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Superhydrophobic Micro/Nanostructured Copper Mesh with Self-Cleaning Property for Effective Oil/Water Separation

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(Dated: Received on October 22, 2018; Accepted on December 18, 2018)

In this work, a simple method was carried out to successfully fabricate superoleophilic and superhydrophobic *N*-dodecyltrimethoxysilane@tungsten trioxide coated copper mesh. The as-fabricated copper mesh displayed prominent superoleophilicity and superhydrophobicity with a huge water contact angle about 154.39° and oil contact angle near 0°. Moreover, the coated copper mesh showed high separation efficiency approximately 99.3%, and huge water flux about 9962.3 $L\cdot h^{-1}\cdot m^{-2}$, which could be used to separate various organic solvents/water mixtures. Furthermore, the coated copper mesh showed favorable stability that the separation efficiency remained above 90% after 10 separation cycles. Benefiting from the excellent photocatalytic degradation ability of tungsten trioxide, the coated copper mesh possessed the self-cleaning capacity. Therefore, the mesh contaminated with lubricating oil could regain superhydrophobic property, and this property of self-cleaning permitted that the fabricated copper mesh could be repeatedly used for oil and water separation.

Key words: Superhydrophobicity, Micro/nanostructure, Tungsten trioxide, Self-cleaning, Oil/water separation

I. INTRODUCTION

Nowadays, more and more attentions have been focused on environmental problems, and oily wastewater is identified as one of the important pollution source all over the world. With the flourishing development of industrialization, lots of industrial processes almost discharged huge organic solvents (such as benzene, toluene, cyclohexane, dichloromethane, and so on) into water and polluted precious water sources. This arbitrary discharge of oily liquid waste and continual occurrence of oil spillages have caused severe environmental pollution and ecological imbalance [1−3]. So how to solve these oily wastewater in an efficiency and environmental friendly way already has become a global challenge. Traditional technologies have been used to treat oily wastewater such as centrifugation, gravity, air floatation, chemical, microbiological methods, and so on [4−8]. However, these technologies also have their own drawbacks and limitations such as low energy efficiency, high energy consumption, using toxic chemical compounds, secondary pollution and large space for installation, and are thus not very feasible for large-scale application [2, 9]. To search a new and efficiency way to selective separately oily wastewater has got more and more attention all over the world.

At present, series of special wettability materials possessing selective permeability to oil and water have been widely used in oil/water separation. In general, selective permeability materials can be summarized into three categories: "water-removing" type materials with superhydrophilicity and superoleophobicity which can selectively separate water from oil and water mixtures, "oil-removing" type materials with superhydrophobicity and superoleophilicity which can selectively filter or absorb oil from oil and water mixtures, and smart responsive separation materials [10−13]. In recent years, because superhydrophobic and superoleophilic surface has the ability to resist contamination, self-cleaning and excellent oil absorption, these materials are widely used in our daily life and production. Inspired by the natural superhydrophobic surfaces such as butterfly wings, lotus leaves, water strider, gecko feet, and rose petals, we could know that the facile combination of micro/nano hierarchical structures with hydrophobic materials or groups is indispensable to realize the superhydrophobicity of membranes surface [14, 15]. Hence, an advisable mesh with superhydrophobic and superolephilic through surface modification was widely used to oil/water separation. For example, Zhang *et al.* fabricated a transparent film by spraying SiO_2 suspension

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DOI:10.1063/1674-0068/cjcp1810233

on any substrate virtually and then drying at ambient environment, the obtained material demonstrated superhydrophobicity and superoleophilicity and showed oil or organic solvent uptake ability [16]. Turco *et al.* fabricated a porous magnetic reusable nanocomposite integrating PDMS sponges with multi-walled CNTs (PDMS-MWNTs) via a facile hard template approach that does not require the use of complex instrumentation [17]. Even though the superhydrophobic-superoleophilic materials could be high-efficiency and massively to selectively separate oil-water mixtures, superoleophilic membranes often fouled by all kinds of organic solvents which restricted this materials application greatly. In practical application, the separating mesh was inevitably used in recurrent oil/water separation and frequent contacted a variety of organic solvents, and correspondingly the flux and separation efficiency of the contaminated mesh would obviously decline.

As for the superoleophilic membranes often fouled by all kinds of organic solvents, lots of correlative scientists attempt to introduce semiconductor light sensitive materials to restore the fouled meshes. Kang *et al.* fabricated nanostructured TiO_2 mesh membrane through a simple electrochemical anodization and heating process, this mesh demonstrated unique self-cleaning and anti-fouling ability which could be used for effective oil/water separation [18]. In this work, we fabricated *N*-dodecyltrimethoxysilane (DTMS)@tungsten trioxide superhydrophobic surface on copper mesh with a simple hydrothermal method (FIG. 1). Success fabricated DTMS@WO_3 copper mesh was ascertained by scanning electron microscopy (SEM), Fourier transform infrared (FT-IR) spectra, X-ray diffraction (XRD), X-ray photoelectron spectroscopy (XPS), and static contact angle measurements. And from the characterization results we knew that this coated copper mesh had micro pores of near 40 μm, on which the tungsten trioxide similar to echinopsis tubiflora grew on the smooth surface of pristine copper mesh. This mesh showed excellent superhydrophobicity with water contact angle (WCA) being above 150° and superoleophilicity with oil contact angle (OCA) of 0°. In consequence, oil could rapidly spread and penetrate this mesh when oil droplets contact the mesh surface, while water was restricted on the surface of the mesh. The as-fabricated DTMS@WO_3 copper mesh exhibited high separation efficiency, good stability, and possessed excellent self-cleaning property towards polluted copper mesh under UV illumination environment. In a word, our findings expounded a simple, fast, environmental friendly, and low-cost strategy to fabricate highly durable and self-cleaning superhydrophobic copper mesh, which could be applied to effective oil/water separation.

II. EXPERIMENTS

A. Materials

Copper meshes (99.9%, 400 meshes) were purchased from Xiancai Metallic Net Co., Ltd. (Anping, China). $NaWO_3 \cdot 2H_2O$, Na_2SO_4, and $H_2C_2O_4$ were purchased from Guangzhou Chemical Company, China. DTMS, oil red, dichloromethane (AR), tetrachloromethane (AR), methylene blue (MB), and hydrochloric acid (HCl) were obtained from Aladdin (Shanghai, China). Ethanol (99.5%, Shanghai Chemical Reagent Co., Ltd) was analytical grade. All chemical reagents were used without further purification.

B. Fabrication of tungsten trioxide coated copper mesh

The original copper meshes were cut into 3 cm×3 cm piece and sequentially rinsed with hydrochloric acid, ethanol, and deionized water. Then the cleaned copper meshes were dried in vacuum oven at about 60 °C. Next, 5 mmol $NaWO_3 \cdot 2H_2O$, 10 mmol Na_2SO_4, and 10 mmol $H_2C_2O_4$ were added into 50 mL of deionized water under drastic stirring for 30 min at environment temperature to synthesize transparent tungsten trioxide precursor solution, then 6 mL of 3 mol/L hydrochloric acid was added to the above solution under stirring for another 30 min. The prepared copper meshes were immersed in the transparent tungsten trioxide precursor solution, and then transferred to a high pressure reactor for 12 h at 180 °C. Afterwards, the coated mesh samples were removed from the high pressure reactor and cleared by deionized water and ethanol. Finally, the as-fabricated meshes were dried in oven at 70 °C for further experiment.

C. Fabrication of superhydrophobic coated copper mesh

The tungsten trioxide coated superhydrophobic copper meshes were fabricated with a simple dipping method. Typically, 3 mL DTMS was added into 50 mL anhydrous ethanol for homogeneous mixing. Then, the coated copper meshes were immersed into the mixture solution for about 4 h at room temperature. Finally, the meshes were dried at 60 °C for further characterization and experiment.

D. Instrumentation and characterization

The surface composition of the coated copper mesh was analyzed by an X-ray photoelectron spectroscopy (XPS). Scanning electron microscope (SEM, TESCAN MIRA3 LMU) was used to analyze the microstructures of coated copper mesh. The Fourier transform infrared (FT-IR) spectra were recorded to confirm the successful coating of DTMS and WO_3. The oil and water contact angle of the samples was measured through a JC 2000D1 apparatus (Zhongchen Digital Equipment Co.

DOI:10.1063/1674-0068/cjcp1810233

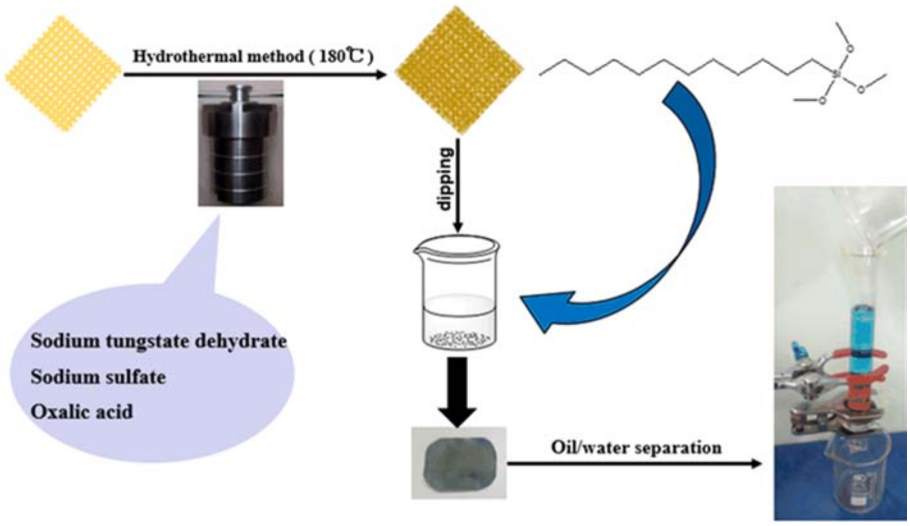

FIG. 1 Schematic description of the preparation for DTMS@WO_3 coated copper mesh.

Ltd., China) at room temperature. For the measurements of the contact angle, the volume of water and oil droplet were about 4 μL and selected at least five different surface positions, respectively. The crystal structure of the coated copper mesh was analyzed by X-ray powder diffraction (XRD, Bruker D8) using Cu Kα radiation.

E. Oil/water separation experiments

Organic solvents and water were colored by oil red and methylene blue for further experiment. Oil/water mixtures were prepared by mixing 10 mL organic solvents and 10 mL water for the separation performance experiments. Typically, the as-fabricated copper mesh was sandwiched in two glass tubes, and organic solvents/water mixtures were poured into the glass tubes. All the separation process experiments were driven only by gravity. The liquid flux F was calculated by the following equation [19, 20]:

$$F=\frac{V}{S\Delta t} \tag{1}$$

Here, V is the volume of the penetrant (L), t is the time of separation process (h), and S is the effective filtration area of the coated copper mesh (m^2).

The oil/water separation efficiency η was gained by the ratio of the volume of organic solvents collected in a beaker V_1 to that added initially V_0 using equation:

$$\eta=\frac{V_1}{V_0}\times 100\% \tag{2}$$

F. Self-cleaning property and stability of the coated copper mesh

To research the self-cleaning ability of the coated copper mesh, this mesh was fouled by the organic solvents (lubricating oil and so on) until the WCA of the coated mesh underwent a decline. Then, the fouled mesh was flushed by deionized water and ethanol, and dried in vacuum oven at 70 °C to measure the water contact angle. The fouled mesh was finally irradiated with a 500 W mercury light (YM-GHX-V, Yuming Instrument Co., Ltd., Shanghai, China). The regenerated copper mesh was measured for the water contact angle again and reused for the organic solvents/water separation experiments to assess the coated copper mesh reusability.

III. RESULTS AND DISCUSSION

A. Morphology of the coated copper mesh

The surface microstructure of pristine copper mesh and coated copper mesh were characterized by scanning electronic microscopy (SEM). FIG. 2 (a−d) revealed the SEM images of pristine copper mesh with different magnifications, the pristine copper mesh displayed a smooth network structure and the pore size was near 85 μm. As shown in FIG. 2 (e−h), the surface of the coated copper mesh was covered by a layer of tungsten trioxide nanowire arrays, as though lots of echinopsis tubiflora grew on the smooth surface of copper mesh. Meanwhile, the pore size of the coated copper mesh was approximately 40 μm, and the small pore size would restrict water to penetrate through the coated copper mesh. These micro/nanostructures similar to *echinopsis tubiflora* greatly increased the surface roughness of coated copper mesh, which is indispensable to prepare robust superhydrophobic separation materials.

DOI:10.1063/1674-0068/cjcp1810233

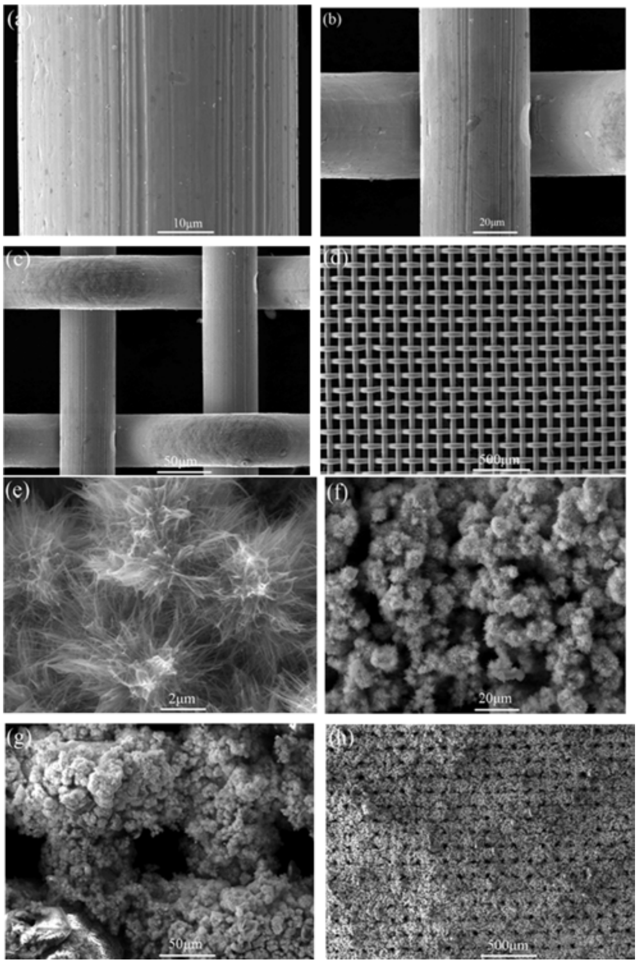

FIG. 2 (a−d) SEM images of pristine copper mesh. (e−h) SEM images of coated copper mesh.

B. Chemical composition of the coated copper mesh

As shown in FIG. 3, FT-IR and XRD measurements were used to distinguish the surface chemical composition of the pristine and coated copper mesh. The FT-IR spectra are shown in FIG. 3(a), the absorption peaks emerging at 600−1000 cm^{-1} were the characteristic vibration absorption peak of WO_3, which was caused by the stretched vibration of W−O−W bond in WO_3 and proved the tungsten trioxide successfully grew on the surface of copper mesh [21, 22]. The absorption peaks around 1690 cm^{-1} were probably created by hydroxyl stretching and bending vibration from water. The absorption peak at 1103 cm^{-1} was the characteristic peak of the Si−O bond, and the absorption peaks around 1307 cm^{-1} and 1375 cm^{-1} were interrelated with the stretching and bending vibrations of methyl groups, which verified the chemical functionalization of coated copper mesh [23]. These results confirmed that the copper mesh was successfully coated by tungsten trioxide and functionalized with DTMS. FIG. 3(b) shows the XRD patterns of the pristine and coated copper mesh.

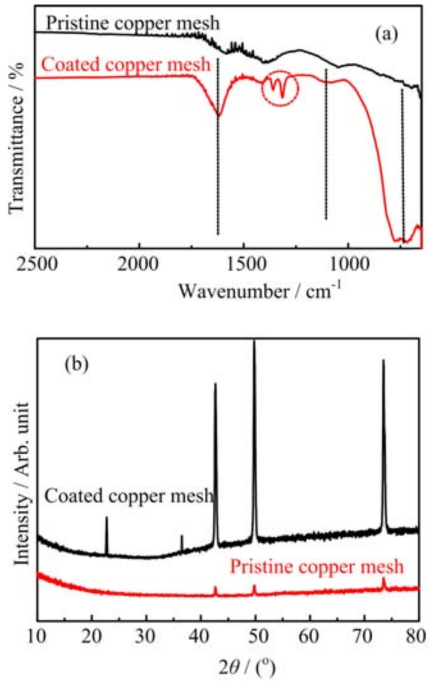

FIG. 3 (a) FT-IR spectra of pristine copper mesh and the coated copper mesh. (b) XRD patterns for pristine copper mesh and the coated copper mesh.

The peaks were highly conformed to the values of the standard card (JCPDS card No.85-2459). For WO_3, diffractions peaks with 2θ at 23.8° and 36.3° were observed, which indicated that the surface of the copper mesh was covered by tungsten trioxide crystal. XPS survey spectra of pristine and coated copper mesh are shown in FIG. 4(a). Cu, O and C elements were found in pristine and coated copper mesh, while Si and W elements only existed in coated copper mesh. The high-resolution spectra of W4f, Si2p, C1s, and O1s signals are shown in FIG. 4 (b−e), respectively. For coated copper mesh, the peaks at 35.8 and 37.0 eV were caused by the binding energy of WO_3 bonds, which demonstrated the former results of FT-IR [21]. According to the Table of signals from elements and common chemical species, from FIG. 4, we know that the binding energies at the peaks of 100.2 eV, 102.6 eV, and 100.4 eV belong Si−O and Si−C bonds, respectively. The binding energies at the peaks of 285.4 and 284.7 eV were attributed to C−O and C−C bonds. As for pristine copper mesh, the binding energies at 285 and 531.8 eV were arisen from C 1s and O 1s bonds [24]. In summary, W−O, Si−C, Si−O, C−O, and C−C bonds were also presented at the coated copper mesh, which adequately proved the WO_3 and DTMS were successfully grown and coated on the surface of copper mesh, and conformed with the conclusion obtained from FIG. 3.

DOI:10.1063/1674-0068/cjcp1810233

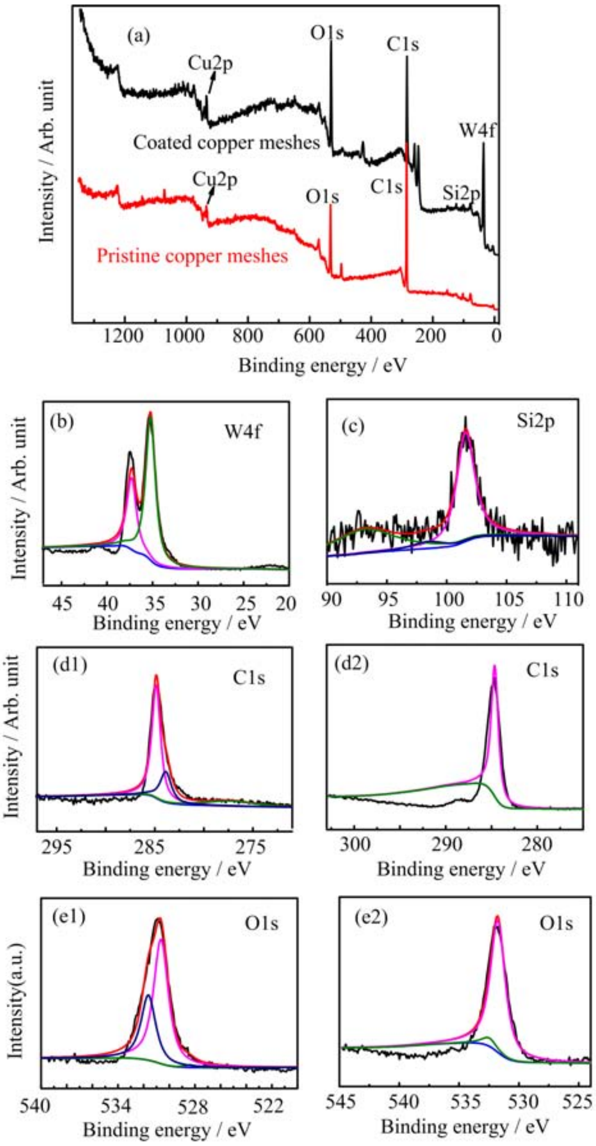

FIG. 4 (a) XPS survey spectra of pristine and coated copper mesh. (b) W 4f high-resolution spectra of coated copper mesh. (c) Si 2p high-resolution spectra of coated copper mesh. (d) and (e) show C 1s and O 1s high-resolution spectra of coated (d1, e1) and pristine (d2, e2) copper mesh.

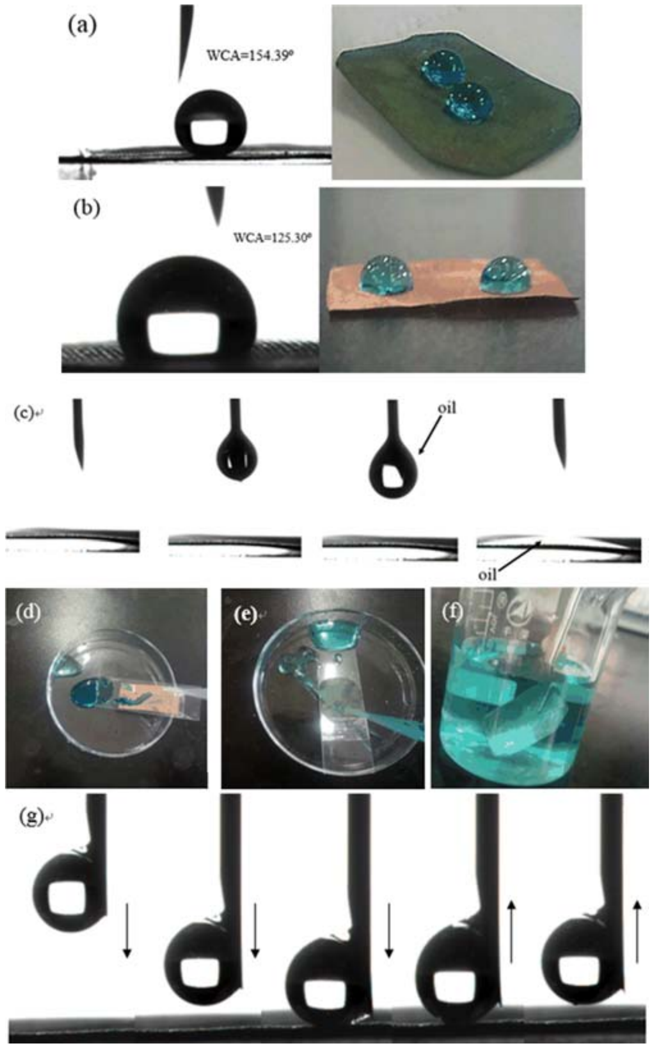

FIG. 5 Water and oil wettability of coated copper and pristine mesh. (a) Static WCA measurements for coated copper mesh. (b) Static WCA measurements for pristine mesh. (c) Procedure of oil contacted the coated copper mesh surface. (d) Photograph of a water column squirted on the pristine copper mesh. (e) Photograph of a water column squirted on coated copper mesh. (f) Photograph of the coated copper mesh immersed in the water. (g) Photographs of contact, and departure processes of a water droplet with respect to the coated copper mesh surface.

C. Wettability properties of the coated copper mesh

The separation materials must possess surface special wettability to satisfy the requirements of effective oil/water separation. In this work, the surface special wettability of pristine and coated copper mesh were researched by oil and water contact angle measurements. It can be seen from FIG. 5(a), the WCA of the coated copper mesh was 154.39°, which showed favorable superhydrophobic of the coated copper mesh. As for the pristine copper mesh, the WCA was 125.30° (FIG. 5(b)), but the ability of hydrophobic was unsatisfied with effective oil/water separation. Furthermore, the measurement of dynamic OCA was made to demonstrate the superoleophilicity of the coated copper mesh, and the measuring results are presented in FIG. 5(c). It could be found that the oil droplet was soaked into the coated copper mesh as soon as it contacted the surface of the coated copper mesh. FIG. 5(d) shows the photograph of a water column squirting on the pristine copper mesh, FIG. 5(e) and Movie S1 (see supplementary materials) showed the process of a water column squirting on the coated copper mesh, this further evidenced that our fabricated coated copper mesh had the ability of superhydrophobic compared with pristine copper

DOI:10.1063/1674-0068/cjcp1810233

mesh. Moreover, the coated copper mesh surface exhibited a clear silver mirror when the coated mesh was immersed into the water by an external force (FIG. 5(f) and Movie S2 in supplementary materials). This phenomenon was ascribed to the mesh being wrapped with a layer of air bubble and a composite solid-liquid-air interface. For this type of phenomenon, the WCA could be described by the Cassie-Baxter equation [25−27]:

$$\cos\theta' = f\cos\theta + f - 1 \quad (3)$$

Here, θ and θ' are the WCA with a water droplet on the smooth and rough substrates surface, f denotes the proportion fraction of coated copper mesh contacting with water. In our work, θ'=154.39°, and θ=125.30°. Hence, f was calculated to be 0.23, indicating that of the coated copper mesh surface, approximately 23% was in touch with water droplet and 77% was in touch with air. In addition, FIG. 5(g) revealed the inadhesion ability of the superhydrophobic coated copper mesh while a water droplet contacted the surface using a syringe needle. It was arresting that the water droplet could absolutely deviate from the surface of the coated mesh under an external force. Therefore, the fabricated superhydrophobic copper mesh could effectively separate oil from oil/water mixtures.

D. Oil/water separation behavior of superhydrophobic coated copper mesh

Because the coated copper mesh had super wettability of superhydrophobic and superoleophilic, it could be used for effective oil/water separation with the continual occurrence of oil spillages and increasing of oily sewage emission. The separation performance of DTMS@WO_3 copper mesh was measured using all kinds of water and organic solvents mixtures, which are shown in FIG. 6 and Movie S3 (see supplementary materials). It could be clearly seen from Movie 3 (supplementary materials), a certain volume mixture of water and organic solvent (dichloromethane) was poured directly into the separating unit, the oil immediately infiltrated through the coated copper mesh, while water was inhibited on the surface of coated mesh. The volume of water after separation was almost no less than that of the pristine, and nearly no water could be observed from the filtered oil phase, which further indicated the obtained DTMS@WO_3 copper mesh could effectively separate oil and water mixtures. FIG. 6(a) shows the separation efficiency for different water and organic solvents mixtures, the separation efficiencies of the coated copper mesh were more than 99% for all kinds of organic solvents/water mixtures. The liquid flux was also used to investigate the oil and water separation property of coated copper mesh (FIG. 6(b)). The tetrachloromethane/water mixtures possessed maximum flux of up to (9962.3±195.15) $L\cdot h^{-1}\cdot m^{-2}$, while the minimum flux was (7657.1±208.03) $L\cdot h^{-1}\cdot m^{-2}$ for

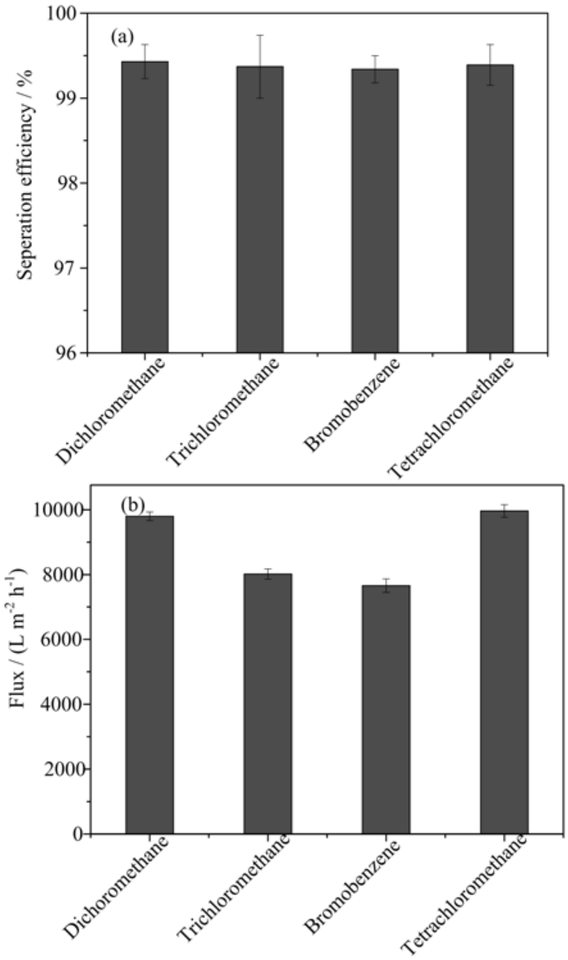

FIG. 6 Experiments of oil/water separation. (a) Separation efficiency of various heavy oil/water mixtures through coated copper mesh. (b) Flux of various oil/water mixtures through coated copper mesh.

trichloromethane/water mixture. In summary, the DTMS@WO_3 copper mesh has outstanding separation efficiency for all kinds of heavy oil and famous flux of organic solvents/water mixtures separation, which showed enormous potential to apply for industrial oil/water separation.

E. Stability and self-cleaning property performances of coated copper mesh

To further investigate the recyclability and stability of the coated copper mesh, a series of experiments were designed, the results are shown in FIG. 7. A water droplet was dripped on the coated copper mesh (FIG. 7(a)), while the WCA almost was no change after 80 min and the volume of the water droplet was diminished only because of the evaporation of water. Then, the dichloromethane/water mixture was used to reveal the oil and water separation efficiency of the coated copper mesh. As shown in FIG. 7(b), the separation effi-

DOI:10.1063/1674-0068/cjcp1810233

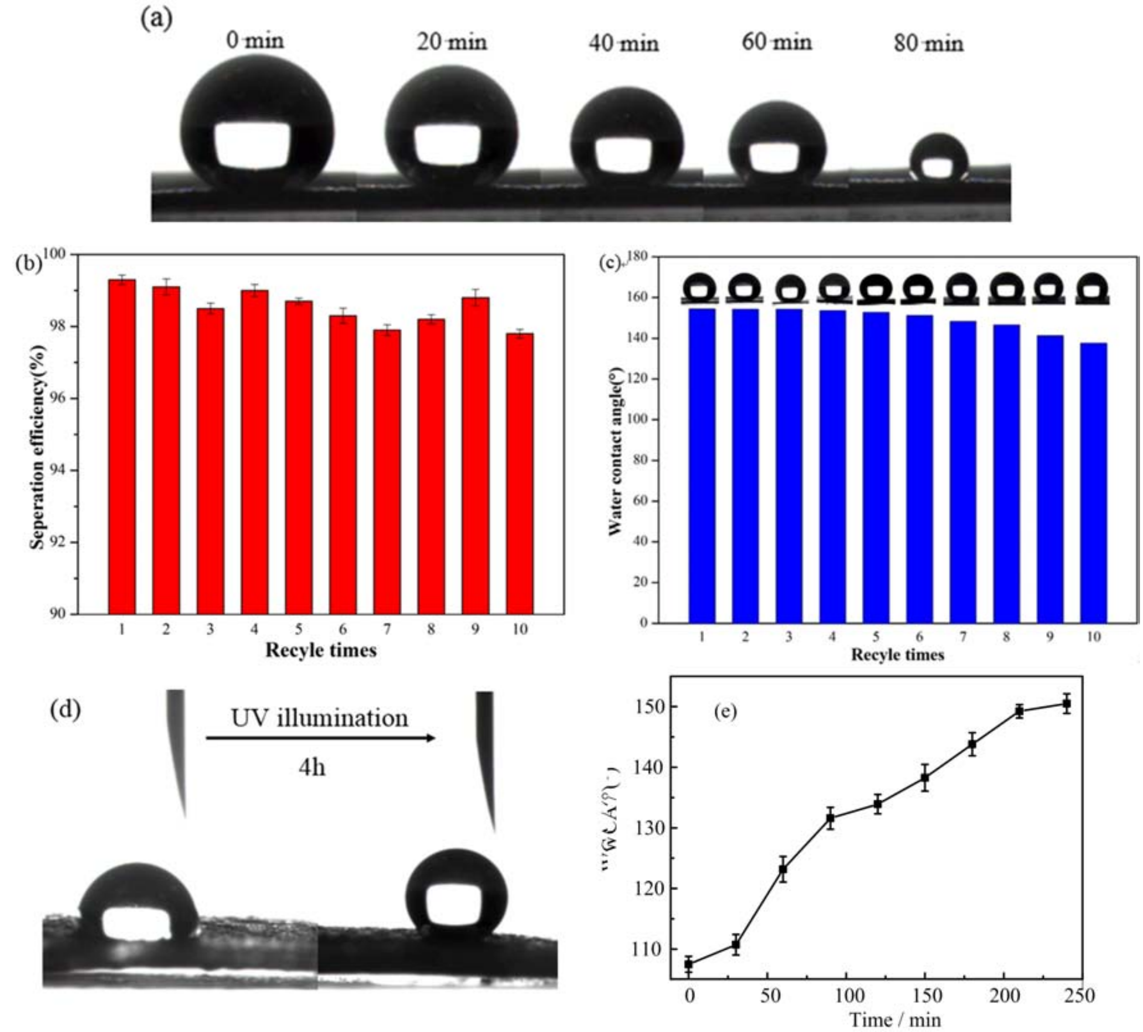

FIG. 7 Stability and self-cleaning performances of coated copper mesh. (a) The time-dependent evolution of the WCA on the coated copper mesh. (b) Separation efficiency of the coated copper mesh of heavy oil/water mixtures at different cycles number (1−10). (c) WCA change of the coated copper mesh at different cycles number (1−10). (d) WCA of the coated copper mesh fouled by lubricating oil before and after UV illumination. (e) The time-dependent evolution of the WCA of coated copper mesh fouled by lubricating oil under UV illumination in air.

ciency remained above 97% after 10 separation cycles. The WCA of coated copper mesh almost had no change after 5 separation cycles, indicating the coated copper mesh possessed admirable recyclability. Nevertheless, the WCA of coated copper mesh was diminished after 10 separation cycles (FIG. 7(c)), which is attributed to the fouled effect organic solvents. During the practical applications of the coated mesh, we have to consider the problem that the separation materials were often contaminated. To decrease the effect of separation materials contaminated by some oils or organic molecules and maintain the continuous and effective separation property of the coated copper mesh, we further investigated the self-cleaning property of the coated copper mesh through the photocatalytic degradation activity of WO_3. As shown in FIG. 7 (d) and (e), the coated copper mesh was fouled repeatedly with a certain amount of lubricating oil, the WCA diminished to 107.5° that cannot be satisfied with the formal oil/water separation. Afterwards, the fouled mesh was irradiated under UV illumination for about 4 h, the WCA was regained to about 150.49° and the separation efficiency approximately reached 90% that we could deem the fueled mesh regained the property of superhydrophobic. We assume that the surface of the coated copper mesh occurred hydroxyl radicals and photo-induced holes with strong oxidation under UV illumination, and this ability of self-cleaning could be attributed to the disintegration of lubricating oil molecules.

IV. CONCLUSION

In this work, a superhydrophobic DTMS@WO_3 copper mesh was successfully fabricated with a simple hy-

DOI:10.1063/1674-0068/cjcp1810233

drothermal method and an immersion method, which showed superhydrophobic (WCA=154.39°) and superoleophilic (OCA=0°). The modified coated copper mesh demonstrated high separation efficiency (99.3%), huge water flux (9962.3 $L\cdot h^{-1}\cdot m^{-2}$) and outstanding self-cleaning property under UV illumination for conveniently regeneration. Because of the excellent superoleophilic and superhydrophobic properties, oil could easily penetrate through the coated copper mesh only driven by gravity, while water was restricted to pass through the surface of copper mesh. Considering high separation efficiency, huge water flux, and outstanding self-cleaning of the coated copper mesh, the mesh could be potentially applied in oily wastewater treatment in practice.

Supplementary materials: Movie S1 showed the process of a water column squirting on the coated copper mesh. Movie S2 displayed that the coated mesh was immersed into the water by an external force. Movie S3 showed the process of the oil/water separation.

V. ACKNOWLEDGMENTS

This work was supported by the National Natural Science Foundation of China (No.21776319 and No.21476269).

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DOI:10.1063/1674-0068/cjcp1810233