Reversible Wettability between Underwater Superoleophobicity and Superhydrophobicity of Stainless Steel Mesh for Efficient Oil–Water Separation

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ABSTRACT: Design and fabrication of smart materials with reversible wettability for oil–water separation have attracted worldwide attention due to the increasingly serious water pollution problem. In this study, a rough oxide coating with micro/nanoscale structures is developed on the 304 stainless steel mesh (SSM) by laser ablation. The smart surface with ethanol immersion and natural drying treatments shows the wetting conversion between underwater superoleophobicity and superhydrophobicity. Based on the wettability transition behavior, both light and heavy oil–water mixtures can be separated with the high separation efficiency. Moreover, after being exposed to various corrosive solutions and high temperatures, the smart surface still shows prominent environmental stability. Switchable surface with excellent properties should be an optimal choice to solve the environmental conditions that need to be addressed urgently.

1. INTRODUCTION

Over the past few decades, water pollution caused by offshore oil exploration, oil tanker leakage, and industrial wastewater discharge has attracted widespread attention worldwide,1 which has led to not only serious environmental pollution but also huge economic loss.2–5 Therefore, a cost-effective method to treat oil wastewater is highly desired. Inspired by the lotus leaf, springtail, and striped scale in nature, special wettability surfaces have recently received extensive attention of researchers because of their significance in diverse fields including self-cleaning,6–8 antifogging9–11 anti-icing12 corrosion resistance,13–15 and drag reduction.16–20 With in-depth research on wettability, some of these materials are widely used in the treatment of oily wastewater.21–23 In general, advanced materials based on special wettability can be classified into two types in the oil–water separation field: the “oil-removing” surface with superhydrophobic/superoleophilic properties can complete the separation of heavy oil–water mixture (heavy oil means that the density of oil is greater than that of water), and the “water-removing” surface with superhydrophilic/underwater superoleophobic properties can finish the separation of light oil–water mixture.24–26 Particularly, the smart interface with switchable wettability is particularly interesting,27–29 which can separate both light and heavy oil–water mixtures using the different circumstance stimuli.30–32 For example, Zhang et al. prepared the nylon membrane via the hydrothermal route, which attained switchable wetting properties upon changing the temperature.31 Du et al. reported a fluorine-free copolymer by disparate pH conditions, and it was compounded by silica nanoparticles/polydimethylsiloxane to prepare a superhydrophobic coating on paper and cotton fabric.32 Sawai et al. proposed the photo-induced underwater superoleophobicity of TiO2 thin film.33 Among these stimuli, there are still many defects such as special equipment, relatively long time, and complex process. Therefore, it is necessary to develop more facile and green approaches to achieve this purpose.

Nanosecond laser is widely used in various materials via etching to obtain microstructures on the surfaces to alter surface wettability,34 which has advantages such as high efficiency, great precision, and easy operation. Nanosecond laser has a higher machining efficiency and a lower cost than femtosecond and picosecond lasers.35 The previous study reported that surface fabricated by a nanosecond laser has good properties such as self-cleaning, anticorrosion, and antifrosting.36 However, there are a few reports on the biomimetic surface, and it was prepared by nanosecond laser to treat oily wastewater. Herein, stainless steel mesh (SSM) has been developed via nanosecond-pulsed fiber laser ablation and ethanol was utilized to attain the reversible wettability to realize oil–water separation. To test the properties of SSM, wettability and separation efficiency are studied. Environmental stability under harsh conditions and mechanical durability also have been investigated.

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2. RESULTS AND DISCUSSION

2.1. Morphology and Chemistry. Figure 1a–d shows the surface morphologies of the original and processed SSMs. The original substrate is nearly smooth, and the average diameter of the untreated mesh (pore size) is approximately 40 μm. By contrast, a number of cross-scale micro/nanoscale structures can be observed evidently on the machined surface that include micron-sized sand structure and nanoscale villi structure; and the average diameter of the mesh pore size is reduced to 33 μm. The reason for the formation of the above microstructure may be that the metal is recast under high temperatures by laser ablation in the processing area.

The chemical compositions and crystal structure of original and processed SSMs are analyzed by energy-dispersive system (EDS) and X-ray diffraction (XRD) spectra, respectively. It can be seen from Figure 2a,c,d that compared with the original substrate, the content of the iron element for the processed SSM is significantly decreased from 65.5 to 56.2%, the oxygen element is obviously increased from 0.5 to 7.1%, and other elements remain stable. Because the laser beam with a higher energy density irradiates the workpiece, the surface absorbs a lot of heat to make the material molten or vaporized to achieve the purpose of removing the material. Therefore, the content of iron is reduced due to the material gasification, and the increase of oxygen content is a result of the oxidation reaction between the substrate material and the oxygen in the air during the laser ablation process. The X-ray diffraction pattern further characterizes the crystal structure on SSM. As shown in Figure 2b, there are four main characteristic peaks on the original substrate, corresponding to the (111), (200), (220), and (311) crystal planes of austenite in the raw material at 43.64, 50.82, 74.66, and 90.62°, respectively. After laser processing, the surface also exhibited four austenite diffraction peaks at 42.44, 44.01, 72.34, and 87.64°, respectively, which are sharper than the original substrate and show the diffraction peak deviation phenomenon. This is due to the lattice distortion caused by solid solution during laser processing, but the crystal structures have not changed and are γ-Fe. The hierarchical structures and chemical composition are necessary for the superhydrophobic surface. Meanwhile, the treatment of oily wastewater is mainly attributed to the existence of the above characteristics.

2.2. Wettability. As shown in the insets of Figure 1b,d, the original SSM shows hydrophobicity/superoleophilicity with static water/oil droplets. For the laser-processed SSM, the superhydrophobic property can be obtained after the processed mesh was exposed to air for 30 days; then, the CA values can reach higher than 150° and the contact angle hysteresis of SSM...
can reach lower than 5° (Figure 1e). This phenomenon can be interpreted that as the sample is placed in the for an increase of time, the decomposition reaction of carbon dioxide still proceeds slowly. As the nonpolar carbon accumulates enough on the rough surface, the wettability of mesh changes from the superhydrophilic after laser radiation to superhydrophobic.\(^{37}\)

When the surface is immersed in water, wettability shows superoleophilicity. As shown in Scheme 1, after immersing this sample in ethanol for 5 s, an underwater oil CA of 150° is achieved. The superhydrophobic surface can be recovered after natural drying, which realizes the conversion between superhydrophobicity and underwater superoleophobicity.\(^{38}\)

Figure 3. (a) Digital images of the bouncing behavior of an oil droplet on the underwater superoleophobic surface. (b) Digital images of the bouncing behavior of an oil droplet on the underwater superoleophilic surface. (c) Underwater oil contact angles and (d) water contact angles during reversible wettability process of the SSM after being immersed in various harsh solutions for 24 h. (e) Underwater oil contact angles and (f) water contact angles of reversible wettability of the SSM changes over five cycles by ethanol immersion and natural drying treatments.

The bouncing behaviors of underwater oil (1,2-dichloro-ethane) droplets on the SSM with ethanol immersion and natural drying treatments are shown in Figure 3a,b and Video S1 (Supporting Information). Before the oil droplet hits the surface of SSM, the falling process is affected by the combined effect of gravity and water resistance, so the shape of the oil droplet is ellipsoidal. As shown in Figure 3a, after the oil droplet hits the surface, it is still ellipsoidal at 48 ms. Then, the oil droplet spreads to the largest diameter at 52 ms and returns to an ellipsoidal shape at 60 ms. At 65 ms, the oil droplet bounces upward from the surface and finally hits the surface again and remained nearly spherical at 69 ms. As shown in Figure 3b, the oil droplet also experiences a fall-impact—bounce-impact again process 71 ms ago. However, unlike the surface immersed in ethanol, the oil droplet continues to spread after impacting again and shows an approximate cone shape after 80 ms. Finally, at 162 ms, the oil droplet is spread almost flat on the surface. The oil droplet stays on the surface stably in a nearly spherical shape,
which demonstrates the superior underwater superoleophobicity. For the naturally dry surface, the oil droplet spreads out on the surface to manifest that the coated mesh shows underwater superoleophobicity. Therefore, the wettability conversion can be achieved by pretreating (with ethanol) and drying surface.

As shown in Figure 3c,d, the CA values of underwater oil and water droplets are studied to evaluate the chemical durability of SSM in a harsh environment. After immersing in the corrosive solution with different pH values (1, 5, 8, 12) and various metal ions (1 M NaCl, 1 M FeCl₃) for 24 h, the contact angles of underwater oil and water droplets have only changed slightly, but both have remained above 150°. The prepared surface still has the ability of wettability conversion via ethanol immersion and natural drying treatment. Therefore, the functional mesh in this work has excellent corrosion resistance and chemical stability. From the energy-dispersive X-ray spectroscopy (EDXS) and XRD results, the substrate chemical compositions and crystal structure have no change during the laser ablation process and there are no new substances generated. According to the previous studies, the austenitic stainless steel has strong corrosion resistance; thus, the hierarchical structures after being immersed in the corrosion solution were not damaged.39 It can be seen from Figure 3e,f that the SSM still has reversible wettability after five reversible cycles, which indicated that the inducing process of wettability conversion via ethanol immersion and natural drying treatments shows good repeatability.

2.3. Oil−Water Separation. The reversible wettability performance of laser-processed SSM is a transition from superhydrophobicity to underwater superoleophobicity, corresponding to the two modes of water-removing and oil-removing, respectively.40 Here, four different oils are selected for oil−water separation testing, including kerosene, lubricant, 1,2-dichloroethylene, and dichloromethane, with densities of 0.8, 0.91, 1.26, and 1.325 g/mL, respectively.

The separation process of the light oil−water mixture (the volume ratio of the two-phase liquid is 1:1) is shown in Figure 4a−d and Videos S2 and S3 (Supporting Information). Kerosene was dyed red, and the lubricating oil was yellow. The processed SSM was prewetted with water and then fixed between the two vessels. It is found that the water quickly passed through the mesh into the beaker, while the oil remained on the surface.

The separation process of the heavy oil−water mixture (the volume ratio of the two-phase liquid is 1:1) is shown in Figure 4e−h and Videos S4 and S5 (Supporting Information). The water was dyed blue and red, respectively. The SSM was
The separation efficiency of various oil–water mixtures (Figure 5a) was more than 96% according to eq 1. As shown in Figure 5b, taking the kerosene oil–water mixture as an example, the separation efficiency can still be maintained above 96% after 20 cycles.

\[ \eta = \frac{m_1}{m_0} \times 100\% \]  

where \( m_1 \) (g) is the weight of water collected in the breaker and \( m_0 \) (g) is the weight of water before the separation test. In addition, the permeation water flux is also discussed to evaluate the mesh separation property. During the 20 cycles, the water flux of the obtained SSM is between 25.47 and 29.87 L/(m² s⁻¹), and the change in the whole experiment process is not obvious, indicating that the mesh has ideal recyclability and good separation ability.

The Laplace equation is used to further explain the oil–water separation mechanism. The intrusion pressure (Δp) can be calculated using the following formula:

\[ \Delta p = \frac{2\gamma}{R} = -\frac{\gamma \cos \theta}{A} \]  

where \( R \), \( l \), and \( A \) represent the side length, perimeter, and cross-sectional area of the mesh pore, respectively; \( \gamma \) is the liquid surface tension; and \( \theta \) is the contact angle value of droplet. The \( \theta \) value is the only variable in the formula, so the positive and negative of Δp can be determined by \( \theta \). As shown in Scheme 2a,b, the surface that is not modified with ethanol is superhydrophobic/superoleophilic. According to eq 2, \( \theta > 150^\circ \) and \( \Delta p > 0 \), indicating that the surface also needs some external pressure and can be wetted by water. For the oil droplet, \( \theta \approx 0^\circ \) and \( \Delta p < 0 \), so the oil can directly pass through the mesh. When the heavy oil–water mixture was poured into a homemade separator, the heavy oil could quickly permeate the surface and enter the beaker, while the water remained in the container. As shown in Scheme 2c,d, the surface that is modified with ethanol is superhydrophilic/underwater superoleophobic. According to eq 2, it can be seen that \( \theta \approx 0^\circ \) and \( \Delta p > 0 \), so water can directly pass through the mesh. For underwater oil droplet, \( \theta > 150^\circ \) and \( \Delta p > 0 \), indicating that the surface also needs some external pressure and can be wetted by oil. Therefore, when the light oil–water mixture was poured into the homemade separator, water could quickly permeate the surface, while the light oil remained on the mesh.

### 2.4. Mechanical Durability

Because mechanical durability plays a vital role in functional materials, sandpaper abrasion tests are carried out to evaluate the physical properties. As shown in Figure 6a, the mesh fixed with the confining layer was put on the sandpaper (1000 grids) and loaded with a 100 g weight. The mesh was moved forward 200 mm as one abrasion cycle. It can be seen that with the increase of abrasion cycle, the sample was worn more seriously. However, after 15 cycles, the hierarchical structures still can be observed and were not completely damaged (Figure 6c–e). Meanwhile, various underwater oil contact angles were measured (Figure 6b), and the results showed that the values decreased slightly but remained above 150°. These phenomena are attributed to the special structures on the SSM. During the wear tests, the SSM was not completely in contact with the sandpaper. The abrasion only damaged the uppermost structure, and the internal structure was protected. Therefore, the SSM can still realize oil–water separation (Video S6 (Supporting Information)) and has good mechanical durability.

### 3. CONCLUSIONS

In summary, a rough oxide coating on the stainless steel mesh is fabricated successfully via laser ablation. The reversible wettability between superhydrophobicity and underwater superoleophobicity of the surface can be obtained by ethanol immersion and natural drying treatments. The smart surface can be used for both light and heavy oil–water mixture separation, and the efficiency of diverse oil–water mixture can maintain above 96% after 20 cycles. The mesh keeps the excellent environmental stability under the rigorous environment and good mechanical durability after sandpaper abrasion. This work offers a novel and sustainable method to obtain the functional surface, which will have a prominent application prospect on the treatment of oily wastewater.

### 4. EXPERIMENTAL SECTION

#### 4.1. Materials

SSM was purchased from HebeiHaiji Metal Wire Mesh Manufacturing Co., Ltd. Acetone, ethanol, Calcium Flavin, NaCl, and methylene chloride were obtained from Beijing Chemical Works. NaOH and HNO₃ were supplied by Yonghua Chemical Co., Ltd. Kerosene was purchased from China Petroleum & Chemical Co., Ltd. Sudan Red III was purchased from Tianjin Guangfu Fine Chemical Research Institute. No. 40 universal lubricating oil was purchased from Jinmei Petrochemical Co., Ltd. Dichloromethane was purchased from Tianjin Tiantai Fine Chemical Co., Ltd. All of the chemicals were utilized without further explanation.

#### 4.2. Sample Fabrication

The SSM was washed using ethanol and deionized water three times before laser processing, respectively. The meshes with a size of 60 mm × 60 mm were fabricated by a nanosecond-pulsed fiber laser marking system (YLP-ST20, Shenzhen DazuLaser GMBH) via a line scanning method (Figure 7), in which pulse width, laser spot diameter, and wavelength are 100 ns, 52.63 μm, and 1064 nm, respectively. The laser beam is generated by the nanosecond laser and irradiates on the surface fixed by the transparent confining layer after passing through a reflector, dual-galvanometer system, and focusing mirror in sequence. The main machining parameters including scanning speed, scanning spacing (parameter \( D \)), and laser power were 500 mm/s, 0.03 mm, and 12 W, respectively.
The obtained samples were cleaned in an ultrasonic cleaner with acetone, ethanol, and deionized water in sequence.

4.3. Experimental Setup. The as-prepared stainless steel mesh was fixed on the filtering device. The oil–water mixtures with 30 mL each of the two phases were poured on the SSM prewetted with water or oil. The above experiments were repeated three times to calculate the average separation efficiency as the final results. A high-speed camera was used to photograph the bounce behavior of oil droplet in water. In addition, the fabricated SSMs were placed in a 1 mol/L HNO₃, 1 mol/L NaOH, and 1 mol/L NaCl solution for 24 h at room temperature, and then the wettability of the SSMs was tested. The wettability conversion experiment of SSM under alcohol immersion and natural drying has been repeated to verify its good sustainability. Moreover, the sandpaper abrasion test was utilized to evaluate the mechanical durability of SSM. The coating mesh of weight 100 g was rubbed with sandpaper (1000 grit) and moved 200 mm as one abrasion cycle, and the morphologies and wettability of SSM after 5, 10, and 15 cycles were characterized.

4.4. Characterization. Observation of surface morphology was carried out by a scanning electron microscope (SEM, ZEISS, EVO25). For the wettability tests, contact angle (CA) values and the bouncing behaviors of oil droplet in water were measured by a self-developed contact angle instrument and a high-speed camera (PCO.dimaxHS), respectively. During this process, the volume of the droplets used was 5 μL, and the final CA values were obtained after averaging the measurements made at three different positions of the same surface. The chemical compositions were checked by energy-dispersive X-ray spectroscopy (EDXS). The crystal structure of coating was detected by an X-ray diffractometer system (XRD, D/Max-2500, Japan). Digital photos and videos were taken by a camera (Canon, EOS, M3).

ASSOCIATED CONTENT
Supporting Information
The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acsomega.0c03369.

Movie S1: Process of underwater oil droplets bouncing on the superoleophobic/superoleophobic surface (MP4)
Movie S2: Separation process of the kerosene—water mixture (MP4)
Movie S3: Separation process of the lubricating—water mixture (MP4)
Movie S4: Separation process of the dichloromethane—water mixture (MP4)
Movie S5: Separation process of the 1,2-dichloroethane—water mixture (MP4)
Movie S6: Separation process of the kerosene—water mixture after 15 cycles sandpaper abrasion tests (MP4)

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Notes
The authors declare no competing financial interest.

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