



Reduced Graphene Oxide/Nanocellulose/Amino-Multiwalled Carbon Nanotubes Nanocomposite Aerogel for Excellent Oil Adsorption

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Abstract

In this work, reduced graphene oxide/amino multi-walled carbon nanotube/nanocellulose (rGO/MWCNTs-NH₂/NC) nanocomposite aerogel has been successfully fabricated through the hydrothermal with freeze-drying and high-temperature annealing method and confirmed by Fourier transform infrared spectroscopy (FTIR), Raman spectroscopy, scanning electron microscope (SEM) and transmission electron microscopy (TEM). The optimal condition for fabrication of rGO/MWCNTs-NH₂/NC nanocomposite aerogel is mass ratio of GO to MWCNTs-NH₂ of 8:1 with NC weight percentage of 0.06%. The adsorption tests and oil/water separation experiments certify that the rGO/MWCNTs-NH₂/NC nanocomposite aerogel owns an excellent adsorption performance for various oil and organic solvents including ethyl acetate ($161.76 \pm 7.69 \text{ g g}^{-1}$), dichloromethane ($156.14 \pm 8.24 \text{ g g}^{-1}$), cyclohexane ($92.73 \pm 5.22 \text{ g g}^{-1}$), acetone ($103.87 \pm 7.83 \text{ g g}^{-1}$), and sesame oil ($124.09 \pm 6.64 \text{ g g}^{-1}$), and achieves an oil/water separation within only 20 s. Our rGO/MWCNTs-NH₂/NC nanocomposite aerogel is a promising material for oil and organic solvent adsorption.

Keywords: Nanocellulose; Graphene Oxide; Amino Multi-Walled Carbon Nanotubes; Nanocomposite Aerogel; Oil Adsorption.

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1. Introduction

Recently, oil spills and organic solvent pollution from industry have caused serious water pollution and are harmful to the people's health.^[1,2] It's required a facile and effective method to treat the oily wastewater.^[3] Owing to the high oil removal efficiency, simple operation, low energy consumption, adsorption is recognized as an efficient method for oily wastewater treatment.^[4,5] In this method, the type of adsorbents plays an important role in treating oily wastewater. Even the common adsorbents like activated carbon,^[6] fly ash,^[7] bentonite,^[8] and zeolite^[9] could be used for the water treatment, the disadvantages such as low oil/water separation efficiency and poor recyclability severely limit their applications. As a result, it is imperatively to find an efficient and fast material for treating oily wastewater.^[10]

Reduced graphene oxide (rGO) aerogel with the

characteristics of high specific surface area for graphene oxide as well as high porosity and ultra-low density of aerogel, exhibits a promising application in the oil/water separation and oil adsorption.^[11] More recently, in order to increase the adsorption property and hydrophobic property of single rGO aerogel, the researchers commonly mixed the rGO with other materials including metal oxide, nanocellulose,^[12] and carbon nanotubes (CNTs).^[13,14] For example, Cai *et al.*^[15] prepared the reduced graphene oxide/amino multi-walled carbon nanotube (rGO/MWCNTs-NH₂) aerogel, which exhibited an adsorption capacity to ethyl acetate of 152.79 g g^{-1} . Xiao *et al.*^[16] used cellulose acetate nanofibers to prevent the sheets from over-stacking of graphene and fabricated graphene oxide/nanofiber aerogel (GNA), which could quickly separate oil-in-water emulsions with an extremely high flux ($>19,000 \text{ L m}^{-2} \text{ h}^{-1} \text{ bar}^{-1}$). Unfortunately, there are few reports on utilization of both nanocellulose (NC) and multi-walled carbon nanotubes (MWCNTs) to enhance the hydrophobic property and adsorption property of rGO aerogels for oil adsorption and oil/water separation.

In this work, we have added the amino-MWCNTs (MWCNTs-NH₂) and nanocellulose into the rGO aerogel to

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manufacture the rGO/MWCNTs-NH₂/NC nanocomposite aerogel for ameliorating the hydrophobic property of rGO aerogel, which further enhances its oil and organic solvent adsorption properties. The scanning electron microscope (SEM), transmission electron microscopy (TEM), Fourier transform infrared spectroscopy (FTIR), and Raman spectroscopy have been conducted to study their microstructures and chemical structures. Moreover, the oil adsorption capacity and oil/water separation capability have been determined.

2. Experimental

2.1 Materials

Graphite powder (~325 mesh, 99%) was purchased from Alfa Aesar Chemical Co., Ltd., Amino multi-walled carbon nanotubes (MWCNTs-NH₂) was purchased from Beijing Deke Daojin Science and Technology Co., Ltd. Cellulose nanofiber (NC, 99.6%) was gained from Guilin Qihong Technology Co., Ltd. Potassium persulfate (K₂S₂O₈, 99%) was provided by Beijing Inoke Technology Co., Ltd. Aichloromethane (CH₂Cl₂, 99.5%) and Sudan Red B (C₂₄H₂₀N₄O, AR) were supplied by Aladdin Reagent Co., Ltd. and Shanghai Maclean Biochemical Technology Co., Ltd., respectively. Phosphorus pentoxide (P₂O₅, 99%), concentrated sulfuric acid (H₂SO₄, 95~98%), hydrogen peroxide (H₂O₂, 30%), *L*(+)-ascorbic acid (C₆H₈O₆, 99.7%), potassium permanganate (KMnO₄, 99.5%), ethanol (C₂H₅O, 95%), ethyl acetate (C₆H₁₂, 99.5%), hydrochloric acid (HCl, 36~38%), cyclohexane (C₆H₁₂, 99.5%) were obtained from Sinopharm Chemical Reagent Co., Ltd. All the chemicals were used as received without any further treatment.

2.2 Preparation of rGO/MWCNTs-NH₂/NC nanocomposite aerogel

The rGO/MWCNTs-NH₂/NC nanocomposites aerogel was fabricated by the following processes. Firstly, the graphite powder was used to prepare graphite oxide (GO) by a modified Hummers method.^[17] Briefly, the graphite powder (3.0 g), K₂S₂O₈ (4.0 g), P₂O₅ (4.0 g) were mixed and put in 20 mL of concentrated H₂SO₄ in a three-neck bottom flask under the magnetic stirring at 80°C in an water bath for 6 h. After cooling down to room temperature, the products were washed with deionized water until the pH of solution was equal to neutral. After filtered and dried, the samples and KMnO₄ (9.0 g) were slowly added into 120 mL of concentrated H₂SO₄ in a three-neck bottom flask, respectively, under the magnetic stirring in an ice-water bath for 30 min. After that, the solution was kept at 35°C in a water bath for 2 h. Later on, the GO was acquired by precipitation with adding 125 mL of deionized water and 30 wt% of hydrogen peroxide, respectively. Secondly, the MWCNTs-NH₂ and GO solution (2 mg mL⁻¹) were mixed and kept at 60°C in the water bath under the mechanical stirring and sonication for 2 h. After cooling down to room temperature, the NC and *L*-ascorbic acid were added into above solution and poured into a 25 mL of beaker with a

sealing cap at 70°C for 4 h to obtain the hydrogel. Afterwards, the hydrogel was dialyzed in a 15% (v/v) ethanol aqueous solution for 4 h and then taken out for freezing of 12 h in a refrigerator. Subsequently, the frozen sample was freeze-dried for 24 h to attain the aerogel. Finally, the rGO/MWCNTs-NH₂/NC nanocomposites aerogel was received with annealing at 150°C for 1 h in an infrared tube furnace (Beijing Huace Instruments Inc). In order to obtain an optimal preparation condition of nanocomposite aerogel, the different NC weight percentages (0.04, 0.06, 0.08, and 0.10 wt%) and different mass ratios of GO to MWCNTs-NH₂ (6:1, 8:1, 10:1, and 12:1) were exploited to prepare the rGO/MWCNTs-NH₂/NC nanocomposite aerogel. In comparison, the pure rGO aerogel, rGO/MWCNTs-NH₂ and rGO/NC aerogel were also manufactured.

2.3 Characterizations

The morphologies of samples were observed on a field emission scanning electron microscope (FE-SEM, Hitachi S-4800 system) and transmission electron microscopy (TEM, Tecnai G2 F20 S-TWIN). The chemical structures of samples were obtained by Fourier transform infrared spectrometer (FT-IR, Thermo Nicolet NEXUS spectrometer) and Raman spectra (Renishaw in Via Reflex Raman spectrometer). Adsorption-desorption isotherm curves of rGO/MWCNTs-NH₂, rGO/NC and rGO/MWCNTs-NH₂/NC nanocomposite aerogel were conducted on the Tristar3020 specific surface area and porosity analyzer. Their water contact angles were performed on a contact angle measuring instrument (Model: JC2000DS1, Shanghai Zhongchen Digital Technology Equipment Co., Ltd).

2.4 Adsorption test

2.4.1 Oil adsorption capacity test

The weighing bottle contained oil or organic solvent was weighed on an electronic balance (m_2). Then, the nanocomposite aerogel was put into this weighing bottle for adsorption of 5 min and taken out quickly with tweezers. After that, the weighing bottle was weighed again (m_1). The adsorption capacity (q) was attained from the weight difference by Equation: $q=(m_2-m_1)/m_{aero}$. The reported q value was the average of three measurements with a deviation of 10%. The q of nanocomposite aerogel to ethyl acetate, dichloromethane, cyclohexane, acetone, and sesame oil was estimated with this equation.

2.4.2 Oil/water separation test

The Sudan Red B was dissolved in the cyclohexane under magnetic stirring. Afterward, this solution was poured into a petri dish without lid that contained deionized water. Then, the rGO/MWCNTs-NH₂/NC nanocomposite aerogel was utilized to do oil/water separation test.

3. Results and discussion

3.1 Optimal condition for preparation of rGO/MWCNTs-NH₂/NC nanocomposite aerogel

With the purpose of exploring the optimal fabrication conditions to produce the rGO/MWCNTs-NH₂/NC nanocomposite aerogels with high adsorption capacity to oil and organic solvent, the different weight percentages of NC (0.04, 0.06, 0.08, and 0.10 wt%) and different mass ratios of GO to MWCNTs-NH₂ (6:1, 8:1, 10:1, and 12:1) have been chosen to prepare rGO/MWCNTs-NH₂/NC nanocomposite aerogels. The obtained SEM images are depicted in Figs. 1 and 2. The porous structure is observed in all the rGO/MWCNTs-NH₂/NC nanocomposite aerogels. However, as the weight percentage of NC is increased from 0.04 to 0.1%, the pores become larger, Figs. 1(a-d), and the adsorption capacity of rGO/MWCNTs-NH₂/NC nanocomposite aerogels to ethyl acetate is 134.39 ± 6.94 , 161.76 ± 7.69 , 132.45 ± 5.11 , and 128.05 ± 6.01 g g⁻¹ for NC weight percentage of 0.04, 0.06, 0.08, and 0.1%, respectively, Fig. 2(a), revealing that the best NC weight percentage for manufacturing rGO/MWCNTs-NH₂/NC nanocomposite aerogel is 0.06%.

After that, different mass ratio of GO to MWCNTs-NH₂ (6:1, 8:1, 10:1, and 12:1) have been selected to produce the rGO/MWCNTs-NH₂/NC nanocomposite aerogel under the NC weight percentage of 0.06%. As the obtained SEM results in Fig. 3, when the mass ratio of GO to MWCNTs-NH₂ is increased from 6:1 to 10:1, the pore structures become larger, Figs. 3(a-c). As the mass ratio of GO to MWCNTs-NH₂ is 12:1, some pores are damaged, Fig. 3(d). The adsorption capacity of rGO/MWCNTs-NH₂/NC nanocomposite aerogel to ethyl acetate is 100.96 ± 7.54 , 161.76 ± 7.69 , 78.11 ± 5.85 , 75.04 ± 6.23 g g⁻¹, separately, Fig. 2(b), demonstrating that the best mass ratio of GO to MWCNTs-NH₂ for preparation of

rGO/MWCNTs-NH₂/NC nanocomposite aerogel is 8:1. In a word, the optimal condition for synthesis of rGO/MWCNTs-NH₂/NC nanocomposite aerogel with a high adsorption capacity is GO to MWCNTs-NH₂ mass ratio of 8:1 with NC weight percentage of 0.06%.

3.2 Structure determination of rGO/MWCNTs-NH₂/NC nanocomposite aerogel

As shown in Figs. 4A-(a) and (b), the diameter and height for cylindrical shape of rGO/MWCNTs-NH₂/NC nanocomposite aerogel is measured to be 1.60 and 1.30 cm, respectively. After gaining its weight on the electronic balance (15.01 mg), its density is estimated to be 5.75 mg cm⁻³. This nanocomposite aerogel could be situated on a dandelion with any deformation, Fig. 4A-(c), indicating a low density of this nanocomposite aerogel.

The FTIR spectra of MWCNTs-NH₂, rGO/MWCNTs-NH₂ aerogel, NC, rGO/NC aerogel, and rGO/MWCNTs-NH₂/NC nanocomposite aerogel are depicted in Fig. 4(B). In the FTIR spectrum of MWCNTs-NH₂, Fig. 4(B)-a, a small broad peak around 1236 cm⁻¹ is attributed to the bending vibration of N-H and stretching vibration of C-N bands in the characteristic peaks of amide group.^[18] In the FTIR spectrum of NC, Fig. 4(B)-c, the absorption peaks at 1054, 1321, and 1419 cm⁻¹ are assigned to the stretching vibration of C-O, bending vibration of -OH, and C-H, respectively.^[2] These characteristic peaks could be observed in the FTIR spectra of rGO/MWCNTs-NH₂/NC nanocomposite aerogel, Fig. 4(B)-e, indicating that the MWCNTs-NH₂ and NC have been successfully introduced into the rGO/MWCNTs-NH₂/NC nanocomposite aerogel.

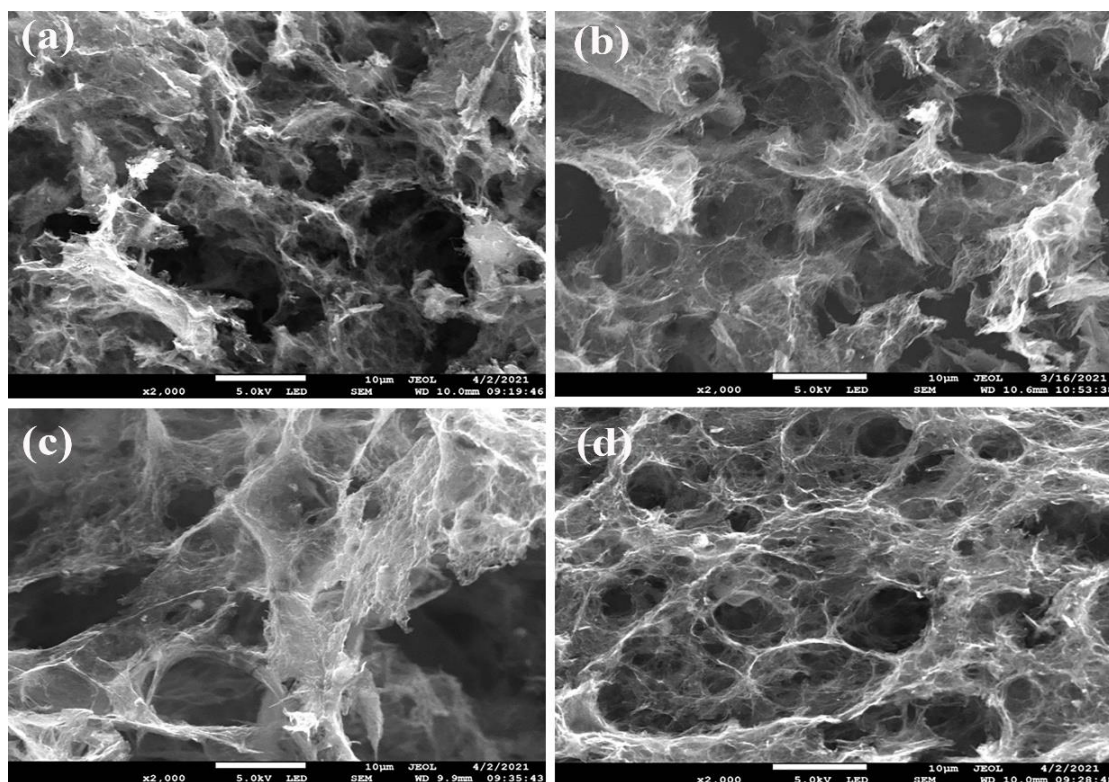


Fig. 1 SEM images of rGO/MWCNTs-NH₂/NC nanocomposite aerogels with NC weight percentages (a) 0.04, (b) 0.06, (c) 0.08, and (d) 0.10 wt%.

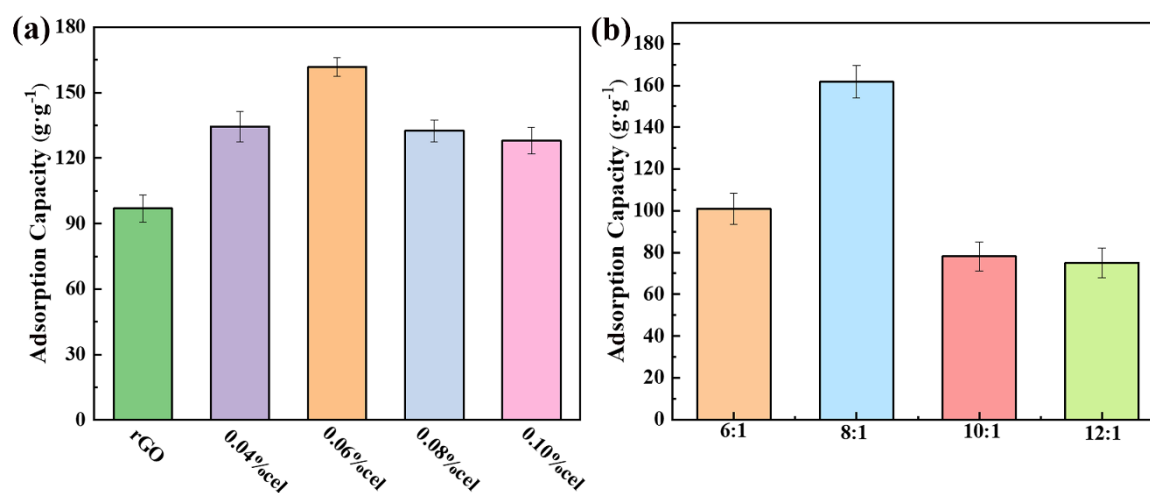


Fig. 2 Adsorption capacity of rGO/MWCNTs-NH₂/NC nanocomposite aerogels with different (a) NC weight percentages, and (b) mass ratio of GO to MWCNTs-NH₂.

In the Raman spectrum, Fig. 4(C), GO mainly has two obvious characteristic peaks, that is, G peak (1575 cm⁻¹, E_{2g} vibration model of sp^2 hybrid carbon atoms) and D peak (1350 cm⁻¹, defect structure of GO edge).^[19] The ratio for intensity of D peak and G peak (I_D/I_G) is computed from the Raman spectra to illustrate the defect in the samples and listed in the Fig. 4(C). It's found that I_D/I_G for GO, rGO aerogel, rGO/MWCNTs-NH₂ aerogel, rGO/NC aerogel, and rGO/MWCNTs-NH₂/NC nanocomposite aerogel is 1.24, 1.07, 1.21, 1.11 and 1.25. This implies that the rGO aerogel has less defect than other samples and the introduction of both MWCNTs-NH₂ and NC could increase the defect of rGO

aerogel.^[20]

Fig. 5 displays TEM images of rGO aerogel, rGO/MWCNTs-NH₂ aerogel, rGO/NC aerogel, and rGO/MWCNTs-NH₂/NC nanocomposite aerogel. It's noted that the TEM images for all the samples are translucent and there are many lamellar folds in the graphene layer. Compared with TEM image of rGO aerogel, Fig. 5(a), the MWCNTs could be discovered clearly in the rGO/MWCNTs-NH₂ aerogel, Fig. 5(b), and rGO/MWCNTs-NH₂/NC nanocomposite aerogel, Fig. 5(d). The MWCNTs is well distributed within the graphene layers without any agglomeration, suggesting that the presence of -NH₂ group

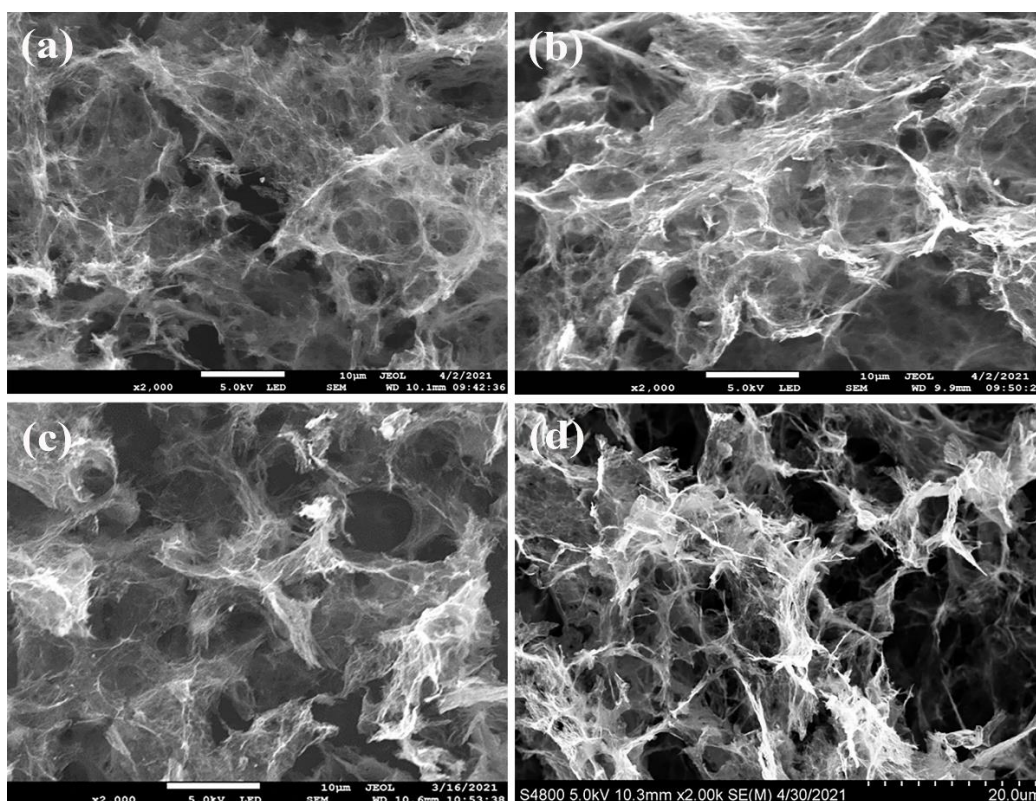


Fig. 3 SEM images of rGO/MWCNTs-NH₂/NC nanocomposite aerogels with mass ratios of GO to MWCNTs-NH₂ of (a) 6:1, (b) 8:1, (c) 10:1, and (d) 12:1.

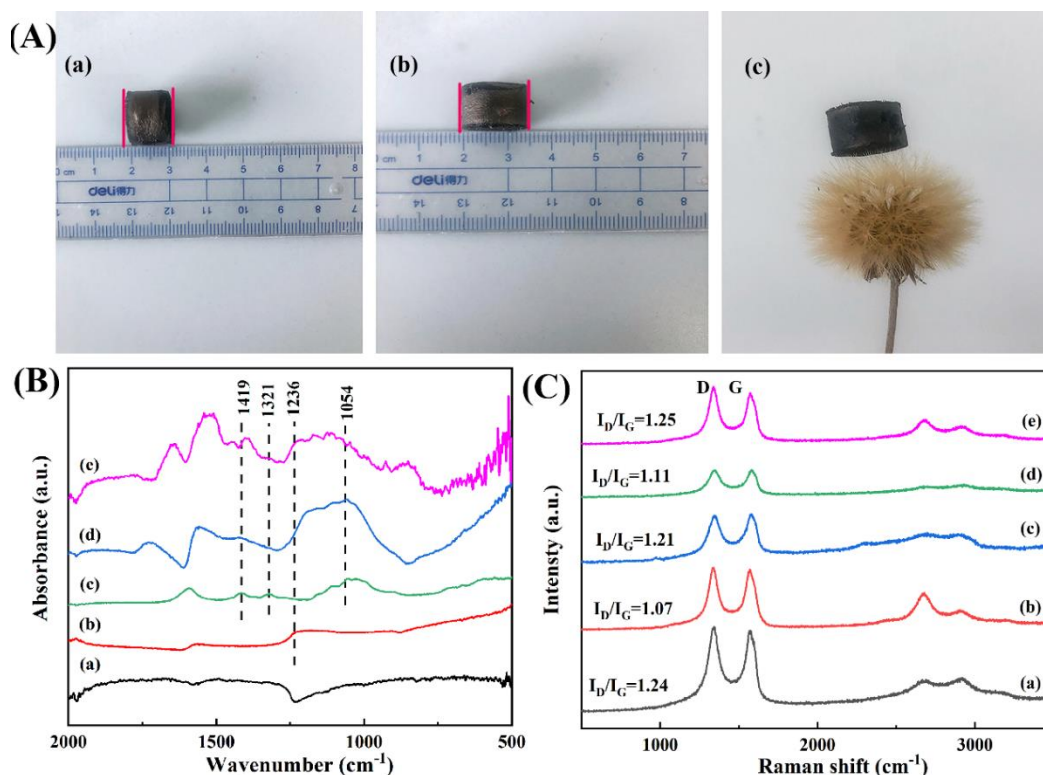


Fig. 4 (A) Digital photos of (a) height, (b) diameter measurements of rGO/MWCNTs-NH₂/NC nanocomposite aerogel, and (c) nanocomposite aerogel was placed on a dandelion; (B) FTIR spectra of (a) MWCNTs-NH₂, (b) rGO/MWCNTs-NH₂ aerogel, (c) NC, (d) rGO/NC aerogel, and (e) rGO/MWCNTs-NH₂/NC nanocomposite aerogel; (C) Raman spectra of (a) GO, (b) rGO aerogel, (c) rGO/MWCNTs-NH₂ aerogel, (d) rGO/NC aerogel, and (e) rGO/MWCNTs-NH₂/NC nanocomposite aerogel.

could help the dispersion of MWCNTs within the graphene layers. In addition, after adopting the NC, the lamellar folds become more, Fig. 5(c), relative to the pure rGO aerogel, Fig. 5(a). This may be due to the fact that the introduction of NC

could affect the dispersion of GO in the aqueous solution, further influencing the graphene layers in the aerogel.^[15]

In the nitrogen adsorption-desorption isotherm curves, Fig. 6, the BET specific surface areas of rGO aerogel, rGO/NC

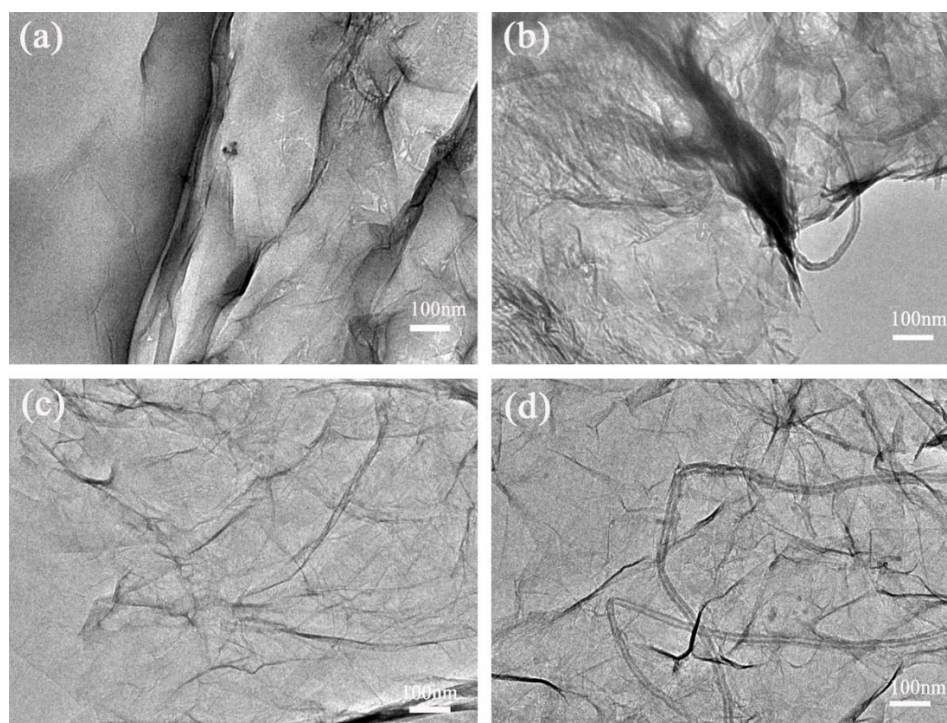


Fig. 5 TEM images of (a) rGO aerogel, (b) rGO/MWCNTs-NH₂ aerogel, (c) rGO/NC aerogel and (d) rGO/MWCNTs-NH₂/NC nanocomposite aerogel.

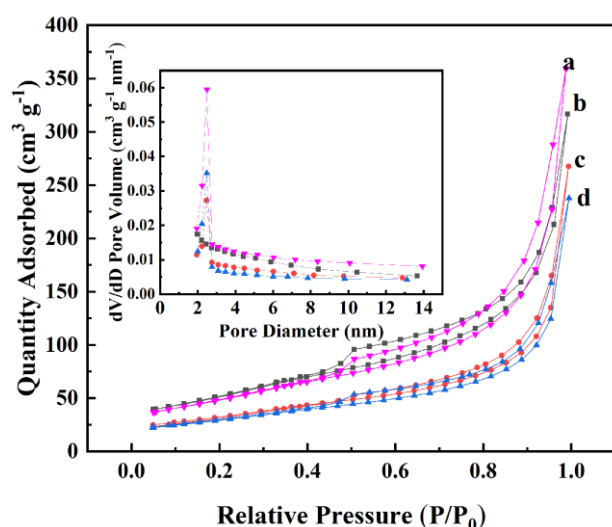


Fig. 6 Nitrogen adsorption-desorption isotherms and pore size distributions of (a) rGO/MWCNTs-NH₂/NC nanocomposite aerogel, (b) rGO aerogel, (c) rGO/NC aerogel, and (d) rGO/MWCNTs-NH₂ aerogel.

aerogel, rGO/MWCNTs-NH₂ aerogel, and rGO/MWCNTs-NH₂/NC nanocomposite aerogel are estimated to be 188.74, 119.15, 107.48, and 176.67 m² g⁻¹, respectively. The pore size distribution of rGO/NC aerogel, rGO/MWCNTs-NH₂ aerogel, and rGO/MWCNTs-NH₂/NC nanocomposite aerogel concentrates around at 2.3 nm, illustrating that their internal

structure possesses a uniform pore size distribution and belongs to the mesoporous materials, whereas that of rGO aerogel has no evident peak, signifying that its internal structure is non-uniform pore size.

3.3 Adsorption performance of rGO/MWCNTs-NH₂/NC

Before measuring adsorption capacity, the water contact angle tests are performed and the results are laid out in Fig. 7(A). The water contact angle for rGO aerogel, rGO/NC aerogel, rGO/MWCNTs aerogel, and rGO/MWCNTs-NH₂/NC nanocomposite aerogel is 91.09° (Fig. 7(A)-a), 91.31° (Fig. 7(A)-b), 95.15° (Fig. 7(A)-c), and 114.55° (Fig. 7(A)-d), respectively. This confirms that the incorporation of both MWCNTs-NH₂ and NC could effectively facilitate the hydrophobicity of rGO aerogel, which may be favorable for the adsorption of oil and organic solvents. The adsorption capacity of rGO/MWCNTs-NH₂/NC nanocomposite aerogel to ethyl acetate, dichloromethane, cyclohexane, acetone, and sesame oil is 161.76 ± 7.69, 156.14 ± 8.24, 92.73 ± 5.22, 103.87 ± 7.83, and 124.09 ± 6.64 g g⁻¹, respectively, as listed in Fig. 7(B). Later on, the oil/water separation experiment by rGO/MWCNTs-NH₂/NC nanocomposite aerogel is conducted in the petri dish without lid by applying Sudan Red B to dye the cyclohexane. The results are outlined in Fig. 7(C). It's noticed that the rGO/MWCNTs-NH₂/NC nanocomposite aerogel could distract cyclohexane from water within 20 s. It's

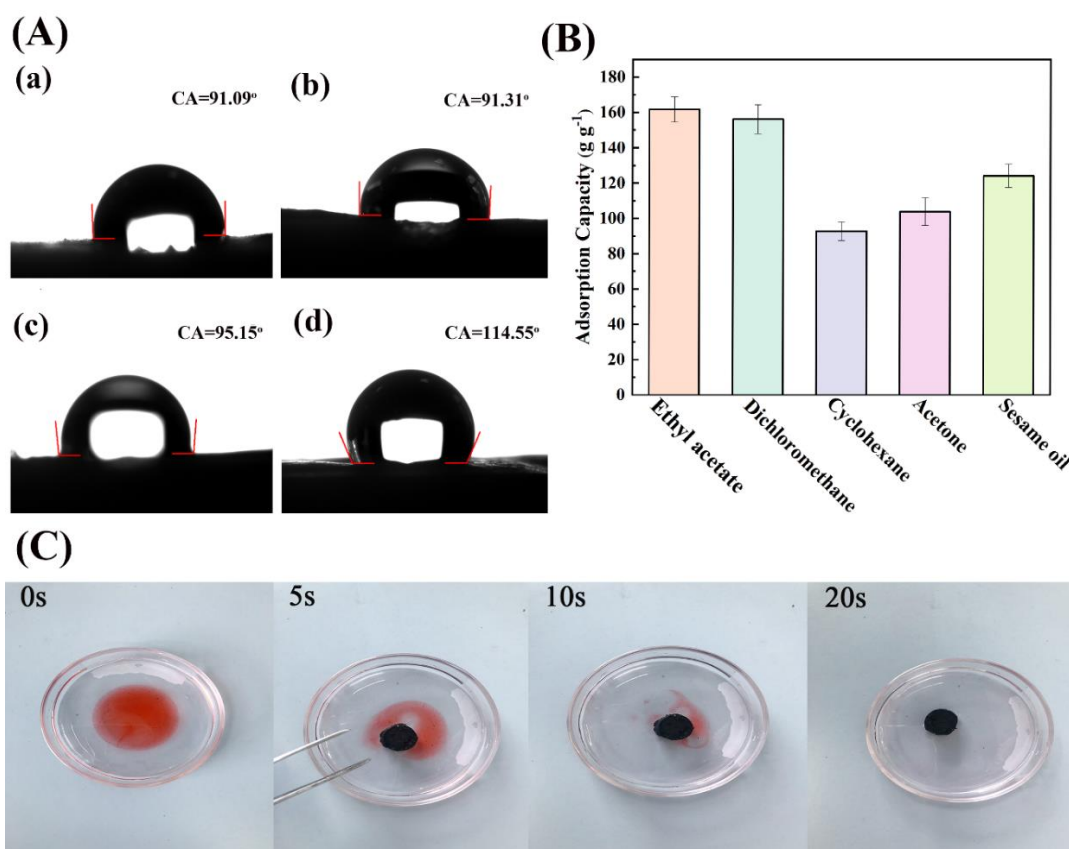


Fig. 7 (A) Water contact angle of (a) rGO aerogel, (b) rGO/NC aerogel, (c) rGO/MWCNTs-NH₂ aerogel, and (d) rGO/MWCNTs-NH₂/NC nanocomposite aerogel; (B) adsorption capacity of rGO/MWCNTs-NH₂/NC nanocomposite aerogel to ethyl acetate, dichloromethane, cyclohexane, acetone, and sesame oil; (C) oil/water separation experimental results of rGO/MWCNTs-NH₂/NC nanocomposite aerogel to cyclohexane.

concluded that our rGO/MWCNTs-NH₂/NC nanocomposite aerogel with fast oil/water separation capability and high oil adsorption capacity could serve as a potential candidate for the oil removal from wastewater.

4. Conclusions

In this work, we have prepared a novel rGO/MWCNTs-NH₂/NC nanocomposite aerogel. The FTIR, Raman, SEM and TEM results verify that the MWCNTs-NH₂ and NC are successfully introduced into rGO aerogel. The optimal condition for preparation of rGO/MWCNTs-NH₂/NC nanocomposite aerogel with high oil and organic solvent adsorption is the mass ratio of GO to MWCNTs-NH₂ of 8:1 with NC weight percentage of 0.06%. The adsorption capacity of rGO/MWCNTs-NH₂/NC nanocomposite aerogel to ethyl acetate, dichloromethane, cyclohexane, acetone, and sesame oil is 161.76 ± 7.69 , 156.14 ± 8.24 , 92.73 ± 5.22 , 103.87 ± 7.83 , and 124.09 ± 6.64 g g⁻¹, accordingly. This rGO/MWCNTs-NH₂/NC nanocomposite aerogel could separate oil and water within 20 s, exhibiting a high oil/water separation efficiency.

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Conflict of Interest

There is no conflict of interest.

Supporting Information

Not Applicable.

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