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Facile fabrication of superhydrophobic copper hydroxide coated mesh for effective separation of water-in-oil emulsions

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A R T I C L E I N F O

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Water-in-oil emulsions
High separation efficiency
Recyclability

A B S T R A C T

Recently, the super-wetting materials for separating oil/water emulsions have attracted great attention. Developing a low-cost and high-efficient filter material that is challenging to separate stable oil/water emulsions efficiently. In this work, the superhydrophobic copper hydroxide coated mesh was successfully fabricated through a facile spraying and surface modification approach. The excellent separation property of the as-prepared material with efficiency greater than 99.0% under the driving of gravity. Furthermore, the as-developed SCM retained the favorable recyclability and excellent stability even after 10 reuses. More importantly, the SCM exhibited excellent mechanical abrasion resistance after 50 abrasion cycles with sandpaper, which could still remove water from emulsions and possessed excellent separation efficiency. Therefore, the newly developed materials with special wettability have excellent potential in practical use and provide a novel perspective for preparation of stable emulsions separation materials.

1. Introduction

Recently, the increasing in discharge of oily sewage and frequent oil spills from industrial accidents have posed serious hazards to marine ecosystems safety and human health, and separation of oily sewage, especially for purifying emulsified oily sewage, becomes a global problem and challenge [1–4]. To resolve these problems, the functional materials with super-wetting have been explored for achieving separation of oil/water mixtures effectively [5–9]. The oil/water mixtures are mainly composed of emulsified and immiscible oil/water mixtures. The various traditional methodologies of oil/water separation, such as oil skimmers, de-emulsification, ultrasonic irradiation, centrifuges, et al. are utilized for immiscible oil/water mixtures separation [10–12]. However, traditional separation techniques for oil/water mixtures have disadvantages of low efficiency, secondary pollution, high energy-consuming, and are not applicable to oil-water emulsions separation, especially for the surfactant-stabilized emulsions [13–17]. The emulsified oil/water mixtures are easy to form with relatively small droplets size and stable structure which were more difficult to separate [18–20]. Therefore, materials with super-wetting are greatly desired to effectively purify oil/water emulsions.

Nowadays, the filters materials with special wettability have gained widespread attention because of their high efficiency in separating emulsions. Generally, such super-wetting materials are mainly ranged into two types: superhydrophobic/superoleophilic (oil-removing) [21–24] orsuperhydrophilic/underwater superoleophobic (water-removing) [25–28]. The oil-removing materials with superhydrophobicity/superoleophilicity can allow oil pass through the materials and repel the water. Jiang et al. firstly prepared superhydrophobicity and superoleophilicity mesh film by a facile spray-and-dry method to separate oil from water, which allowed the oil permeated through the mesh freely while the water phase to be repelled above the mesh completely [29]. Inspired this work, Zhang and co-workers fabricated all-inorganic membranes on copper mesh via a chemical oxidation method, and these superhydrophilic and underwater superoleophobic membranes could remove oil from water with excellent separation property [30]. Although many super-wetting materials have been investigated, they were incapable of separating surfactant-stabilized water-in-oil emulsions. The diameters of emulsified water droplets in surfactant-stabilized emulsions were usually below 10 μm which were difficult to separate [31–33]. Subsequently, other kinds of separation materials with special wettability were explored to

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separate surfactant-stabilized water-in-oil emulsions efficiently. For instance, Zeng et al. gained enlightenment from the special topography of the Stenocara beetle's back, fabricated SBS-SSM filter via an electrostatic self-assembling and spin-coat process which was utilized for separating surfactant-stabilized water-in-oil emulsions [33]. Zhang et al. fabricated a poly (vinylidene fluoride) (PVDF) membrane through a phase-inversion method which as-fabricated membrane exhibited high separation efficiency and recyclability for separating highly stable water-in-oil emulsions [34]. Cai and co-workers reported that the double-layer stainless steel mesh (DSSM) was prepared via modifying approach of different polymers coating which could achieve the separation of emulsified water-in-oil mixtures [35]. These materials exhibited excellent separation property, however, the synthesis of these materials normally relates high energy consumption and complexity of fabrication approaches, which can be very challenging for large-scale membrane fabrication. Therefore, designing a facile, low-cost superwetting separation films to effectively separate different types emulsions became an urgent demand, especially the separation of surfactant-stabilized emulsions under gravity.

Herein, in order to realize low-cost and efficient separation of surfactant stabilized water-in-oil emulsions, the superhydrophobic copper hydroxide coated mesh (SCM) was fabricated via a facile spraying and modifying approach. The hydrophilic copper hydroxide coated mesh functionalized with n-dodecanethiol was selected as a coating material due to its stable superhydrophobicity. The obtained SCM was capable of efficiently separating different sorts of surfactant-stabilized water-in-oil emulsions at ambient pressure. The separation efficiency of the as-prepared SCM for a wide range of highly stable emulsions was more than 99.0% under the driving of gravity. Furthermore, the as-fabricated SCM still retained the favorable recycling ability even after 10 cycles test. More importantly, the SCM exhibited excellent superhydrophobicity and robustness after 50 abrasion cycles with sandpaper. Thus, we anticipate that the SCM has potential uses in industrial oily sewage remediation.

2. Experimental

2.1. Materials

Sodium hydroxide and copper (II) chloride dehydrate was supplied from Guangzhou Jinhuada Chemical Reagent Co., Ltd. Acetone and ethanol were obtained from Shanghai Chemical Reagent Co., Ltd. The stainless steel meshes (1000 mesh size) were provided by a local hardware store. Waterborne PU was bought from Sinopharm Chemical Reagent Co., Ltd. Span 80 was purchased from Shandong LaiyangShuangshuang Chemical Co., Ltd. China. The n-dodecanethiol was gained from shanghai Sinopharm Chemical Reagent Co., Ltd. Oils (kerosene, diesel, petroleum ether, hexane, and heptane) were supplied by Guangdong Guanghua Sci-Tech Co., Ltd. The stainless steel meshes were washed with acetone and ethanol by ultrasonication to remove the pollutants on the surfaces before use. All the reagents were analytical grade.

2.2. Preparation of the superhydrophobic copper hydroxide coated mesh

First, the stainless steel meshes (1000 mesh size) was cut up into 4 cm × 4 cm substrates and then cleaned with acetone and ethanol for 10 min by ultrasonication, respectively. After that, 2.0 g dehydrated copper (II) chloride and 1.05 g sodium hydroxide were dispersed in 50 mL distilled water. The amount of 2–3 drop waterborne polyurethane (PU) was dissolved in prepared solution with stirring magnetically for 20 min. The as-prepared solution was sprayed onto the mesh utilizing a spray gun. The obtained meshes were sufficiently dipped in a solution of n-dodecanethiol and ethanol for 8 min, and subsequently the meshes were cleaned in ethanol to remove the excess n-dodecanethiol. Ultimately, the superhydrophobic copper hydroxide coated meshes (SCM) were dried at room temperature for 1 h before characterizations.

2.3. Characterization

The surface morphology of the as-fabricated mesh was characterized using field emission scanning electron microscopy (FE-SEM, Zeiss). EDS spectra were collected on the Zeiss Ultra Plus equipped with an EDS detector (Oxford, Aztec-X-80). The FT-IR spectroscopy was conducted with Bio-Rad FTS-165 equipment and the data was collected using the KBr method as the transmission mode. The static water contact angle (CA) and sliding angle (SA) were evaluated with 3 μL of water droplets using a SL200KB apparatus at ambient temperature. The water CA was the average of at least five measurements which obtained on different locations of the sample surface. The error of the contact angle is ± 2. The Karl Fischer Titrator (SN-WS200A) was used to measure the water content in the collected oil in oil filter. Optical microscopy images were taken on an inverted fluorescence microscope IX51 (Olympus, Japan) by dropping the emulsions separated before and after on biological counting board. Dynamic light scattering (DLS) measurements which were performed on a Zetasizer Nano ZS (Malvern, UK) to probe droplet size distribution.

2.4. Separation of surfactant-stabilized water-in-oil emulsions

A series of surfactant-stabilized emulsions were fabricated by following steps: Firstly, 0.15 g Span-80 was mixed with the five kinds of 100 mL oils and subsequently 2 mL water was added drop by drop into the oils. The mixtures formed stable milky emulsions by intensive stirring for more than 6 h. In this work, five different types of oil including kerosene, diesel, hexane, petroleum ether and heptane were chosen as the oil phase in the emulsions and the as-prepared emulsions could be stable for more than 10 days. Ultimately, the emulsified water/oil mixtures were poured onto the as-prepared SCM, the oil phase passed through the mesh while the water phase was blocked by the mesh. Simultaneously, the transparent oils were collected into the backer under the driving of gravity. The utilized mesh with alcohol treatment can be recycled and used to separate water-in-oil emulsions.

2.5. Robustness tests

The robustness of the obtained mesh was studied via the sandpaper abrasion test. The SiC sandpaper (800 Grit) was used as the friction surface and the obtained mesh surface was placed face down on the sandpaper in mechanical abrasion resistance tests. The sample under a 100 g weight was pull back and forth, in which the abrasion length is 10 cm. Subsequently, the water contact angle and separation efficiency were carried to evaluate robustness of the obtained mesh.

3. Results and discussion

A spraying and modifying process was used in this work to fabricate the SCM, as the schematic diagram exhibited in Fig. 1. The as-prepared materials used stainless steel meshes as substrate due to its good mechanical strength and low-priced. The mixtures of sodium hydroxide, PU and copper (II) chloride dehydrate were sprayed onto the surfaces of the mesh to gain hydrophilic copper hydroxide coated mesh. The reaction could be illustrated as follow:

$$CuCl_2 + 2NaOH \rightarrow Cu(OH)_2 + 2NaCl$$

Subsequently, the hydrophilic copper hydroxide coated mesh was reacted with solution of n-dodecanethiol and ethanol to obtain a superhydrophobic surface with low surface energy. The chemical equation of reaction was illustrated by means of the following equation [36–37]:

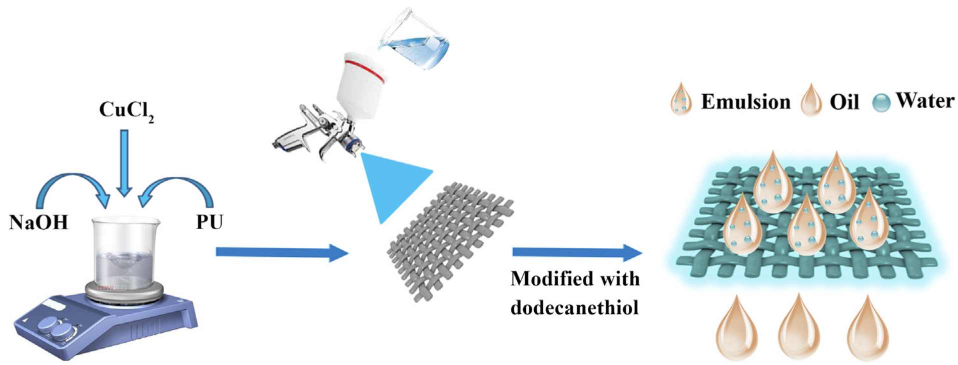

Fig. 1. Schematic diagram of the fabrication of superhydrophobic copper hydroxide meshes and the separation of emulsions.

$Cu(OH)_2 + 2C_nH_{2n+1}\text{-}SH \rightarrow Cu(SC_n H_{2n+1})_2 + 2H_2O$

Then, the superhydrophobic meshes were tested with experimental device, which could permit oil to pass through and water to be blocked by the superhydrophobic meshes. Hence, the copper hydroxide mesh with superhydrophobicity/superoleophilicity could be successfully constructed and realize stabilized emulsions separation.

The surfaces morphological images of the pristine and modified hydrophilic copper hydroxide coated meshes were presented in Fig. 2. The morphological of as-prepared SCM surface obviously differ from raw hydrophilic copper hydroxide coated mesh surface. As exhibited in Fig. 2a and b, the surface of mesh was entirely overlapped by hydrophilic copper hydroxide, forming a rough surface. The inset of Fig. 2b depicted the rough structure of nano-filamentous on hydrophilic copper hydroxide coated mesh. In this work, the nano-filamentous structure of hydrophilic copper hydroxide coated mesh is consistent with previous reports [36]. In Fig. 2c, it also obviously seen that the mesh was overlapped by a dense layer superhydrophobic copper hydroxide after treatment with n-dodecanethiol. Fig. 2d and the high magnification image in the inset of Fig. 2d further exhibited that the surface of SCM possessed micro-nano papillary rough structure, which possesses average pore size about 64 nm (Fig. S1).

The surface chemical composition of hydrophilic copper hydroxide coated mesh and the as-prepared SCM was confirmed by EDS. As exhibited in Fig. 3a, the chemical element includes C, O, Cu and Au. Compared with Fig. 3b, the S peak was found in the spectrum of as-prepared SCM. Therefore, the result indicated the dodecanethiol molecules was assembled on the copper hydroxide [37]. Furthermore, the element content of hydrophilic copper hydroxide coated mesh before and after treating with n-dodecanethiol was compared, the percentage of Cu atomic decreased from 19.20% to 10.94% and the percentage of Oatomic percentage reduced from 29.88% to 1.55%. The results confirmed the n-dodecanethiol was successfully assembled on the surface of copper hydroxide coated mesh. The appearance of Au elements is due to the treatment of spray-gold.

The XRD results of the coated copper hydroxide on the mesh was displayed in Fig. 3c, all diffraction peaks ascribed to the copper hydroxide, which was consistent with the previously report [38]. In addition, the FT-IR spectra of hydrophilic copper hydroxide coated mesh before and after modification with n-dodecanethiol were displayed in Fig. 3c. In the spectrum of superhydrophobic copper hydroxide, the absorption bands at 2920 cm^{-1}, 2850 cm^{-1} are ascribed to the C—H asymmetric and symmetric stretching vibrations [39]. The absorption peak at 1460 cm^{-1} is ascribed to S—CH_2 deformation vibration. Meanwhile, the absorption peak at 721 cm^{-1} could be accounted to the stretching vibration of S—C. However, the S-H stretching vibration of weak peak at 2600 cm^{-1} is not found in the infrared spectrum of the hydrophilic copper hydroxide. The results further indicated that n-dodecanethiol molecules were grafted onto original copper hydroxide

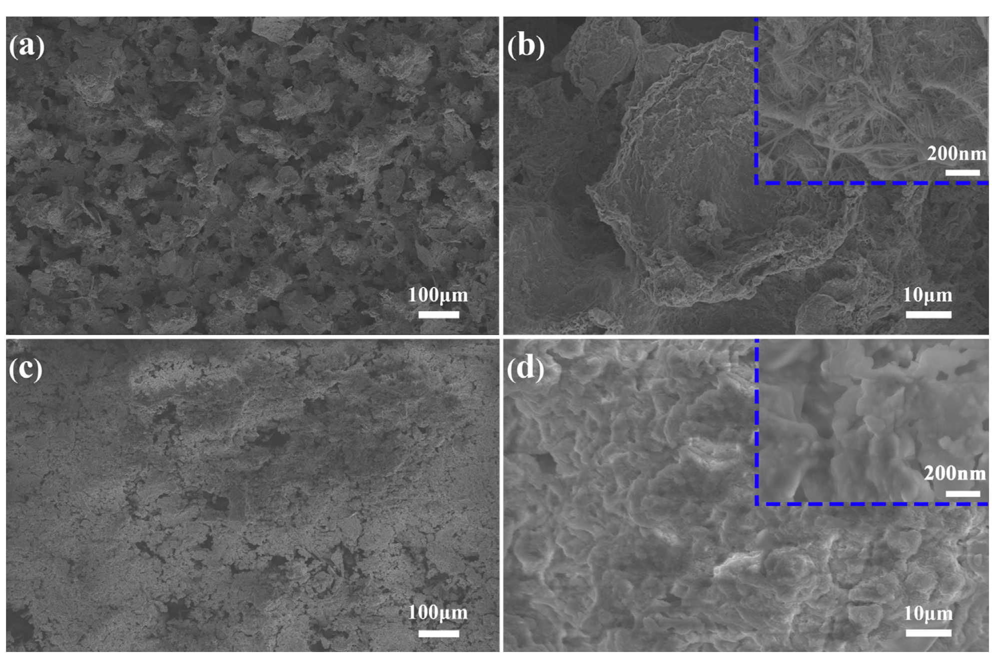

Fig. 2. (a-b) FE-SEM images of the hydrophilic copper hydroxide coated mesh. (c-d) FE-SEM images of the as-prepared SCM modified by n-dodecanethiol.

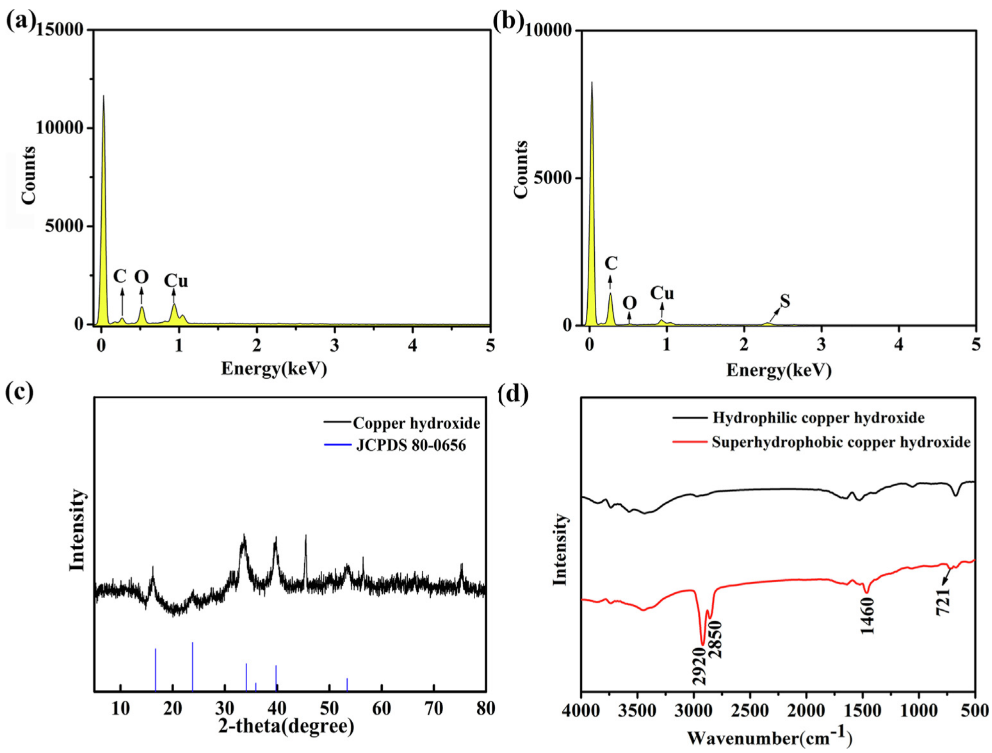

Fig. 3. EDS patterns and elemental content results of the (a) hydrophilic copper hydroxide coated mesh and (b) hydrophilic copper hydroxide coated mesh modified by n-dodecanethiol. (c) XRD analysis of the coated copper hydroxide on the mesh. (d) FT-IR spectroscopy of the (black line) hydrophilic copper hydroxide coated mesh and the (Red line) hydrophilic copper hydroxide coated mesh modified by n-dodecanethiol. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

mesh. As mentioned above, the hydrophilic copper hydroxide coated mesh was successfully modified with the low energy material.

The surface-wetting behaviors of the obtained SCM were investigated by contact angle (CA) measurements. As displayed in Fig. 4a and b, owing to the cooperation of the n-dodecanethiol molecules assembled and the nano-filamentous structures, the water droplets kept spherical shape when the water droplets touched the surface of as-prepared SCM with water CA of 154.4 ± 2°. While the oil droplet (kerosene) touched the surface of as-prepared SCM, it quickly spread on the surface of as-prepared SCM and the oil CA was approximately 0°, as presented in Fig. 4c and d. Therefore, the obtained SCM exhibited excellent superhydrophobic/superoleophilic properties.

Owing to SCM surfaces possess excellent superhydrophobic property and especial micro-nano papillary structure, it could efficiently remove the water from emulsions. Many different kinds of stable water-in-oil emulsions were fabricated to estimate the separation capability of the SCM such as water-in-hexane, water-in-kerosene, water-in-petroleum ether, water-in-diesel and water-in-heptane. Taking the separation of Span-80 stable water-in-kerosene emulsions as examples. Compared with pristine water-in-kerosene emulsions, the emulsions turned to the clear state (Fig. 5a). Furthermore, it obviously seen from the optical microscopy that the phase composition of original emulsions and the collected filtrate were remarkably distinct. As Fig. 5a presented, the feed emulsions include many water droplets in the optical images, whereas any droplet was not detected in the collected filtrate over the entire image, indicated that the as-prepared SCM could efficiently

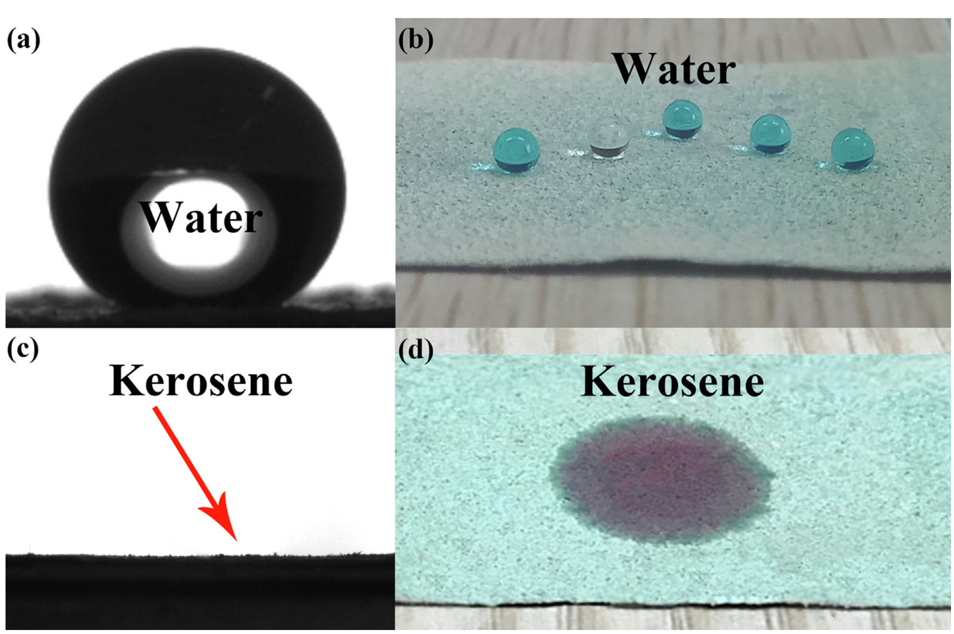

Fig. 4. (a) Photographs of water contact angle on the as-prepared SCM in air. (b) The photograph of water droplets (dyed with methylene blue) on the surface of the as-prepared SCM. (c) Photographs of oil contact angle on the as-prepared SCM in air. (d) The photograph of oil droplet (dyed with Oil Red O) on the surface of the as-prepared SCM. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

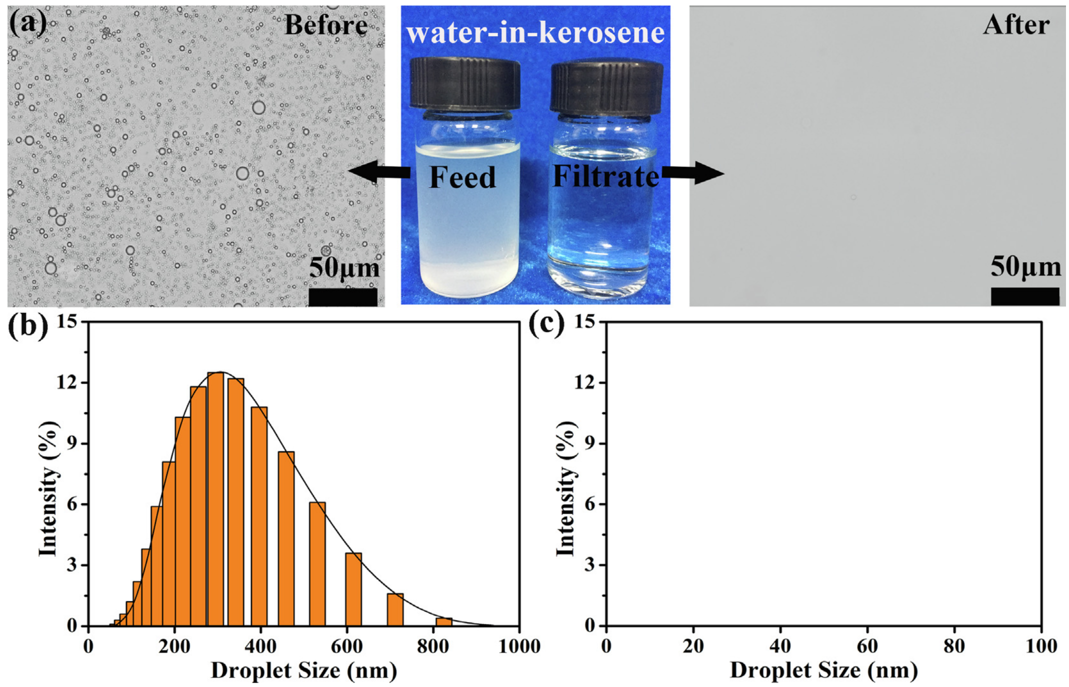

Fig. 5. (a) The optical images and digital photos of water-in-oil emulsions before and after separation. (b-c) The droplet size analysis of water-in-kerosene emulsions before and after separation.

separate water droplets from water-in-kerosene emulsions. Moreover, the droplet diameters distributions of the various kinds of emulsions were measured by DLS. Fig. 5b exhibited the droplet size of water droplets dispersed in kerosene were smaller than 1000 nm before separation. Furthermore, it was obviously seen from Fig. 5c that no droplet was found in the collected filtrate. In addition, other different types of stabilized emulsions contain water-in-hexane, water-in-petroleum ether, water-in-diesel and water-in-heptane emulsions also achieved similar effective separation (Fig. S2). The results confirmed that many densely packed water droplets of different stabilized water-in-oil emulsions were efficiently separated. The separation efficiency and flux were further measured to estimate separation property of the as-prepared SCM for various water-in-oil emulsions. The separation efficiency (E) was computed by the formula (1):

$$E = (1 - C_f/C_o) \times 100\% \tag{1}$$

where, E is the separation efficiency. C_f and C_o are the water content of the collected filtrate and the original water-in-oil emulsions, respectively. As presented in Fig. 6a, the separation efficiency of the SCM were greater than 99.0% for a series of water-in-oil emulsions. Moreover, flux is also a significant index to evaluate filter materials separation ability. The fluxes (F) were calculated by the equation (2):

$$F = V/ST \tag{2}$$

where V is the volume of the filtrate, S is the effective contact area of emulsions and SCM, T stands for the time that it takes to separate a certain amount of emulsions. As displayed in Fig. 6b, the flux of five sorts of water-in-oil emulsions was up to 16 $L\,m^{-2}h^{-1}$. The as-fabricated SCM could separate the stabilized emulsions through sieving effect by the driving of gravity. Therefore, the flux of separation was relatively lower. Because when the surfactant was existed in the emulsions, the water droplets scarcely gathered, and a filter cake formed on the surface of the separation mesh, which would improve the separation efficiency on some level. Nevertheless, the cake would also terribly clog the surface pores and lowered the effective filtration area of the membranes, which resulted in a quick decrease in the permeation flux [40]. Moreover, the recyclability property of the filter material is significant parameters in oily wastewater remediation. The separation efficiency versus cycle numbers was further investigated by taking water-in-kerosene emulsions as instance. As shown in Fig. 6c, the separation efficiency of as-prepared SCM still remained above 99.0% after 10 cycles, demonstrating the favorable recyclability and excellent stability of the as-prepared SCM.

The robustness of the superhydrophobic materials surface is of importance factor which limit broad applications [41]. In order to

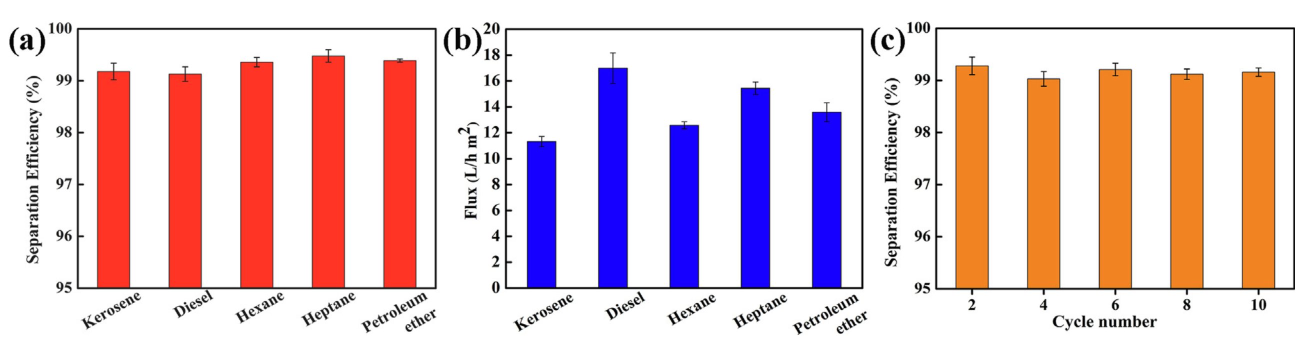

Fig. 6. (a) The separation efficiency of a series of stabilized water-in-oil emulsions. (b) The separation flux of different stabilized water-in-oil emulsions. (c) The separation efficiency versus cycle numbers by taking water-in-kerosene emulsions as instance.

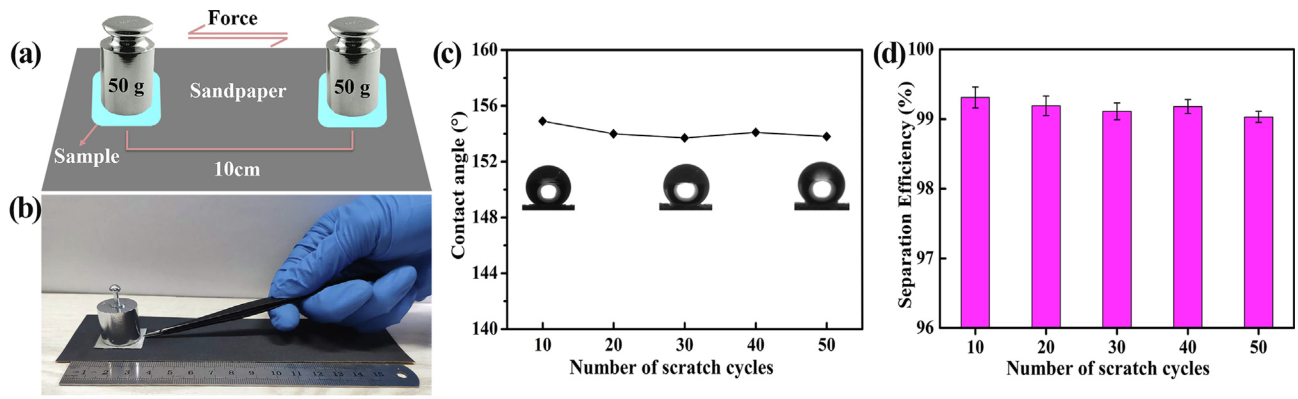

Fig. 7. (a) The schematic diagram of the sandpaper abrasion test. (b) The photograph of the sandpaper abrasion test. (c) The variation of water CAs after every ten abrasion tests. (d) The separation efficiency of as-fabricated SCM after sandpaper abrasion test.

Table 1
Comparison between separation efficiency and mechanical robustness of copper hydroxide material materials.

Material	Method	Mechanical Robustness	Separation type	Efficiency/Oil content	Reference
$Cu(OH)_2$ nanowire haired membrane	Surface oxidation	None	Oil-in-water emulsions	< 60 ppm	[42]
$Cu(OH)_2$ coated copper mesh	Spraying	Sand powder impact	Oil-water mixture	> 99%	[43]
Copper foam	Solution-immersion	None	Oil-water mixture	> 98.9%.	[44]
$Cu(OH)_2$ Nanoneedles Mesh	Surface oxidation	Stretch	Oil-water mixture	> 98%	[45]
Superhydrophobic copper mesh	Chemical oxidation	None	Oil-water mixture	> 97%	[46]
$Cu(OH)_2$ coated stainless steel mesh	Spraying	Sand paper abrasion 50 cycles	Water-in-oil emulsions	> 99%	This work

Table 2
Comparison between separation efficiency and mechanical robustness of various superwetting separation materials.

Material	Method	Mechanical Robustness	Separation type	Efficiency	Reference
SSM/CNFs-PDMS membrane	Vacuum-based filtration	Sand paper abrasion 20 cycles	Water-in-oil emulsions	None	[47]
Carbon nanofibers membrane	Assembling	Sand paper abrasion 20 cycles	Water-in-oil emulsions	None	[48]
HBPU/F-SiO_2 membranes	Electrospinning	None	Water-in-oil emulsions	> 99%.	[49]
Cu@Ag@DDT film	Immersing	Sand paper abrasion 10 cycles	Water-in-oil emulsions	> 98%	[50]
WO_3/TiO_2 membrane	Chemical deposition	Sand paper friction	Oil-in-water emulsions	> 98.5%	[51]
Superhydrophobic SiO_2 microspheres	Template	Sand paper abrasion 30 cycles	Oil-in-water emulsions	None	[52]
PFDT/PDA/PI nanofibrous membranes	Immersing	Sand paper abrasion 5 cycles	Water-in-oil emulsions	> 99%	[53]
$Cu(OH)_2$ coated stainless steel mesh	Spraying	Sand paper abrasion 50 cycles	Water-in-oil emulsions	> 99%	This work

evaluate robustness of the obtained mesh, the sandpaper abrasion test was carried utilizing SiC sandpaper (800 Grit). The schematic diagram and photograph of the sandpaper abrasion test were presented in Fig. 7a and b respectively. As exhibited in Fig. 7c, the water contact angles of the SCM were still more than 150° after 50 abrasion cycles. The results indicated that the as-prepared SCM exhibited excellent and stable superhydrophobicity. Moreover, the separation efficiency after abrasion tests was further investigated by taking water-in-kerosene emulsions as instance. As shown in Fig. 7d, the separation efficiency of as-prepared SCM maintained above 99.0% after 50 abrasion cycles, demonstrating that the as-fabricated SCM possess outstanding mechanical abrasion resistance. However, the previously reported materials that used to separate oil/water emulsions have certain defects in stability and durability. The separation efficiency and mechanical robustness of separation materials were compared in Table 1 and 2, further illustrated that the obtained SCM exhibited excellent separation efficiency, stability and mechanical robustness.

4. Conclusion

In summary, we have designed a copper hydroxide coated mesh with superhydrophobic/superoleophilic property via facile method of spraying and modifying. The as-fabricated SCM exhibited excellent selective wettability for oil and water. Meanwhile, the as-developed SCM could efficiently separate various surfactant-stabilized water-in-oil emulsions with high separation efficiency (above 99.0%) under the driving of gravity. Furthermore, the separation efficiency of the as-developed SCM was more than 99.0% after 10 cycles, suggesting the favorable recyclability and excellent stability of the as-prepared SCM. More importantly, the SCM exhibited excellent robustness and superhydrophobicity even after 50 sandpaper abrasion cycles. Therefore, the newly developed materials with special wettability are promising candidates in oily wastewater treatment, which offer a new perspective on preparation of emulsified oil/water mixtures separation materials.

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Appendix A. Supplementary material

Supplementary data to this article can be found online at https://

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