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Facile preparation of superhydrophobic and superoleophilic porous polymer membranes for oil/water separation from a polyarylester polydimethylsiloxane block copolymer

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Abstract To fabricate superhydrophobic membranes for oil/water separation, a mesh serving as a support is usually required for improving mechanical properties and rendering a micro-scale porous structure. Herein, we develop a simple spraying-coating process for producing superhydrophobic and superoleophilic porous polymer membranes without meshes as supports from a fluorinated polyarylester polydimethylsiloxane block copolymer (PAR-*b*-PDMS). It is exciting that the surface morphology of the PAR-*b*-PDMS membranes can be controlled by the copolymer solution concentration. The PAR-*b*-PDMS membranes are endowed with promising superhydrophobicity and superoleophilicity, displaying excellent oil affinity and low water adhesion behaviors. Significantly, the PAR-*b*-PDMS membranes exhibit fast and efficient separation for oil/water mixtures with separation efficiency as high as 99 %. And after 50 cycles of oil/water separation, the as-prepared membranes can still maintain stable superhydrophobicity and high separation efficiency, indicating good recyclability and durability. Moreover, the fabrication approach presented here can be easily scaled up for producing polymer membranes on a large scale, which makes it very promising for practical oil/water separation.

Introduction

The discharge of oil-contaminated water from the daily life of people as well as from industries can result in serious environmental pollution, and the frequent oil spills resulting from oil transport accidents may be destructive for marine and aquatic ecosystems. Therefore, oil/water separation has always been an important and exigent task in the fields of chemical engineering and environmental protection [1, 2]. Although many methods such as chemical and biological treatments, gravity separation, as well as the use of absorbance and electric field, have been developed and successfully employed as oil/water separation technologies, the limitations of low separation efficiency, energy-cost, and complex separation instruments have caused difficulty in taking advantage of these methods [3]. Thus, it is necessary to develop effective technologies and new materials for oil/water separation.

Recently, considerable interest has been focused on the design and preparation of novel materials with special wettability for oil/water separation [4–11]. Thus far, a myriad of materials with such special wettability have been widely applied for the separation of oil and water [4]. In general, the wettability of material surfaces depends on the surface chemistry and the geometrical roughness. Hence, materials with superhydrophobicity and superoleophilicity could be obtained by the combination of reduced surface energy and enhanced surface roughness [12, 13]. In 2004, Jiang et al. were the first to fabricate an “oil-removing” type filtration membrane by both spraying a polytetrafluoroethylene (PTFE) emulsion on a stainless steel mesh and high-temperature baking [7]. The results showed that the PTFE-coated mesh exhibited superhydrophobicity and superoleophilicity and could successfully separate mixtures of diesel oil and water solely by gravity. Subsequently, various

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approaches have been applied towards the manufacture of "oil-removing" type filtration membranes, including chemical deposition, electrospinning, plasma treatment, template synthesis, and phase separation method [6, 14, 15]. Nevertheless, most of these methods may not be practically viable as they involve multi-step processes and sometimes harsh conditions, as well as exhibit limitations for the large-scale fabrication of such functional membranes and poor selectivity and recyclability.

As a cost-effective and well-established technique, spray coating is capable of achieving superhydrophobic surfaces with different morphologies; it involves the spraying of a low-concentration suspension consisting of a solvent, inorganic particles, and a polymeric binder [16–18]. In previous studies, our group has reported the simple approach for directly fabricating superhydrophobic polymer coatings with hierarchical micro and nano-structures via spraying polymer solutions on flat substrates with various solvent compositions [19, 20]. Conventionally, by utilizing the spray coating method to fabricate superhydrophobic membranes for oil/water separation, a mesh (metal mesh and fabric) serving as a support is required for improving mechanical properties and rendering a micro-scale porous structure [3, 7, 21, 22]. However, up to date, polymeric materials have been rarely used by themselves in the preparation of the "oil-removing" type filtration membrane based on this method.

In this contribution, hydroxyl-terminated fluorinated polyarylester (PAR), α,ω-dihydroxy polydimethylsiloxane (HO-PDMS), and 4,4′-diphenylmethane diisocyanate (MDI) were used as the monomers for preparing novel PAR-*b*-PDMS through condensation. We developed a simple spraying-coating process for producing superhydrophobic and superoleophilic porous polymer membranes from the PAR-*b*-PDMS without meshes or fabrics as supports. The surface morphology of the PAR-*b*-PDMS membranes could be controlled by the concentration of the copolymer solution. The as-prepared membranes not only exhibited excellent superhydrophobicity and superoleophilicity, but also a good mechanical property. The surface chemical composition, and oil sorption of the resultant membranes were investigated, and persuasive examples demonstrating the rapid and efficient oil/water separation performance were also presented. In addition, the details of fabrication, characterization, and properties of the PAR-*b*-PDMS membranes were described herein.

Experimental

Materials

α,ω-Dihydroxy polydimethylsiloxane (HO-PDMS) with molecular weight of about 4200 and 4,4′-diphenylmethane diisocyanate (MDI) were supplied by Hangzhou Silong Material technology Co., Ltd., China. Bisphenol AF, Terephthaloyl chloride (TPC), Isophthaloyl chloride(IPC), and Benzyltriethylammonium chloride (BTEAC) were purchased from Sinopharm Chemical Reagent Co., Ltd. General other chemicals such as tetrahydrofuran(THF), N,N-dimethylformamide (DMF), and dichloromethane in reagent grade were used as received from Kemiou Chemical Co., Tianjin, China.

Synthesis of PAR

PAR was prepared by interfacial polycondensation (Fig. S1, Supporting Information). Typically, bisphenol AF (8.40 g), NaOH (2.04 g), BTEAC (0.005 g), and H_2O (80 mL) were mixed together with stirring for 2 h and then the mixture was cooled to 6 °C. The solution of TPC (2.91 g), IPC (2.91 g) and dichloroethane (160 g) was added drop-by-drop within 1 h. After stirring for 2 h at 6 °C, the mixture was heated up to 15 °C and maintained at that temperature under constant stirring for another 2 h. The reactant was washed with HCl (5 wt%) and water three times, and then slowly added to methanol to precipitate the polymer. The white precipitated PAR was collected by filtration and dried in vacuum oven.

Synthesis of PAR-*b*-PDMS

The synthesis of PAR-*b*-PDMS via condensation reaction is similar to the previous report [23]. In a typical experiment, HO-PDMS(0.89 g), MDI (0.11 g), and dibutyltin dilaurate (0.006 g) were dissolved in 20 mL of THF under dry nitrogen at room temperature and refluxed at 75 °C for 4 h. The hot solution made up of PAR (7.41 g) and THF (100 ml) was then added, and the reaction was carried out at 75 °C for 5 h (Fig. S2, Supporting Information). At the end, the mixture was cooled to room temperature. The solvent was then evaporated under reduced pressure at 40 °C. The PAR and PAR-*b*-PDMS were characterized by Fourier transform infrared (FT-IR) and gel permeation chromatography (GPC) (Figs. S3, S4, Supporting Information).

Fabrication of PAR-*b*-PDMS membranes

A certain amount of the obtained PAR-*b*-PDMS copolymer was dissolved in a mixture of THF and DMF with a volume ratio of 9/1 to form 1, 5, and 10 wt% solutions, respectively. Prior to spray coating, aluminum substrates were cleaned with acetone and water. The PAR-*b*-PDMS membranes prepared by uniformly spraying the copolymer solution on these flat aluminum substrates at an air pressure of 0.8 MPa were peeled off from the substrates after drying at room temperature. The sample thickness was 150 ± 5 μm. During spraying, the nozzle of spray gun having a diameter of approximately 0.8–2 mm was placed at a certain distance

(~15 cm) from the substrate. Room temperature and relative humidity were 25 ± 2 °C and 50 ± 10 %, respectively. The PAR membrane was fabricated under the same condition for comparison.

Characterization

FT-IR spectra of samples were recorded between 4000 and 500 cm^{-1} in a Bio-Rad FTS-135 spectrophotometer. The molar mass distribution was determined in a Waters 1515 gel permeation chromatography (GPC) with THF as the eluent flowing at 1 mL/min. The surface morphology was observed with a scanning electron microscope (SEM, FEL Nova Nano 430). The surface composition was measured by X-ray photoelectron spectroscopy (XPS, Perkin-Elmer PHI 5000C ECSA) using at a 90° take-off angle. Contact angles and sliding angles on the surfaces at 25 ± 2 °C were measured with an optical contact angle meter system (Dataphysics OCA40), and the measurements were carried out with 4 µL water droplet and 2 µL oil droplet, respectively. The optical contact angle meter system with a high-speed CCD camera was also utilized to observe the profiles of individual oil droplets on sample substrates at 25 °C in a sorption process. The sorption process of oil droplet was recorded by the high-speed CCD camera at a speed of 25 f/s. Oil/water separation experiments were carried out with a filtration system. The as-prepared membranes were fixed between two glass tube with a metal clamp and 100 g mixture of oil and water (3/7, v/v) were poured onto the upper glass tube.

Results and discussion

The superhydrophobic and superoleophilic porous polymer membranes were basically prepared by spraying the copolymer solution on a flat aluminum substrate, and after drying at room temperature, the as-prepared PAR-*b*-PDMS membranes were easily peeled off from the substrate (Fig. 1). Figure 2 displays the SEM images of the PAR and PAR-*b*-PDMS membranes prepared from a 5 wt% solution in THF/DMF (9:1, v/v); rough surfaces are observed for both membranes. However, there is a noticeable difference between the two surfaces: The PAR membrane surface consists of irregular protrusions (grooves), while the PAR-*b*-PDMS membrane surface consists of micro- and nano-porous structures.

The overall XPS spectra of the surfaces of PAR and PAR-*b*-PDMS membranes prepared from a 5 wt% solution are shown in Fig. 3. The atomic percentage of carbon, oxygen, and fluorine in the PAR membrane is estimated to be approximately 67.1, 18.7, and 14.2 %, respectively. The ratio of fluorine to oxygen from the XPS data (14.2%:18.7% ≈ 0.76:1) is less than the theoretical value of the PAR molecule (≈1.78:1). Hence, it can be concluded that although the hexafluoroisopropyl groups of the PAR molecule exhibit low surface energy, these groups do not easily migrate to the surface of the PAR membrane because of the restriction of the stiff molecular chain of PAR. On the other hand, the atomic percentage of carbon, oxygen, fluorine, and silicone in the PAR-*b*-PDMS membrane is estimated to be approximately 63.7, 17.6, 7.5, and 11.2 %, respectively. Due to the migration of the flexible PDMS segments onto the PAR-*b*-PDMS membrane surface, there is a higher ratio of silicone to oxygen in the XPS data (11.2:17.6 ≈ 0.64:1) than the theoretical value of PAR-*b*-PDMS molecule (≈0.28:1), which might play an important role in achieving superhydrophobic surfaces. Static contact angles (CA) and sliding angles (SA) were measured to characterize the wettability of the membrane surfaces. The water contact angle (WCA) on the spray-coated PAR membrane is 147° ± 3.1° and its sliding angle (SA) is more than 12° (inserts in Fig. 2), which cannot be

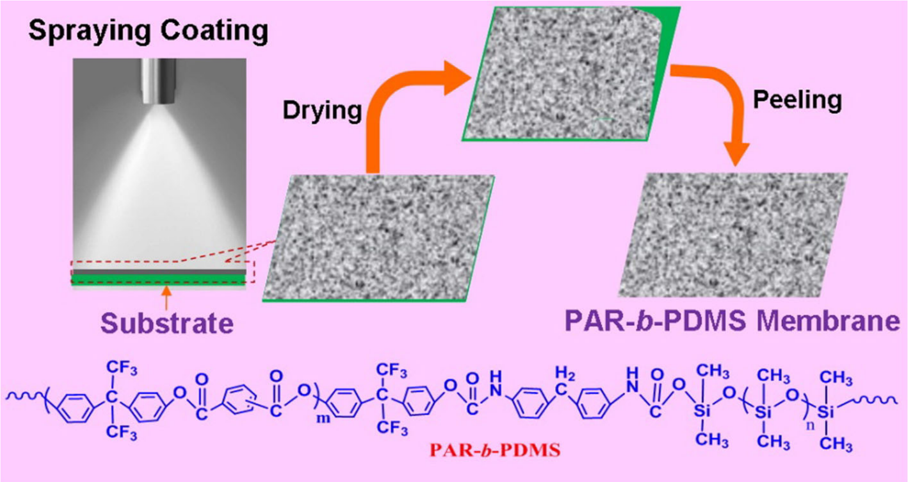

Fig. 1 Schematic of the fabrication of PAR-*b*-PDMS membranes via spray coating method

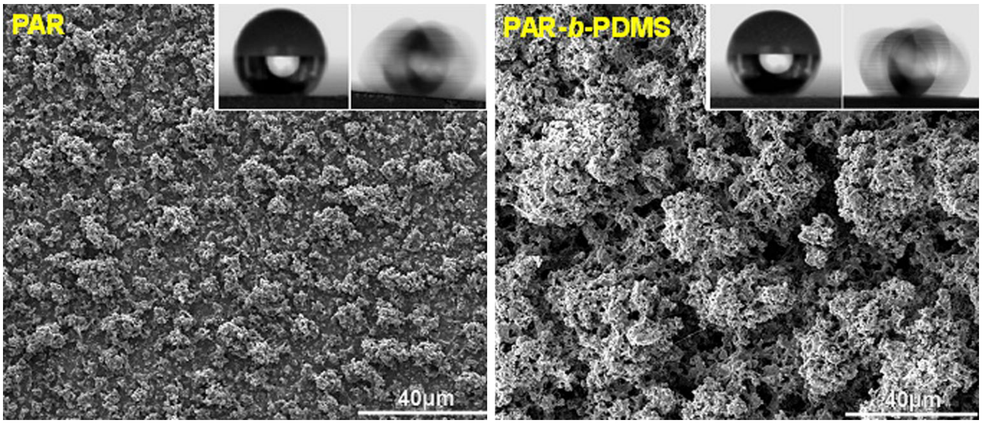

Fig. 2 SEM images of the PAR and PAR-*b*-PDMS membranes prepared from a 5 wt% solution in THF/DMF (9:1, v/v). Insets show water contact angles and sliding angles. The contact angle value of PAR membrane is 147° ± 3.1°, and the sliding angle value is more than 12°. The contact angle value of PAR-*b*-PDMS membrane 163° ± 2.3° is 163° ± 2.3°, and the sliding angle value is less than 2°

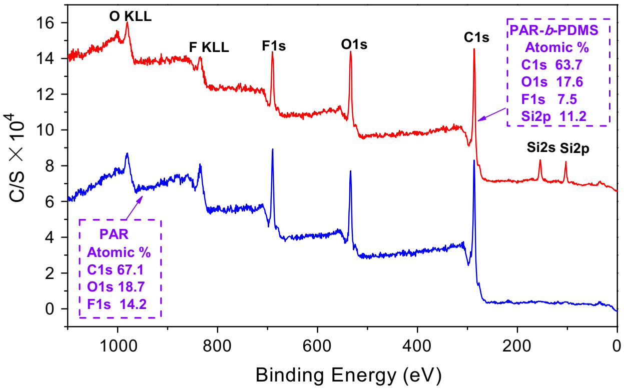

Fig. 3 The overall XPS spectra of the surfaces of PAR and PAR-*b*-PDMS membranes prepared from a 5 wt% solution

classified as a superhydrophobic material. However, after modification with PDMS, the WCA on the spray-coated PAR-*b*-PDMS membrane increases to 163° ± 2.3° and the sliding angle (SA) is less than 2°, exhibiting significant superhydrophobicity.

Moreover, SEM micrographs of the PAR-*b*-PDMS membranes prepared from the solution concentrations of 1, 5, and 10 wt% are depicted in Fig. 4. Of note, the surface morphologies of the as-prepared membranes can be controlled by adjusting the concentration of the copolymer solution. As shown in Fig. 4a, b, the surface of PAR-*b*-PDMS membrane prepared from a 1 % copolymer solution consists of many pores, having a diameter of 0.1–0.8 μm and a small quantity of nano-protrusions. With increase in the copolymer solution concentration from 1 to 5 wt%, numerous connected irregular protrusions appear on the PAR-*b*-PDMS membrane surface, resulting in the formation of a continuous porous structure (Fig. 4c). More exhilaratingly, the high-magnification SEM image of the PAR-*b*-PDMS membrane (Fig. 4d) clearly shows that there are innumerable nanopores with diameter of 40–120 nm on every protrusion (groove). The result might be caused by the thermally induced phase separation and vapor-induced phase separation in the process of spraying, and it is in agreement with the previous reports prepared by electrospinning method [24, 25]. As the solution concentration further increases to 10 wt%, the higher copolymer solution can enhance the number of copolymer chain entanglement and reduce the number of liquid droplets atomized during spray coating [19, 21], ultimately leading to the coexistence of more nanofibers and a few beads on the surface of the PAR-*b*-PDMS membrane (Fig. 4e, f) [26, 27]. In a word, the surface morphology of the membrane might be affected by several factors including polymer chemical composition and structure, polymer solution concentration, solvent evaporation rate, spraying condition (temperature, pressure, and humidity). As can be observed from the insets of Fig. 4, the WCA values on the PAR-*b*-PDMS membrane prepared from 1, 5, and 10 wt% copolymer solutions are 133° ± 4.1°, 161° ± 1.7°, and 155° ± 2.6°, respectively, suggesting that the WCA values of the as-prepared membranes are different, caused by their different surface morphologies.

For examining the wetting behavior of oil on the PAR-*b*-PDMS membrane prepared from a 5 wt% solution, a high-speed camera system was used to record the adsorption of an oil droplet (Fig. 5). The wetting behavior of oil on the PAR membrane was also measured for comparison. The time between the first droplet touch and stability is known as the sorption time. It can be clearly seen that when the oil (dichloromethane) droplet (2 μl) contacts the membrane surface, the oil droplet decreases rapidly because of the sorption of membrane. After a sorption time of 0.44 s, the oil droplet stabilizes on the PAR membrane, a portion of the oil droplet is left on the PAR membrane, and the oil CA

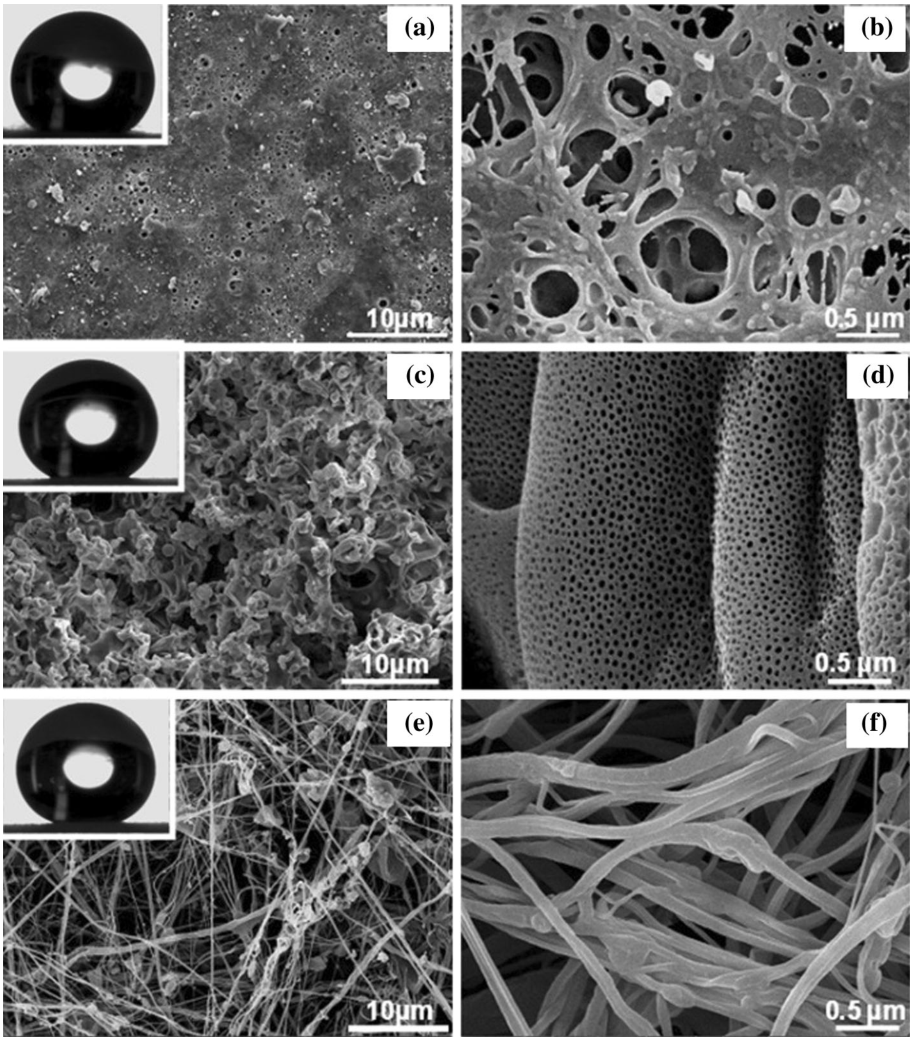

Fig. 4 SEM micrographs of the PAR-*b*-PDMS membranes prepared from the copolymer solution concentrations of **a** 1, **c** 5, and **e** 10 wt%. **b**, **d**, and **f** are the magnification corresponding to **a**, **c**, and **e**, respectively. *Insets* show water contact angles. The WCA on the PAR-*b*-PDMS membrane prepared from the copolymer solution concentrations of 1, 5, and 10 wt% is 133° ± 4.1°, 161° ± 1.7°, and 155° ± 2.6°, respectively

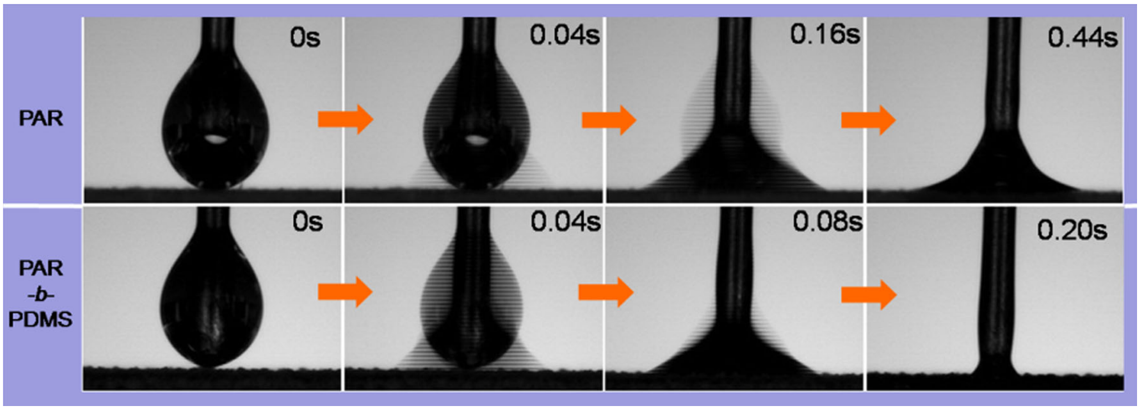

Fig. 5 Photographs of the profiles of individual oil droplets on the PAR and PAR-*b*-PDMS membranes prepared from a 5 wt% solution in the whole sorption process were captured by a high-speed camera system

is 11° ± 3.2° (Fig. S5a). As compared to that of the PAR membrane, a shorter sorption time of only approximately 0.20 s is required for the PAR-*b*-PDMS membrane, and the oil droplet is adsorbed completely (that is, the oil CA is 0°, Fig. S5b), confirming a superior property of the PAR-*b*-PDMS membrane for oil wetting.

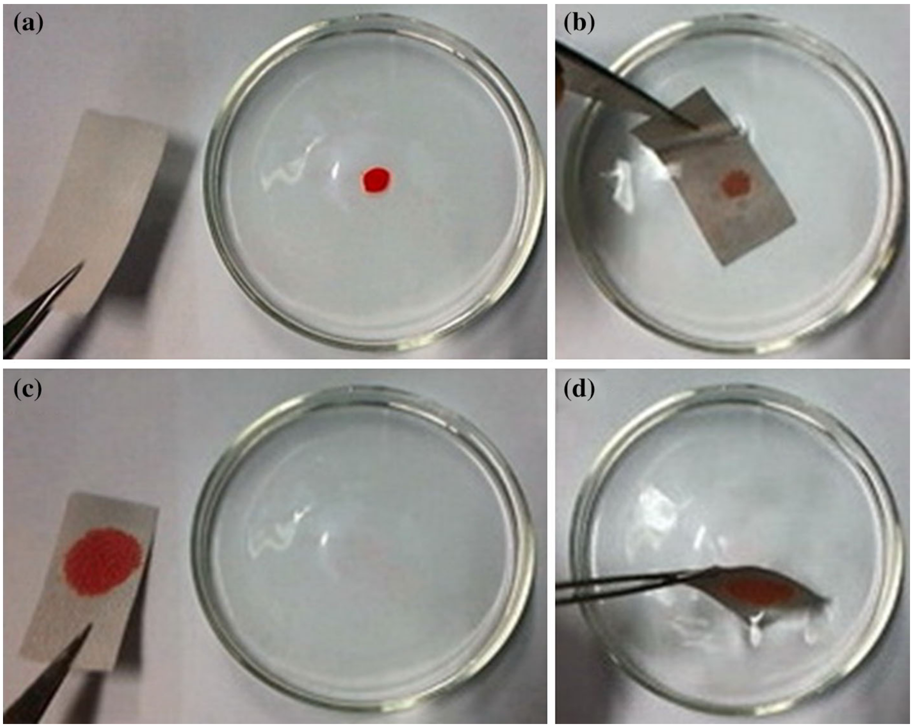

Fig. 6 Typical digital photographs of the removal processes of dichloromethane **a**–**d** from water

In addition, the PAR-*b*-PDMS membrane selectively absorbs oil (dichloromethane) from water (Fig. 6). When the PAR-*b*-PDMS membrane was inserted into water to approach oil, the oil droplet was immediately sucked up by the membrane underwater, and water imbibition was not observed during oil absorption (Fig. 6a–d), further indicating excellent oil affinity and low water adhesion behaviors of the as-prepared sample, which could be an important factor for oil/water separation.

From a practical viewpoint, large-area as well as different-shaped PAR-*b*-PDMS membranes without meshes as supports can be easily fabricated by this simple spray coating method and the PAR-*b*-PDMS membranes are of good mechanical property (tensile strength is about 19 ± 2.4 MPa) (Fig. 7a). As mentioned, the PAR-*b*-PDMS membrane prepared from a 5 wt% solution showed selective wettability and nano-porous structure, which is considered as a very promising candidate for oil/water separation. The oil/water separation of the PAR-*b*-PDMS membrane was carried out as shown in Fig. 7b. As oil (dichloromethane)/water mixtures are poured on the PAR-*b*-PDMS membrane, only oil rapidly passes through the membrane and falls into an Erlenmeyer flask below by gravity, while water is repelled and kept above the membrane (Movie S1, Supporting Information). The separation efficiency of the PAR-*b*-PDMS membrane is greater than 99 %, as calculated by the previously reported methods [3]. Therefore, by means of a simple filtering method, the PAR-*b*-PDMS membrane can successfully separate oil/water mixtures with high efficiency.

We also examined the recyclability and durability of the obtained PAR-*b*-PDMS membrane. After every cycle of oil/water separation, the PAR-*b*-PDMS membrane was thoroughly dried at 80 °C. Figure 8a shows the relationship of the WCA, separation efficiency, and recycle numbers. Although the WCA on the PAR-*b*-PDMS membrane has a slight decrease after 50 cycles of oil/water separation, the obtained membrane maintains superhydrophobic performance, which demonstrates good durability of our membrane. Meanwhile, we did not observe that repeated separation experiments could be a noticeable effect on the separation efficiency of the PAR-*b*-PDMS membrane. The separation efficiency varied slightly during the repeated experiments and always kept above 98 %, further displaying good recyclability of the as-prepared membrane. Besides the mixture of water and dichloromethane, good separation is observed for mixtures of water and other solvents including carbon tetrachloride, dimethylbenzene, hexadecane, and diesel oil.

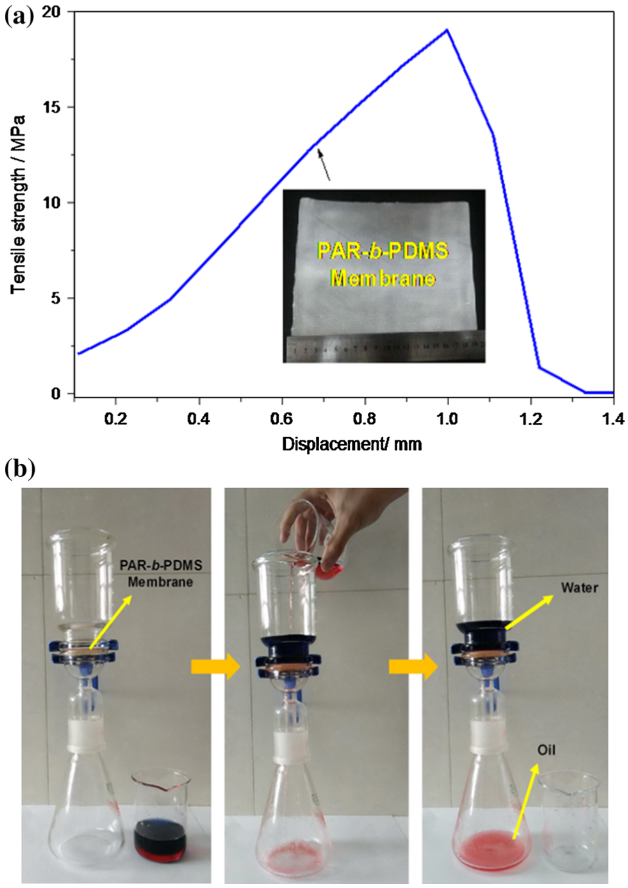

Fig. 7 **a** Stress–displacement curve of the PAR-*b*-PDMS membrane from a 5 wt% solution by spraying (sized at 5 cm × 1 cm) measured on a testing machine (5560, Instron, US), and the *inset* is a photograph of the as-prepared PAR-*b*-PDMS membranes. **b** The facile oil/water separation using the PAR-*b*-PDMS membrane, the oil and water were dyed by oil red and black ink, respectively

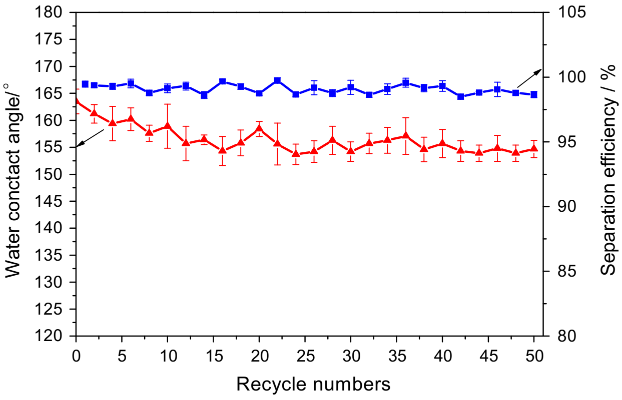

Fig. 8 Water contact angle and separation efficiency of the PAR-*b*-PDMS membrane as function of recycle numbers during the repeated separation experiments

Conclusions

In summary, we have successfully developed a simple procedure to fabricate a novel superhydrophobic and superoleophilic porous polymer membrane through spraying a PAR-*b*-PDMS solution. The surface hierarchical structure of the PAR-*b*-PDMS membrane can be controlled by adjusting the copolymer solution concentration. The PAR-*b*-PDMS membrane with a multi-scale porous structure prepared from a 5 wt% solution in THF/DMF (9:1, v/v) exhibits superhydrophobicity with a WCA of 163° ± 2.3° and superoleophilicity with an oil CA of 0°. The obtained PAR-*b*-PDMS membrane can selectively absorb oil from water, indicating outstanding oil affinity and low water adhesion behaviors. Significantly, the PAR-*b*-PDMS membranes can effectively separate oil/water mixtures, and a notable oil/water separation efficiency of 99 % was achieved. This approach can be repeated for at least 50 times with stable superhydrophobicity and high separation efficiency. Besides, the results obtained from this study can allow for the easy fabrication of large-scale superhydrophobic and superoleophilic polymer membranes with a good mechanical property. Therefore, we anticipate that the membrane developed herein will have high potential in practical application of oil/water mixtures due to the oil affinity and water repellency, the high oil/water separation efficiency, the recyclability and durability, and the good mechanical property.

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Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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