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ORIGINAL RESEARCH

Robust and durable superhydrophobic cotton fabrics via a one-step solvothermal method for efficient oil/water separation

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Abstract Traditional superhydrophobic cotton fabrics (CF) demonstrate various advantages such as self-cleaning, water repelling and efficient oil/water separation capacity but limited practical applications due to their poor mechanical robustness and environmental durability. In this work, robust and durable superhydrophobic polydivinylbenzene (PDVB) coated CF (PDVB@CF) are fabricated via a simple one-step, fluorine-free, and inorganic nanoparticle-free solvothermal method. The PDVB@CF displays excellent chemical resistance without losing superhydrophobicity after immersing in various organic solvents, strong acid and alkali solutions for a long time. The PDVB@CF shows excellent mechanical robustness to resist sandpaper abrasion and ultrasonication treatment. Furthermore, the PDVB@CF exhibits outstanding high and low temperature resistances since the contact angle of either acidic or alkali droplets is greater than 150° after isothermal treatment at 200 °C or immersion in liquid nitrogen for 3 h. The PDVB@CF is very efficient in separating water mixtures with various oily compounds with separation efficiency of higher than 98% and flux up to 56 k L m^{-2} h^{-1} depending on characteristics of the oils. In addition, the PDVB@CF shows excellent recyclability with superhydrophobicity and separation efficiency that remained after 10 separation cycles. The PDVB@CF with excellent robustness and durability exhibits potential utility in oil/water separation even under some harsh conditions.

Keywords Superhydrophobicity · Polydivinylbenzene · Cotton fabric · Durability · Oil/water separation

Introduction

Oil/water separation materials are greatly desired as the increase in industrial oily wastewater and frequent oil-spill accidents (Ivshina et al. 2015; Sun et al. 2018; Wang et al. 2015a; Xu et al. 2015; Zhou et al. 2016). Superhydrophobic materials with different affinities towards oil and water are regarded as the most promising oil/water separation materials as they are able to selectively absorb or directly separate oil from water (Cheng et al. 2018a; Chu et al. 2015; Ghobashy and Elhady 2017; Li et al. 2018; Piltan et al. 2016; Wang et al. 2015b). Inspired by the lotus effect, artificial superhydrophobic surfaces can be fabricated

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by rough surface structure creation and subsequent low surface energy modification (Bellanger et al. 2014; Cheng et al. 2018b; Feng et al. 2002; Gao and McCarthy 2006; Xie et al. 2017). In this regard, superhydrophobic surfaces have been constructed on to various substrates such as metal mesh, sponge, membrane, filter paper and fabric for potential oil/water separation (Jian et al. 2018; Ouyang et al. 2016; Wu et al. 2018; Zhang et al. 2017; Zhou et al. 2017). Among these substrates, cotton fabric (CF) demonstrates great potential due to the various advantages such as renewability, good permeability, high flexibility and easily scalable fabrication (Li et al. 2015a). Unfortunately, CF cannot be used to separate oil/water mixtures directly due to its hydrophilic and oleophilic performance (Gao et al. 2017; Guo et al. 2017; Li et al. 2017b; Nyström et al. 2009).

Recently, many improved methods such as surface grafting (Gao et al. 2016; Li et al. 2015b), dip-coating (Chen et al. 2017; Cheng et al. 2017; Jin et al. 2015; Wu et al. 2016a), and spray coating (Sasaki et al. 2016; Zeng et al. 2017) have been utilized to fabricate superhydrophobic cotton fabrics for oil/water separation. It is noted that most of the methods include several complex and lengthy steps, making them sophisticated, time-consuming, and hard to scale up. In addition, expensive and toxic fluorine-containing reagents are usually required to lower the surface free energy, which would reduce environmental safety and increase cost of the final materials (Wang et al. 2013; Wu et al. 2016b; Yang et al. 2018). Furthermore, most of the fabricated superhydrophobic surfaces have poor mechanical robustness and environmentally durability. The surface rough structure often damaged when exposed to mechanical abrasion, high temperature, or corrosive substances, which results in the loss of surface superhydrophobicity permanently (Li et al. 2014; Roach et al. 2008; Xue and Ma 2013; Zhou et al. 2013). Therefore, increasing endeavors have been devoted to develop robust and durable superhydrophobic cotton fabric for oil/water separation (Chen et al. 2018; Si et al. 2016; Wu et al. 2016a). For instance, Zhou et al. fabricated a PANI-PTES coated superhydrophobic cotton fabric, showing high separation efficiency, stable recyclability, and excellent durability (Zhou et al. 2013). Robust superhydrophobic or underwater superoleophobic TiO_2 coated cotton fabric showed high efficiency for oil/water separation as well as good mechanical robustness as reported by Huang et al. (2015) and Zheng et al. (2015). Cao et al. prepared robust fluorine-free PDMS-ormosil coated cotton fabric with good physical and chemical durability as well as high oil/water separation efficiency (Cao et al. 2016). Guo's group prepared a polydopamine@SiO_2 coated cotton fabric for oil/water separation, which exhibited excellent stability, good boiling-water resistance and high separation efficiency (Guo et al. 2017). Although the robustness and durability of superhydrophobic cotton fabric can be improved significantly, the problems of complicated and lengthy fabrication or expensive and toxic fluorine containing reactants were still not avoided in those fabrication techniques. Therefore, facile and safe strategies are still highly desirable for fabrication of robust and durable superhydrophobic cotton fabrics.

Herein, we report a superhydrophobic cotton fabric prepared through a facile one-step solvothermal polymerization of divinylbenzene (DVB) with 2,2-azobis(2-methylpropionitrile) (AIBN) and ethyl acetate as the initiator and solvent, respectively. The rough surface with low free energy was constructed onto CF by deposition of hydrophobic PDVB particles formed through solvothermal polymerization. No expensive harmful inorganic nanoparticle and fluorine-containing compound was used during the one-step and simple fabrication process. The superhydrophobic PDVB@CF with excellent mechanical robustness, environmental durability, high oil/water separation efficiency and stable recyclability should find much more extensive applications especially in some harsh conditions.

Experimental section

Materials

Divinylbenzene (DVB), mixture with isomers (55%) and 2,2-azobis(2-methylpropionitrile) (AIBN) (98%) were purchased from Adamas Reagent Co., Ltd. Decane was received from Shanghai Titanchem Co., Ltd, and silicon oil was received from Aladdin Industrial Corporation. Furthermore, ethyl acetate, absolute ethanol, chloroform, petroleum ether and toluene were obtained from Chuandong chemical plant (Chongqing, China). Cotton fabric was brought from a local store.

Fabrication of PDVB@CF

The superhydrophobic PDVB@CF was prepared by one-step solvothermal polymerization. 1.6 g DVB and 0.04 g AIBN were dissolved in 80 ml ethyl acetate (EA) to get a DVB in EA solution. The solution was then transferred into an autoclave. Meanwhile, the purified cotton fabric was immersed into the solution. The autoclave was placed in a 100 °C oven for 24 h to perform the solvothermal polymerization. After cooling to room temperature, PDVB@CF was taken out of the autoclave, washed with ethanol and finally dried at 80 °C for 1 h.

Characterization

The morphology was observed with a field emission scanning microscope (FESEM, JSM-7800F) at an accelerating voltage of 2 kV. The samples were sputtered with a layer of gold before measurement. The wetting properties of water droplets on the treated cotton fabric were measured by an optic water contact angle meter system (OSA100S-T) with a 5 μL distilled-water droplet at ambient temperature. In addition, the average values of water contact angle were obtained by measuring at least five different positions on the same sample.

Mechanical robustness measurement

Sandpaper abrasion and ultrasound tests were adopted to assess the mechanical robustness of the superhydrophobic PDVB@CF. For the sandpaper abrasion test, the superhydrophobic cotton fabric was faced down on sandpaper with 800 meshes and moved 10 cm along the ruler under a 200 g weight by an external force, which is defined as one abrasion cycle. Water contact angle (WCAs) were measured after every five abrasion cycles. For the ultrasound test, the cotton fabric was treated with ultrasound in ethanol for 60 min, and the wetting behavior was measured after every 5 min of ultrasonic treatment.

Environmental durability measurement

The solvent resistance was evaluated by immersing PDVB@CF in water, ethanol, chloroform and DMF for 7 days. The acid and alkali resistance was tested by immersing PDVB@CF in H_2SO_4 (pH 1) and NaOH (pH 14) solution for 72 h, respectively. For all above chemical resistance measurement, the sample was taken out of the medium after predetermined time, washed and dried and then subjected to water contact angle measurement. High and low temperature resistance was measured by isothermal treatment at 200 °C or immersed in liquid nitrogen for 3 h, respectively. After the treatment, the contact angle of water droplet with different pH value was measured and compared with original PDVB@CF.

Oil/water separation

Oil/water mixture (100 mL) with volume ratio of 1:1 was used as the mimic oily wastewater. Various organic solvents such as cyclohexane, decane, toluene, chloroform, petroleum ether, and silicon oil were selected as the oil phases. The PDVB/CF was as a filter membrane located between two glass wares during oil/water separation. When the mimic oily wastewater was poured into the separation system, separation occurred with water remaining in the upper container and oil penetrating the fabric and falling into the lower receipting container. The separation efficiency (*W*) was calculated by the following equation:

$$W = \frac{M_2}{M_1} \times 100\% \tag{1}$$

where M_1 and M_2 are the weight of the initial oil and the collected oil after separation, respectively. Flux (*F*) of different oils was assessed by measuring the time spent in collecting the permeated oil, and calculated by:

$$F = \frac{V}{St} \tag{2}$$

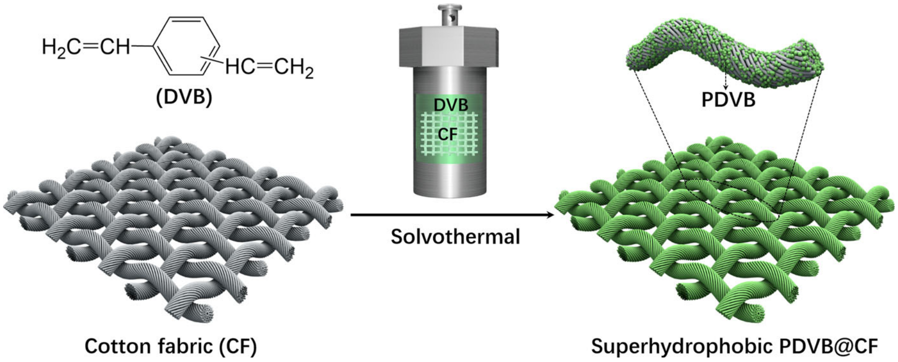
where *V* is the volume of the permeated oil, *S* represents the effective surface area of superhydrophobic cotton fabric, and *t* is the time.

Results and discussion

Preparation and characterization of superhydrophobic PDVB@CF

As mentioned in experimental section, superhydrophobic cotton fabric was fabricated by a one-step solvothermal polymerization of DVB with AIBN and

Fig. 1 Schematic illustration of the fabrication of PDVB@CF via a one-step solvothermal polymerization



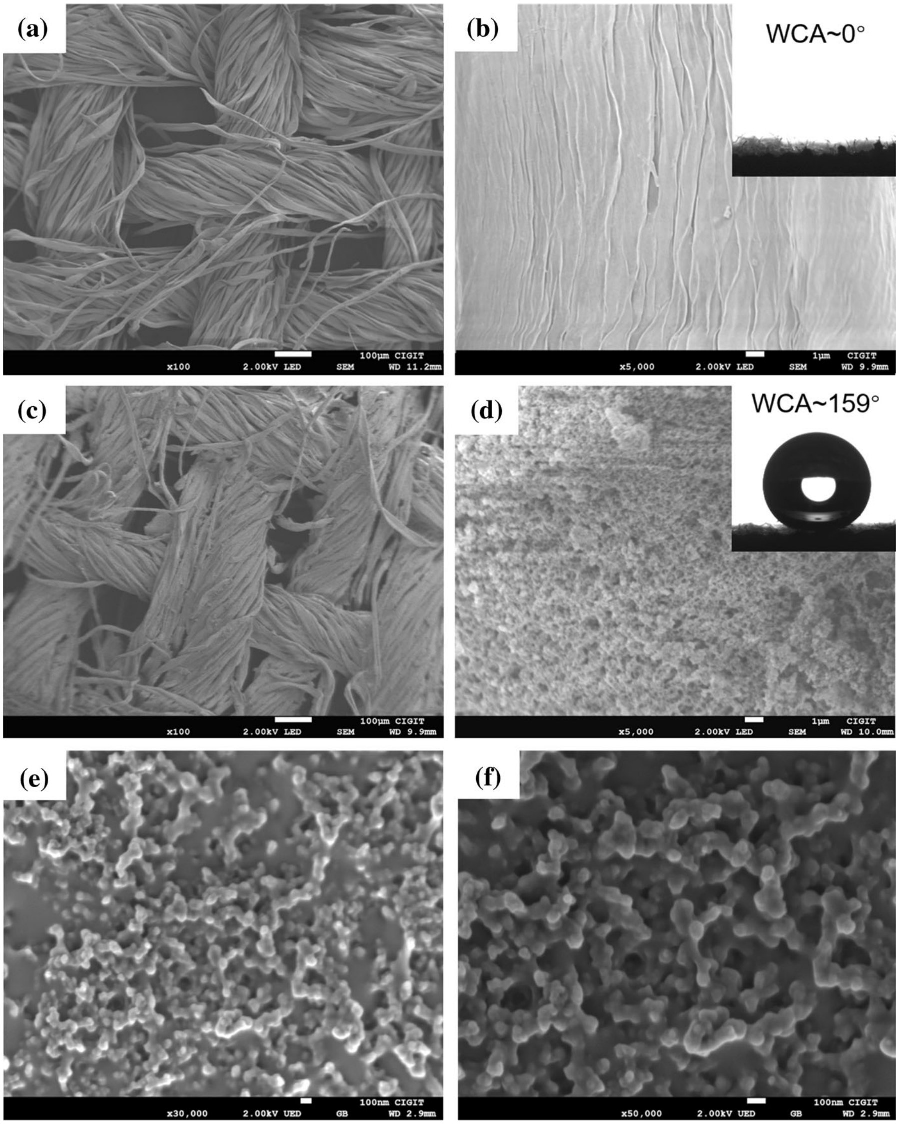


Fig. 2 FESEM and WCA images of pristine CF (**a**, **b**), PDVB@CF (**c**, **d**) and PDVB particles (**e**, **f**)

EA as the initiator and solvent, respectively, as shown in Fig. 1. Hydrophobic PDVB cross-linking networks generated and deposited on CF substrate through solvothermal polymerization, leading to rough surface with low free energy. Therefore, PDVB@CF without additional processing would exhibit superhydrophobicity. The thickness of PDVB layer on CF fabric was 14.8 µm and the loading of PDVB was 5.8 wt%.

The surface morphology and wetting behavior of pristine CF and PDVB/CF were characterized by FESEM and contact angle meter, respectively. The pristine CF possesses a porous network weave structure (Fig. 2a) with somewhat smooth cellulosic fibers (Fig. 2b). The water droplet spread quickly and wetted the pristine cotton fabric completely with a water contact angle (WCA) of 0° (inset in Fig. 2b), indicating a hydrophilic wetting behavior. PDVB@CF also shows porous network weave structure (Fig. 2c), which is similar to pristine CF. However, the magnified micrograph of PDVB/CF (Fig. 2d) demonstrates the surface of individual fiber is completely covered with PDVB particles, which enable formation of hierarchical micro- and nanoscale rough structures onto cotton fabric, resulting in a superhydrophobic surface with a water contact angle of 159° (inset of Fig. 2d). Figure 2e, f present the field emission scanning electron microscopy (FE-SEM) images of the PDVB particles under different magnifications. The size of PDVB particles almost ranged from 40 to 80 nm. The nanoscale PDVB particles aggregated and

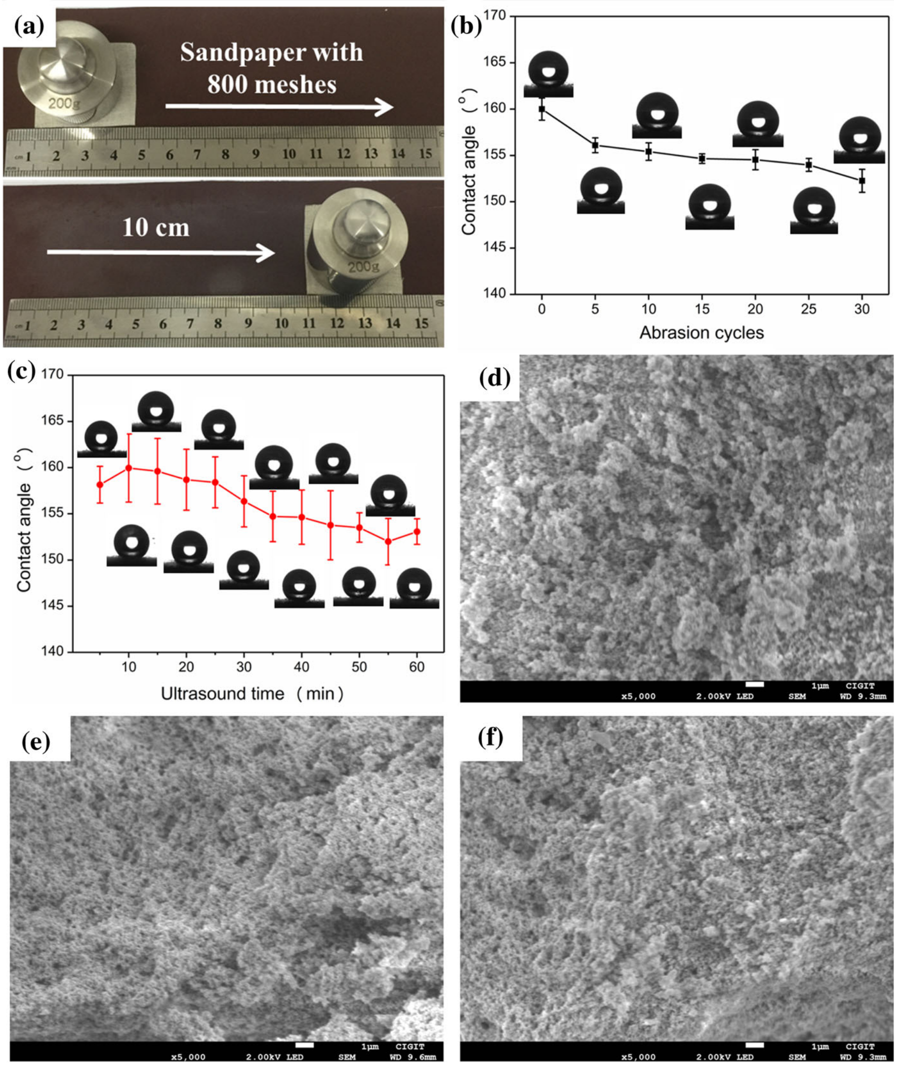


Fig. 3 **a** Photographs showing the process of abrasion test, **b** variation of WCA versus abrasion cycle during sandpaper abrasion resistance measurement, **c** variation of WCA versus ultrasound time during ultrasound resistance measurement, and SEM micrographs of original PDVB@CF (**d**), PDVB@CF after abrasion for 30 times (**e**), and PDVB@CF after ultrasonication for 60 min (**f**)

formed numerous porous structures on fibers after coated on CF.

Mechanical robustness and environmental durability

The micro/nanoscale hierarchical structure of superhydrophobic surface may be destroyed to lose superhydrophobicity during practical application when exposure to mechanical abrasion, chemical corrosion, and high or low temperature condition (Gao et al. 2016; Hou et al. 2018; Liu et al. 2017). The mechanical robustness of PDVB@CF was characterized by sandpaper abrasion and ultrasonic treatment. Figure 3a demonstrates mechanical abrasion measurement by moving PDVB@CF on sandpaper under 200 g loading. Figure 3b shows the variation of WCA versus abrasion cycle for the superhydrophobic PDVB@CF. Obviously, WCA only decreased slightly with the increase of abrasion cycles and the superhydrophobicity maintained even after abrasion for 30 times. Figure 3c illustrates the variation of WCA versus ultrasound time for the superhydrophobic cotton fabric. Although the WCA decreased slightly with increasing ultrasound time, the superhydrophobicity remained even after ultrasonication for an hour. The surface morphology of original PDVB@CF, PDVB@CF after abrasion for 30 times and PDVB@CF after ultrasonication for 60 min was observed by SEM, as shown in Fig. 3d–f. It is interesting that the rough structure didn't change obviously after abrasion or ultrasonication, indicating strong adhesions between CF substrate and PDVB particles formed through solvothermal polymerization, which endows the PDVB@CF with excellent

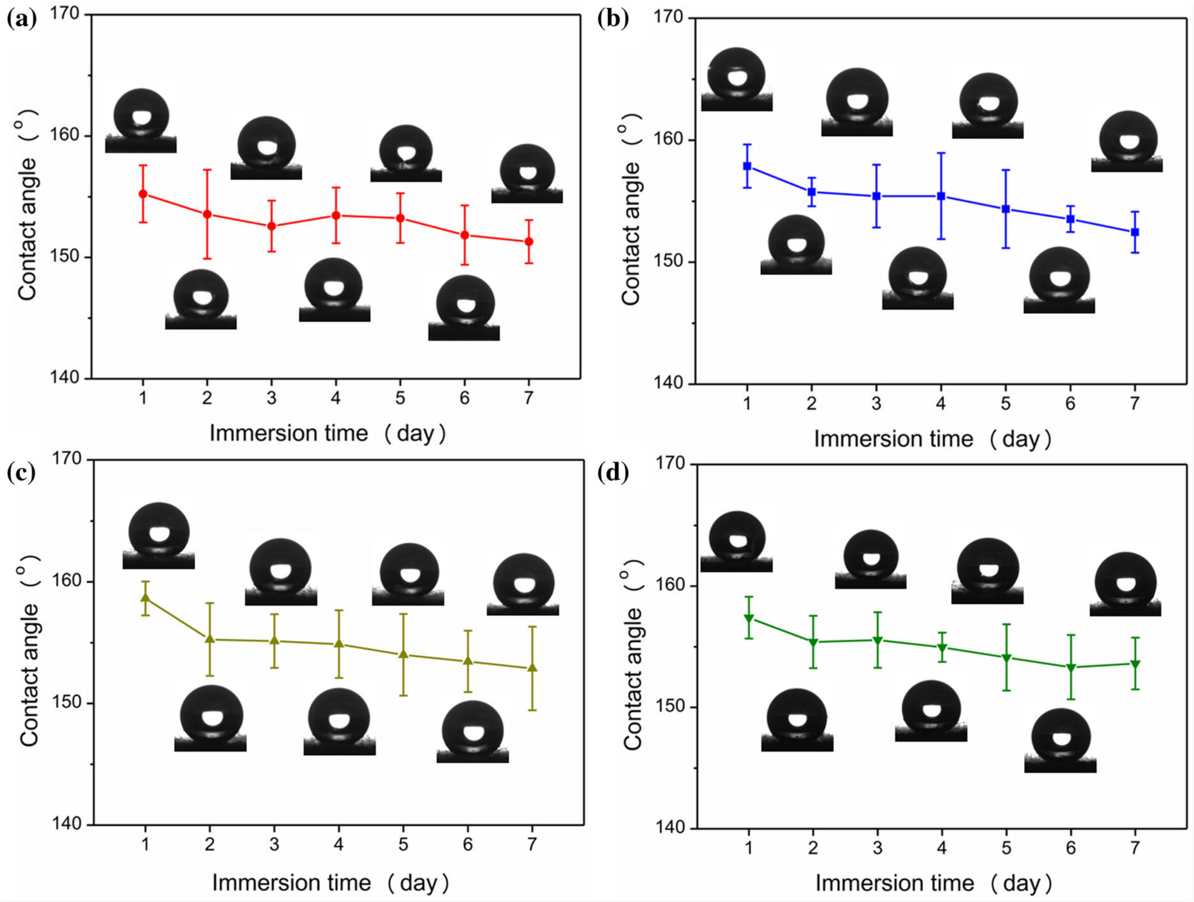


Fig. 4 Variation of WCA versus immersion time for PDVB@CF with different solvents: water (**a**), ethanol (**b**), chloroform (**c**) and DMF (**d**). Insets are pictures of water droplets (5 μL) on the PDVB@CF surface after immersion for different time

mechanical robustness to resistant sandpaper abrasion and strong ultrasonication.

Except for mechanical abrasion, oil/water separation materials may suffer from various chemical corrosions such as organic solvent, acid and alkali during practical application. Therefore, it is necessary to measure the chemical resistance of the superhydrophobic PDVB@CF. The solvent resistance of PDVB@CF was evaluated by measuring the WCA after immersing in various solvents for several days. Four solvents with different polarities including water, ethanol, chloroform and *N*,*N*-dimethylformamide (DMF) were used in this study. Figure 4 shows the variation of WCA with immersion time in the different solvents. Obviously, the superhydrophobicity remained after immersion in all the solvents for at least 1 week as the WCA was greater than 150° irrespective of the natures of solvent, which demonstrates an excellent solvent resistance of PDVB@CF.

Superhydrophobic materials may be exposed to high/low temperature and acid/alkali conditions during practical applications. Therefore, the contact angle toward water droplets of different pH values was measured after exposure at 200 °C or immersion in liquid nitrogen for 3 h, respectively, to demonstrate the high/low temperature and acid/alkali resistances of the superhydrophobic PDVB@CF. The contact angles to water droplet of different pH values for the original PDVB@CF were measured for comparison. As Fig. 5a shows, no obvious diversification in contact angles was found for the original PDVB@CF with contact angle greater than 158° over a wide pH range from 1 to 13, indicating excellent permanent superhydrophobicity toward not only pure water but also corrosive acid and alkali solution. After immersion in liquid nitrogen for 3 h, the contact angles did not show apparent fluctuation under various pH conditions with the values higher than 158°, indicating the excellent resistance to low temperature. After exposure to 200 °C oven for 3 h, the surface superhydrophobicity was also maintained with contact angles toward water droplets with various pH values greater than 157°. The acid and alkali resistances of the superhydrophobic PDVB@CF were further measured by immersion in H_2SO_4 aqueous solution (pH 1) and NaOH aqueous solution (pH 14) for 72 h. Figure 5b shows the variation of WCA versus immersion time of PDVB@CF. It is obviously that the superhydrophobicity of PDVB@CF remained after immersion in acid and alkali solutions with WCA greater than 153° and 155°, respectively.

The above results indicate that PDVB@CF exhibits excellent mechanical robustness and environmental durability, due to the outstanding stability of PDVB with cross-linked structure toward various extreme conditions and the strong adhesion between CF substrate and the cross-linked PDVB particles formed via solvothermal polymerization. With excellent mechanical robustness and environmental durability, the superhydrophobic PDVB@CF can find widespread applications even under some extreme conditions.

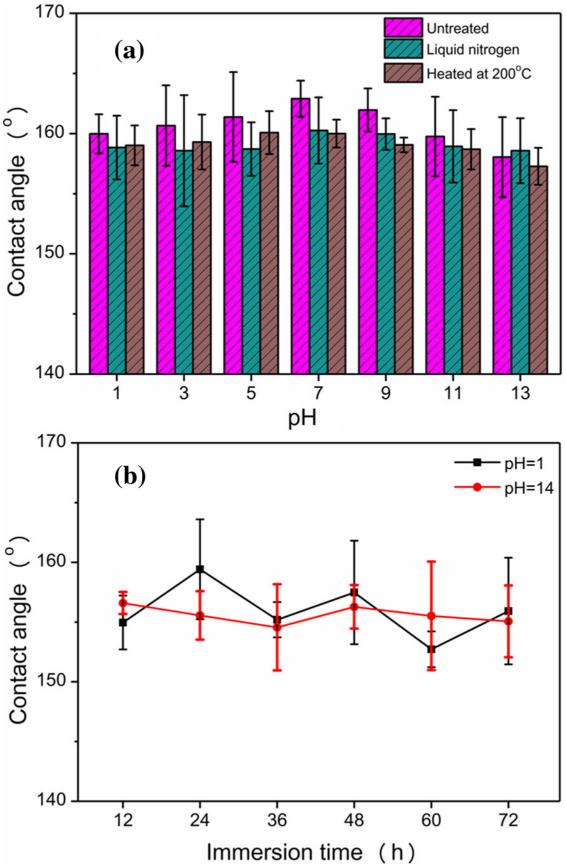


Fig. 5 Contact angles toward water droplets with different pH values for untreated and low/high temperature treated PDVB@CF (**a**) and WCA versus immersion time for acid/alkali resistance measurements of PDVB@CF (**b**)

Oil/water separation

For oil/water separation, the superhydrophobic PDVB@CF was used as filter fabric located between two glass tubes, as shown in Fig. 6. If oil is denser than water, their mixtures can be separated with vertically placed apparatus (Fig. 6a), and the mixtures with oil less dense than water can be separated with aslant placed apparatus (Fig. 6b). Oils were dyed with oil red and water was colored with dispersed blue for clear observation. When oil/water mixture was poured into the separation setup, the water was inhibited above while the oil phase straightly penetrated through PDVB@CF driven by gravity. The separation

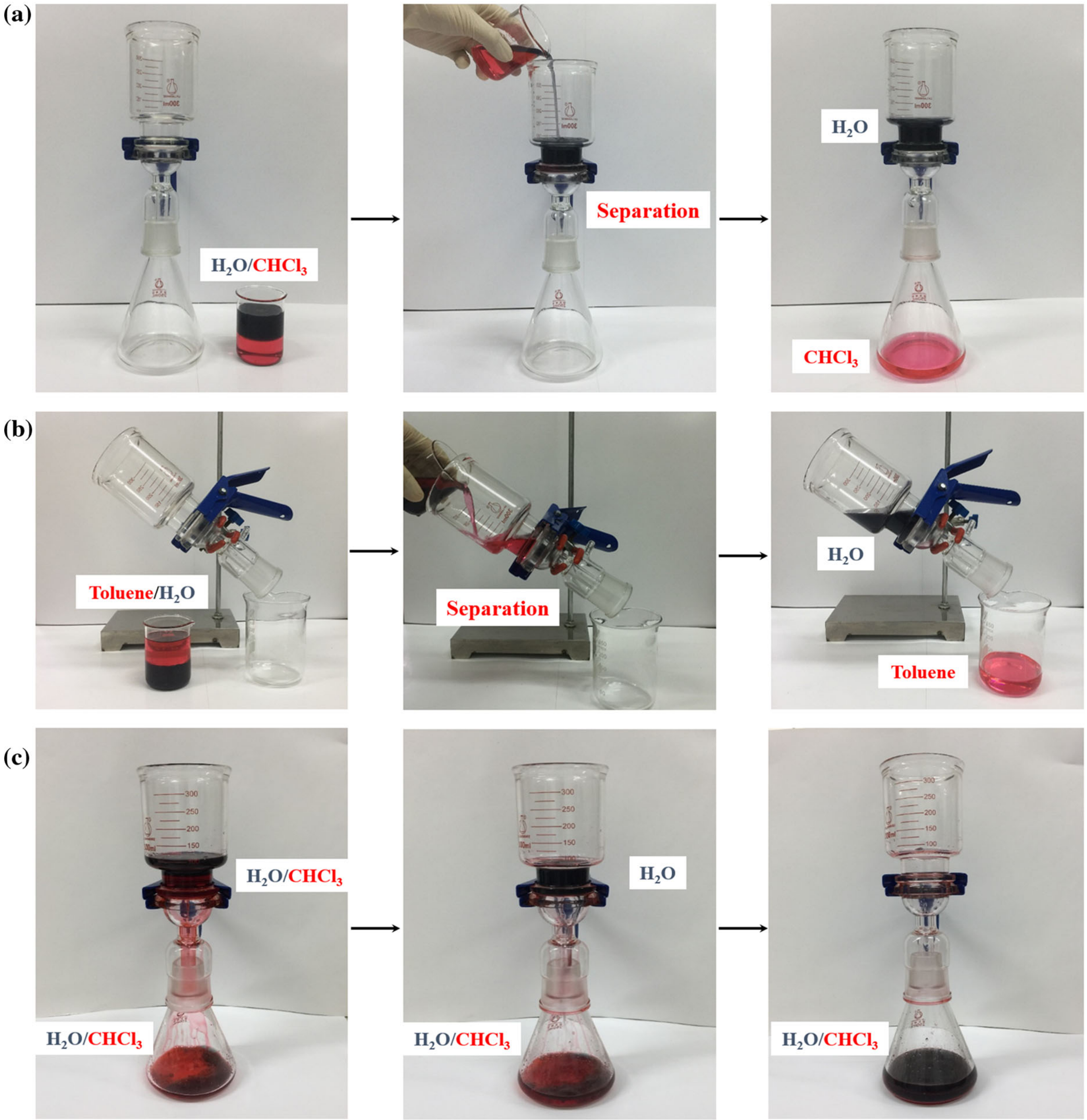


Fig. 6 Photographs for separation process of water (dyed with dispersed blue) mixtures with chloroform (dyed with oil red) (**a**) and water (dyed with dispersed blue) mixtures with toluene (dyed with oil red) (**b**) to PDVB@CF, and water (dyed with dispersed blue) mixtures with chloroform (dyed with oil red) to pristine CF (**c**). (Color figure online)

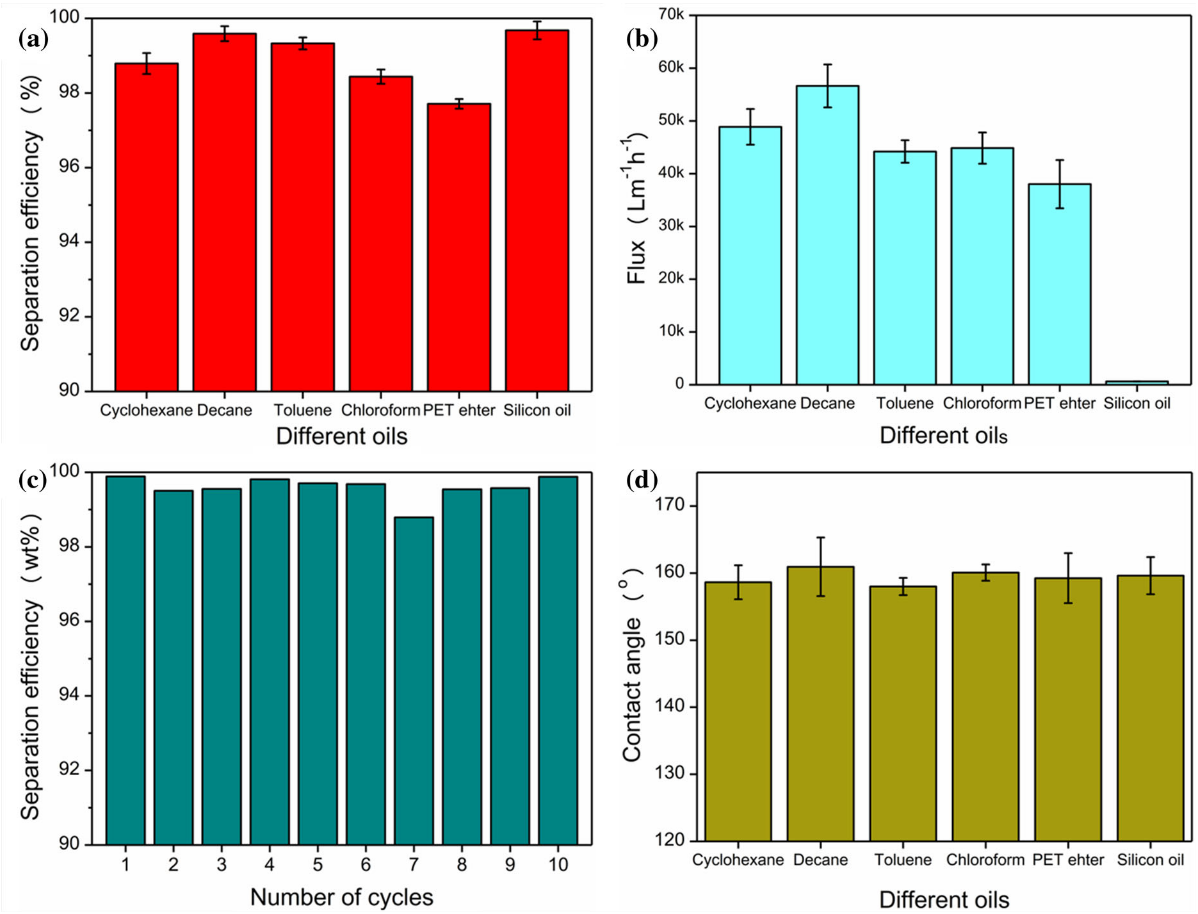


Fig. 7 Separation efficiency (**a**) and flux (**b**) of PDVB@CF toward different oil/water mixtures, separation efficiency of PDVB@CF toward decane/water mixture for 10 cycles (**c**), and water contact angle of PDVB@CF after separation of different oil/water mixtures separation for 10 cycles (**d**)

performance of PDVB@CF are determined by the oil layer that blocks the surface pores which presents inconsistent gating behaviors towards water and different oils at the same time, due to superhydrophobic and superoleophilic nature of the PDVB@CF (Dou et al. 2017; Li et al. 2017a). For comparison, the oil/water separation performance of CF was also conducted (Fig. 6c). When oil/water mixture was poured into the separation setup equipped with pristine CF, both oil and water both penetrated through pristine CF simultaneously driven by gravity, indicating non-selective separation of the pristine CF toward oil/water mixture.

The oil/water separation efficiency of and flux of oil through PDVB@CF were investigated to evaluate the separation performance quantitatively. The water mixtures with several different oils such as chloroform, toluene, petroleum ether, hexane, decane and silicon oil were chosen as the model oil/water mixtures. It is interesting that the separation efficiency of PDVB@CF towards all the oil/water mixtures is higher than 98%, as shown in Fig. 7a. In the case of flux, the oils with low viscosity such as cyclohexane, decane, toluene, chloroform and petroleum ether exhibit high flux with value in the range of 38–56 k L m^{-2} h^{-1}, while the high viscous silicon oil shows a low flux with 635 L m^{-2} h^{-1}, as shown in Fig. 7b. It is reasonable since liquid with high viscosity has low flow velocity.

The reusability of PDVB@CF was evaluated by separating decane/water mixtures for 10 cycles and the separation efficiency for each cycle was recorded, as shown in Fig. 7c. The separation efficiency for each cycle was higher than 98.5%. The WCA of

PDVB@CF after each separation was measured after washing with ethanol and drying, as shown in Fig. 7d. It is interesting that the values were greater than 158° regardless of the reuse times. The results indicated PDVB@CF exhibits an excellent recyclability due to its excellent mechanical robustness and environmental durability.

Conclusions

In summary, superhydrophobic cotton fabric was fabricated via a facile one-step solvothermal reaction with DVB and AIBN as the monomer and initiator, respectively. The polymerized PDVB particles with cross-linked structure coated on the surface of cotton fabric very tightly, endowing the superhydrophobic PDVB@CF with excellent stability. The PDVB@CF showed excellent mechanical robustness with surface morphology and superhydrophobicity remained when suffering from mechanical abrasion and ultrasonication. The PDVB@CF exhibited excellent resistance to chemical corrosions without obvious loss in contact angle after immersion in various solvents and acid/alkali solutions. In addition, the PDVB@CF had outstanding high/low temperature resistance with contact angle to water droplets with various pH values similar to original PDVB@CF even after exposure to liquid nitrogen and 200 °C oven. Furthermore, PDVB@CF was very effective in separation of water mixtures with various oils with separation efficiency higher than 98% and showed stable recyclability without loss in separation efficiency during reusing. The mechanism for the excellent separation efficiency of PDVB@CF has been proposed.

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