Fabrication of durable superhydrophobic/oleophilic cotton fabric for highly efficient oil/water separation

M. E. Mohamed and B. A. Abd-El-Nabey

ABSTRACT

In the present work, dopamine is self-polymerized on cotton fabric by a simple deep-coating method and followed by modification with an ethanolic solution of palmitic acid: a superhydrophobic/oleophilic cotton fabric was obtained. The as-prepared cotton fabric exhibits a superhydrophobic character with a water contact angle of 157°. The absorption capacity of as-prepared superhydrophobic/oleophilic cotton fabric in n-hexane, petroleum ether, and silicone oil was determined. The results show that silicone oil has the highest absorption capacity while n-hexane has the lowest value. The absorption capacity is nearly constant even after ten cycles, indicating the efficient recyclability of the as-prepared superhydrophobic/oleophilic cotton fabric for oil separation. The as-prepared superhydrophobic/oleophilic cotton fabric shows excellent separation efficiency, high flux rate, and excellent chemical and mechanical stability.

Key words | dopamine, oil/water separation, oleophilic, palmitic acid, superhydrophobic

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HIGHLIGHTS

- A superhydrophobic/oleophilic cotton fabric was fabricated by immersion of the cotton fabric in dopamine and palmitic acid solutions.
- The superhydrophobic cotton fabric shows excellent separation efficiency with high flux rate.
- The superhydrophobic cotton fabric shows excellent chemical and mechanical stability.
- The superhydrophobic cotton fabric shows high durability and recyclability.

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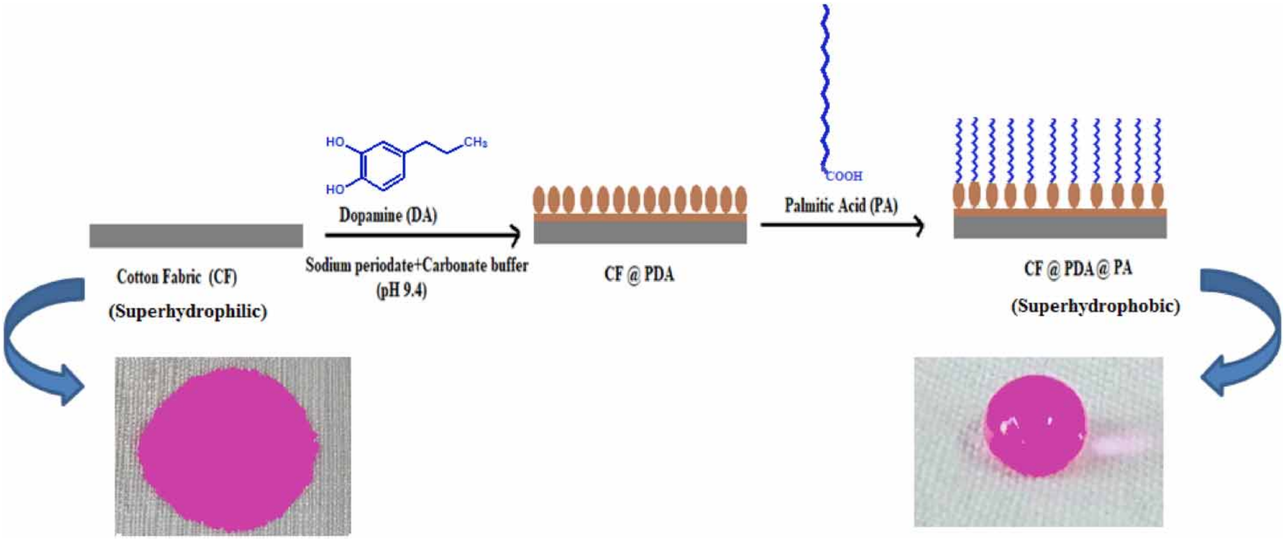
GRAPHICAL ABSTRACT



INTRODUCTION

Numerous industries, such as mining, textiles, food, petrochemicals, and metal/steel industries, generate massive quantities of oily wastewater that has become an extremely frequent pollutant worldwide, and has become a significant international environmental issue (Chen & Xu 2013; Gupta *et al.* 2017). Besides, regular oil leakage/spillage during shipping or oil processing is a possible marine environment disaster (Lu *et al.* 2014). The separation of oil/water, therefore, has gained significant attention. Thus there is an increasing demand for functional materials that can effectively separate a mixture of water and oil.

Recently, the use of superhydrophobic materials for oil/water separation has gained considerable attention due to their massively different wettability to water and oils (Chen & Xu 2013; Lu *et al.* 2014; Guo *et al.* 2016; Gupta *et al.* 2017; Sriram & Kumar 2019). Many of the superhydrophobic materials are oleophilic or even superoleophilic; that is, the superhydrophobic materials can be easily wetted by oils because of their low surface tension (Kang *et al.* 2019). Thus, there is a great chance to prepare excellent superhydrophobic/superoleophilic materials for oil/water separation by introducing materials of low surface energy and proper surface topography (Chen & Xu 2013; Lu *et al.* 2014; Guo *et al.* 2016; Gupta *et al.* 2017; Kang *et al.* 2019; Sriram & Kumar 2019). Numerous methods have already been developed to produce a superhydrophobic surface, such as plasma etching (Hou *et al.* 2020), chemical etching (Attar *et al.* 2020), dip-coating (Gupta *et al.* 2017), sol-gel technique (El Fouhaili *et al.* 2019), chemical vapor deposition (Kang *et al.* 2019), anodic oxidation (Zang *et al.* 2019), and electrodeposition (Mohamed & Abd-El-Nabey 2020).

There are in reality many superhydrophobic materials used to separate oil from water, such as textiles (Zhou *et al.* 2013), membranes (Chen & Xu 2013), sponges (Wang *et al.* 2015), metal meshes (Guo *et al.* 2016), filter paper (Sriram & Kumar 2019), glass wool (Kang *et al.* 2019), and conjugated polymer (Xiao *et al.* 2017). Unfortunately, there are many defects of these materials, such as low efficiency in oil/water separation, low stability and low recyclability. Therefore, there is a high demand for further improvement of cost-effective, environmentally friendly, recyclable materials, and effective oil/water separation techniques that can purify high quantities of oil/water blends, with high efficiency and high flux rate.

Palmitic acid provides greater hydrophobicity than stearic acid because of the short chain of palmitic acid, which enables charging more nanoparticles in a similar volume, giving higher hydrophobicity. Palmitic acid is more available and has a simpler production processes than stearic acid so we used palmitic acid in our study as a low surface energy material (Mohamed & Abd-El-Nabey 2020).

Moreover, considering the weak adhesive force of coating layers on the substrate material, it is an urgent requirement for researchers to fabricate an enhanced adhesive force between the coating layer and the substrate. Recently, mussel adhesive proteins (MAPs) excreted by marine mussels have attracted much attention because of their ability to form strong adhesive interaction with various substrates in a wet environment (Wang *et al.* 2020; Yan *et al.* 2020). In the present work, dopamine is self-polymerized on cotton fabric by a simple deep-coating method and followed by modification with an ethanolic solution of palmitic acid. Polydopamine has strong adhesion to materials and can adhere to almost all materials, including Teflon, metal, wood, fabric, and glass (Lee *et al.* 2006, 2007).

This study aims to fabricate superhydrophobic film on cotton fabric for oil/water separation by immersion in dopamine solution to produce surface roughness with high adhesion to the substrate followed by modification with low surface energy material by immersion in an ethanolic solution of palmitic acid. The oil absorption capacity, oil-water separation performance, chemical stability, and mechanical stability of the modified cotton fabric were investigated.

EXPERIMENTAL

Materials

Dopamine hydrochloride (purity, 98%), palmitic acid, sodium periodate, sodium hydroxide, sulphuric acid (98%), sodium carbonate, sodium bicarbonate, methyl red, n-hexane, petroleum ether, and silicone oil were purchased from SIGMA ALDRICH. Pure cotton fabric was gained from a local store.

The fabrication of superhydrophobic/oleophilic cotton fabric, CF @ PDA @ PA

2.0 g of dopamine hydrochloride (DA), 3.351 g of sodium bicarbonate, 1.071 g of sodium carbonate, 2.1 g of sodium periodate were added to 991.5 g of deionized water (pH = 9.4). Then, the mixture was stirred at room temperature for 0.5 hour. Cotton fabric (circle, with a diameter of 42.5 mm) was immersed in the above mixture for 2 hours under magnetic stirring, and then the cotton fabric was washed with deionized water and then dried in an oven at 60 °C.

The modified cotton fabric with polydopamine (PDA) was immersed in 0.1 M ethanolic solution of palmitic acid (PA) for 24 hours. Finally, a superhydrophobic/oleophilic cotton fabric (CF @ PDA @ PA) was obtained after drying at 60 °C for 1.0 hour. The scheme of fabricating superhydrophobic/oleophilic cotton fabric, CF @ PDA @ PA, is shown in Figure 1.

Characterization

The morphology of cotton fabric samples before and after finishing was tested by a scanning electron microscope (SEM)

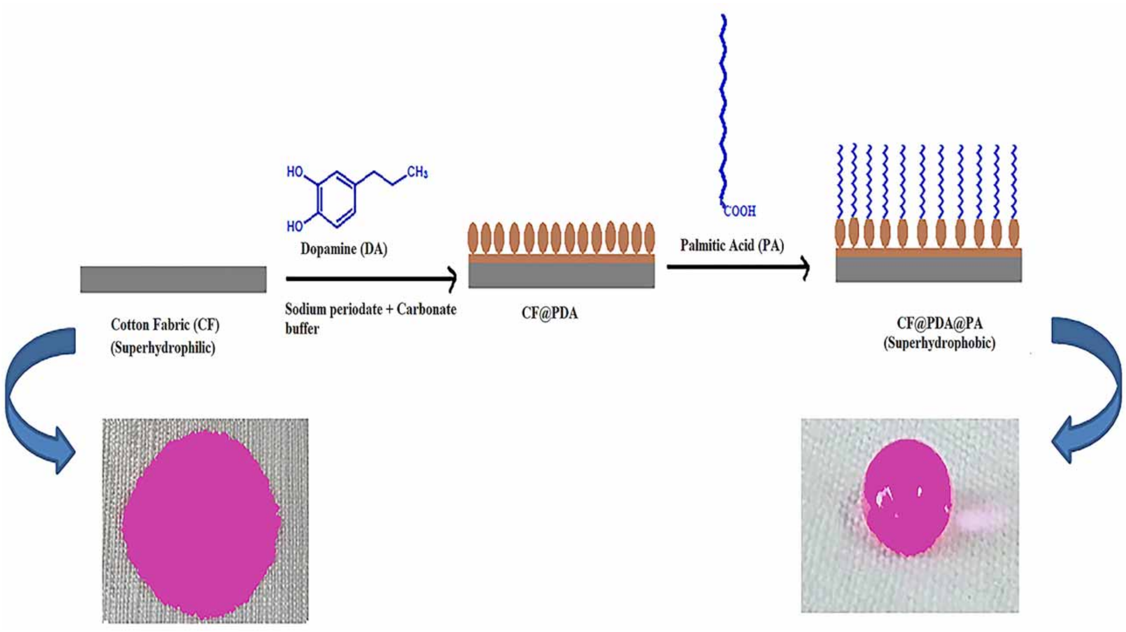


Figure 1 | Schematic diagram of the fabrication process of superhydrophobic/oleophilic cotton fabric, CF @ PDA @ PA.

(model JSM-IT 200). The elemental compositions were measured by energy dispersed X-ray (EDX) microanalyzer attached to the SEM. The FT-IR spectroscopy was studied by Fourier transform infrared spectrophotometer (FT-IR LX 18-5255 Perkin Elmer). The spectra were recorded in the wavenumber range of 4,000–500 cm^{-1}. The static water contact angles were tested with 5 μL water droplets using an optical contact angle meter (OCALS plus). The reported values of contact angles are the averages of five measurements, which are performed on different sites on the surface of the as-prepared superhydrophobic cotton fabric. The pH of the water droplets was adjusted using sodium hydroxide and sulfuric acid. The chemical stability of the as-prepared superhydrophobic cotton fabric was tested at a pH range from 1 to 13. The mechanical properties of the as-prepared superhydrophobic cotton fabric were estimated using the scratch test. The reported chemical and mechanical results are the averages of two experiments.

Absorption capacity measurements

The absorption capacity of the as-prepared superhydrophobic/oleophilic cotton fabric sample was tested as follows: a piece of the superhydrophobic/oleophilic cotton fabric sample was immersed in n-hexane, petroleum ether, or silicone oil at room temperature, and then taken out after 1 minute, drained for several seconds, and wiped with filter paper to remove the excess organic solvents. The n-hexane, petroleum ether, and silicone oil absorption capacity (k) was calculated from weights measured at room temperature according to the following equation (Zhang *et al.* 2017):

$$k = M_1/M_0 \tag{1}$$

where M_1 is the weight of the sample after organic solvent absorption and M_0 is the weight of the modified superhydrophobic cotton fabric sample.

Measurements of oil/water separation performance

Oil/water mixture (50 mL) with a volume ratio of 1:1 was used as the oily wastewater. The used distilled water was dyed with methyl red. Various organic solvents such as n-hexane, petroleum ether, and silicone oil were selected as the oil phases. The as-prepared superhydrophobic/oleophilic cotton fabric was used as a filter membrane during oil/water separation. When the oily wastewater was poured into the separation system, the separation occurred with water remaining in the upper container and oil penetrating the modified cotton fabric and falling into the lower receiving container. The separation efficiency (W) was calculated by the following equation (Kang *et al.* 2019):

$$W = (M_2/M_3) \times 100\% \tag{2}$$

where M_3 and M_2 are the weights of the initial oil and the collected oil after separation, respectively.

The flux of the modified cotton fabric was calculated using the following equation (Cao *et al.* 2017):

$$Flux = V/(S \times t) \tag{3}$$

where, V is the volume of permeating liquid, S is the effective area of the film, and t is the permeating time.

RESULTS AND DISCUSSION

Surface morphology and chemical composition of pristine and modified cotton fabric

Figure 2 shows the SEM micrographs of the pristine and modified cotton fabric. Figure 2(a) and 2(b) show that the pristine cotton fabric exhibits relatively smooth surfaces. Figure 2(c) and 2(d) show the modified cotton fabric with the polydopamine, the fibers were clad in micro/nano polydopamine aggregates, and the surface roughness of the fibers was significantly improved, which is a crucial parameter for attaining superhydrophobicity. Figure 2(e) and 2(f) show the cotton fabric modified by polydopamine after treatment with palmitic acid, the surfaces of the cotton fabric still maintained the micro/nano structure with appearance of new structures, which are attributed to adsorption of low surface energy palmitic acid which is considered as the second necessary parameter to attain superhydrophobicity, the first parameter being surface roughness. To further confirm the successful hydrophobization process, elemental compositions were measured by an EDX technique. Figure 3 shows the elemental analysis of the surface of pristine and modified cotton fabrics obtained from the EDX technique. Figure 3(a) shows the EDX analysis of the pristine cotton fabric, which contains a carbon and oxygen peaks only, a nitrogen peak appeared in the micrograph of the cotton fabric modified with polydopamine, see Figure 3(b). This nitrogen peak confirms that the dopamine was polymerized on the surface of the cotton fabric.

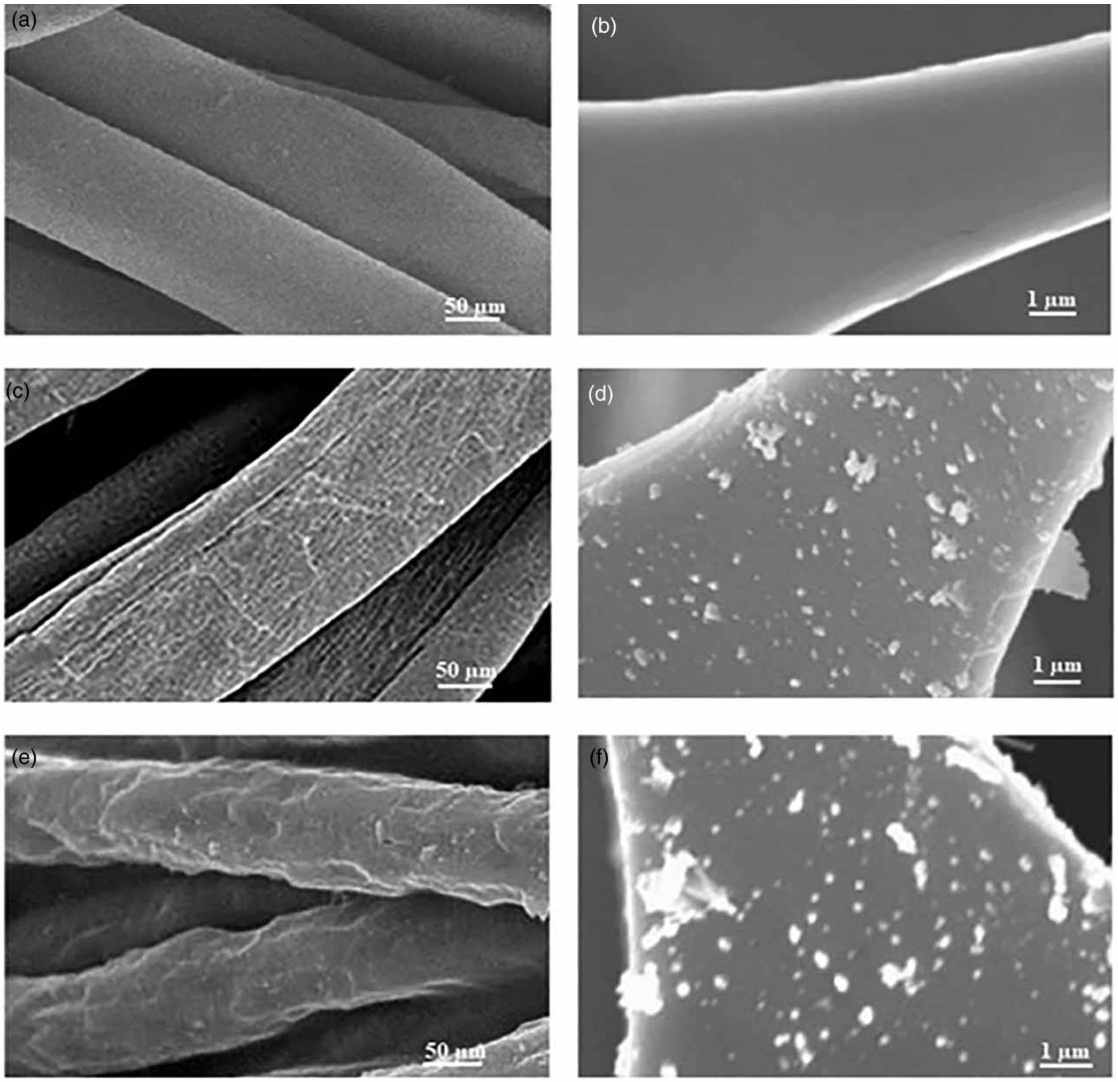


Figure 2 | SEM images of pristine cotton fabric (a, b), modified cotton fabric with PDA (c, d), and modified cotton fabric with PDA and PA (e, f).

Figure 3(c) gives the EDX analysis of the modified superhydrophobic cotton fabric, the micrograph shows carbon, oxygen and nitrogen peaks; furthermore, the wt. % of the nitrogen decreases from 5.12 to 1.67 indicating the adsorption of palmitic acid on the modified cotton fabric with polydopamine.

The FT-IR spectra were used to trace the chemical changes of the cotton fabric surface functionalized with PDA and PA. The FT-IR spectra of pure cotton fabric, cotton fabric modified with polydopamine, and cotton fabric modified with polydopamine and palmitic acid are shown in Figure 4. As displayed in Figure 4, for a pure cotton fabric a broad band corresponding to the hydroxyl groups of cellulose is observed at 3,320 cm^{-1}, the band observed at 2,900 cm^{-1} corresponds to stretching vibrations of $-CH_2$, and the band observed at 1,000 cm^{-1} corresponds to the bending vibration of C–O. While after being modified with polydopamine, absorption peaks at 1,260 cm^{-1} and 1,390 cm^{-1} corresponding to C-OH stretching vibration and O-H deformation vibration respectively besides a broad band corresponds to the stretching vibrations of O-H and N–H groups in the polydopamine is observed at 3,379 cm^{-1}, this confirming the existence of polydopamine on the surface of cotton fabric (Belal *et al.* 2020) While after being modified with palmitic acid, absorption peaks at 2,849 cm^{-1} and 2,917 cm^{-1} corresponding to the C–H symmetric and asymmetric stretching vibrations of palmitic acid, and an absorption peak at 1,400 cm^{-1} ascribed to O-H deformation vibration and an absorption peak at 1,075 cm^{-1} corresponding to the C-OH stretching vibration, and a broad

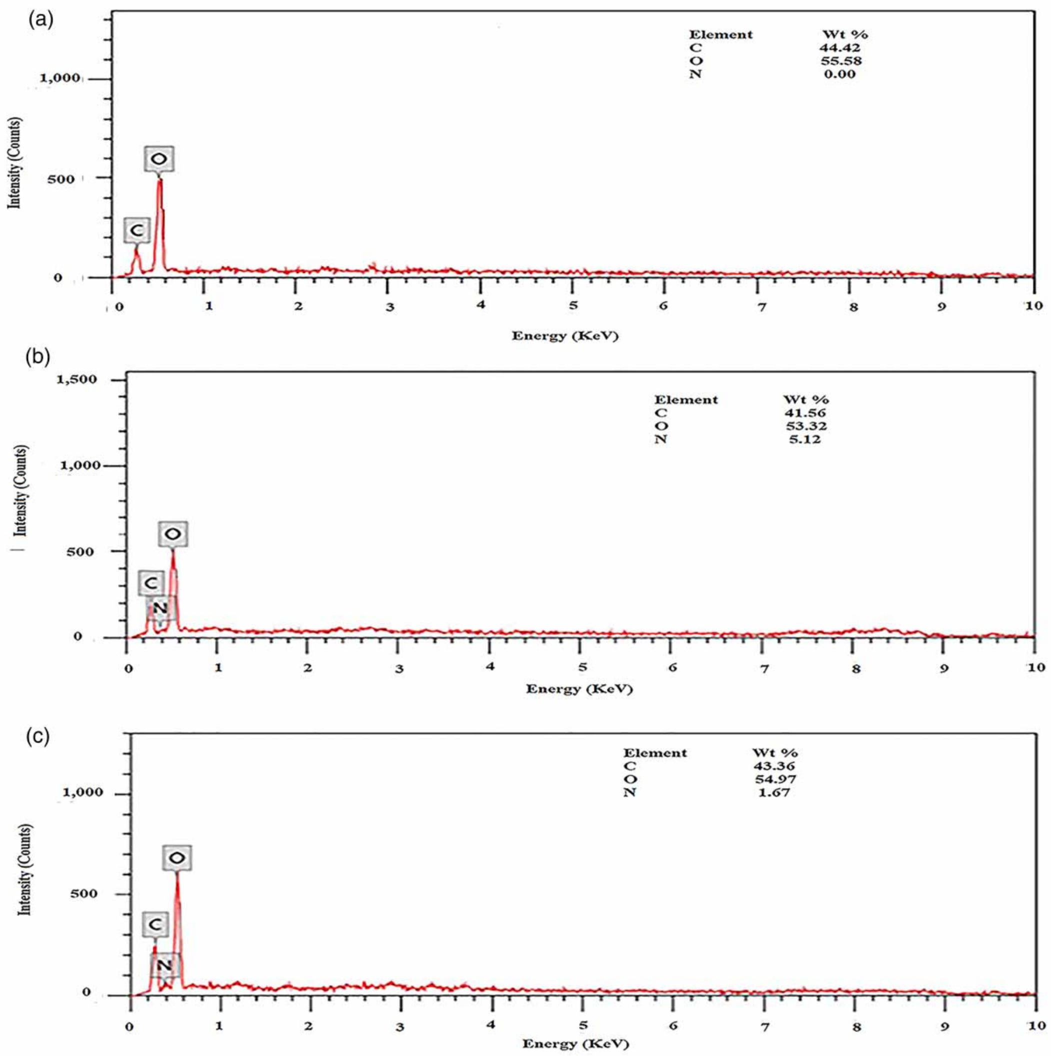


Figure 3 | EDX analysis of the surface of pristine cotton fabric (a), modified cotton fabric with PDA (b), and modified cotton fabric with PDA and PA (c).

band corresponding to the hydroxyl groups is still appeared at 3,320 cm^{-1}, this indicating that palmitic acid is successfully assembled onto the modified cotton fabric with polydopamine.

Wettability of the pristine and modified cotton fabric

It was observed that each of pristine and modified cotton fabric with polydopamine did not show any contact angle with water droplets, due to the superhydrophilic surface behavior, but after immersion in an ethanolic solution of palmitic acid the water contact angle reaches 157^o. Due to the superhydrophilic and oleophilic properties of the cotton fabric, both water and oil can penetrate through it. However, owing to the superhydrophobic/oleophilic properties of the as-prepared cotton fabric, oil can penetrate through it but the water was held above, thus realizing oil/water separation.

Oil absorption capacity

The oil absorption capacity for n-hexane, petroleum ether, and silicone oil was measured. Although both water and oil were absorbed by the pristine, material which has hydrophilic/oleophilic characters, only oil was selectively absorbed by the as-prepared superhydrophobic/oleophilic modified cotton fabric. The fast absorption of organic solvents to the as-prepared modified cotton fabric is attributed to the combination of its low density, high porosity, being superhydrophobic/oleophilic, and capillary force

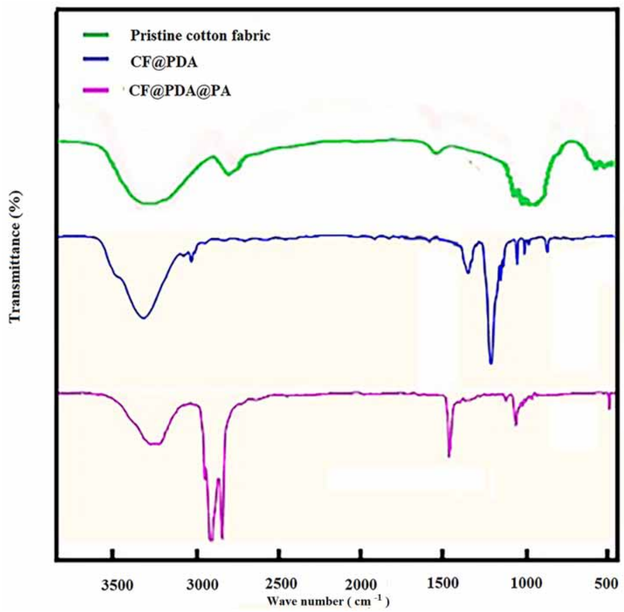


Figure 4 | FT-IR spectra of pristine cotton, modified cotton fabric with PDA, and modified cotton fabric with PDA and PA.

(Wang *et al.* 2017). The absorption and desorption processes were repeated 10 times. After each cycle, oil absorption capacity was measured. As shown in Figure 5, silicone oil has the highest absorption capacity while n-hexane has the lowest value. This result may be attributed to the difference in densities of these organic solvents, the solvent of higher density exhibits greater absorption capacity. The absorption capacity is nearly constant even after ten cycles, indicating the efficient recyclability of the prepared superhydrophobic cotton fabric for oil separation. The superhydrophobicity of the cotton fabric sample was not obviously destroyed even after 10 cycles, which was confirmed by contact angle measurements, CA $= 157 \pm 2.6$. The as-prepared superhydrophobic/oleophilic cotton fabric exhibits high absorption capacity toward the three examined organic solvents as large as 6.1, 6.3, and 8.6 g/g for n-hexane, petroleum ether and silicone oil respectively, which is much larger than the pristine one (1.1, 1.3, and 1.5 g/g for n-hexane, petroleum ether and silicone oil respectively). Additionally, in comparison, these absorption capacities (wt/wt) are superior to those of reported absorbents, such as commercial polyester textile modified with silicon (2.92 g/g) (Zhang & Seeger 2011), a nano-fibrous porous non-woven mat of polyvinylidene fluoride (PVDF) is coated on the cellulosic substrate (maximum value of 4.1 g/g) and after modification with silicone (maximum value of 8.6 g/g) (Gore *et al.* 2016), and a textile was coated by a diamond-like carbon film (maximum value of 5.1 g/g) (Cortese *et al.* 2014).

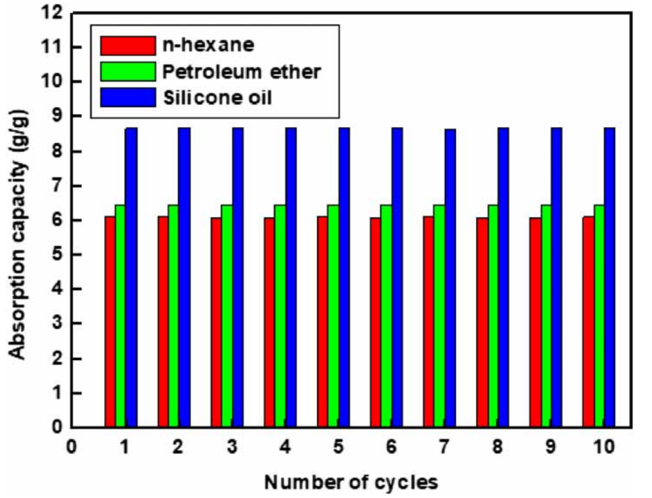


Figure 5 | Absorption capacity for different oil/water mixtures with the number of cycles.

Oil/water separation performance

The separation efficiency results are shown in Figure 6. The superhydrophobic cotton fabric shows high separation efficiency of different oil/water mixtures. The separation efficiency is maximum for n-hexane and minimum for silicone oil. So the superhydrophobic/oleophilic cotton fabric shows high selectivity for different oils.

Figure 7 shows the separation efficiency of modified cotton fabric for n-hexane for ten cycles. The figure shows that the separation efficiency in the first cycle is the highest. After ten cycles of oil/water separation, the separation efficiency was still higher than 95%. In the separation process, the small loss of the collected oil is mainly caused by the oil adhesion to the funnel and the sample. Besides,

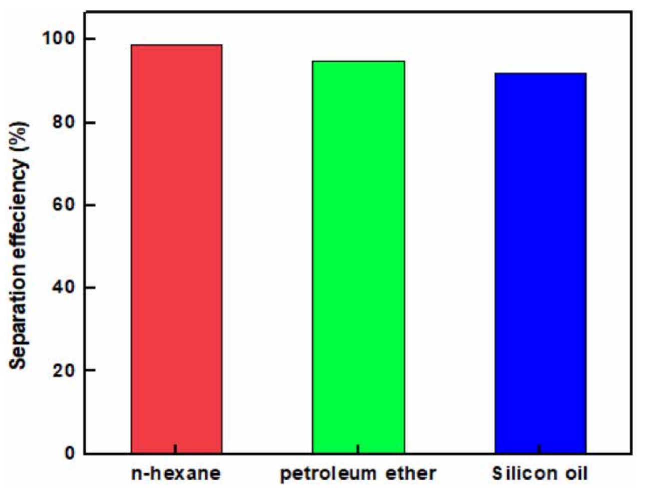


Figure 6 | Results of separation efficiency for different oil/water mixtures.

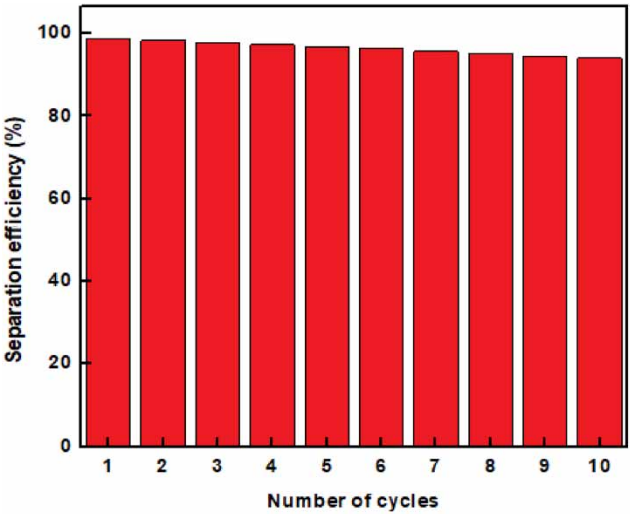


Figure 7 | Separation efficiency for n-hexane/water mixtures with number of separation cycles.

the volatility of the oil should also contribute to the reduction of the separation efficiency (Xu *et al.* 2019).

The oil permeates through the modified cotton fabric due to its porosity and the rough structures make it easy to form oil channels, which contribute to a high oil flux. The flux of the modified cotton fabric was calculated by using Equation (3). As shown in Figure 8, the superhydrophobic cotton fabric exhibited a high oil flux rate; n-hexane has the maximum flux value while silicone oil has the minimum value. The difference in flux values of these organic solvents is due to the difference in the viscosities for these organic solvents; the flux is inversely proportional to the liquid viscosity (Zhang *et al.* 2013). Therefore, the as-prepared

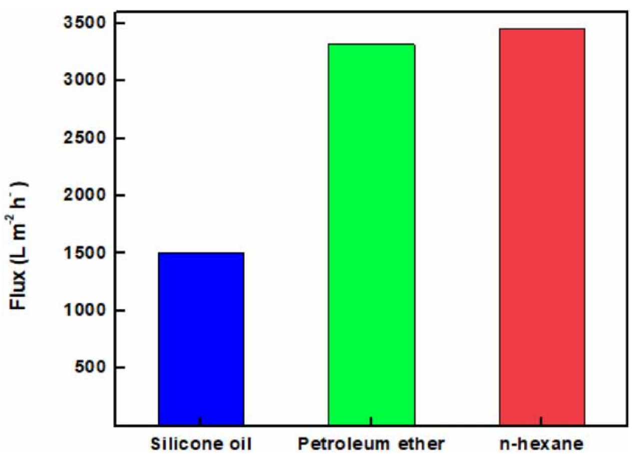


Figure 8 | Flux values for different oil/water mixtures.

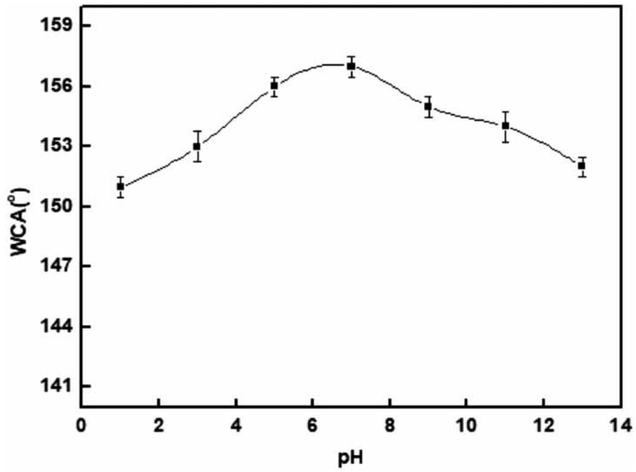


Figure 9 | Influence of pH values of water droplet on the contact angles of the superhydrophobic cotton fabric. Error bars represent standard deviation.

superhydrophobic cotton fabric can be used to separate oil and water effectively. This result indicates that the surface wettability enable the superhydrophobic cotton fabric to separate oil/water mixtures.

Chemical stability

It is well known that the stability of superhydrophobic surfaces is very important for their practical applications. In order to study the chemical stability of the as-prepared superhydrophobic cotton fabric, the impact of acid and alkali solutions on the water contact angle was studied. The superhydrophobic cotton fabric samples were immersed in aqueous solutions of pH from 1 to 13. The water contact angle was measured and the relation between contact angle and pH is shown in Figure 9. The water contact angle values is above 150° at all tested pH values, indicating that the superhydrophobic cotton fabric is still exhibiting superhydrophobic behavior.

The superhydrophobic cotton fabric also showed long term stability in n-hexane, petroleum ether and silicone oil in terms of superhydrophobicity (Figure 10). The water contact angle is above 150° after being kept in n-hexane, petroleum ether and silicone oil for 20 days. This can be attributed to the inherent stability of the polydopamine nanoparticles and the palmitic acid layer towards oils.

Mechanical stability

The sandpaper abrasion experiments were conducted to evaluate the mechanical resistance capability. The modified

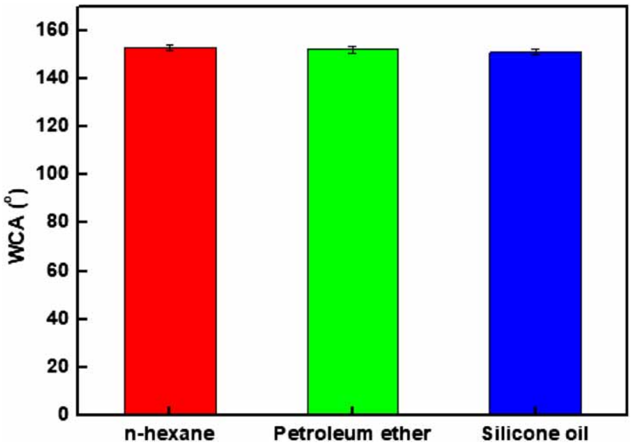


Figure 10 | Water contact angle after 20 days' immersion of superhydrophobic cotton fabric in different oil/water mixtures. Error bars represent standard deviation.

cotton fabric was dragged for 20 cm backwards and forwards as a cycle under a pressure of 8 k Pascal. Water contact angle was measured after every 20 abrasion cycles. The resultant surface still kept outstanding superhydrophobicity (WCA = 150.1°) even after 120 abrasion cycles.

CONCLUSION

1. A superhydrophobic/oleophilic cotton fabric has been fabricated by polymerization of dopamine followed by modification with low surface energy palmitic acid.
2. The polymerization process of dopamine generated a lot of polydopamine nanoparticles on the surface of cotton fabric, which greatly enhanced the surface roughness.
3. The immersion of modified cotton fabric by dopamine in ethanolic solution of palmitic acid efficiently reduced the surface energy and the cotton fabric exhibited a superhydrophobic character with a water contact angle of 157°, so oil could penetrate through the superhydrophobic cotton fabric but water was held above, thus realizing oil/water separation.
4. The as-prepared superhydrophobic cotton fabric shows excellent separation efficiency, 98.95 for n-hexane, 95.51 for petroleum ether and 94.50 for silicone oil.
5. The separation efficiency of modified cotton fabric for n-hexane after ten cycles was higher than 95%, indicating high recyclability of the manufactured superhydrophobic cotton fabric.
6. The as-prepared superhydrophobic cotton fabric shows excellent chemical and mechanical stability.

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DATA AVAILABILITY STATEMENT

All relevant data are included in the paper or its Supplementary Information.

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