



Facile fabrication of superhydrophobic filtration fabric with honeycomb structures for the separation of water and oil

Kunquan Li, Xingrong Zeng*, Hongqiang Li, Xuejun Lai, Hu Xie

College of Materials Science and Engineering, South China University of Technology, No. 381, Wushan Road, Tianhe District, Guangzhou 510640, China

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ABSTRACT

The superhydrophobic filtration fabric (SFF), with a water contact angle (WCA) higher than 150°, was prepared by a facile dip-coating method through decorating the surface with poly(dimethyl siloxanes) (PDMS)/silica (SiO₂) composite coating. To improve the roughness of the surface, toluene and ethanol was used as co-solvent to induce microphase separation. When the mass ratio of ethanol to toluene was 0.6, the SFF showed a high WCA of 152° with some porous honeycomb structures distributed on the surface. Moreover, the as-prepared SFF showed both superhydrophobicity and superoleophilicity, and could effectively separate the mixture of water and oil. Based on the study, the separation mechanism was also proposed.

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1. Introduction

Due to the frequent oil spill accidents and the increase of oily wastewater in industrial production, water/oil separation has become one of the most important issues in environment protection. Recently, surfaces with hydrophobicity and oleophilicity are considered as promising candidates for water/oil separation [1–3]. The surfaces with special wettability are always achieved by decorating the surfaces with low surface energy materials and hierarchical roughness [4,5]. Various functional membrane materials that can be utilized to effectively separate water and oil mixture have been developed, such as stainless-steel mesh [6], ceramic microfiltration membranes [7], filter paper [8], and nanofibrous membranes [9]. Jiang, et al. [1] fabricated a novel superhydrophobic/superoleophilic film through coating a stainless-steel mesh with fluorinated polymer, and found that this functional mesh could be used for the separation of water and oil. Shang, et al. [10] adopted electrospinning technology to prepare a nanofibrous membrane, and the membrane showed superhydrophobicity and superoleophilicity after modifying with fluorinated polybenzoxazine and silica nanoparticles, which endowed the membrane with good performance in water/oil separation. To improve the separation efficiency, a silicone-modified hierarchically porous monolith was synthesized via a sol-gel and phase separation process, and was applied in cleaning oil away from water [11].

Although lots of methods have been reported to fabricate hydrophobic surface for the separation of water and oil, those approaches

are always involved in expensive materials, special equipments, complicated and time-consuming process. Meanwhile, the disadvantages of these coatings, such as the poor mechanical stability of filter paper and weak flexibility of ceramic membranes, have limited their applications in practice. Moreover, the mechanism of separation process is unclear, and needs to be improved [12].

Herein, a simple and facile way for fabricating superhydrophobic filtration fabric (SFF) was presented through dip-coating in the mixture of poly(dimethyl siloxanes) (PDMS) and SiO₂ toluene dispersion. The SFF, made from polyester fiber, was thought to be have good mechanical stability and flexibility [13]. To further increase the roughness on the surface, the mixture of toluene and ethanol was used as co-solvent to induce microphase separation. The condensation reaction between hydroxyl-terminated poly(dimethyl siloxane) (H-PDMS) and butyl titanate (TBOT) was confirmed by Fourier transform infrared spectroscopy (FTIR). The wetting behavior and surface morphology were studied by contact angle analyzer and scanning electron microscope (SEM), respectively. Finally, the mechanism of separation was proposed.

2. Experimental

Materials: Hydroxyl-terminated poly(dimethyl siloxane) (H-PDMS) (Sylgard 107) was obtained from Jiangxi Xinghuo Organic Silicone Plant (China). Butyl titanate (TBOT) was purchased from Tianjin Kemiou Chemical Reagent Company (China). Dibutyltin dilaurate (DBTDL) was got from Shanghai Reagent Factory (China). Toluene and ethanol were acquired from Guangzhou Chemical Reagent Factory (China). Nano-SiO₂ toluene dispersion (CR-23-MB) was provided by Zhangjiagang Churen New

* Corresponding author. Tel./fax: +86 20 87114248.
E-mail address: psxrzeng@gmail.com (X. Zeng).

Material Technology Company (China). Filtration fabric (polyester textiles, 200 mesh) was gained from Guangzhou Qianhui Bose Instrument Company (China). All of the reagents were used as received.

Fabrication of superhydrophobic filtration fabric: First, 0.5 g H-PDMS, 0.01 g catalysis of DBTDL and 0.1 g curing agent of TBOT were added into the co-solvent of 10 g toluene and a certain amount of ethanol, and the mixture was allowed to magnetic stirred for 0.5 h at room temperature. After that, 4 g SiO₂ toluene dispersion was mixed into the above mixture with further stirred for another 0.5 h. Then, the filtration fabric, washed with aqueous alcohol, was immersed into the PDMS/SiO₂ mixture and the SFF was fabricated by dip-coating. Finally, the SFF was dried at 80 °C for 1 h to promote the crosslinking of H-PDMS and TBOT.

Characterization: Fourier transform infrared spectroscopy (FTIR) spectra were recorded on a Bruker Tensor 27 spectrometer (Bruker Optics, Germany) with a spectral resolution of 4 cm⁻¹. Per spectrum was scanned for 16 times in the range from 4000 to 400 cm⁻¹. Samples were coated on the KBr pellets and dried before testing. The microscopic investigation was carried out using scanning electron microscopy (SEM, FEI NOVA NANOSEM 430,

Netherlands). The water contact angle (WCA, 5 μL water) was acquired on a contact angle analyzer (DSA100, Germany). The WCA was a mean value measured from five places of the sample. The digital photos were taken by SLR camera (NIKON D90, Japan).

3. Results and discussion

To confirm the condensation reaction of H-PDMS and TBOT shown in Fig. 1(a), the FTIR spectra of H-PDMS and PDMS was presented in Fig. 1(b). From the spectra of H-PDMS, the peaks at 3463 cm⁻¹ and 1100 cm⁻¹ were attributed to the vibration of Si-OH and Si-O from H-PDMS, respectively. The bands located at 1259 cm⁻¹ assigned to the symmetric deformation of the -CH₃ group in -Si(CH₃)₂, and the peaks near 871 cm⁻¹ and 800 cm⁻¹ were due to the vibration of Si-C and Si-O [14], respectively. Compared with the spectra of H-PDMS, the peak near 3463 cm⁻¹ in the spectra of PDMS became weak. Moreover, the characteristic peak of Ti-O-Si near 942 cm⁻¹ appeared [15], indicating that the condensation reaction between -SiOH of H-PDMS and -TiOC₄H₉ of TBOT occurred.

The surface wettability and morphology of the filtration fabric (FF) before and after treatment with PDMS/SiO₂ coating were shown in Fig. 2. In Fig. 2(a), the uncoated FF with porous diameter of about 80 μm was composed by fibrous stripes, and its WCA was 71°. In Fig. 2(a₁), it seemed that the surface of the fibrous stripes was smooth, and no obvious lumps and protrusions could be observed. After covering with PDMS/SiO₂ coating without ethanol as solvent, some of the protrusions with micron-sized appeared on the surface of FF, and the wetting behavior of coated FF had changed from hydrophilic to hydrophobic with a WCA of 128°, which was shown in Fig. 2(b) and (b₁). For the addition of ethanol to the mass ratio of 0.6, in Fig. 2(c) and (c₁), the surface of fibrous stripes became rather rough with some honeycomb micro-scaled structures distributed on it, which was caused by the microphase separation of PDMS in toluene and ethanol and the uneven dispersion of SiO₂ particles in PDMS. Meanwhile, the WCA increased to 152°. When the mass ratio increased to 1.2, the WCA dropped to 142°, the surface micro-morphology investigation, shown in Fig. 2(d) and (d₁), revealed that most of the honeycomb structures on the surface had been destroyed, owing to the diluting effects of excess ethanol.

The separation process of water and oil was shown in Fig. 3. In Fig. 3(a), it was obvious that the SFF showed both superhydrophobicity

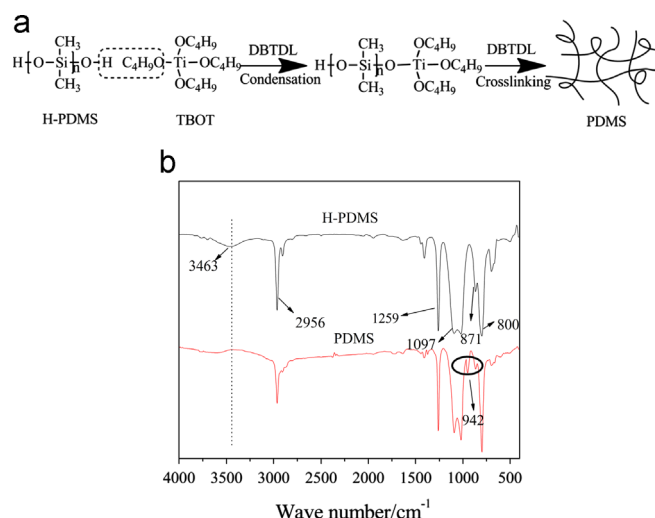


Fig. 1. Schematic of condensation reaction (a), and FTIR spectra (b) of H-PDMS and PDMS.

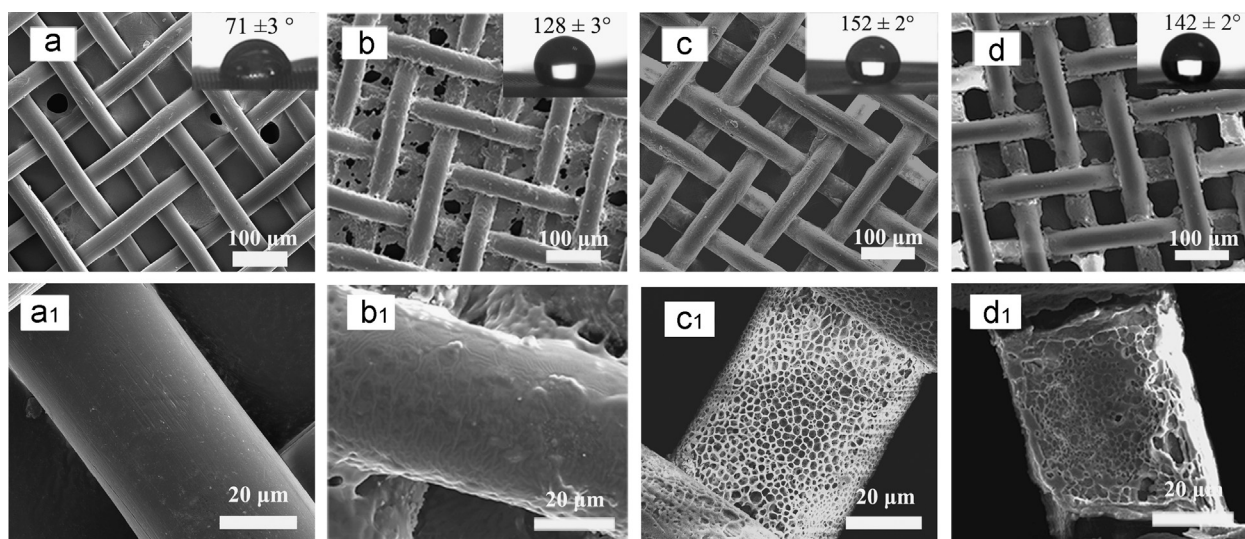


Fig. 2. SEM images and WCAs of filtration fabric before and after coating under different mass ratio of ethanol to toluene. ((a)-(a₁)): filtration fabric; ((b)-(b₁)): 0; ((c)-(c₁)): 0.6; ((d)-(d₁)): 1.2.

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