Filter Paper with Selective Absorption and Separation of Liquids that Differ in Surface Tension

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ABSTRACT Superhydrophobic and superoleophilic filter paper was successfully prepared by treating commercially available filter paper with a mixture of hydrophobic silica nanoparticles and polystyrene solution in toluene. Applications of the filter paper in separating liquids with low surface tensions such as oil and ethanol from water were investigated in detail. The oil uptake ability of the superhydrophobic filter paper was evaluated and the results show that the filter paper can selectively adsorb oil floating on a water surface or in aqueous emulsions. Furthermore, filtration of mixtures of oil and water through the paper can reduce the water content in the oil. Additionally, the filter paper can also extract ethanol from homogeneous aqueous solution.

KEYWORDS: superhydrophobic coating • filter paper • surface tension • oil adsorption • separation

1. INTRODUCTION

Wettability is important for various kinds of solid surfaces (1, 2). Superhydrophobic surfaces typically have a water contact angle >150° and a low sliding angle (<10°). Such surfaces are often present in nature on plant leaves (3, 4), on legs of water striders (5), and in troughs on the elytra of desert beetles (6). On these surfaces, water droplets bounce up and rapidly slide off while washing away powderlike contamination, thereby rending superhydrophobic surfaces with a self-cleaning ability. Studies of natural examples have revealed that this hydrophobicity depends on the chemical composition and the geometric structure of the surface (7−10). Various artificial superhydrophobic surfaces have been fabricated by constructing special hierarchical roughness and modifying surfaces using materials with low free energy (11−16). For instance, Pauporté and co-workers fabricated zinc oxide nanowire arrays on a fluorine-doped tin oxide substrate. The as-grown layers were converted from superhydrophilic to superhydrophobic surfaces by derivatization with stearic acid (15). Fréchet et al. developed a method to copolymerize butyl methacrylate and ethylene dimethacrylate in the presence of a porogenic solvent; the resulting porous polymer surface showed superhydrophobic properties due to micro- and nanoscale roughness (16).

Besides experimental investigations, theoretical studies have revealed that liquids with different surface tension γ_{lv} exhibit varying wettability on the same surface. For example, a water droplet ($\gamma_{lv} = 72.1$ mN/m) easily slips over the superhydrophobic surface of a lotus leaf, whereas a drop of liquid with lower surface tension, such as hexadecane ($\gamma_{lv} = 27.5$ mN/m), spreads quickly over the surface, showing a superoleophilic property (17). Special wetting phenomena on rough surfaces have been theoretically explained by two commonly accepted models, the Cassie−Baxter model (10) and the Wenzel model (9).

Because of its low surface tension, oil can easily wet and penetrate many superhydrophobic surfaces. It has been reported that superhydrophobic and oleophilic manganese oxide nanowire membrane can selectively collect oil on a water surface (18). In spite of potential toxicity, their study suggested that fiberlike microstructures combined with low-free-energy surface modifications possess oil−water separation capability. Some attempts to use superhydrophobic and superoleophilic meshes to separate oil−water mixtures have been made (19−23). For instance, Jiang et al. coated copper meshes with composite coatings consisting of polytetrafluoroethylene and polyvinyl acetate (19). More recently, Lin et al. etched copper meshes with nitric acid followed by modification with 1-hexadecanethiol (20). The as-prepared superhydrophobic and superoleophilic meshes were used to effectively separate oil and water. However, there are few reports on the separation efficiency of such approaches. Filter paper is widely used as a microtextured and porous material in solid−liquid and solid-air separations. Fabricating filter papers with the capability for liquid−liquid separation or adsorption of low-surface-tension liquid from aqueous emulsions, even from miscible liquids, by the coating of superhydrophobic materials would be of both academic and practical significance. In this work, we made use of a facile and inexpensive approach to fabricate superhydrophobic and superoleophilic composite coatings on filter paper. The selective adsorbing capacity of the treated filter paper and its separation effect toward liquids with different surface tensions were investigated. Interestingly, the treated filter

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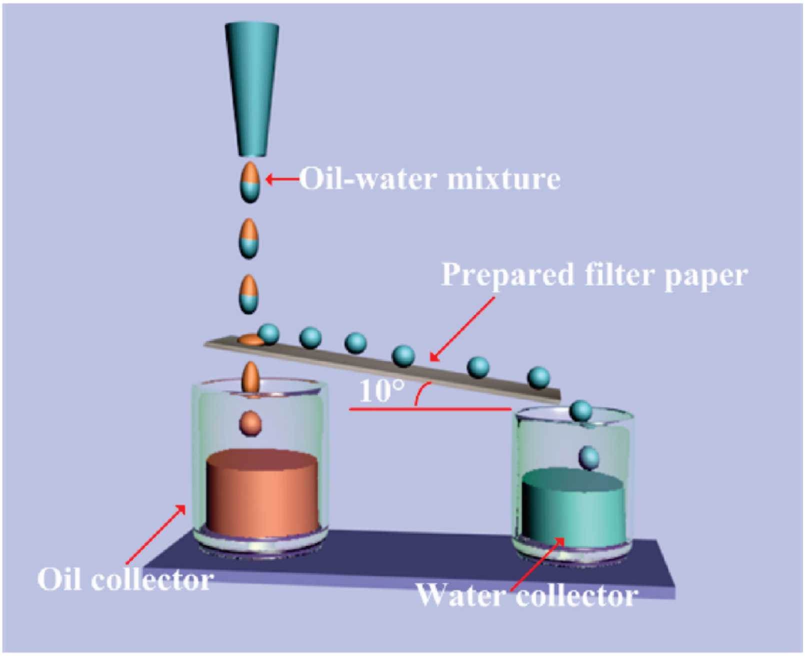

FIGURE 1. Experimental setup for the separation of oil and water.

paper also could be used in the extraction of ethanol from homogeneous aqueous solution. This work is helpful to gain an insight into controlling surface wettability to realize separation of liquids and our results reveal that the filter paper treated with a superhydrophobic coating renders it able to separate a mixed solution with liquids of different surface tensions.

2. EXPERIMENTAL SECTION

2.1. Preparation of Superhydrophobic and Superoleophilic Filter Paper. Polystyrene grains (PS, MB_{wB} 170 000 g mol^{-1} P, average particle diameter 2 mm, BASF) or Polyethylene terephthalate grains (PET, M_w 30 000 g mol^{-1}, average particle diameter 1 mm, DuPont) were dissolved in toluene and then hydrophobic silica nanoparticles (H-SiO_2, silica modified with polydimethylsiloxane, average particle size 14 nm, Evonik Degussa) were ultrasonically dispersed for 0.5 h in the solution. The filter paper was steeped in the mixed solution for approximately 15 s, removed, and dried in an oven at 60 °C for several hours to evaporate the solvent.

Separation of oil and water was carried out in the setup as illustrated in Figure 1. A superhydrophobic filter paper was placed over two beakers at a tilt angle of 10°. When a mixture of oil and water was dropped onto the filter paper, the diesel oil penetrated through the paper and fell into the beaker beneath it, whereas the water droplets rolled along the paper surface and dropped into the other beaker.

2.2. Characterization. The morphology of the resulting surfaces was investigated using a Hitachi-S4500 scanning electron microscope (SEM) at an accelerating voltage of 15 kV. The contact angles were measured using an OCA 20 contact angle system (Dataphysics Instruments GmbH, Germany) at ambient temperature. Droplets of 4 μL of ultrapure water or diesel oil were dropped onto the samples using an SNS 021/011 needle on a microsyringe. The average of five measurements taken at different positions on each sample and calculated under ellipse fitting mode was adopted as the static contact angle. The sliding angle was defined as the angle between the tilted sample and the horizontal plane when an 8-μL water drop just started to roll off from a slowly inclined surface. The content of water in oil was measured using a Mitsubishi CA-200 moisture meter with VA-210 vaporization unit. The refractive index of water–ethanol mixtures was recorded on a BM-2W Abbe refractometer. The diameter of the emulsified oil was determined using a Malvern Zetasizer Nano S photon correlation spectroscope.

3. RESULTS AND DISCUSSION

3.1. Superhydrophobic Composite Coatings on Filter Paper. Filter paper usually consists of wood fibers and can quickly adsorb both water and oil on account of its high surface free energy. Therefore, the toluene solution of polystyrene (PS, 1 wt %) containing suspended hydrophobic SiO_2 nanoparticles (H-SiO_2, the weight ratio of H-SiO_2 to PS is 1:1) can quickly spread and penetrate the filter paper, and subsequent drying yielded a composite coating on the filter paper with superhydrophobic properties. As shown in Figure 2, a water droplet could sit on the filter paper surface with a water contact angle of 157 ± 2° (Figure 2a). An 8 μL water droplet easily rolled off the surface when the paper was tilted by only 4° (Figure 2b), indicating a Cassie–Baxter nonwetting state. By contrast, diesel oil, a liquid with low surface tension, spread quickly on the modified filter paper and permeated it thoroughly (Figure 2c), showing highly oleophilic properties. Scanning electron microscopy (SEM) images (Figure 3) reveal that the H-SiO_2 nanoparticles were densely distributed on the surface of microscale paper fibers, forming nanoscale roughness on the three-dimensional netlike topography and leading to superhydrophobicity of the resulting filter paper (24). In the rough coating, PS acts as a binder to adhere the nanoparticles on the paper fibers.

PS is slightly hydrophobic and the water contact angle on a smooth PS surface is approximately 98° (25), whereas diesel oil spreads quickly, showing oleophilic properties. Wenzel et al. indicated that surface roughness could amplify the wettability of materials, making a hydrophobic surface more hydrophobic and an oleophilic surface more oleophilic

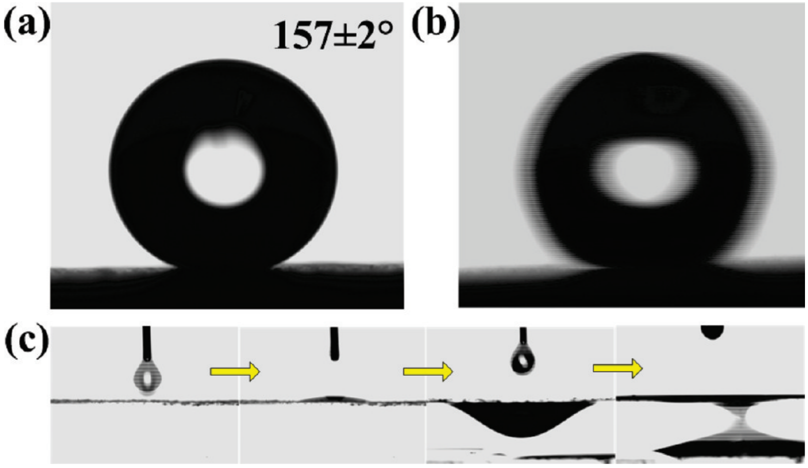

FIGURE 2. (a) Water droplet sitting on the superhydrophobic filter paper. (b) Eight-microliter water droplet sliding off when the paper was tilted by 4°. (c) Diesel oil droplet spreading on and permeating through the paper.

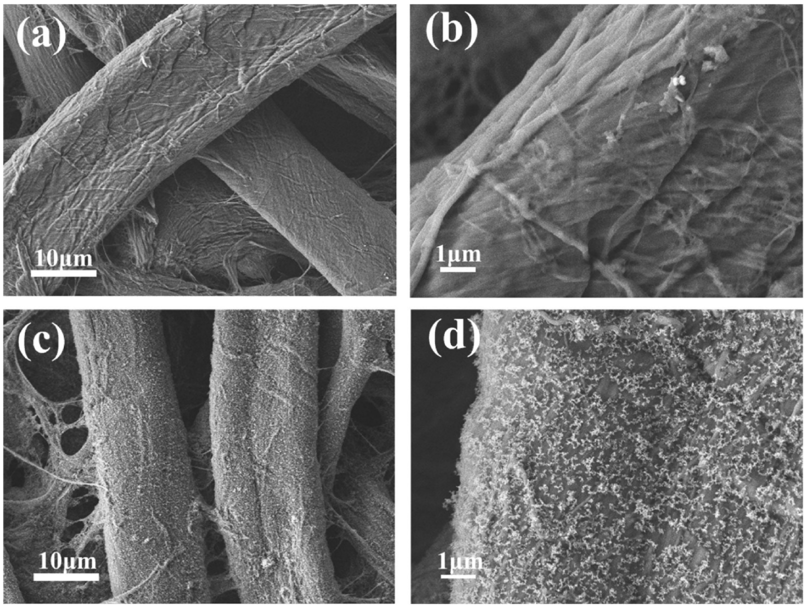

FIGURE 3. SEM images of (a, b) raw filter paper surface and (c, d) superhydrophobic filter paper surface treated with H-SiO_2 nanoparticles.

(9). Filter paper coated with PS (from a 3 wt.% solution in toluene) was highly hydrophobic because of the hydrophobicity of PS and the microscale roughness of the filter paper fibers. The water contact angle on this surface was 143 ± 2° (Table 1). It has been reported that PS membranes fabricated by electrospinning can have a water contact angle as high as 160° (26). However, the sliding angle of a water droplet on the surface has not been reported. In the present study, PS-coated filter paper was so sticky that a water drop was pinned on the surface even when the paper was turned upside down. Jiang and co-workers prepared a strongly adhesive surface consisting of an aligned PS nanotube layer with a water contact angle >150° (27). They proposed that the adhesion was mainly caused by intimate contact between PS and water. Johnson and Dettre theoretically predicted that an increase in the air fraction at the interface between a water drop and a solid surface could lead to a switch in the dominant wetting state from a Wenzel to a Cassie–Baxter regime (28). Accordingly, we introduced H-SiO_2 nanoparticles into the composite coating to improve the amount of microprotrusions and further decrease the surface energy, which are believed to be helpful in trapping air bubbles at the interface. In our experiment, when the weight ratio of H-SiO_2 to PS increased from 0.05:1 to 0.25:

Table 1. Water Contact Angle (WCA) and Sliding Angle (SA) for Filter Paper Treated with PS Solutions with Different Concentrations

	PS coating	
PS (wt %)	WCA (deg)[a]	SA (deg)[b]
3	143 ± 2	>90
1	139 ± 2	>90
0.5	135 ± 1	>90
0.375	129 ± 3	>90
0.25	127 ± 2	>90
0.125	0	>90

[a] An angle of 0° indicates that the water drop spread on the surface in 20 s. [b] An angle >90° indicates that the water drop was pinned on the surface, even when the paper was turned upside down.

Table 2. Water Contact Angle (WCA) and Sliding Angle (SA) for Filter Paper Treated with PS-H-SiO_2 Suspensions with Different Raw Materials Ratios and Concentrations

PS (wt %)	H-SiO_2 (wt.%)	PS-H-SiO_2 coating	
		WCA (deg)[a]	SA (deg)[b]
1	0.05	139 ± 2	>90
1	0.125	140 ± 1	>90
1	0.25	144 ± 1	33
1	0.5	152 ± 2	5
1	1	157 ± 2	4
3	3	158 ± 1	4
0.5	0.5	152 ± 1	6
0.375	0.375	149 ± 2	35
0.25	0.25	142 ± 2	>90
0.125	0.125	0	>90

[a] An angle of 0° indicates that the water drop spread on the surface in 20 s. [b] An angle >90° indicates that the water drop was pinned on the surface, even when the paper was turned upside down.

1, the water contact angle on the treated filter paper slightly increased from 139 ± 2° to 144 ± 1° and the sliding angle of an 8 μL water drop decreased sharply to 33° (Table 2). A further increase in weight ratio to 0.5:1 resulted in a significant decrease in sliding angle to 5°, reflecting a Cassie–Baxter nonwetting state.

We also investigated the influence of the weight fraction of PS (the weight ratio of H-SiO_2 to PS was 1:1) on the wettability of the modified filter paper (Table 2). Dilution of the coating suspension from 3 to 1 wt % PS with toluene did not lead to an obvious change in water contact angle. However, after a further dilution to 0.25 wt % PS the superhydrophobicity disappeared. Moreover, when the weight fraction of PS was decreased to 0.125 wt %, a water droplet spread on the surface within 20 s.

Evaluation of the stability revealed that water contact angles were in the range 151–159° when the superhydrophobic surface was exposed to aqueous solutions of pH 1–14 (see Figure S1 in the Supporting Information). These results demonstrate that the superhydrophobic surface is inert to corrosive liquids and is tolerant to acidic and basic conditions.

In addition, by steeping filter paper in a toluene solution of polyethylene terephthalate (PET, 1 wt %) containing suspended H-SiO_2 nanoparticles (the weight ratio of H-SiO_2 to PET was 1:1) and drying, a superhydrophobic and superoleophilic composite coating was also obtained.

3.2. Selective Adsorption of Oil. As mentioned above, the modified filter paper showed superhydrophobic and highly oleophilic properties. When the paper was immersed into water, it strongly repelled water and remained dry after being taken out. However, when there was a layer of diesel oil on the water surface, the modified filter paper selectively adsorbed the diesel oil with a capacity of 3.4 g/g as soon as it touched the oil surface (Figure 4 and Video S1 in the Supporting Information). Quéré deduced that liquid with an intermediate contact angle between 0 and 90° could invade textured substrates. The driving force of the so-called "hemi-wicking phenomenon" came from the decrease in surface energy as a result of the displacement of a solid/vapor interface to a liquid/vapor interface (29). In our case, the low surface energy and large water contact angle of the modified filter paper resulted in high repellence of water, whereas complete wetting by oil renders an oil film that quickly develops from a drop and spreads over the whole paper.

In addition, the modified filter paper could adsorb oil and a collection of nonpolar organic solvents such as hexane, octane, and dodecane. As important criteria for real applications, the stability and recyclability of the modified filter paper were also evaluated. After the first saturated adsorption of diesel oil and organic solvents, the modified filter paper was dried thoroughly in an oven at 80 °C for several hours. The water contact angle and the oil uptake capacity were measured again. The process was repeated four times and the results are shown in Figure 5. The water contact angle after four experiments remained unchanged and the amount of oil and organic solvent adsorbed was almost constant.

Selective removal of oil from aqueous emulsions is an important process in many industry applications such as oil extraction and water purification (30, 31). To investigate the selectivity of the modified filter paper, an emulsion with 6 vol % diesel oil and 1.5 wt % sodium lauryl benzenesulfate

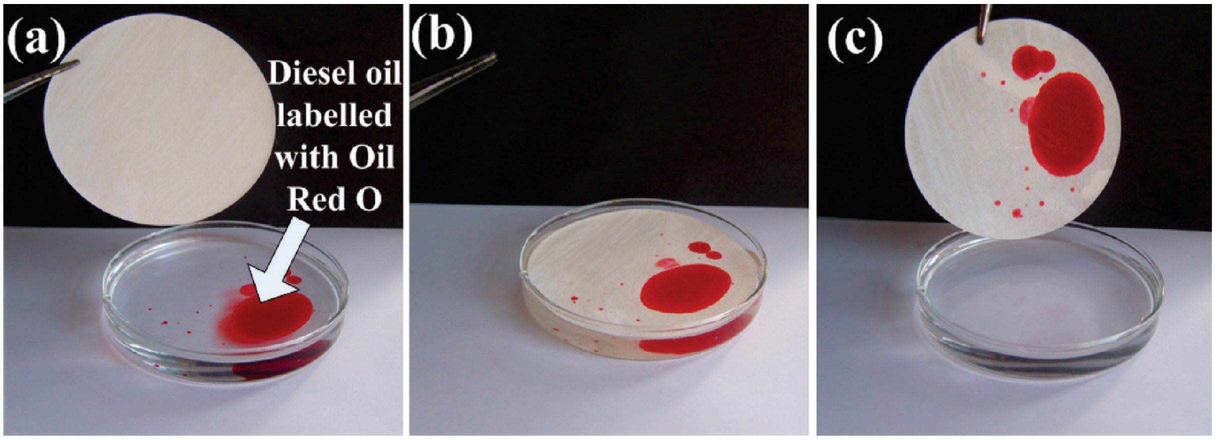

FIGURE 4. Removal of diesel oil from a water surface. The oil was labeled with Oil Red O for easy observation.

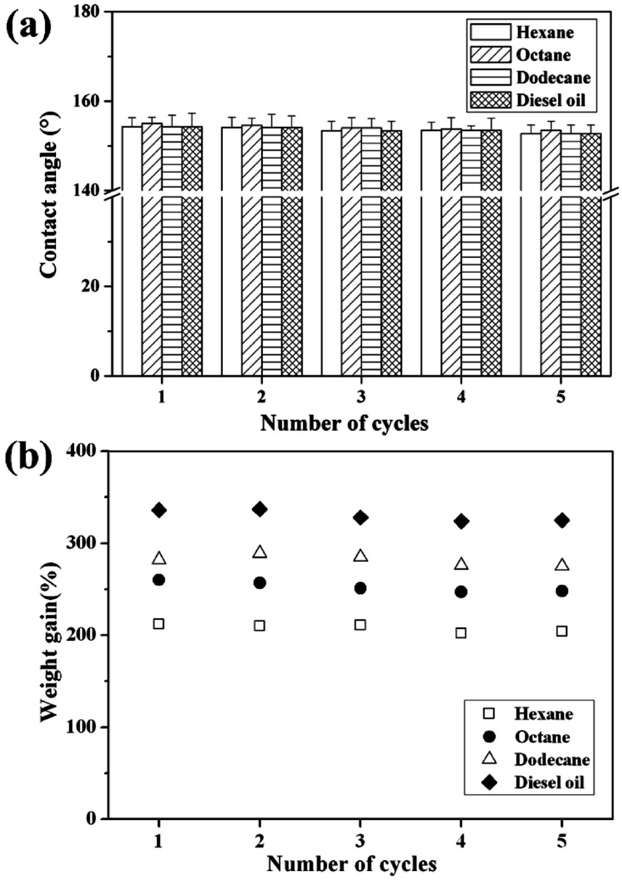

FIGURE 5. (a) Water contact angle showing the filter paper remains superhydrophobic properties after oil absorption. (b) Percentage weight gain after adsorption and evaporation of different organic solvents for five cycles illustrating the oil uptake capacity of the filter paper is stable.

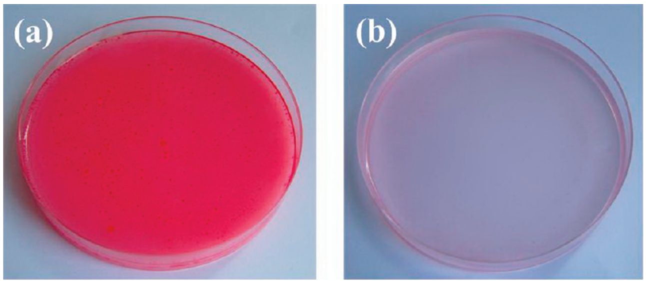

FIGURE 6. Removal of emulsified diesel oil from water. (a) Optical image of diesel oil (6 vol %) emulsified by addition of sodium lauryl benzenesulfate (1.5 wt %) in water. (b) Optical image after removal of the emulsified oil by placing superhydrophobic filter paper in the emulsion for 90 s.

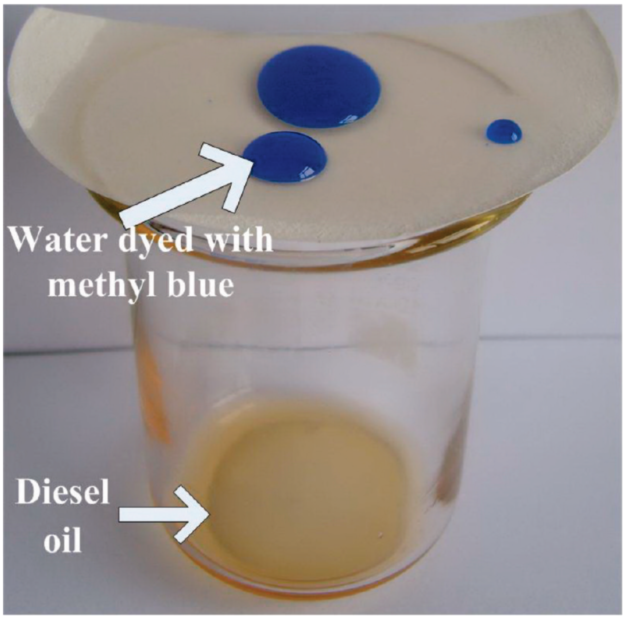

FIGURE 7. When a mixture of water (dyed with methyl blue for easy observation) and diesel oil was poured on the filter surface, diesel oil passed through the surface, whereas water droplets were held on the surface.

in water was prepared. The average diameter of an emulsified oil drop was 225 nm. When the filter paper was inserted into the emulsion, the emulsion became clear within 90 s as a result of selective oil adsorption by the superhydrophobic filter paper (Figure 6; Videos S2−1 and S2−2 in the Supporting Information). Because of the global scale of water contamination arising from oil spills and other industrial organic pollutants, there is an increasing demand for porous materials that can selectively remove oil and organic pollutants. Its special adsorption ability and reproducible and stable properties mean that the superhydrophobic filter paper is an ideal candidate for potential applications in the field of oil spill clean-ups. Moreover, the coating process is relatively facile and can be easily extended to various types of waste paper and porous fibers at very low cost.

For comparison, we also examined two commercial membranes, polyvinylidene fluoride (PVDF) and Nylon membranes with water contact angles of 143 and 47°, respectively (see Figure S2 in the Supporting Information). These could not remove emulsified oil from water at all (see Figure S3 in the Supporting Information). PVDF membranes are hydrophobic and oleophobic (oil contact angle 72°), whereas Nylon membranes are hydrophilic and oleophilic (oil contact angle 0°). The small differences in oil and water wettability for these membranes might contribute to the absence of selevtive adsorption. Therefore, our research on removal of emulsified oil from water is especially useful for practical applications.

3.3. Separation of Oil and Water. When a mixture of diesel oil and water (dyed with methyl blue for easy observation) was poured onto the superhydrophobic and superoleophilic filter paper, only the oil could pass through the paper and water droplets were held on the surface, as shown in Figure 7. This result implies that the special surface wettability enabled the modified filter paper to separate oil−water mixtures with a high oil ratio. The separation efficiency of the filter paper was further investigated. When an oil−water mixture was dropped onto a superhydrophobic filter paper inclined at a tilt angle of 10°, only the oil passed through the filter paper. The water that flowed off the surface was collected and weighed. The separation efficiency was defined as the ratio of the weight of water collected to that initially added (23). The results are listed in Table 3. The separation efficiency was >96% for diesel oil/water volume ratios ranging from 1:15 to 1:1.

Table 3. Separation Efficiency of the Superhydrophobic Filter Paper for Different Oil−Water Mixtures

oil:water volume ratio	separation efficiency (wt %)
1:15	96.1
1:12	96.6
1:9	96.5
1:4	97.7
1:1	98.2

Although a few studies have demonstrated that superhydrophobic surfaces can separate oil and water (32), the water content in oil after separation still needs detailed analysis. Here, we measured the water content in oil passing through the modified filter paper after separating a 1:1 (v/v) mixture of diesel oil and water. To investigate the influence of the mixing process, we prepared three samples. First, water and diesel oil were ultrasonically mixed for 5 min and rested for 3 days. Second, water and diesel oil were mixed by vibration for 2 min and rested for 3 days. Third, water and diesel oil were mixed and vibrated for 2 min. Then the mixtures were separated using superhydrophobic filter paper and the water content of the diesel oil collected was analyzed. The whole process was repeated three times. As shown in Figure 8, the first separated oil sample contained the highest water content (280 ppm) and the water content of all samples decreased to ∼80 ppm after several separation cycles. The water content in the second and third separated oil samples obviously decreased thanks to selective penetration. The separation efficiency and selective penetration mean that the modified filter paper has potential applications in oil separation and purification.

3.4. Extraction of Ethanol from Homogeneous Aqueous Solution. In this work, we explored the separation effects of the modified filter paper on miscible liquids. Unlike diesel oil, ethanol is miscible with water because of hydrogen bonding between these two kinds of molecules. However, because of its low surface tension (22.3 mN/m) (33), ethanol can quickly spread on a superhydrophobic surface, which we believe can be used to separate it in homogeneous aqueous solution. Aqueous ethanol solutions showed a gradually decrease in contact angle on the superhydrophobic filter paper with increasing ethanol content (Figure 9). When the ethanol content reached 40 vol %, the mixed solution spread on the filter paper.

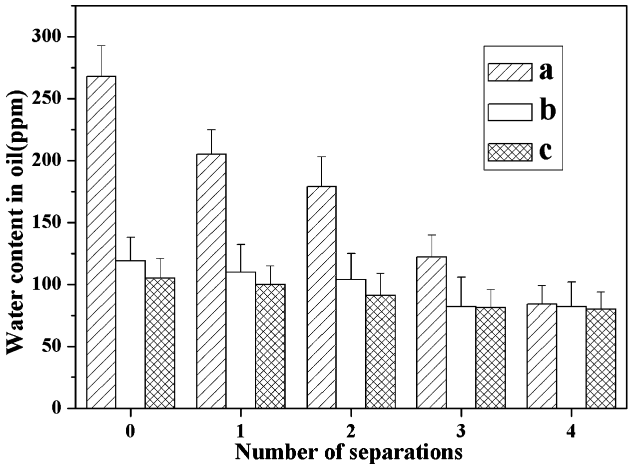

FIGURE 8. Water content in oil before and after separation. (a) Water and diesel oil were ultrasonically mixed for 5 min and the mixture was allowed to rest for 3 days. (b) Water and diesel oil were mixed, vibrated for 2 min, and then allowed to rest for 3 days. (c) Water and diesel oil were mixed, vibrated for 2 min, and immediately subjected to separation.

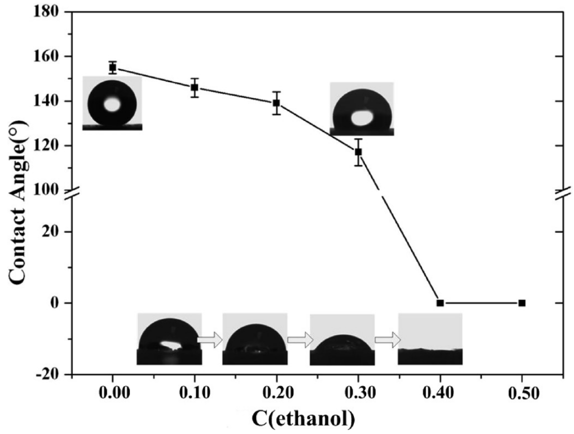

FIGURE 9. Contact angle of aqueous solutions containing different amounts of ethanol on the superhydrophobic filter paper.

In addition, when an aqueous ethanol solution (ethanol content 10−35 vol %) was added dropwise to a tilted superhydrophobic filter paper, a fraction of the solution passed through the filter paper, but most of the mixture droplets rolled along the paper surface and were collected. The refractive index of all collected solutions was 1.338, indicating that the ethanol content had been reduced to approximately 8 vol % according to a calibration curve of refractive index vs ethanol content (see Figure S4 in the Supporting Information). The solution that passed through the filter paper was also collected and measured after 3-fold dilution, which revealed that the ethanol content reached 87 vol %. This result demonstrates that the superhydrophobic filter paper can also extract ethanol from an aqueous solution with a relatively high ethanol content. This can be attributed in part to invasion of ethanol into the filter paper owing to its low surface tension when droplets of the solution roll off the superhydrophobic surface. For the 8 vol % solution, ethanol is tightly held by water in the droplets, leading to incomplete separation. Although the mechanism still requires further investigation, these intriguing results suggest that ethanol can be partially extracted from water using a porous superhydrophobic filter paper instead of traditionally energy-consuming processes such as distillation and pervaporation. Surfaces with specific wettability can find applications in separating liquids even in miscible systems as long as the liquids have large differences in surface tension.

4. CONCLUSIONS

We used a simple dip-coating approach to create filter paper with both superhydrophobic and superoleophilic properties. The specific wettability was used to separate liquids with low surface tension, such as diesel oil, from water. The treated filter paper could remove not only diesel oil floating on a water surface, but also emulsified oil in aqueous suspensions. In particular, it exhibited reproducible selective adsorption of a collection of organic solvents such as hexane and dodecane that reached 2.0−3.4 times its original weight. Moreover, the superhydrophobic filter paper was effectively used to separate oil and water, leading to a decrease in water content in the oil. The filter paper was also able to partly extract ethanol from aqueous solutions. Its reproducible and stable properties mean that the superhydrophobic filter paper is an ideal candidate for applications in the cleanup of oil pollutants and the separation of oil and water. Considering the global scale of severe water contamination arising from oil leakage, this study provides a useful strategy for the design and application of superhydrophobic porous films with specific adsorption abilities to meet these issues.

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Supporting Information Available: Videos showing the uptake of oil from the water surface and an aqueous emulsion with the superhydrophobic filter paper (.AVI); contact angle of aqueous solution with different pH values on the superhydrophobic filter paper, SEM images and profiles of water drops on a PVDF and a Nylon membrane, optical images showing the emulsified oil still in water after a PVDF or a Nylon membrane treatment, refraction index of ethanol aqueous solutions (PDF). This material is available free of charge via the Internet at http://pubs.acs.org.

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