Ultra-Light GO@KGM Aerogels for Oil−Water Separation Based on CVD Modification

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ABSTRACT: Nowadays, oil pollution of water caused by illegal discharges or accidental events occurs frequently, and the waste of resources and environmental pollution cannot be ignored, so effective oil−water separation methods are needed to cope with such incidents. To solve these problems, this paper investigated an aerogel made from a plant polysaccharide, konjac glucomannan (KGM), supplemented with graphene oxide (GO), to improve the mechanical properties. Finally, a hydrophobic layer was attached to the surface and interior of the aerogel via chemical vapor deposition to improve its selectivity toward oil. Through a series of characterization methods such as infrared, X-ray photoelectron spectroscopy, and X-ray diffraction, it was demonstrated that KGM and GO were successfully cross-linked, resulting in excellent mechanical properties and directional absorption properties on oil. This composite polysaccharide aerogel could absorb oil 48 times its own weight. In addition, due to its strong mechanical properties, the gel can be reused many times, and the maximum recovery rate can be maintained at 96% after 10 cycles. Furthermore, the absorption of oil from water was conducted in a continuous mode, demonstrating the diversity of application scenarios. Generally, the results observed in this work have shown that the KGM aerogels have great potential for applications in oil−water separation.

1. INTRODUCTION

With industrialization, oil spills at sea and oily effluents from the oil industry pose a non-negligible threat to the natural environment and human health. Various oil−water separation methods are applied to this field, including in situ combustion, microbial degradation, physical absorption, and so forth. Physical absorption is the primary means of treating oil and water contaminants. Because it is efficient, low cost, environmentally friendly, and energy efficient. Three-dimensional (3D) materials such as foams, sponges, and aerogels, with their rich porous structures, are ideal absorption materials.

Since aerogel was first produced in 1931,7 it has been widely utilized in many fields due to its excellent properties, such as low density, high porosity, high specific surface area, and low thermal conductivity. The application fields of aerogels include insulating layers with ultra-low conductivity,2 acoustic thermal insulation materials,3 sensors,4 catalyst/catalyst carriers,5 electrode materials,6 and absorption materials.7 As an absorption material, the highly porous structure of the aerogel results in great absorption capacity. Hence, they can be applied to absorb multiple substances, for instance, carbon dioxide, benzene, and other toxic gases and wastewater. In recent years, aerogels as a new type of highly efficient oil-absorbing material have attracted much attention.

Konjac is a perennial herb of the monocotyledonous genus Konjac in the family Tenaxaceae, widely grown in Hubei, Yunnan, and Sichuan in China. Its tubers are rich in konjac glucomannan (KGM), which is a kind of natural plant polysaccharide with sufficient raw materials, biodegradable, and chemically modifiable functional groups. Thus, KGM is an ideal natural material for preparing aerogels. At present, research on KGM is mainly concentrated in the food industry8−10 and biomedicine fields,11−13 but there is little research on KGM in the field of oil−water separation.14−19

As a classical two-dimensional lamellar material, graphene oxide (GO) has been widely used for the preparation of aerogels.20−22 GO has a large number of oxygen-containing groups such as hydroxyl groups, aldehyde groups, epoxide groups, and carboxyl groups at its base and edges. These hydrophilic polar groups allow GO to be self-assembled by hydrogen bonding to prepare aerogels23 or compounded with
polymeric materials to increase the active sites and improve mechanical properties.

Although KGM has been used in the oil–water separation field, pure KGM aerogels were not able to be reused because of their inherent structural fragility and lack of selective absorption. In this work, an environmentally friendly biomass aerogel was prepared using KGM as a raw material and freeze-drying technique with the participation of GO, in an attempt to improve the mechanical properties of the aerogels by extrusion and to achieve reuse of the aerogels. In addition, the aerogels were modified with methyltrimethoxysilane (MTMS) to enhance hydrophobicity.

2. EXPERIMENTAL SECTION

2.1. Materials. KGM powder (99%) was provided by Hubei Yizhi Konjac Technology Co., Ltd. Graphite powder (99%) and MTMS were purchased from Aladdin Reagent (Shanghai) Co., Ltd. Anhydrous sodium carbonate (Na₂CO₃), ethanol, cyclohexane, N,N-dimethylformamide (DMF), dichloromethane, silicone oil, and methylene blue were supplied by Chengdu Cologne Reagent Co., Ltd. Rapeseed oil was purchased from China Oil and Foodstuffs Corporation. Pump oil was purchased from Shanghai McLean Biochemical Technology Co., Ltd. Diesel oil was supplied by China National Petroleum Corporation. Sudan red was purchased from Tianjin Komiu Chemical Reagent Co., Ltd. All the reagents used in this work were of analytical grade and had no further purification.

2.2. Preparation of GO@KGM Aerogel. Nanosheets of GO were synthesized by a modified Hummer method using natural graphite as the raw material.

Generally, GO nanosheets were first dispersed with 20 mL of pure water to acquire a homogeneous solution with various mass concentrations (0.5, 1, and 1.5 mg/mL). Then, a certain amount of KGM powder (9 mg/mL) was subsequently dissolved in the GO dispersion. The pH of the mixture was adjusted to 10 with a Na₂CO₃ solution (5 wt %). The fully stirred mixture was transferred to a polytetrafluoroethylene reactor to carry out a hydrothermal reaction at 90 °C for 4 h. After that, a deacetylated GO/KGM hydrogel was obtained. Finally, the hydrogels were placed into a vacuum freeze dryer and dried for 30 h to obtain the GO@KGM aerogels.

2.3. Preparation of GO@KGM@MTMS via Chemical Vapor Deposition. The hydrophobic modification of the GO@KGM aerogel surface was obtained via chemical vapor deposition (CVD) treatment. Specifically, the samples were placed into a sealed box together with two bottle caps, containing 1 mL of MTMS and 1 mL of pure water, respectively. Then, the box was put into a vacuum oven, where the temperature was kept at 70 °C for 2 h. For clarity of presentation, GK-1, GK-2, and GK-3 are used to denote three different scaled samples, and GKM-1, GKM-2, and GKM-3 are used to denote the hydrophobic modification products of the three scaled samples, respectively.

2.4. Characterization. The size and morphology of samples were characterized using a scanning electron microscope (JEM-2100F, Japan Electron Optics Laboratory Co., Ltd). Also, the elements on surfaces were characterized using another scanning electron microscope (EVO MA15, Zeiss) equipped with an energy dispersive X-ray spectrometer for mapping and an X-ray photoelectron spectroscopy (XPS, Thermo Fisher Escalab 250Xi, Al Kα ray, HV = 1486.6 eV). Functional groups of samples were detected by Fourier transform infrared spectroscopy (WQF520; the test wavelength range was 500–4500 cm⁻¹, the resolution was 4 cm⁻¹, and the number of scans was 32). The crystal structure of aerogels was characterized by X-ray diffractometer (X Pert PRO MPD, where the X-ray emission source was copper Ka, wavelength λ = 0.154 nm). The mechanical properties of GO@KGM aerogels were tested at a speed of 2 mm/min on a microcomputer-controlled electronic universal testing machine (C42.S03Y) for a total of 10 cycles. In order to test the hydrophobicity of the material, the contact angles of the samples were measured using a contact angle tester (HARKE-SPCAX2, Beijing HARKE Testing Instrument Factory). All the measurements were conducted at room temperature (15 °C), taking the average value of five samples.

2.5. Absorption Performance Test. Generally, 10 mL of the liquid is poured into a weighing bottle. Then an aerogel, weighed in advance with an initial mass (denoted as w₀), was placed into the weighing bottle with the bottle cap covered. After 30 min, the fully infiltrated aerogel was taken out and weighed (denoted as w₁). In order to calculate the liquid absorption capacity (Q), three experiments were performed for each sample and averaged.

The liquid absorption formula is as follows

\[ Q = \frac{w_1 - w_0}{w_0} \]

To evaluate the regeneration and reuse performance of the GO@KGM@MTMS aerogel, the organic solvent and oil in the saturated aerogel were removed by the extrusion method, and 10 absorption–extrusion cycle experiments were carried out. By measuring the weight of the GO@KGM@MTMS aerogel before and after absorption, the liquid absorption capacity (Q) was calculated. All the absorption experiments were performed three times, and the average value was calculated.

3. RESULTS AND DISCUSSION

3.1. Structural and Chemical Analysis. Figure 1 illustrates the preparation process of GO@KGM@MTMS aerogels and the deacetylation process of KGM. A widely accepted view is that water solubility is conferred by the presence of acetyl groups on the molecule, and the steric...
hindrance effect of the acetyl group hinders the formation of intramolecular and intermolecular hydrogen bonds.\textsuperscript{25,26} Deacetylation of KGM was carried out by the hydrothermal method at 90 °C to obtain more −OH, which can form hydrogen bonds with GO, resulting in a jelly-like GO@KGM hydrogel. After removing the water from the hydrogel in the vacuum freeze dryer, the GO@KGM aerogel with a 3D network structure was obtained. The CVD hydrophobic modification of GO@KGM with MTMS resulted in GO@KGM@MTMS aerogels with a superhydrophobic surface.

As shown by the scanning electron microscopy (SEM) image, Figure 2a,b, there was a 3D network structure formed by the interaction of many strip-shaped materials in the interior of GK-2, which provided space for oil absorption. Hydrophobic modification was a necessary step as the material was not selective for oil and water due to the hydroxyl groups on the surface of GO and KGM.

After modification, the internal structure of aerogels did not change according to Figure 2c,d. Many small particles appeared inside the pores, which might be attributed to the self-polymerization of MTMS molecules on the aerogel surface during modification.

In order to further confirm that the particles in the SEM images were the products of the hydrophobic modification process, the samples were analyzed using another scanning electron microscopy equipped with an energy dispersive X-ray spectrometer. As shown in the energy dispersive X-ray spectrometry mapping image (Figure 3), the silicone atoms in the MTMS molecules were uniformly distributed on the surface of the aerogel, indicating that the hydrophobic coating formed by the MTMS molecules was successfully loaded on the surface of the sample.

The crystal structures of GO, KGM, and GKM-2 were analyzed by X-ray diffraction (XRD). As shown in Figure 4,

![Figure 2](image)  
Figure 2. SEM images of GK-2 and GKM-2. (a,b) Internal SEM image of GK-2 and its magnification. (c,d) Internal SEM image of GK-2 and its magnification.

![Figure 3](image)  
Figure 3. Electron microscope image and element distribution map of the GKM-2 surface. (a) SEM image of the aerogel surface; (b-d) X-ray energy spectrum of Si, O, and C on the surface of the aerogel.

![Figure 4](image)  
Figure 4. XRD patterns of KGM, GO, and GKM-2.
there was a broad peak near 2θ = 19.9°, which could be attributed to the amorphous structure of KGM. Also, a narrow and strong peak at 2θ = 11.2° of GO could be observed. However, two peaks of GKM-2 were determined at 2θ = 11.7° and 2θ = 20.1°. It indicated there was an entanglement between GO and KGM, which led to the change of the nanolayer spacing of GO; and hydrogen bonding occurred within the molecule after the deacetylation of KGM, transforming the substance from an amorphous structure to a more ordered 3D network structure.

According to the infrared spectra (Figure 5), GO included a broad peak for the O−H vibration (3405 cm⁻¹), a C=O vibration peak for the carbonyl and carboxylic groups (1714 cm⁻¹), a skeletal C=C vibration peak for the phenolic ring (1623 cm⁻¹), a bending vibration peak for the O−H group (1378 cm⁻¹), and a respiratory vibration peak of the epoxide group (1052 cm⁻¹). For KGM, there were three absorption peaks for O−H, namely the stretching vibration peak (3417 cm⁻¹), the bending vibration peak (1646 cm⁻¹), and the variable angle vibration peak (1108 cm⁻¹). Moreover, the peaks at 2926 and 1735 cm⁻¹ corresponded to C−H in ethanol and C=O in acetyl, respectively. In addition, the peaks at 873 and 802 cm⁻¹ were the characteristic vibrations of the mannose unit in KGM. After the hydrothermal treatment, the peaks of the O−H stretching vibration and bending vibration peaks of GK-2 near 3400 and 1646 cm⁻¹ became smaller, indicating that GO and KGM directly produce hydrogen bonding. Also, the peak at 1735 cm⁻¹ disappeared, indicating that the deacetylation of KGM was successful by the hydrothermal method. These were the basis of the 3D structure. Compared with GK-2, GKM-2 produced stronger absorption peaks at 778 and 1272 cm⁻¹, which were due to the generation of the Si−O−Si bond and the C−Si−O bond after CVD modification, respectively.

The XPS test of GKM-2 confirmed the successful hydrophobic modification of the sample by CVD. As shown in Figure 6, C 1s, O 1s, and Si 2p peaks were observed in the XPS spectrum of GKM-2. By analyzing Figure 6d, the high-resolution Si 2p spectrum, it was found that it can be decomposed into two Gaussian components: Si−O (102 eV) and Si−C (103 eV). This suggested that the hydroxyl groups on the GK-2 skeleton reacted with MTMS and formed a silicone coating on the surface after CVD hydrophobic modification.

3.2. Mechanical Properties. Direct extrusion is currently the most cost-effective method of oil recovery and material recovery. KGM was used as the skeleton material and GO as

Figure 5. Infrared spectra of GO/KGM/GK-2/GKM-2.

Figure 6. XPS spectra of GKM-2. (a) Full spectrum of GKM-2; (b) spectrum of C 1s; (c) spectrum of O 1s; and (d) spectrum of Si 2p.
the support material. The composite material constitutes a porous material with a 3D mesh structure that had a certain mechanical strength and can undergo certain elastic deformations. When the GKM-2 in Figure 7a was compressed to 50% of its original height, it could immediately return to its original height once the pressure was released. More sophisticated compression performance tests were carried out on a microcomputer-controlled electronic universal testing machine, and stress–strain curves were obtained for the samples. Figure 7b shows the stress–strain curve for GKM-2 at 40% deformation for the 1st and 10th times, which demonstrates its ability to recover the deformation after 10 cycles of extrusion. This indicates that the GO@KGM aerogel can recover both oil and adsorbed materials by simple extrusion after oil absorption. The advantage of this recovery method over the combustion recovery method is that the extrusion recovery method can recover the oil and is more energy and time efficient compared to the distillation recovery method.

3.3. Hydrophobic Performance. Excellent hydrophobicity is the basis of water–oil separation. Figure 8 illustrates the hydrophobic performance before and after CVD treatment, which clearly exhibits apparent improvement. CVD treatment does not require sophisticated equipment, nor does it require large amounts of organic solvents. Its process requires a short time and a mild temperature. More importantly, this simple and gentle hydrophobic modification method has been proven to be very efficient. For ease of observation, water and oil were previously stained with methylene blue and Sudan red, respectively.

Pure KGM aerogels failed to absorb liquids selectively, as shown in Figure 8a. To make matters worse, the KGM skeleton tended to collapse after contact with water droplets. After CVD treatment, it had obvious selective absorption of oil droplets and water droplets. As shown in Figure 8b, the water contact angle of the sample at room temperature was measured using a contact angle instrument, indicating that the sample had good hydrophobicity.

3.4. Absorption Capacity and Reusability. The absorption capacity of modified aerogels in both common oils and organic solvents was investigated. All the oils used in experiments are very common, readily available, and come in different densities and viscosities. Figure 9a shows the absorption capacity of samples. In brief, the absorption capacity was roughly proportional to the density of the oil. The lowest absorption capacity was 14 g/g (ethanol) and the highest was 48 g/g (silicon oil). When the densities of the liquids under test were similar, the absorption rate was related to the viscosity. For example, compared to dichloromethane (1.33 g/cm³), GKM-2 has a higher absorption capacity for silicone oil (1.08 g/cm³). Because the viscosity of silicone oil is much greater than that of dichloromethane, it can adhere more to the surface of GKM-2. With GO doped in different proportions, the absorption capacity of GKM-2 and GKM-3 was significantly better than that of GKM-1. This was because the addition of GO improved the structure of the aerogel and increased the absorption sites of the aerogel. With the further increase in concentration, there was no significant difference between GKM-2 and GKM-3. It may be attributed to the smaller pore channels of the aerogels as the total concentration of GO and KGM increased, resulting in a slight decline in the absorption multiplicity.

The reusability of aerogel is a key factor in practical applications. For the pure KGM aerogel, it is deformed permanently once squeezed and thus cannot be reused. Fortunately, the addition of GO greatly improved the mechanical properties of the aerogel and the reuse of the GO@KGM became possible. After the modified aerogel absorbed oil, it could be regenerated by a simple extrusion method. Figure 9b records the cyclic absorption effects of aerogel on four kinds of oils. After 10 cycles of absorption of diesel oil, rapeseed oil, pump oil, and silicone oil, the
absorption capacity went down to 88.79, 92.57, 96.93, and 96.63% of the first absorption, respectively, demonstrating excellent reusability.

3.5. Oil−Water Separation Performance. Because of the excellent hydrophobicity, GO@KGM@MTMS aerogels were able to separate oil and water by selective absorption of the oil in the oil−water mixture. To facilitate observation, the oil in the experiment was stained with Sudan red. Figure 10a,b show the absorption of cyclohexane and dichloromethane on the samples within a few seconds. Also, no oil re-osmosis was observed. Therefore, considering another possible situation, when the oil volume is large due to the recyclability of the sample, after removing the oil from the sample by extrusion, intermittent absorption and separation of the oil−water mixture can be performed.

In addition to the excellent oil absorption capacity and reusability, it was amazing that GO@KGM@MTMS aerogels were capable of continuous separation of oil−water mixtures. As shown in Figure 10c, GKM-2 was able to perform continuous absorption of cyclohexane in the left beaker with the involvement of a peristaltic pump. The flux rate of oil was calculated to be $8.6 \times 10^3$ L/(m$^2$ h) during continuous separation. During the collection process, no oil release and no absorption of sample water were observed. Moreover, after the
separation was complete, no residual cyclohexane was visible on the water surface. This demonstrates the potential of the sample to provide continuous separation of oil–water mixtures. Thus, as a biopolymer-based konjac dextran aerogel, it can be used as a lightweight device for the treatment of oil spills or organic contaminants.

4. CONCLUSIONS

In this work, a deacetylated KGM@GO aerogel was prepared to form an elastic backbone structure. Furthermore, the surface was hydrophobically modified by MTMS through the CVD method. The maximum oil absorption capacity of 48 g/g was obtained for GO@KGM@MTMS aerogels. In addition, the aerogels also exhibited excellent reusability. The absorption capacity could be maintained at 96.93% of the first absorption after 10 cycles. Also, the composite aerogel could also continuously separate oil–water mixtures when connected to a peristaltic pump. In summary, a kind of natural polysaccharide named KGM was used to prepare a reusable oil–water separation aerogel, indicating a promising application of KGM in oil–water separation.

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Notes

The authors declare no competing financial interest.

ACKNOWLEDGMENTS

This work was financially supported by the Sichuan Science and Technology Program (2021YJ0534 and 2021YJ0540), the National Natural Science Foundation of China (No. 51874255 and 52174209), supported by the scientific research starting project of SWPU (No. 2021QHZ004), and the Project funded by China Postdoctoral Science Foundation (2020M683362).

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