MoS$_2$-PVP aerogel.
Fig. 2 (a-b) XRD patterns of MoS$_2$, MoS$_2$-PVP aerogel and PVP. (c-d) FTIR spectra of MoS$_2$, MoS$_2$-PVP aerogel and PVP.
Fig. 3 (a) Photograph of the 3D MoS$_2$-PVP aerogel stands on a flower surface. (b) Photograph of the WCA of 3D MoS$_2$-PVP aerogel. (c) The WCA of 3D MoS$_2$-PVP aerogels with different concentrations of MoS$_2$. (d) Photograph of mirror-reflection, which further confirms the hydrophobicity of the 3D MoS$_2$-PVP aerogel.
Fig. 4 Absorption performance of the 3D MoS\(_2\)-PVP aerogel. (a) Photographs of absorbed pump oil by the 3D MoS\(_2\)-PVP aerogel. (b) The absorption capacity of the 3D MoS\(_2\)-PVP aerogel for various organic solvents and oils.
Fig. 5 Recycle of 3D MoS$_2$-PVP aerogel. (a) The photographs show the process of adsorption-squeezing. (b) Adsorption-squeezing recycles of the 3D MoS$_2$-PVP aerogel for the absorb octadecene. (c) Adsorption-distillation recycles of the 3D MoS$_2$-PVP aerogel for the absorb heptane.
| Adsorbent materials          | Absorbed substances                      | Sorption capacity (g g⁻¹) | Cost | Ref. |
|-----------------------------|------------------------------------------|---------------------------|------|-----|
| Wool-based nonwoven         | Diesel, crude oil, SN 150                | 9-15                      | Low  | 48  |
| Graphene/CNT foam           | Compress oil, organic solvents           | 80-140                    | High | 53  |
| Vegetable fiber             | Crude oil                                | 1-100                     | Low  | 49  |
| Graphene sponge             | Oils and organic solvents                | 60-160                    | High | 54  |
| Exfoliated graphite         | Heavy oil                                | 60-90                     | Low  | 51  |
| Carbon nanotube sponges     | Oils and organic solvents                | 80-180                    | High | 50  |
| Magnetic exfoliated graphite| Oils                                     | 30-50                     | High | 52  |
| Graphene-based sponges      | Oils and organic solvents                | 60-160                    | High | 46  |
| Reduced graphite oxide foam | Oils and organic solvents                | 5-40                      | High | 55  |
| SMF foam                    | Oils and organic solvents                | 78-172                    | High | 79  |
| CNT sponge doped graphene foam| Oils and organic solvents               | 25-125                    | High | 57  |
| BCM sponge                  | Oils and organic solvents                | 86-201                    | High | 80  |
| CMB aerogel                 | Oils and organic solvents                | 56-188                    | Low  | 59  |
| GMF aerogel                 | Oils and organic solvents                | 60-140                    | Low  | 60  |
| OCA aerogel                 | Oils and organic solvents                | 81-171                    | Low  | 61  |
| MCF aerogel                 | Oils and organic solvents                | 88-228                    | Low  | 62  |
| SMS sponges                 | Oils and organic solvents                | 82-159                    | Low  | 63  |
| MCGA                        | Oils and organic solvents                | 80-197                    | High | 64  |
| FGN/PU sponge               | Oils and organic solvents                | 25-44                     | High | 81  |
| HAP nanowire aerogel        | Oils and organic solvents                | 83-156                    | Low  | 65  |
| Silylated wood sponge       | Oils and organic solvents                | 16-41                     | Low  | 66  |
| Graphene foams              | Oils and organic solvents                | 120-250                   | High | 67  |
| EVOH NFAs                   | Oils and organic solvents                | 45-102                    | Low  | 68  |
| GCTs                        | Oils and organic solvents                | 250-400                   | High | 69  |
| TCF aerogel                 | Oils and organic solvents                | 50-192                    | Low  | 70  |
| Carbon aerogel              | Oils and organic solvents                | 80-161                    | High | 71  |
| 3C aerogels                 | Oils and organic solvents                | 33-70                     | High | 72  |
| Co-C/CF sponge              | Oils and organic solvents                | 85-200                    | High | 73  |
| CMA                         | Oils and organic solvents                | 78-348                    | Low  | 45  |
| 3D MoS₂-PVP aerogel         | Oils and organic solvents                | 195-649                   | Low  | This work |

SMF: superhydrophobic melamine-formaldehyde, BCM: biomass-decorated carbonaceous melamine, CMB: Carbon microbelt, GMF: Oleophylic carbon aerogel, MCF: Microfibrillated cellulose fibers, SMS: Superoleophilic MoS₂ nanosheet sponge, MCGA: Modified cellulose/graphene aerogels, FGN/PU: functionalized graphene/PU, HAP: Hydroxyapatite, EVOH NFAs: Poly (vinyl alcohol-co-ethylene) nanofiber aerogels, GCTs: Giant carbon tubes, TCF: Twisted carbon fibers, 3C: Compressible and conductive carbon, CF: Carbon foam, CMA: Carbon microtube aerogel.