

Facile fabrication of underwater superoleophobic TiO_2 coated mesh for highly efficient oil/water separation



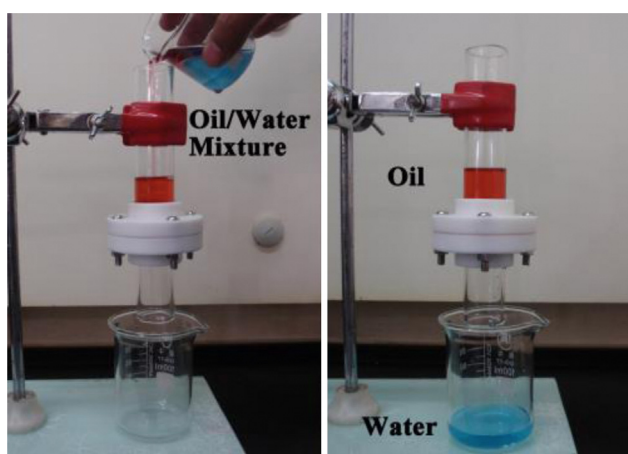
Jian Li^{*}, Long Yan, Wenfang Hu, Dianming Li, Fei Zha, Ziqiang Lei

Key Laboratory of Eco-Environment-Related Polymer Materials, Ministry of Education of China, Key Laboratory of Gansu Polymer Materials, College of Chemistry and Chemical Engineering, Northwest Normal University, Lanzhou 730070, China

HIGHLIGHTS

- Underwater superoleophobic TiO_2 mesh was fabricated by a facile spray-coating process.
- The mesh was used to separate a series of oil from water with the separation efficiency up to 99.0%.
- The mesh can be reused more than 40 times with the separation efficiency still above 97.5%.

GRAPHICAL ABSTRACT



The underwater superoleophobic TiO_2 coated meshes can separate oil from water with the separation efficiency up to 99.0%.

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ABSTRACT

The removal of oil and organic pollutants from water is highly desired because of the frequent oil spill accidents and increase of industrial oily wastewater. Here, underwater superoleophobic TiO_2 coated mesh was fabricated through spraying TiO_2 nanoparticles and polyurethane mixtures onto stainless steel mesh. The underwater superoleophobic TiO_2 coated mesh was then used to separate water from oil/water mixtures, where only the water permeates through the mesh while the oil is repelled on the mesh. In addition, the separation efficiency of the coated mesh is as high as 99.0%. Furthermore, the TiO_2 coated mesh still maintained high separation efficiency over 97.5% and stable recyclability after 40 separation cycles. Thus, such superhydrophilic and underwater superoleophobic TiO_2 coated mesh is a very potential material for the application of oil spill cleanup and industrial oily wastewater remediation.

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1. Introduction

With the development of industry and social economy, oil spillage and industrial discharge of organic solvents become more

^{*} Corresponding author. Fax: +86 931 7971989.
E-mail address: lijian@lcp.cas.cn (J. Li).

and more serious, which have caused severe environmental and ecological damage [1]. The environmental and economic highly need for functional materials that can separate water/oil mixtures efficiently. Because oil/water separation is an interfacial phenomenon, using special wettability to design novel membranes is a facile and effective way [2–5]. To utilize the special wettability to separate oil/water mixtures, the used membranes commonly possess an opposite wettability to water and oil. Jiang et al. first reported a PTFE coated mesh with superhydrophobicity and superoleophilicity for separation of oil and water [6]. From then on, the films with both superhydrophobic and superoleophilic properties (termed as “oil-removing” types of films) have been extensively investigated and used to remove oils from water effectively [7–18]. As water usually has a higher density than oils, it tends to form a barrier layer to prevent oil permeation. In addition, these “oil-removing” films are easily fouled or even blocked up by adsorbed oils, which results in the quick decrease of flux and separation efficiency as well as secondary pollution. From a practical point of view, superoleophobic and superhydrophilic films may be more suitable for effective oily wastewater separation and long-term use; however oleophobic surfaces are often hydrophobic because of the water surface tension being higher than that of oil in air [19–21]. Recently, the oil-repellent ability of fish scales in aqueous media has attracted increasing interest [22]. Inspired by fish scales, films with hydrophilic and underwater superoleophobic properties have been developed as “water-removing” types of materials, rendering an alternative route to address some of the aforementioned issues in oil/water separation [23–35]. Hydrogen and polymer materials were broadly applied to achieve superhydrophilic and underwater superoleophobic properties, and the “water-removing” method has been developed to separate oils from oil/water mixtures effectively [36–43]. However, the main obstacle of these hydrogen and polymeric materials are their weak environmental adaptability, due to their characteristic of swelling in water and/or metamorphosis of the polymeric membrane under long-term scouring by water. In addition, restrictions in scalable fabrications hindered their further applications. Therefore, it is of great significance to develop functional materials with stable underwater superoleophobicity for oil/water separation in a simple, economical and scalable approach.

Herein, underwater superoleophobic TiO_2 coated meshes were fabricated by spraying TiO_2 nanoparticles (NPs) and waterborne polyurethane (PU) mixtures on stainless steel mesh. The spray-coating method is a fairly facile, low-cost and commercially available process, which is not specific to a particular substrate and can be easily applied to large surface area [44,45]. In addition, the TiO_2 coated mesh shows an excellent water affinity and strong underwater superoleophobicity. Since water permeated through the mesh quickly and oil remained exclusively on the coated mesh surface, thus it could be used for the gravity-driven oil/water separation experiments and showed 99.0% water/oil separation efficiency. Furthermore, the coated mesh maintained high separation efficiency and stable recyclability after 40 separation cycles with the surface morphology of the TiO_2 coated mesh nearly unchanged. This work provides an effective strategy for oil/water separation and would be suitable for future practical treatment of oily wastewater.

2. Experimental

2.1. Materials

TiO_2 nanoparticles (NPs) and waterborne polyurethane (PU) were purchased from Sinopharm Chemical Reagent Co., Ltd. and used without further purification. The stainless steel mesh sub-

strates (300 mesh size) was precleaned sequentially with acetone and ethanol before use.

2.2. Preparation of underwater superoleophobic mesh

In a typical process, the TiO_2 NPs (0.3 g) and 20 ml acetone were placed into a round-bottomed flask. After adding 0.05 g waterborne polyurethane (PU), they were stirred for at least 1 h and the resulting were sprayed onto the stainless steel mesh substrate with 0.2 MPa compressed air gas using a spray gun. The spray-coating was repeated several times and the TiO_2 coated mesh was dried at ambient temperature for 1 h to allow the acetone to evaporate completely.

2.3. Oil/water separation

A series of studies were operated to test the oil/water separation capacities of the as-prepared mesh. Some oils and organic solvents (including kerosene, hexane, petroleum ether, toluene and rape-seed oil) were mixed with water respectively, which indicated the mesh to be suitable for separating diversified oil/water mixtures with high stability and separation efficiency. Beforehand the oils and organic solvents were colored with oil red O and the water was colored with methylene blue in order to clearly distinguish. The underwater superoleophobic TiO_2 coated mesh was completely wetted by water and tightly fixed with a special device. Then mixtures of oil and water (50% v/v) were poured slowly into a test tube. It could be seen that water immediately pass through the TiO_2 coated mesh while the oil was repellent on the mesh. The driving force during the separation process is its own gravity. The separation efficiency was calculated according to $\eta = (m_1/m_0) \times 100$, where m_0 and m_1 were the mass of the oil before and after separation process, respectively [46].

2.4. Characterization

The SEM images were obtained by field emission scanning electron microscopy (FE-SEM, Zeiss). The crystal phase of the samples were confirmed by an X-ray diffractometer (XRD) (Rigaku Corp., D/max-2400) equipped with graphite monochromatized $\text{Cu K}\alpha$ radiation. The water and oil contact angle (CA) and sliding angle (SA) were measured on SL200KB apparatus at ambient temperature. The volume of the individual water and oil droplets in all measurements was 5 μL . The average value of five measurements acquired at different positions on the same sample was adopted as the oil contact and oil sliding values.

3. Results and discussion

In this work, we selected TiO_2 NPs for the fabrication of superhydrophilic surface, because this material is commercial available and inexpensive, in addition to exhibiting remarkable superhydrophilicity. The waterborne PU was added in order to increase the binding force between the TiO_2 NPs and stainless steel mesh. XRD analysis of the TiO_2 NPs, shown in Fig. 1, reveals that all diffraction peaks can be assigned the anatase structure of TiO_2 (JCPDS No: 84–1286), which was produced as a product.

The surface morphologies of the original and the coated meshes were characterized by FE-SEM. As shown in Fig. 2a, the original mesh has an average pore diameter of about 50 μm (300 mesh size), and the magnified view in the inset of Fig. 2a reveals that the original wires have smooth surface. After coated with the mixture of TiO_2 NPs and PU, it can be seen that the original mesh has been densely covered by randomly distributed TiO_2 NPs with the microscale (Fig. 2b and c). The high magnified FE-SEM image of Fig. 2d shows that the TiO_2 NPs are relatively aggregated together

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