Superhydrophobic graphene-decorated mesh gauze: recycling oils and organic solvents enhanced by large-diameter capillary action

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ABSTRACT A novel recyclable, superhydrophobic and superoleophilic graphene-based textile sorbent was prepared for the removal of oil spills and organic pollutants, by functionalizing cotton mesh gauze with reduced graphene oxide (RGO) using dip-coating and hydrothermal reduction approach. The RGO-gauze composite textile showed efficient water/oil separation, and the amount of oil absorbed by the sorbent could be up to 11 times of its own weight. About 50(±5)% absorbed oil could be recycled by mechanical squeezing and it was mainly extracted from the reserve of large-diameter capillary action. The RGO-gauze maintained high structural stability and outstanding recyclability after eight cycles of oil removal. This RGO-gauze consumes a small quantity of graphene (0.6 g m⁻²) and can be produced in large scale, very suitable for application in the cleanup of oil spill and organic solvent effluent.

Keywords: superhydrophobic, graphene, mesh gauze, capillary action, oil removal

INTRODUCTION

Our water resources face serious threat from the spillage of oil and its derivatives during their exploration, transportation, storage and usage [1,2]. The most urgent countermeasure to deal with the emergency spread of these pollutants is collecting them from water with the assistance of sorbents [3–7]. To achieve highly efficient oil-water separation, superhydrophobic, superoleophilic and highly porous sorbent materials are developed in recent years, which involve metal meshes [8–11], textiles

[12–17], foams [18–23], membranes [24–26], aerogels and so forth [27,28]. Particularly, textiles are the most attractive candidates in large-scale applications, like dozens of miles of protection belt used in marine oil spillage, due to their abundance, good mechanical durability, high flexibility and some other excellent properties. Superhydrophobic textiles were generally fabricated by roughening their skeletons/surfaces with micro/nanomaterials, such as ZnO and SiO₂ nanoparticles [29–33], layered double hydroxide microcrystals [34,35], carbon nanotubes and reduced graphene oxide (RGO) as modifiers [36,37]. Among these decorations, RGO is the promising one not only because of its large specific surface area and oleophilic features, but also because its graphene oxide precursor behaves like macromolecules that can wrap each fiber in yarns by self-assembly. In addition, RGOs themselves in foam and aerogel structures have demonstrated remarkable performance in oil adsorption and recovery, although their cost, output and mechanical properties are largely behind application requirement [38,39]. Hence integrating textiles and RGO together to combine their advantages allows us to develop high-quality and mass production-scale sorbents.

The uptake of oil or organic solvents by porous sorbents is a complex process, probably through adsorption, absorption and capillary action mechanism [40]. In adsorption, organic molecules are in general attracted and restricted to the surface of the sorbent from the solution-adsorbent interface. Absorption is a process in which the molecules

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transport from liquid into sorbent phase and migrate beneath at least several nanometers. Capillary action occurs in pores with diameters ranging from nanometer to centimeter scale where the solute flows into the pores without the assistance of external force. The interaction force between the solute and sorbent in a capillary pore is inversely proportional to the pore diameter. Regarding oil recovery from sorbents by squeezing or twisting, small liquid-solid interaction is preferred if the sorption amount is not reduced. Hence it is of great interests to prepare oil sorbents using large pore size textiles to low liquid-solid interaction.

In this study, we selected cotton mesh gauze (warp: weft $21\text{ s} \times 32\text{ s}$) with average pore size of 0.6 mm as the substrate to prepare graphene-gauze sorbent. This gauze has downy surface because many cotton fibers protrude from yarns as illustrated in Scheme 1. The gauze was dipped into GO solution to allow GO nanosheets wrapping each fiber spontaneously. Then the dried GO-gauze was reduced in hydrothermal condition with the aid of hydrazine hydrate. The resulting RGO-gauze replicated the template morphology well and presented superhydrophobic and superoleophilic properties as well as water-oil separation capability. The first-run sorption capacitance of RGO-gauze for soybean oil is 11 g g^{-1} and about $20(\pm 2)\%$ of which is ascribed to large-diameter capillary action. The capillary part that bridged by mesh could be easily recovered by squeezing. More importantly, our RGO-gauze consumes a small quantity of graphene (0.6 g m^{-2}) and can be produced in large scale, very suitable for future industrial production.

EXPERIMENTAL SECTION

Preparation of RGO-gauze composite

GO aqueous dispersion was prepared as described in our previous work, whose concentration was controlled to be

Scheme 1 The preparation method of graphene coated meshed gauze.

1 mg mL^{-1} [41]. A piece of commercial cotton gauze (purchased from Xinxiang Hongda Textile, the yarn count was warp: weft $21\text{ s} \times 32\text{ s}$ with average pore size of 0.6 mm) with area of 1.5 m^2 was cleaned by sonication in deionized (DI) water before immersed into 1000 mL GO solution. Then the wet gauze was taken out and dried in oven at 70°C for 10 min . The dip-coating process was repeated for 5 times. Then the dried GO-gauze composite was put into a 1000 mL Teflon-lined autoclave with 600 mL DI water and 10 mL hydrazine hydrate and then reduced at 90°C for 12 h . Finally, the obtained RGO coated mesh gauze was washed with DI water for three times to remove the residuals and dried at 70°C .

Absorption capacity test

The absorption capacity test was taken by a piece of RGO-gauze ($5\text{ cm} \times 6\text{ cm}$) that was put into various organic solvents and oils until saturation. The sorption capacity (k) was defined as the mass ratio of sorbed oil (m_{ii}) to the dry RGO-gauze (m_{i}).

Characterization

The products were characterized by scanning electron microscopy (SEM, Hitachi S-4800), Fourier transform infrared spectrometer (FTIR, Bruker VECTOR-22), X-ray photoelectron spectroscopy (XPS, VersaProbe PHI5000) and Raman spectroscopy (Renishaw in Via Raman Microscope with an argon-ion laser at an excitation wavelength of 523 nm). Contact angle (CA) was measured with $3\text{ }\mu\text{L}$ droplets of water and soybean oil using a Krüss DSA 100 (Krüss Company, Germany) apparatus at ambient temperature (The CA was measured from seven samples). The optical images were captured by a digital camera (Sony) and an optical microscope (Nikon ECLIPSE LV100N POL).

RESULTS AND DISCUSSION

Fig. 1a shows the photograph of the pristine cotton mesh gauze and RGO decorated gauze, whose sizes are $1.5\text{ m} \times 1\text{ m}$. White gauze was turned to black when RGO was coated. More interesting, the RGO-gauze could float on water surface while the raw gauze sank into water (Fig. 1b). On the RGO-gauze surface, a water droplet almost kept sphere morphology and stayed stable for a long time while an oil droplet was quickly absorbed in a few seconds (Fig. 1c). The detail wettability of the pristine and RGO-decorated gauze was analyzed by water contact angle measurement, given in Figs 1d and e. Quite different wetting processes were observed for these two gauzes. Pristine gauze is hydrophilic and can absorb water quickly. In contrast,

Figure 1 (a) Photograph of the gauze and RGO-gauze with size of 1.5 m × 1 m; (b) original gauze (down) and RGO-gauze (up) after being placed in water; (c) a water droplet (left) and an oil droplet (right) dyed with Sudan II on the RGO-gauze; (d and e) video snapshots of the contact angle of a water droplet placed onto the surface of gauze and RGO-gauze.

RGO-gauze is superhydrophobic with contact angle of $155.3(\pm 0.3)^{\circ}$. In order to understanding the role of RGO, the same treatment was also carried out for a neat cotton gauze as that for GO-gauze. The FTIR, oil and water adsorption tests clearly indicate that without RGO coating, the gauze features the unchanged surficial functional groups and wettability before and after the hydrazine treatment. All these results indicate that after RGO coating, gauze is tuned from hydrophilicity to superhydrophobicity (Fig. S1, Supplementary information). In addition, the superhydrophobic RGO-gauze also shows superoleophilic property (Fig. S2) as expected, which favors its application in oil/water separation.

The microstructure of the gauze before and after RGO coating was observed by SEM. Figs 2a–c show the SEM images of the pristine gauze. It is seen that the meshes in gauze are knitted from warp and weft yarns with pore size of 0.6 mm. Each yarn is composed of dozens of loosely twisted fibers with diameter of 10–15 μm so that they produce bulk and space among the fibers. Specifically, the space in porous fiber bundles can entrap numerous air pockets,

which is necessary to achieve surface superhydrophobicity. After RGO coating, the structure of gauze does not show any obvious change (Figs 2d–e). But for fibers, their surfaces become rough because of the wrinkles of RGO sheets that inherits from GO (Fig. S3). The Brunauer–Emmett–Teller (BET) surface areas of the pristine gauze and RGO-gauze are 3.24 and 4.69 $\text{m}^2 \text{g}^{-1}$, indicating that the surface area is increased by RGO decorating. A significant change after RGO coating is that the hydrophilic hydroxyl groups on cellulose fibers was screened. The FTIR spectra, Raman spectra and XPS analysis (Figs S4 and S5) clearly indicate that RGO on fibers with little amount of oxygen containing groups was obtained, forming a hydrophobic surface. The water contact angle of flat RGO/glass film is $70.2(\pm 0.2)^{\circ}$ (Fig. S6). Hence the superhydrophobic features owned by RGO-gauze should result from the synergic effect of porous fiber bundles and their hydrophobic surface.

To understand the sorption capacity of RGO-gauze, several kinds of oils and organic solvents were evaluated in this study, including pump oil, soybean oil, diesel oil, glycol,

Figure 2 SEM images of the gauze (a, b and c) and RGO-gauze (d, e and f). (b) and (e) are SEM images of fibers in gauze and RGO-gauze, respectively. (c) and (f) are the corresponding enlarged images of (b) and (e).

Figure 3 (a) The absorption capacity of RGO-gauze on different oils and organic solvents; (b) absorption capacity calibrated by the mass loading of RGO; (c and d) optical microscope images of capillary action of water (c) and soybean oil droplet (d) within two parallel RGO-decorated yarns.

acetone, dimethyl formamide (DMF) and chloroform. The RGO-gauze can efficiently recover oil or insoluble organic solvents from water, indicating the good oil/water absorption selectivity (Fig. S7). In general, RGO-gauze sorbs the liquids at 5 to 11 times of its own weight (Fig. 3a). Since

the loading mass of RGO was as little as 0.6 g m^{-2} , the sorption efficiency calibrated by graphene will be as high as 650 to 1300 times to the weight of the RGO (Fig. 3b), much greater than neat carbon-based materials [26,32,33,42–46]. As mentioned above, capillary action plays a main role in

oil sorption. This could be well understood by the following demonstration. Figs 3c and d show the capillary of water and soybean oil on two parallel yarns that extracted from RGO-gauze. The space of these two parallel yarns was controlled to be 600 μm . The liquid droplets were bridged by the parallel yarns. The water droplet is in convex meniscus and none penetrates into the superhydrophobic fibers. In contrast, a large part of soybean oil droplet is absorbed into the fiber bundles and the remained generate concave meniscus between the yarns. To roughly evaluate the amount of oil taken by capillary from the large pores of RGO-gauze, a piece of RGO-gauze (2 cm $\times$ 2 cm) was disassembled to separated yarns (Fig. S8). By calculating

the difference of sorption amount between the unbroken gauze and separated yarns, the oil uptake contributed from large-diameter capillary is about 20($\pm$ 2)%.

The recyclability of oil and reusability of absorbents are important properties required for oil recovery. The recycling oils and organic solvents as well as the regeneration of RGO-gauze are illustrated in Figs 4a and b by squeezing and acetone washing regeneration, respectively. For these small molecule organic solvents such as acetone and DMF, the removal efficiency of RGO-gauze almost keeps the same as the fresh one due to the self-regeneration by solvent evaporation. But for those oils with large viscosity and high evaporation temperature, only about 50($\pm$ 5)% absorbed oils

Figure 4 (a) Recyclability of RGO-gauze; (b) repeat runs of soybean oil removal by acetone washing regeneration; (c and d) optical microscope images of RGO-gauze with the sorption of soybean oil before (c) and after squeezing (d); (e) contact angle of RGO-gauze after 8 times cycling tests; (f) SEM image of the RGO coated fibers in the regenerated RGO-gauze.

can be recovered by mechanical squeezing. The oil recovery efficiency also keeps constant of 50(±5)% in recycling use tests. Figs 4c and d show the photographs of soybean oil saturated RGO-gauze before and after mechanical squeezing. It is seen that the oil in holes is removed by squeezing and the yarns could be easily distinguished, suggesting that a large amount of recycling oil should come from that kept by capillary action. Indeed, the oil uptake by large-diameter capillary makes the oil recovery easy, which allows RGO-gauze suitable for practical application. The full regeneration of the used RGO-gauze could be achieved by washing them in acetone. The absorption ability of RGO-gauze remained essentially identical after 8 cycles of testing. In addition, the wettability of the clean RGO-gauze still keeps superhydrophobicity with contact angle of 152.7(±0.5)° (Fig. 4e) and each fiber is well coated by RGO (Fig. 4f), indicating the outstanding mechanical stability owned by this absorbent.

CONCLUSIONS

Large scale superhydrophobic and superoleophilic RGO-gauze was developed by taking advantages of the porous fiber bundles of gauze and hydrophobic property of RGO coating. The RGO-gauze delivered excellent oil absorption properties and recyclability, structural flexibility and robustness, and little graphene consumption (0.6 g m⁻²). This sorbent can selectively remove oils and insoluble organic solvents from water. About 20(±2)% oil uptake comes from the capillary action of the large-diameter holes in gauze mesh, which allows 50(±5)% absorbed oils that can be recovered by mechanical squeezing. Moreover, the efficiency was retained over several cycles of the absorption/squeezing process. Our results indicate that the advanced textile sorbent not only using the adsorption from the superhydrophobic yarns but also employing the capillary action of holes in mesh. The high-performance RGO-gauze offers great potential for treatments of oil spills and industrial effluent.

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超疏水石墨烯修饰纱布: 通过大孔径毛细作用提高油与有机溶剂的回收能力

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摘要 本论文采用浸渍涂覆和水热还原方法将还原氧化石墨烯沉积于棉纱布纤维表面, 制备了一种新型可回收的超疏水、超亲油性石墨烯基织物吸附材料. 该石墨烯基织物具有高效的油/水分离能力, 其吸油量可达自身质量的11倍以上. 约50(±5)%的吸附油可通过机械挤压方式回收, 其中大部分来源于石墨烯纱布网格间的大孔径毛细作用. 经过8次吸油/回收反复过程, 石墨烯纱布仍然具有良好的吸油能力, 表现出高稳定性. 该方法可大规模制备石墨烯纱布, 石墨烯活性材料用量少(0.6 g m⁻²), 特别适用于油污和有机污水的回收应用.