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ORIGINAL RESEARCH

An acid–alkali–salt resistant cellulose membrane by rapidly depositing polydopamine and assembling $BaSO_4$ nanosheets for oil/water separation

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Abstract With the increase of oil spills, leakage of chemical products and discharge of oily wastewater, oil–water separation has become an urgent problem. In this study, polydopamine (PDA) was first coated on the surface of paper fibers (PF@PDA) by $CuSO_4$/H_2O_2 triggering to accelerate the reaction rate. Then, the $BaSO_4$ nanosheets were deposited on the surface of PF@PDA membrane by alternating soaking process (ASP) to obtain superhydrophilic/underwater superoleophobic membrane (PF@PDA/$BaSO_4$) for oil/water separation. PF@PDA/$BaSO_4$ shows good separation efficiency (more than 99%) and high flux (more than 550 L m^{-2} h^{-1}) for various oil–water mixtures. Importantly, the membrane shows excellent acid, alkali and salt resistance that maintains the high separation performance even under acid, base and salt solution environments. Moreover, the PF@PDA/$BaSO_4$ membrane has excellent antifouling property and outstanding reusability. Our PF@PDA/$BaSO_4$ with low cost, easy preparation and high performance has a good application prospect for oil/water separation even in harsh environments.

Keywords $BaSO_4$ nanosheets · Superhydrophilicity · Underwater superoleophobicity · Acid–alkali–salt resistance · Oil/water separation

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Introduction

Water pollution has become a serious global environmental problem (Yang et al. 2014; Dai et al. 2018; Xie et al. 2019a; Xie et al. 2020a, b). Increasing oil spills, chemical products leakage and oily wastewater emissions seriously threaten human survival and ecological environment (Wang et al. 2019; Xie et al. 2019a). Traditional oily wastewater treatment methods, such as oil skimming, coalescence, flotation, combustion and adsorption have been used to treat oily wastewater (Ao et al. 2017; Xie et al. 2020a, 2020b). However, the drawbacks of low separation efficiency and high energy consumption have always limited their extensive application. Therefore, the development of efficient strategies or materials to solve oily wastewater is a challenging task.

Recently, especially wettable membranes have been widely used for oil/water separation (Long et al. 2018; Xie et al. 2019e; Xie et al. 2019b; Cui et al. 2019a). Mainly, the especially wettable membranes fall into include two categories: superhydrophobic/superoleophilic and superhydrophilic/superoleophobic. However, the separation efficiency and reusability of superhydrophobic/superoleophilic membranes were easily affected by membrane surface contamination due to its good lipophilicity (Mengke et al. 2020; Maphutha et al. 2013). On the contrary, the superhydrophilic/superoleophobic membranes allowed water to permeate through while repelled the oil on the surface, which could effectively prevent oil from contaminating the membrane surface (Meihua et al. 2012; Kota et al. 2012). So far, various materials such as GO coated wire mesh (Yu-Qing et al. 2015) and NiOOH coated metal mesh (Li et al. 2015) have been used for oil/water separation. Liu et al. discovered dopamine-modified graphene oxide membrane for oil–water separation (Liu et al. 2018). Zhong et al. developed a new type of superhydrophilic/underwater superoleophobic hydrogel-coated membrane for achieving underwater superoleophobicity (Zhongxin et al. 2011). However, these materials have the disadvantages of complex preparation, high cost and poor corrosion resistance. Moreover, these materials have difficulty when separating oil/water mixtures in harsh (e.g., acid or alkaline) environments. Therefore, it is urgent to develop superhydrophilic membrane materials with low cost, simple preparation, harsh environments resistance, high separation efficiency and environmentally friendly.

Cellulose is the most abundant natural macromolecule on the earth and an inexhaustible natural renewable resource (Zheng et al. 2015). Cellulose has the characteristics of good biodegradability, light weight, easy availability, low cost, easy chemical modification, etc. It is a promising raw material for oil–water separation. For instance, a polyurea coated cellulose membrane for oil/water separation was developed by Li et al. (Li et al. 2019). Chen et al. prepared UV-driven anti-fouling paper fibers membranes for efficient oil/water separation (Chen et al. 2019). Xie et al. reported a superhydrophobic cellulose membrane via a sol–gel strategy for efficient oil/water separation (Xie et al. 2019c). However, to the best of our knowledge, few works have focused on acid–alkali-resistant cellulose membranes for oil/water separation.

Polydopamine (PDA) deposition technology provides a new method for modification of various materials (Tang et al. 2018; Zhao et al. 2019). PDA has been used as a surface immobilization medium to generate and immobilize nanoparticles on the surface of membranes (Yang et al. 2015; Xie et al. 2019d). However, traditional methods have shown the low formation rate of PDA coatings, and the coating process usually required several hours to several days. In addition, the PDA coating by traditional methods was not resistant to harsh environments. Zhang et al. have reported a new strategy to accelerate the deposition rate of PDA coating using $CuSO_4/H_2O_2$ as a trigger (Zhang et al. 2016). In this case, $CuSO_4$ acts as an oxidant to oxidize dopamine, Cu^{2+} and H_2O_2 produce reactive oxygen species (ROS) to trigger rapid polymerization of dopamine showing acid and alkali-resistance. Thus, this rapid PDA deposition method may show the advantages of low cost and fast preparation in the construction of acid–alkali–salt-resistant cellulose membranes (Yanlan et al. 2014).

To date, many inorganic nanomaterials have been modified onto membranes for oil–water separation (Dai et al. 2020). For instance, a chitosan-cellulose acetate-TiO_2 membrane was prepared for oil/water separation (Yu et al. 2019). TiO_2/CuO dual-coated copper mesh with underwater superoleophobicity and self cleaning function was developed for oil/water separation (Yuan et al. 2017). Cui et al. reported SiO_2 and NiCo-LDH modified PVDF composite membrane for oil/water emulsion separation (Cui et al. 2019b, c). However, these membranes were not resistant to acid, alkaline and salt environments. It is well known that $BaSO_4$ is insoluble in acid and alkaline solution and could be deposited on the surface of cellulose membranes by alternating immersion method (ASP) to build micro-nano structures (Jin et al. 2015; Taguchi et al. 1999). Thus, $BaSO_4$ modified membrane may show superhydrophilicity and acid–alkali–salt resistance.

Here, we prepared a stable cellulose membrane with acid, alkali and salt resistance for the separation of a series of oil/water mixtures. PDA was first coated on the surface of paper fibers (PF@PDA) by $CuSO_4$/H_2O_2 triggering to accelerate the reaction rate. Then, the $BaSO_4$ nanosheets were deposited on the surface of PF@PDA membrane by alternating soaking process (ASP) to endow membranes with hydrophilicity and acid, alkali, salt resistance. The as-prepared membrane shows high separation efficiency (above 99%) for various oil/water mixtures. The prepared membrane can maintain the performance that the separation efficiency of various oil/water mixtures is more than 99% even under 0.1 M acid, alkali, or salt solution. In addition, the membrane has excellent reusability. Our cellulose-based membranes with low cost, high efficiency and acid, alkali, salt resistance may have good application prospects in the treatment of oily water.

Experimental

Materials and chemicals

Soybean oil and the paper were purchased from Kaiyuan Supermarket. Dopamine hydrochloride (DA, 98%), 2-Methylimidazole, tris (hydroxymethyl) aminomethane (Tris),petroleum ether (AR, bp 90–120 °C) and methylene blue were received from Aladdin Reagent Co., Ltd. Ammonium sulfate, Barium chloride, Toluene ($\geq$ 99.5%), 1, 2-Dichloroethane ($\geq$ 99.5%), *n*-hexane (> 99%) and anhydrous ethanol were purchased from Sinopharm Chemical Reagent Co., Ltd. (Shanghai, China).

Synthesis of PF@PDA membrane

Firstly, paper (0.3 g) was added to deionized water (100 mL) and strongly stirred for 30 min. Then dopamine hydrochloride (2 mg/mL), Tris (pH = 8.5, 50 mM) and $CuSO_4$ (5 mM)/H_2O_2 (19.6 mM) were poured into the above dispersion solution stirring for 30 min at 25 °C. Finally, the polydopamine (PDA) coated PF (PF@PDA) membrane was obtained through a vacuum filter drying in vacuum oven at 60 °C.

Synthesis of PF@PDA/$BaSO_4$ membrane

Firstly, 0.05 M $BaCl_2$ and $(NH_4)_2SO_4$ aqueous solution were prepared separately before mineralization. Then alternate soaking process (ASP) was used for surface mineralization. A complete cycle of ASP contains four steps: First, the PF@PDA membrane was immersed in $BaCl_2$ aqueous solution at 25 °C for 60 s, then cleaned with deionized water for 60 s, then immersed in $(NH_4)_2SO_4$ aqueous solution at 25 °C for 60 s, and the membrane was cleaned again with deionized water for 60 s. Finally, the resultant membrane was dried at 40 °C. The mineralized PF@PDA membranes with ASP cycles of 0, 4, 7 and 10 were named as PF@PDA/$BaSO_4$, PF@PDA/$BaSO_4$-4, PF@PDA/$BaSO_4$-7, PF@PDA/$BaSO_4$-10, respectively.

Oil/water separation tests

The PF@PDA/$BaSO_4$ was first wetted with water, then the membrane was clamped in two Teflon flanges with a diameter of 0.8 cm to form an oil/water separation device. Finally, the oil/water mixtures of 20 mL (V_{oil}/V_{water} = 1/1) were poured into the separation device to separate oil/water mixtures under gravity. Petroleum ether (light oil) and 1,2-dichloroethane (heavy oil) were selected as representatives. The separation efficiency (R) was calculated by the following equation:

$$R(\%) = \left(1 - C_p/C_f\right) \times 100,$$

where C_f and C_p are the water concentration of the water/oil mixtures and the filtration after one separation, respectively.

The flux was calculated by the following equation:

$$J = \frac{V}{S\Delta t}$$

where J (L m^{-2} h^{-1}) is flux, V (L) is the filtrate volume, S (m^2) is the effective area, Δt (h) is the penetrating time.

After that, the oil/water separation tests were carried out in an acid, base, salt environments by same procedures: replace the water in the above experiment with the acid, alkali or salt (0.1 M) solution.

Results and discussion

The morphology of the samples was analyzed by SEM. From Fig. 1a, the paper fibers with smooth

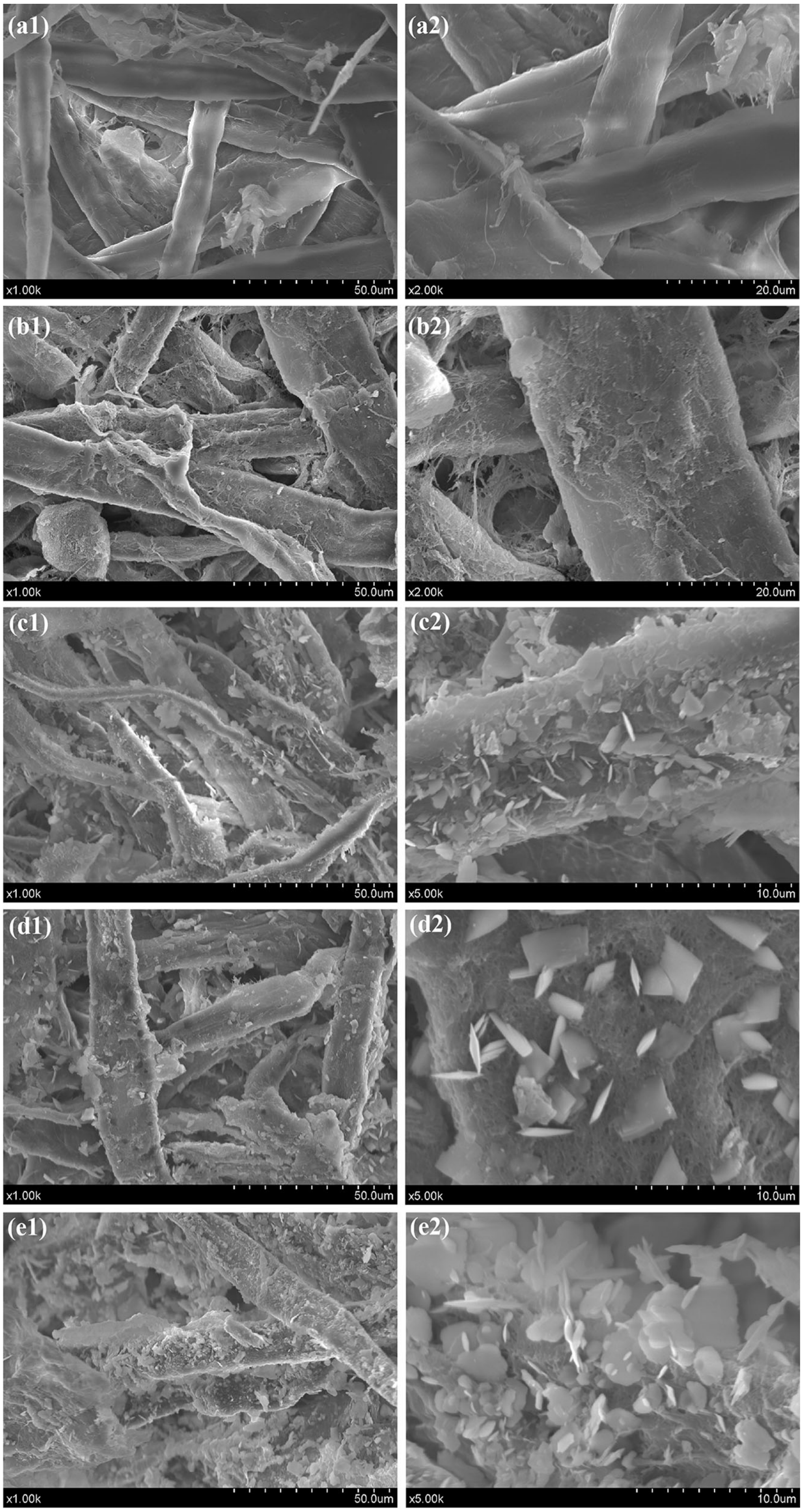

Fig. 1 SEM images of **a** PF **b** PF@PDA, **c** PF@PDA/$BaSO_4$-4, **d** PF@PDA/$BaSO_4$-7, **e** PF@PDA/$BaSO_4$-10

surface showed a 3D framework structure. As depicted in Fig. 1b, the fibers surface of PF@PDA membrane became rough due to the polydopamine deposition. Figure 1c–e showed that $BaSO_4$ nanosheets were anchored on the fibers surface of the PF@PDA/$BaSO_4$ membrane. Importantly, the number and size of $BaSO_4$ nanosheets increased with the number of ASP cycles from 4 to 10 times. $BaSO_4$ nanosheets coated on the fibers surface of the membrane may make the membrane more hydrophilic and acid, alkali, and salt resistant.

Figure 2 showed XRD patterns of PF, PF@PDA and PF@PDA/$BaSO_4$. The XRD pattern of PF displayed two broad peaks at 16°, 20.5° attributing to the characteristic diffraction peaks of cellulose (Chen et al. 2019). The PF@PDA showed the similar peaks, indicated that polydopamine has no effect on the crystallinity of cellulose. In addition, the XRD pattern of PF@PDA/$BaSO_4$ showed the new characteristic peaks at 26°, 29°, 44° belong to the characteristic peaks of $BaSO_4$ (Jin et al. 2015). The results indicated the $BaSO_4$ nanosheets had successfully integrated on the fibers surface.

The chemical composition of samples was analyzed by XPS. The C 1s peak, O 1s peak and N 1s peak were detected on the PF@PDA. Similarly, the C 1s peak, O 1s peak and N 1s peak were discovered on the surface of PF@PDA/$BaSO_4$. However, the XPS spectrum also showed the existence of Ba 3d peak in the PF@PDA/$BaSO_4$ (Fig. 3a). Figure 3b showed the high resolution XPS spectrum of N 1s, it could be fitted by three peaks at 400.2, 399.6 and 399.2 eV assigning to NH_2, C–NH–C and C–N (Xie et al. 2019d), respectively. The Ba 3d spectrum (Fig. 3c) strongly indicated the presence of Barium in the PF@PDA/$BaSO_4$. These results proved the PF@PDA/$BaSO_4$ has been prepared successfully.

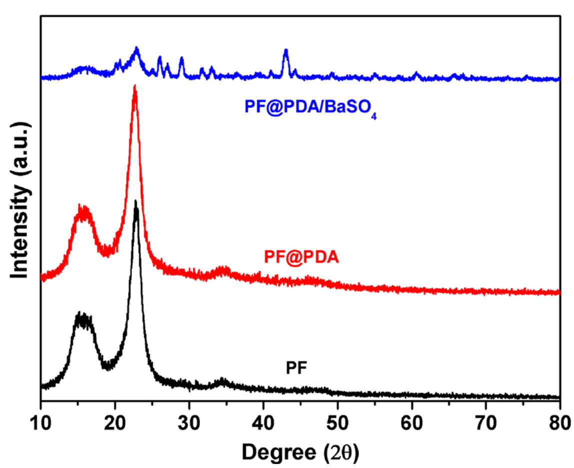

Fig. 2 XRD patterns of PF, PF@PDA and PF@PDA/$BaSO_4$

The surface wettability of membranes was evaluated by oil contact angle (OCA) underwater, here 1, 2-dichloroethane was chosen as a model oil. As can be seen from Fig. 4a, the OCA underwater of original PF is 133°, while the OCA underwater of PF@PDA is 143°, showing PDA coating improved underwater oleophobicity of membrane. Importantly, underwater OCA of the PF@PDA/$BaSO_4$ membranes are 144°, 154° and 162°, respectively. The results showed that underwater oleophobicity of membranes gradually strengthened with the increases of ASP cycles. Figure 4b exhibited the separation efficiency and water flux of different membranes. Obviously, the separation efficiency gradually increased and the water flux gradually decreased with the increases of ASP cycles. Comprehensive consideration of flux and separation efficiency, PF@PDA/$BaSO_4$-7 was selected for subsequent analysis and separation experiments (Notes: hereinafter PF@PDA/$BaSO_4$ referred to the PF@PDA/$BaSO_4$-7).

Figure 5a showed that water droplet could easily permeate the surface of PF@PDA/$BaSO_4$ with WCA of 0° in air, indicating superhydrophilicity of the PF@PDA/$BaSO_4$. The OCA underwater of the PF@PDA/$BaSO_4$ is about 156° indicating underwater superoleophobicity of the PF@PDA/$BaSO_4$ (Fig. 5b). As shown in Fig. 5c, some oil droplets underwater on the surface of PF@PDA/$BaSO_4$ membrane exhibited near-spherical indicating good oil resistance. Moreover, Fig. 5d showed that the oil droplet could be completely detached from the superoleophobic surface even in the case of severe deformation and repeated contact on the surface, indicating that the low oil adhesion of the membrane. The above experimental results indicated that PF@PDA/$BaSO_4$ has excellent superhydrophilicity/underwater superoleophobicity. Figure 5e-g indicated PF@PDA/$BaSO_4$ membrane remained underwater superoleophobicity with OCA underwater of above 150° in acid, base and salt solution (0.1 M).

Figure 6 showed the separation device for light oil (petroleum ether)/water (Fig. 6a) and heavy oil (dichloroethane)/water (Fig. 6b) mixture. As shown in Fig. 6b, the oil density is higher than water to cause

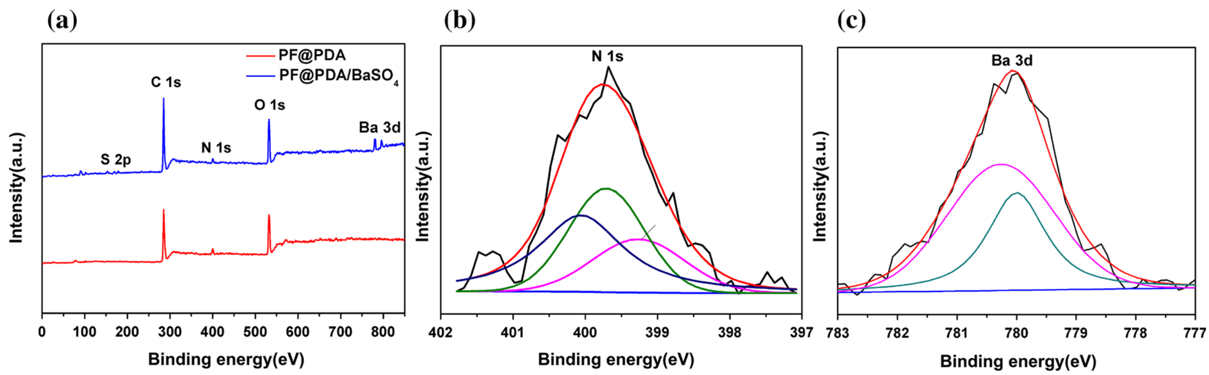

Fig. 3 XPS survey spectra of the PF, PF@PDA and PF@PDA/$BaSO_4$ (**a**), high resolution spectrum of N 1 s (**b**) and Ba 3d (**c**) of PF@PDA/$BaSO_4$

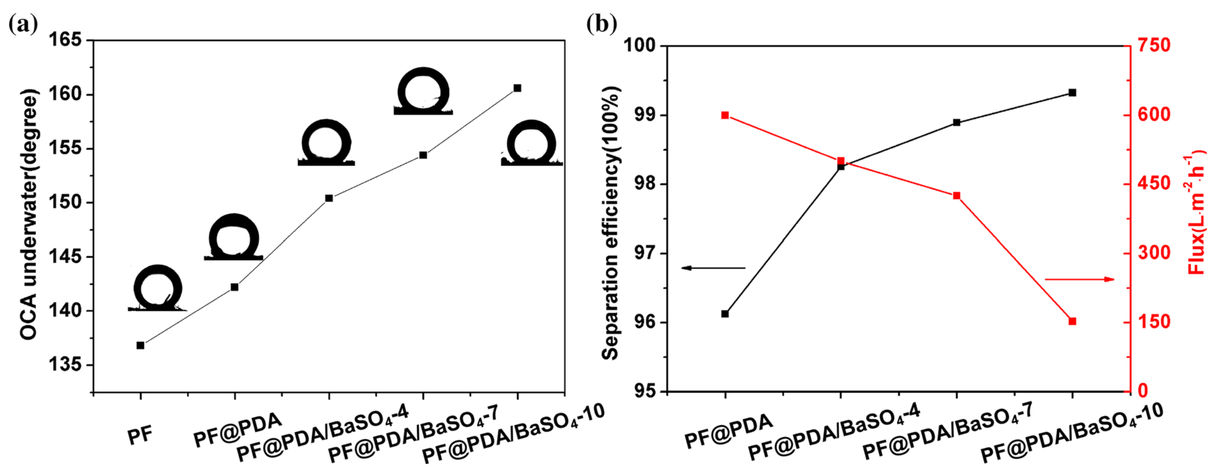

Fig. 4 **a** OCA underwater, **b** separation efficiency and water flux of membranes

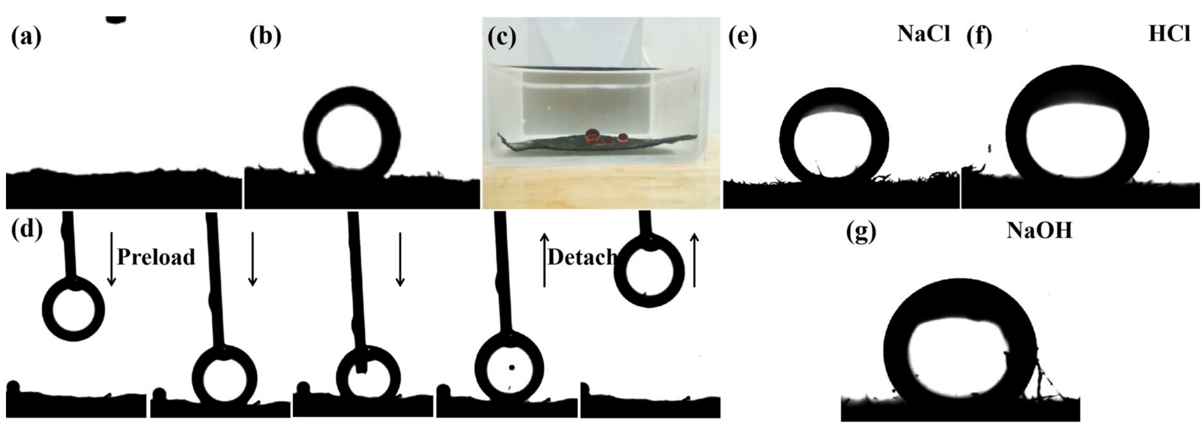

Fig. 5 **a** WCA and **b** OCA underwater of PF@PDA/$BaSO_4$, **c** a photograph of underwater oil droplets (red) on the membrane surface, **d** dynamic oil adhesion process on the PF@PDA/$BaSO_4$ underwater, OCAs underwater in acid (**e**), base (**f**) and salt (**g**) environments

aggregation, the oil/water mixture could not be separated effectively, so the separation device was tilted by 15° for separating the dichloroethane/water mixture. After pouring oil/water mixture into the device, the water quickly penetrated through the

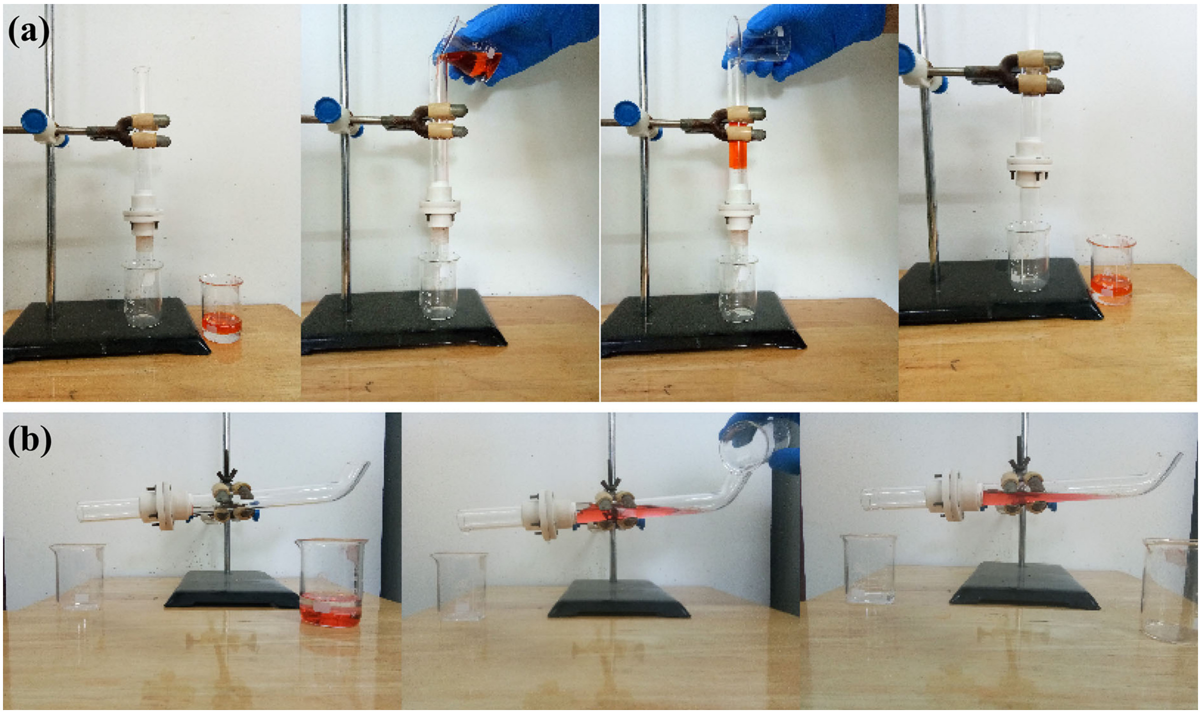

Fig. 6 Photographs of oil/water separation device: **a** petroleum ether/water and **b** dichloroethane/water

membrane under the gravity, but the oil was repelled on the membrane surface.

Figure 7a showed the separation efficiency and water flux of PF@PDA/$BaSO_4$ for various oils/water mixtures. The PF@PDA/$BaSO_4$ membrane displayed high separation efficiency of above 99% for various oil/water mixtures. In addition, the PF@PDA/$BaSO_4$ membrane showed good flux of above 400 L m^{-2} h^{-1} except soybean oil/water mixture due to high viscosity of soybean oil. The durability of PF@PDA/$BaSO_4$ membrane was further investigated. More importantly, the Fig. 7b shows the separation efficiency and flux have no obvious decline even in acid, base and salt environments, demonstrated good acid, alkali and salty resistance of PF@PDA/$BaSO_4$ membrane.

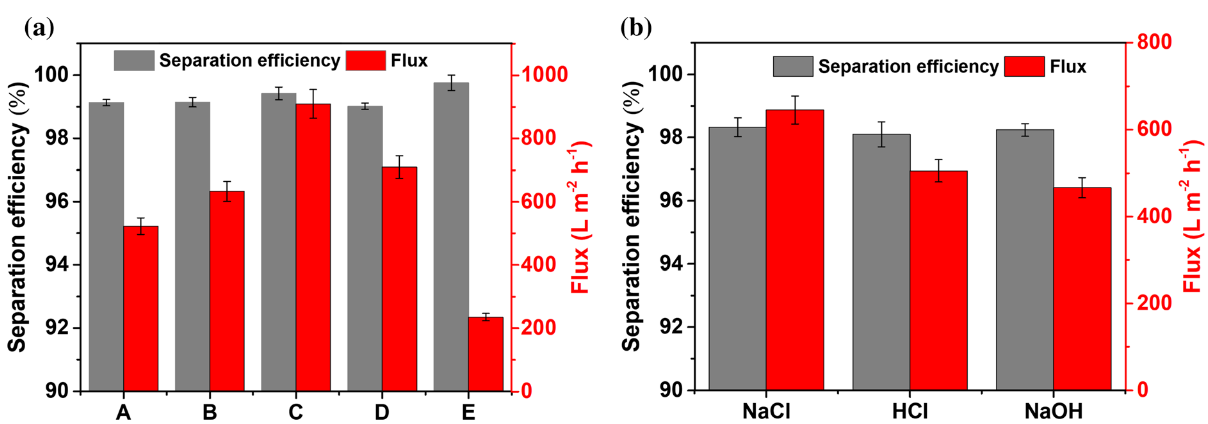

Fig. 7 **a** Separation efficiency and water flux of PF@PDA/$BaSO_4$ for different oil/water mixtures (A–E represent petroleum ether, hexane, toluene, soybean oil, dichloroethane and water mixtures), **b** Separation efficiency and water flux of PF@PDA/$BaSO_4$ in acid, base and salt environments

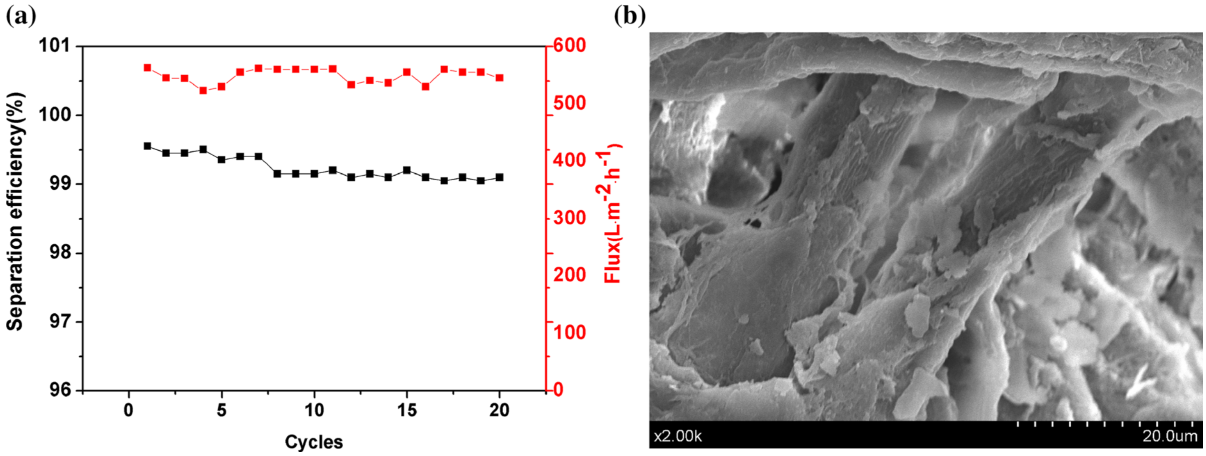

Fig. 8 **a** Separation efficiency and water flux, **b** the SEM image of PF@PDA/$BaSO_4$ after 20 cycles petroleum ether/water separation

The stability and antifouling performance are significant to superhydrophilic/underwater superoleophobic membrane for oil/water separation. As shown in Fig. 8a, the PF@PDA/$BaSO_4$ remained a high flux (above 99%) and separation efficiency (above 550 L m^{-2} h^{-1}) even after 20 cycles. Furthermore, the microstructure of PF@PDA/$BaSO_4$ after 20 cycles was observed by SEM. As shown in Fig. 8b, the $BaSO_4$ nanosheets were still firmly bonded to the fiber surface, which may be due to the strong adhesion of polydopamine. The above results demonstrated the PF@PDA/$BaSO_4$ membrane has excellent antifouling properties and outstanding reusability.

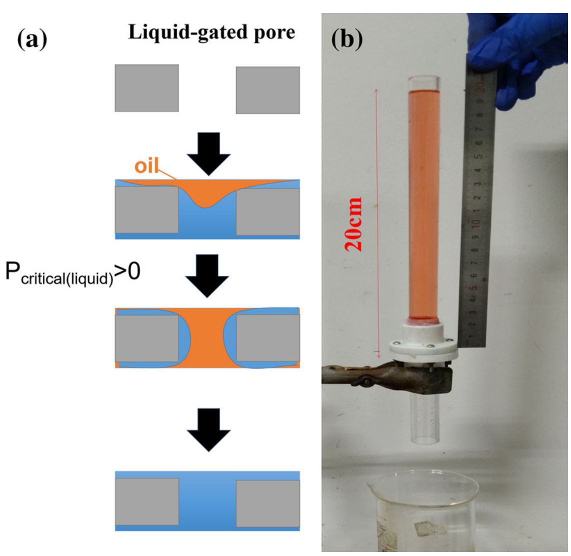

Fig. 9 **a** The separation mechanism of PF@PDA/$BaSO_4$, **b** an oil (red) intrusion pressure of PF@PDA/$BaSO_4$

Figure 9a showed a hypothetical model of superhydrophilic/underwater superoleophobic membrane for oil/water separation process. The pores were filled with a continuous layer of water. The surface of the membrane has a greater affinity for water than oil, thus water will preferentially pass through the membrane and oil cannot, so a specific critical pressure ($P_{pritical}$) was required to pass through the pores for oil (Xu et al. 2015). We usually think that the $P_{pritical}$ was intrusion pressure indicating maximum height of the oil column that the membrane can withstand. The calculation formula $P_{pritical(oil)} = \rho\, g\, h_{max}$ (Gondal et al. 2014) was used, where ρ (g/cm^3) is the density of petroleum ether, g (9.8 N/kg) is the gravitational acceleration, and $P_{pritical}$ is the critical pressure from which the height was measured. Figure 9b showed the maximum critical height of the oil column (about 0.2 m), thus the maximum intrusion pressure value is 1254 Pa according to the above formula. Therefore, if the hydrostatic pressure of the oil was greater than the $P_{pritical}$, the oil could penetrate though the membrane; if the hydrostatic pressure was less than the $P_{pritical}$, the oil would hold on the membrane surface.

Conclusions

In conclusion, a PDA deposited cellulose fiber membrane with $BaSO_4$ nanosheets was reported for oil/water separation. The $CuSO_4$/H_2O_2 accelerated the

PDA reaction rate and stability, and $BaSO_4$ nanosheets were deposited on the surface of cellulose membrane by facile alternating soaking process (ASP) to construct micro-nano structures. The as-prepared superhydrophilic/underwater superoleophobic PF@PDA/$BaSO_4$ membrane showed high separation efficiency and flux for various oil/water mixtures. Importantly, the prepared membrane maintained the high separation efficiency and flux even under acid, base and salt solution, indicated the excellent acid, alkali and salt resistance of PF@PDA/$BaSO_4$ membrane. Moreover, the PF@PDA/$BaSO_4$ membrane has excellent antifouling property and outstanding reusability due to the strong adhesion of polydopamine. In view of its advantages of low cost, easy preparation and high performance, our PF@PDA/$BaSO_4$ is expected to be a new membrane material for oil/water separation even in harsh environments.

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