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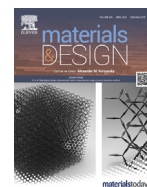
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Superhydrophobic and superoleophilic membranes for oil-water separation application: A comprehensive review

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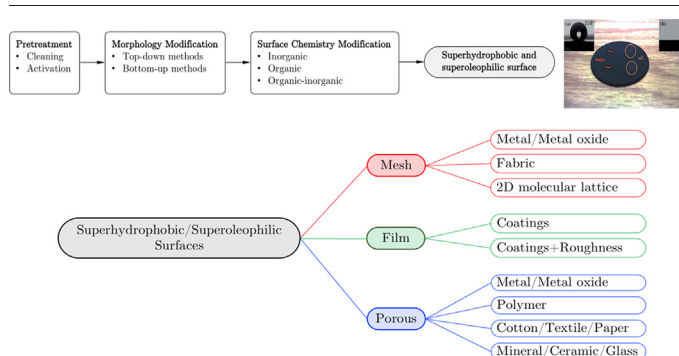
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HIGHLIGHTS

- This review paper focuses on fabrication and characterization of SHSO surfaces.
- The efficiency of SHSO mesh, porous, and film-based membranes for oil-water separation is discussed.
- Different methods to modify surface roughness and chemistry for SHSO wetting conditions are assessed.
- Insights on the future developments and applications of SHSO membranes are given.
- The aspects that need further attention in the future research studies are highlighted.

GRAPHICAL ABSTRACT



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ABSTRACT

Superhydrophobic and superoleophilic (SHSO) membranes have gained remarkable attention, particularly in selective and efficient oil-water separation applications. This paper provides a comprehensive review of the SHSO membranes, fabrication and characterization methods, the advantages and disadvantages of the fabrication techniques, current status and prospects of SHSO surfaces, and potential future research directions. Chemicals such as silanes, thiols, fatty acids, carbon nanotubes, and polyethylene-based polymers are commonly used to adjust the surface energy for the SHSO membranes. These membranes have been proven successful in selectively separating oil from oil-water mixtures with oil separation efficiency >99%. Although there are limited studies on the short-term stability assessment of the SHSO coatings upon exposure to acid and alkaline environments, the effects of temperature, and adsorption of heavier components of the crude oils (such as asphaltenes, and resins) on the separation efficiency have not been adequately investigated yet. Moreover, the fouling performance of the SHSO membranes with water-derived fouling is surprisingly missing in oil-water separation applications in the literature. With the breakthrough in technology, the use of 2D materials such as graphene and high-resolution 3D printers to create hierarchical features on membranes, research on application of SHSO membranes in oil-water separation processes can be further advanced.

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1. Introduction

Oil-water separation becomes important in applications such the treatment of oily wastewater and oil spill removal. A considerable volume of industrial oily wastewater is produced in textile [1], food [2], leather [3], metal processing [4], oil and gas [5], and mining [6] industries. The oil spill can occur in oil exploration and production [7], refining [8], and transportation [9] phases in the oil industry, imposing severe environmental and economic impacts [10–12]. Despite the global awareness about the adverse environmental and health effects of the oil spills incidents, they only account for less than 10% of the oil entering the oceans [13]. A comprehensive review on different oil-water separation technologies is available in the literature [6]. Various methods such as gravity settling [13–15], centrifugation [16], gas flotation [17], electric field [18], coagulation [19], membrane filtrations [20], and electrochemical [21] technologies are commonly used for oil-water separation. However, low selectivity, long separation time [13–15], high energy input [16,22], large land requirement [23], and the production of secondary pollutants [24,25] are among the drawbacks of the conventional oil-water separation strategies.

Membrane filtration with special wetting conditions has found tremendous attention in the last ten years. For aqueous phase filtration, hydrophilic and hydrophobic membranes are developed. The hydrophilic and hydrophobic membranes are used in oil-water mixtures by selectively blocking the oil and water phases, respectively [26]. Due to low surface energy of the hydrophobic membranes, the water droplets exhibit a high contact angle with the solid surface. On the contrary, water droplets can wet the surface of the hydrophilic membranes [27]. A special wetting condition that is suitable for oil-water separation is achieved by engineering the surface chemistry and surface roughness characteristics [28].

Surfaces with extreme wetting (e.g., superhydrophilic/superoleophilic) or extreme non-wetting conditions (e.g., superhydrophobic/superoleophobic) have been fabricated and used in various industrial sectors dealing with oil-water separation. A simultaneously superhydrophobic

and superoleophilic (mesh-based) membrane was first introduced in 2004 by Feng et al. [29]. In 2010, the catastrophic *Deepwater Horizon* oil spill occurred in the Gulf of Mexico where 100 million barrels of oil were leaked from a faulty valve [30]. Less than 10% of the water surface oil contamination was recovered by mechanical methods [31]. Such a low recovery efficiency revitalized research on superhydrophobic and superoleophilic membranes and sorbents as the selective tools to effectively capture the oil by rejecting water [32].

Feng et al. [29] used a stainless steel (SS) mesh as a base material, and spray coated the clean mesh with a suspension of polytetrafluoroethylene (PTFE, 30 wt%), polyvinyl acetate (PVAC, 10% as an adhesive), polyvinyl alcohol (PVA, 8% as a dispersant), water (50% as a diluent), and sodium dodecylbenzenesulfonate (SDBS, 2% as a surfactant). The coated mesh was cured at 350 °C. They noticed a water contact angle (WCA) of 156.2°, an OCA of zero for the diesel oil, and a sliding angle of 4°. The general procedure of fabricating superhydrophobic and superoleophilic membranes includes three main steps of: 1) surface preparation involving cleaning and activation, 2) surface roughness modification, and 3) surface chemistry modification. In addition to abrasion and sandblasting as the physical methods for removal of loose materials [33], successive cycles of chemical cleaning (with and without ultrasonication) are applied through using deionized water, acetone, and ethanol to eliminate the contaminations [34]. To prepare the surface for superhydrophobic and superoleophilic coating, surface activation methods such as acid/oxidizers (e.g., H₂SO₄/H₂O₂, H₂SO₄/H₂CrO₄, and H₂SO₄/CrO₃), plasma (air or oxygen), ultraviolet (UV)/ozone, and corona treatments have been used in previous research and engineering activities [35,36]. The micro- and nano- surface roughness types are required for the superhydrophobic and superoleophilic wetting conditions. These hierarchical micro- and nano-roughness schemes are created by top-down methods such as lithography, etching, laser ablation, annealing, and sandblasting [37–45], and/or bottom-up methods such as layer-by-layer (LbL) assembly, hydrothermal, anodizing, electrodeposition, colloidal assembly, rough polymer films, templating, casting and replication, and 3D

printing [38,39,41–43,46,47]. To attain proper surface energy for superhydrophobic and superoleophilic wettability conditions, organic, inorganic, and organic-inorganic composite chemicals are employed to reduce the surface energy [39,42]. Among the inorganic chemicals, silanes are the most widely used chemical solutions to lower the surface energy of superhydrophobic and superoleophilic membranes [48–63], while in the category of organic coatings, stearic acid [23,34,58,64–71], and a variety of thiols [72–81] appear to be dominant chemicals.

Superhydrophobic and superoleophilic functionalized SS [34,50,54,65–68,72,75,82–90] and copper (Cu) [52,53,69,76–80,91–95] mesh-based membranes have found more attention by researchers and engineers, because of their mechanical strengths, and lower pressure drop (compared to the porous membranes). The mesh pore opening and thickness control the flow rate of permeate for a given oil. For example, Shi et al. [96] used a single wall carbon nanotube (CNT) film with a thickness of 30–120 nm and obtained up to three orders of magnitude higher permeate flux (up to 100,000 L/(m².h.bar)), compared to conventional filters. With the recent advancements in 2D materials such as graphene, they are expected to play a key role in fabrication of ultra-thin filters with application to oil-water separation. Creating the hierarchical micro- and nano-roughness is an important step in achieving the super wetting or super non-wetting conditions. Although specific hierarchical micro- and nano-roughness structures are created using lithography [97], femtosecond laser ablation [98], templating [99], casting [100], and 3D printing [101] based on the literature, there are still cost- and scaling limitations to overcome, implying that more research and engineering activities are needed to address these aspects. Methods on the basis of electrochemistry and crystal growth are also used to create superhydrophobic and superoleophilic surfaces with hierarchical micro-nano structures such as spikes [102], flake [34], flower-like [90], coral [95], and pillars [102]; they can be effective alternatives to construct complex roughness patterns. The facile approaches such as colloidal assembly or applying a rough polymer film [49,52,53,55,56,59–61,64,72,75,85–87,89,91,103–111] include reduced number of process steps; they also appear to be as effective as the complex methods for creating hierarchical superhydrophobic and superoleophilic surfaces.

We organize the structure of this review paper in eight sections. Following the introduction, a general background on oil contamination states and available oil-water separation technologies is given in Section 2. In Section 3, the theoretical foundation of different wetting states is provided. Section 4 reviews different methods for modifying surface morphology and chemistry. Section 5 briefly describes existing experimental methodologies to fabricate superhydrophobic and superoleophilic surfaces. In Section 6, we present three forms of superhydrophobic and superoleophilic surfaces that are used in oil-water separation, including film, mesh-based membranes, and porous membranes. Section 7 provides a brief discussion on current challenges and future perspectives of the superhydrophobic and superoleophilic membranes employed in oil-water separation operations. Finally, in Section 8, concluding remarks are listed.

2. Oil contamination and separation technologies

2.1. The source of oil contaminations and their potential hazards

Although unexpected oil spills gain more attention (due to their short- and long-term intensive impacts on the environment), they only account for about 10% of the oil entering the ocean. A majority of the pollutants come from natural seeps, motor oil leakage, run-off oil from paved urban areas, and untreated industrial oily wastewater systems [13]. In terms of size, the oil contaminations can be categorized as free oil, dispersed oil, emulsified oil, and dissolved oil. Free oil can be easily removed by gravity settling and floatation compared to other types of dispersed or emulsified oils [112,113].

The primary sources of the industrial oily wastewater are from food, metal processing (where cooling is required [113]), mining, textile, oil and gas, and chemical industries [6,114]. In general, the concentration of oil in industrial oily wastewater systems varies in the range 10 ppm to 200,000 ppm [112,115]. The development/design of the effluent treatment systems to meet the regulations of discharging oily wastewater is, therefore, imperative [115]. Governmental agencies have established quantitative (e.g., mg/L) and qualitative (no visible sheen in wastewater) measures for the intensity of oil contaminations in water [116]. These standards, may however, differ from one country to another. For example, the United States Environmental Protection Agency limits the daily discharge of oily wastewater up to 42 mg/L for oil and gas industries [117], while this limit is 10 mg/L in China [23].

The oily wastewater can cause odor annoyance, pipeline corrosion, and interference with the proper sewage treatment process, prompting potential health risks by utilizing dissolved oxygen of water [24,118]. Bio-assay data demonstrate that oily wastewater brings acute and chronic toxicity to aquatic invertebrates [119–121]. For example, long-term exposure of both embryo and larvae at an oil concentration of 0.06 mg/L led to a significantly higher mortality rate due to a greater surface area-to-volume ratio [122]. Moreover, exposure of shrimp larvae to an Arctic crude oil of 0.015 mg/L and 0.06 mg/L resulted in a higher mortality and developmental time with increased oil concentration [123].

The hazardous materials found in industrial oily wastewater can affect human health, as well. Detrimental dermatologic and pulmonary effects are reported among the oil industry workers due to exposure to the barium that is used in drilling fluids [124,125]. Furthermore, prolonged exposure to gasoline and its additives can cause cancer and central nervous system toxicity [124]. Due to the hazardous and toxic by-products in the oil and gas industry, the produced water treatment should be considered before discharging the oil contaminations into surface waters [125]. These wastewater sources can also include heavy metals and chemicals used in hydraulic fracturing [126,127]. The Environmental Protection Agency has identified over 1000 chemicals in hydraulically fractured wells [128,129]. Most of these carcinogenic compounds have potential to act as endocrine disrupting chemicals [130,131], which can interfere with hormonal activities [126,132].

2.2. Oil-water separation technologies

In this section, we summarize numerous techniques used for oil-water separation, including gravity settling, centrifugation, gas floatation [15,133], coagulation (and electrocoagulation) [134–138], adsorption, and membrane filtration [114]. Physical, chemical, and biological methods of oil-water separation are the main treatment categories in various industrial and municipal sectors [139]. The chemical methods usually have higher operating costs, demand skilled operators, and require reliable process monitoring and control [1,23]. Gas floatation methods, such as sparging or dissolved gas floatation (pressure-swing mode), can be employed to buoy the oil contamination droplets in a continuum of water. In the gas floatation systems, the gas bubbles (either injected or exsolved) adhere to the dispersed oil droplets to make agglomerates that can float. Researchers have also suggested use of surfactant to increase the removal efficiency of the oil droplets from water in a gas floatation system. In a sparging system, air is usually used in gas sparging due to its abundance. The gas floatation technique is more efficient for oil concentrations <1000 mg/L [15]. The centrifugation increases the driving force to separate oil from oil-water mixture, which is especially beneficial when the oil and water have similar density values; however, the centrifugation is energy-intensive [16,22]. Coagulation is a technology with high adaptability that has been widely used for treating oily wastewaters. This method can also be used for emulsified oil or dissolved oil, where colloids and suspended solids aggregate to form bigger flocs; the precipitated flocs can be removed from the system through sedimentation [140]. Despite the success of coagulation approach, the choice of coagulant and its concentration depend on the

Table 1
Conventional methods for separation of oil from oily wastewaters.

| Treatment | Advantages | Disadvantages | Driving force(s) | Screen Criteria/Remarks | Ref. |
|----------------------|---|---|---|---|----------------------|
| Gravity Settling | <ul style="list-style-type: none"> - Separation of bulk oils - Low energy consumption - Economical | <ul style="list-style-type: none"> - Not efficient for high-density oil | <ul style="list-style-type: none"> - Density difference | <ul style="list-style-type: none"> - In American petroleum institute (API) tanks: oil droplets >150 μm - In plates: oil droplets >50 μm | [13–15] |
| Centrifugation | <ul style="list-style-type: none"> - Efficient for free and dispersed oil - Fast separation | <ul style="list-style-type: none"> - Produces low-quality oil - High energy demand - Fouling - Time-consuming - Expensive maintenance - Space limitations | <ul style="list-style-type: none"> - Relative centrifugal force | <ul style="list-style-type: none"> - Suitable for separating oil droplets with a size of 1 to 15 μm and oil contamination concentration of 20 to 30 mg/L | [13,15,147,148] |
| Gas Flotation | <ul style="list-style-type: none"> - Effective separation - Energy efficient - Simple operation | <ul style="list-style-type: none"> - Requires large air volume - Slow separation | <ul style="list-style-type: none"> - Solubility (in dissolved air floatation) - Density difference | <ul style="list-style-type: none"> - More efficient with a smaller gas bubble, saltier wastewater, and oil droplet >20 μm | [13,15] |
| Electrocoagulation | <ul style="list-style-type: none"> - Effective separation - Economical - Simple operation | <ul style="list-style-type: none"> - Initial high expenditure - Anode passivation - High energy consumption | <ul style="list-style-type: none"> - Voltage | <ul style="list-style-type: none"> - Efficient up to 40 V and 1 cm distance between the electrodes - Energy consumption reduced to half, using voltage pulsation mode | [113,149] |
| Coagulation | <ul style="list-style-type: none"> - Good separation - Flexibility to be combined with floatation for higher separation efficiency | <ul style="list-style-type: none"> - High operating costs - Skilled operator dependent - Secondary pollution problem - Composition dependent | <ul style="list-style-type: none"> - Density difference | <ul style="list-style-type: none"> - More efficient with oil droplets $\geq 50 \text{ mg/L}$ | [23,136,137,150,151] |
| Membrane Filtrations | <ul style="list-style-type: none"> - Fast separation - Pressure dependent | <ul style="list-style-type: none"> - Fouling - High energy demand - High operating costs | <ul style="list-style-type: none"> - Size | <ul style="list-style-type: none"> - Polymeric membranes can be degraded under high temperatures >50 °C | [13,15,112,149] |
| Electrochemical | <ul style="list-style-type: none"> - Controlled coating thickness | <ul style="list-style-type: none"> - Deterioration of electrodes | <ul style="list-style-type: none"> - Oxidation-reduction reactivity - Voltage | | [152] |
| Adsorption | <ul style="list-style-type: none"> - Low chemicals consumption - High removal of oil and chemical oxygen demand - Low cost and low-energy consumption process - Natural sorbents are environmentally friendly | <ul style="list-style-type: none"> - Low hydrophobicity - High water uptake - Low efficiency - High retention time - Secondary pollutant in regeneration stage | <ul style="list-style-type: none"> - Intermolecular and chemical forces (Coulombic, Debye, Kiesom, ion-ion, ion-dipole, covalent, and hydrogen bond) | <ul style="list-style-type: none"> - Not recommended for oil concentrations >50 mg/L - Not suitable for emulsified oil | [15] |

composition of wastewater; this treatment technique is expensive and also produces secondary pollution that can harm aquatic species [24,25]. Electrocoagulation is suggested to improve the separation driving force through coagulation process. The electric field increases the rate of coalescence and accelerates the agglomeration of smaller droplets that move towards the electrodes; these larger agglomerates can be separated easier under gravity forces [141]. Adsorption is another method that is used for oil-water separation, featuring low cost, high efficiency, and small space requirement [117]. Conventional adsorbents, including wool [142], zeolites [143], and activated carbon [144] may have some disadvantages (e.g., low selectivity, low absorption capacity, and difficulty in recycling) in the oil-water separation process [145]. Factors such as pH, temperature, suspended oil, concentration of heavy metal, organic chemical, organic-metal complex, and salinity influence the performance of adsorbents [117].

Filtration is an important strategy for oil-water separation that can separate oil from water based on size and capillarity. Membranes are semi-permeable surfaces that can be natural, synthetic, neutral, and charged that are suitable for separation of suspended solids, macromolecules, multivalent ions, and dissolved and ionic materials, respectively. Their thickness varies from several hundred micrometers to less than 10 nm. Pressure, temperature, and concentration gradients between the feed and permeate are usually the main factors for transferring phases through the membrane [146]. Over the past decade, membrane filtration has become an essential separation technique because of lower energy consumption and lower potentials of producing secondary pollutants. Based on the size of membrane opening, they are classified into microfiltration (MF), nanofiltration, ultrafiltration (UF), and reverse osmosis (RO). Membranes can also be useful to remove stable emulsified oils from water. Typically, the concentration of oil in wastewater is in the range 0.1% to 1%; using MF or UF, an oil separation efficiency of 40% to 70% is usually obtained [1]. Natural oils and fats impose permeate flux impairment, and increased fouling risk [1].

A summary of the driving forces, screening criteria, advantages, and disadvantages of different technologies used for oil-water separation is given in Table 1. Limitations, such as low separation efficiency, generation of secondary pollutants, and presence of oil droplets with various sizes have motivated researchers to develop different effective methods. In recent years, advanced filtration technology with functionalized membranes has gained considerable attention for efficient water purification [112].

3. Surface wetting phenomena

3.1. Wetting states

The surface wetting characteristic is critical in oil-water separation application. The state-of-wetting is commonly characterized by contact angle of liquid on the solid surface in the presence of another fluid (e.g., gas). The equilibrium contact angle was derived from the thermodynamics framework, which relates the contact angle to the interfacial tension according to Young's eq. [153]:

$$\cos \theta_Y = \frac{\gamma_{SG} - \gamma_{LS}}{\gamma_{LG}} \quad (1)$$

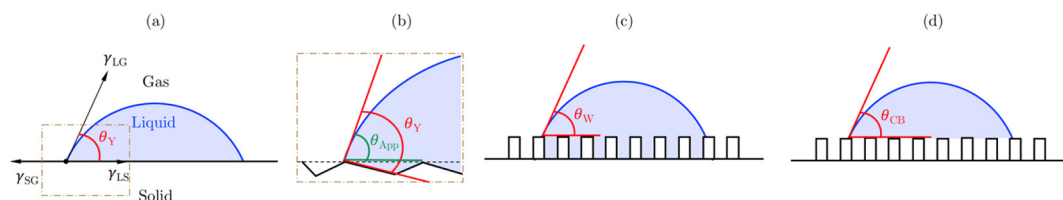


Fig. 1. Three different contact conditions between surfaces and liquids based on (a) Young's state, (b) Wenzel state, and (c) Cassie-Baxter state. The corresponding equations based on these three theoretical approaches help measure the contact angle between the oil droplet and solid surface.

where θ_Y refers to the equilibrium static contact angle of the liquid in the presence of a solid and gas, as depicted in Fig. 1(a); and γ_{SG} , γ_{LS} , and γ_{LG} stand for the solid-gas, liquid-solid, and liquid-gas interfacial tension, respectively.

Wenzel accounted for the effect of surface roughness on contact angle [154]. Let r be the ratio of the actual rough surface area to that of the horizontal projected (smooth) area. The apparent contact angle, which is measured (see Fig. 1(b)), can be correlated to the actual equilibrium contact angle through Wenzel's model [154]:

$$\cos \theta_{App} = r \cos \theta_Y \quad (2)$$

In Eq. (2), θ_{App} represents the apparent contact angle and r denotes the surface roughness parameter. The extreme wetting and non-wetting states are shown in panels (c) and (d) of Fig. 1, respectively; Fig. 1(c) illustrates the Wenzel state, while Fig. 1(d) shows the Cassie-Baxter state [155]. In Eq. (2), the apparent contact angle can be replaced from the Wenzel model (e.g., $\cos \theta_W = r \cos \theta_Y$). In the Cassie-Baxter state, pockets of gas (e.g., air) are trapped below the liquid surface interface; these trapped air pockets do not allow the rough solid surface to be wetted by the liquid. The contact angle on the basis of the Cassie-Baxter model (θ_{CB}) is correlated to equilibrium contact angle from Young's model through the following correlation:

$$\cos \theta_{CB} = f_1 \cos \theta_Y - f_2 \quad (3)$$

where f_1 and f_2 are the areas of the solid and gas under the liquid drop per unit projected area below the drop, respectively. According to Milne and Amirfazli [156], the simplified form of the Cassie-Baxter model that is conventionally used in the literature is only valid for a limiting case where the pillar top surfaces (exposed to the liquid drop) are flat. This means that in general, $f_1 + f_2 \geq 1$ [156]. The surface roughness of lotus leaves provides Cassie-Baxter non-wetting state to water droplets where only about 2–3% of the water droplets becomes wetted by the leaves.

With regard to water droplets, this extreme non-wetting state is known as the lotus effect, super non-wetting or superhydrophobicity. It has been theoretically shown that both the Wenzel and Cassie-Baxter models are valid when the size of liquid droplet is much larger than the size of surface roughness (or heterogeneities) [157]. Kim et al. concluded that regardless of the wetting state, the contact angle at local minimum is correctly estimated from the theory when the size of the liquid drop is 40 times (or more than) the characteristics length of roughness [158]. Intermediate wetting state (so-called Marmur state, mixed state, and penetrating state) is also possible where the air pockets in the rough pore spaces below the liquid drop are partially wetted by the liquid. Moreover, the co-existing of the Wenzel-Cassie states and the transition from one state to another are possible, suggesting that the measured contact angle may be a meta stable condition, which can be perturbed (e.g., through vibration) towards an equilibrium stable condition [159].

For a water droplet on a solid surface, in the presence of air, the contact angle of 90° is the threshold for wetting (hydrophilic) and non-wetting (hydrophobic) states. Surfaces with a static WCA greater than

90° are hydrophobic and those with a static WCA < 90° are hydrophilic. In general, two criteria are required for a surface to be hydrophobic: 1) high contact angle and 2) low rolling angle [160]. A comprehensive review of the hydrophobic surfaces by Li et al. is available in the literature [39]. The term *superhydrophobic* is used for surfaces with extreme non-wetting condition for water. Initially, *superhydrophobicity* was used in 1996 by Onda et al. [161]; it is since accepted as a common term among the scientific community [162]. The superhydrophobic surfaces are generally identified with a static WCA > 150° and a contact angle hysteresis < 10° [163]. In contrast, *superhydrophilicity* refers to a state in which the surface is completely wetted with water. The terminology *superhydrophilicity* was first used in 2000 by Fujishima et al. [164]. This extreme wetting condition is characterized by a static WCA < 10° [165]. Likewise, oleophilic and superoleophilic states are used for surfaces wetted by the oil phase; oleophobic and superoleophobic terms are used for surfaces that cannot be wetted by oil.

3.2. Wetting states at molecular level

At molecular level, the functional groups control the wettability of the surface. For instance, $-\text{OH}$, $-\text{COO}^-$, $-\text{COOH}$, $-\text{NH}_2$, $-\text{NH}_3^+$, $-\text{OSO}_3^-$, and $-\text{OSO}_3\text{H}$ can increase the surface energy and exhibit hydrophilic features, while fluorocarbon, hydrocarbon or silicone-based polymers decrease the surface energy, promoting hydrophobicity [166]. An excellent review of the hydrophobic surfaces was conducted by Drelich et al. [167]. From a molecular perspective, the most hydrophilic surface is obtained when the exposed functional group is capable of forming hydrogen bonding, such as $-\text{OH}$, $-\text{COOH}$, and $-\text{POOH}$; however, without surface roughness, a zero contact angle of water is not observed on these surfaces [167]. Similarly, the ionizable functional groups also provide hydrophilicity. They will dissociate to form highly hydrated ions, such as carboxylate, sulfonate, and alkyl ammonium ions [168]. For these functional groups, the wettability will be significantly affected by the pH; the surface will become more wetted, in general, if the functional groups are more ionized [169]. Thus, for the acidic and basic moieties, the surface will become wetted at higher and lower pH values, respectively. It should be noted that pH has no effect on the wetness characteristic when the functional group is not ionizable [169]. Furthermore, molecules with aliphatic (linear) chains have better hydrophobicity than branched or aromatic molecules, owing to the steric effect of the

neighboring branches and aromatic ring that reduces the hydrophobic interactions with the water molecules [170]. Generally, surfaces tend to become more hydrophobic, and temperature also magnifies the hydrophobicity [170]. Oils typically are non-polar that may only contain a few polar groups and feature a low dielectric constant. As a result, they may not interact through van der Waals or hydrogen bonding to wet the surfaces with polar functional groups [170]. On the other hand, the hydrophobic surfaces have less interaction with water as a result of non-polar functional groups (e.g., F) at the surface [170]. In general, the hydrophobicity is affected by the length and shape of the functional groups [170]; by increasing the length of an alkyl chain, the surface would be more hydrophobic [171,172].

Brown and Bhushan [173] used LbL surface modification and achieved all four states of the wetting (e.g., superhydrophobic-superoleophilic, superhydrophobic-superoleophobic, superhydrophilic-superoleophilic, and superhydrophilic-superoleophobic), as depicted in Fig. 2. In their study, the glass surface was used as the building block onto which polydimethylsiloxane (PDMS) was deposited as a binder, a layer of 7 nm SiO_2 linked between the nanoparticles (NPs) and the functional layer (FL). Without any FLs, the surface with binder and NPs exhibited superhydrophilic and superoleophilic for which the contact angles of water and hexadecane were both zero [173]. Using methyltrichloro silane as the FL, the surface exhibited superhydrophobic and superoleophilic where the contact angle of water was 161°, while that of hexadecane was zero. By using 1H, 1H, 2H, and 2H-perfluorosilane in the FL, the surface becomes superhydrophobic and superoleophobic where the contact angles of water and hexadecane were 163° and 157°, respectively. Finally, utilizing an amphoteric fluorosurfactant in the FL (DuPont™ Capstone™ FS-50), the surface showed superhydrophilic and superoleophobic properties characterized with a contact angle of <5° for water and a contact angle of 157° for hexadecane.

Similar to the wettability states presented in Fig. 2, the combined affinities of a surface for water and oil allow for four different types of membranes, based on wettability. These membrane types are demonstrated in Fig. 3. The first category is oleophilic and hydrophilic (simultaneously), as displayed in Fig. 3(a). This type of membranes permeates both water and oil, and is not common in oil-water separation application but can be used to separate solids. The second category is hydrophilic and oleophobic, as shown in Fig. 3(b). This type of

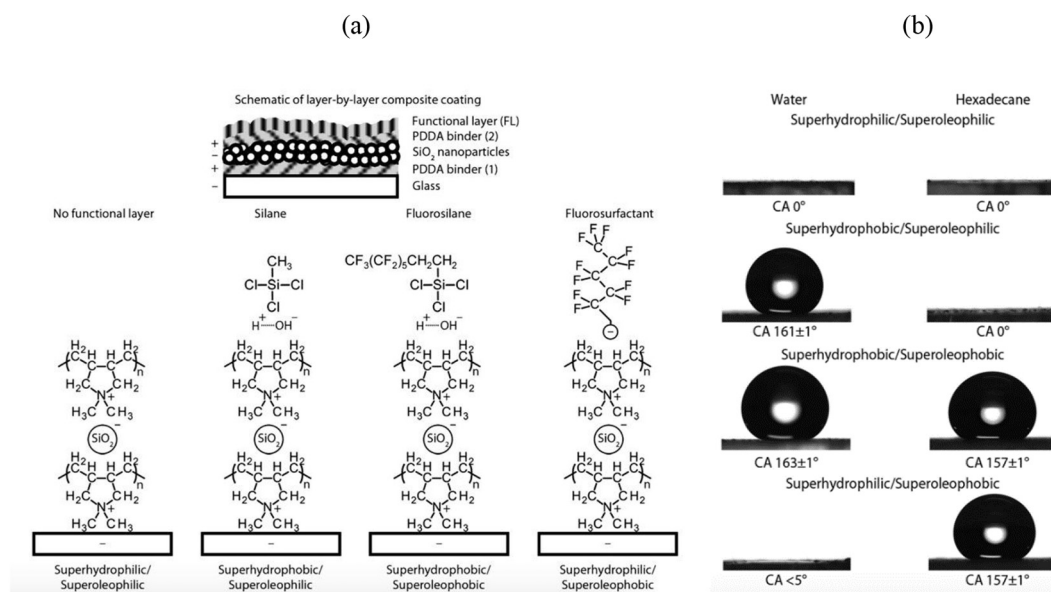


Fig. 2. A comparison of the effect of final surface coating in the LbL modification of a glass surface, using binder PDMA, SiO_2 NPs, and FLs, such as silane, fluorosilane, and fluorosurfactant: (a) chemical representation of the layers and (b) contact angle of water and hexadecane on different surfaces [173].

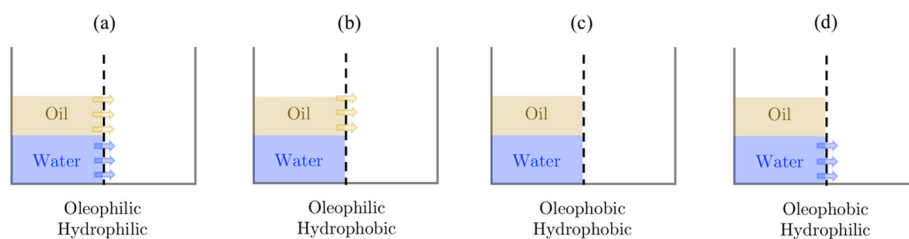


Fig. 3. Illustration of membrane types for oil-water separation based on wettability: (a) oleophilic and hydrophobic; (b) oleophobic and hydrophilic; (c) oleophilic and hydrophilic; and (d) oleophobic and hydrophobic.

membranes was introduced in 2004 by Feng et al. [174]; they have since found great applications in oil-water separation for oil removal. The third category is oleophobic and hydrophobic (see Fig. 3 (c)); these membranes are also not common in oil-water separation, but they can be potentially employed to separate the gas phase. The oleophobic and hydrophilic membranes are the last category. These membranes have been conventionally used in oil-water separation to separate oil by removing water from an oil-water mixture.

3.3. Superhydrophobic and superoleophilic wettability state

The WCA and OCA on membrane surface are important characteristics for oil-water separation applications [175]. The membrane wetting is governed by surface geometry (morphology) and surface free energy [74]. The effect of surface free energy of the interacting phases on the contact angle is given by Young's eq. [153]. The condition for superhydrophobicity is commonly identified with WCA $> 150^\circ$ and small contact angle hysteresis. The first superhydrophobic surface was fabricated in 1996 using fractals and alkyl ketene dimer for which WCA = 174° was achieved [161]. This superhydrophobic condition cannot be achieved solely by the modification of the surface chemistry. In fact, for obtaining WCA $> 120^\circ$, hierarchical micro- and nano-surface roughness is required [176]. The role of surface

roughness is assessed via the liquid contact angle on a flat substrate (θ_Y). Wenzel's eq. [154] predicts that the wetting is enhanced by the surface roughness when $\theta_Y < 90^\circ$ (hydrophilic conditions); the water wettability is lowered by the roughness when $\theta_Y > 90^\circ$ (hydrophobic conditions).

The Cassie-Baxter wetting condition can result in the superhydrophobicity when air is trapped in the micro- and nano-channels, causing the extreme non-wetting condition. Due to the difference between the surface tension of water-air ($\gamma_{WA} = 72 \text{ mN/m}$) and that of the oil-air (usually $\gamma_{OA} < 35 \text{ mN/m}$), it is possible to control the wettability of a surface according to Young's eq. [153]. Hydrophilicity and oleophilicity refer to the conditions in which the surface energy of a substrate is higher than both oil and water phases. Hence, wetting the substrate by either oil or water is unavoidable. Conversely, a substrate with surface energy less than oil and water demonstrates hydrophobicity-oleophobicity properties. Because of this surface energy contrast for water and oil (usually $\gamma_{OA} = 20\text{--}30 \text{ mN/m}$), most hydrophilic surfaces are also oleophilic [28,37]. Most of the low surface energy materials that are usually hydrophobic still show a greater surface energy than oil ($> 35 \text{ mN/m}$); these surfaces tend to be oleophilic ($\theta_{OA} < 5^\circ$) [38,177]. To meet the superhydrophobic and superoleophilic condition, the surface energy of the final coating should be in the range $30 \text{ mN/m} < \gamma < 72 \text{ mN/m}$ [28].

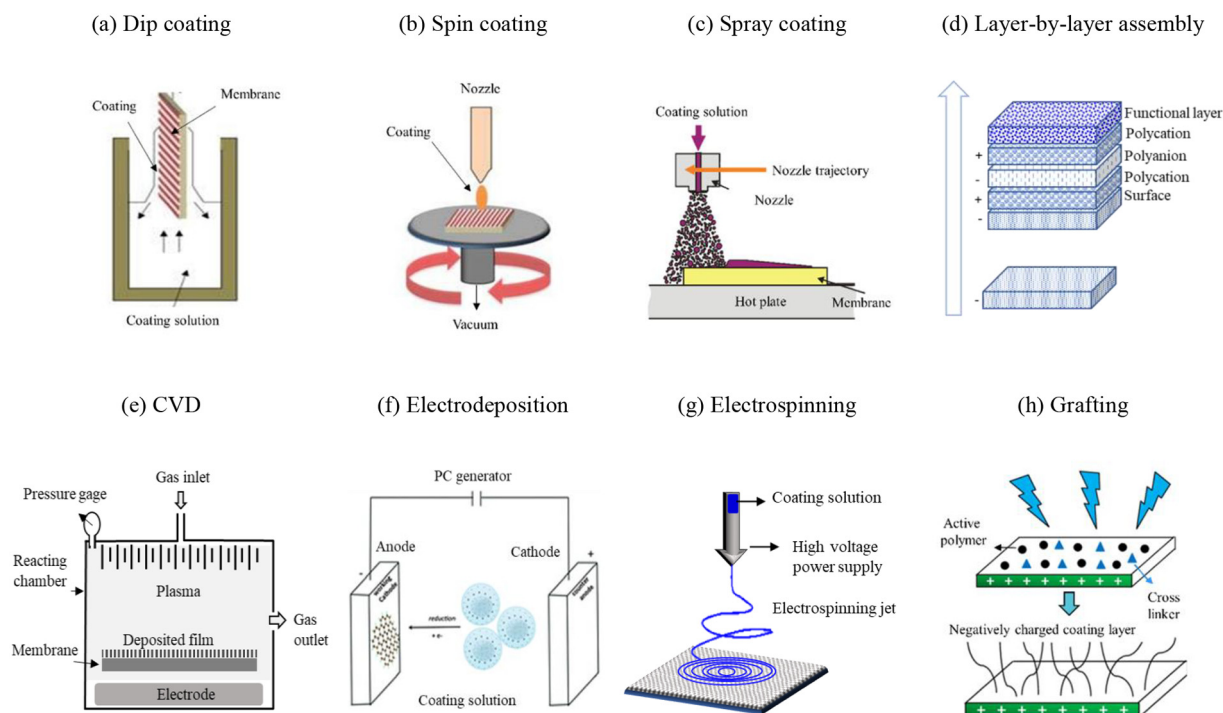


Fig. 4. Schematic of conventional coating mechanisms: (a) dip coating, (b) spin coating [213], (c) spray coating [214], (d) layer by layer assembly, (e) chemical vapor deposition, (f) electrodeposition [215], (g) electrospinning [216], and (h) grafting [217] used for the construction of superhydrophobic-superoleophilic membranes.

4. Surface wetting modification methods

Different methods have been used to alter the wettability towards superhydrophobic and superoleophilic. In this section, some known techniques for construction of superhydrophobic and superoleophilic are briefly discussed. These common methods include dip coating [48,49,103,178], spray coating [91,179,180], spin coating [181–184], sol-gel [50,185,186], LbL [187,188], vapor pressure deficit (VPD) [189–191], chemical vapor deposition (CVD) [82,83,192,193], electro-deposition [72,194], electrospinning [73,104,105,195], acid-base treatment [196,197], grafting [198–200], thermal [74,106,201], plasma [51,202–204], ion beam irradiation [205–207], and femtosecond laser [166,208]. Typically, fabricating of superhydrophobic and superoleophilic surfaces can be achieved via either using low surface energy materials or adding roughness or both. The performance of a membrane is affected by important features, such as pore size, wettability, and surface energy [209]. The wettability of a solid surface is controlled by its geometry and chemical composition [73,210–212]. Fig. 4 depicts a schematic of eight coating techniques, including dip coating, spin coating, spray coating, layer-by-layer assembly, CVD, electrodeposition, electrospinning, and grafting.

Dip, spray, and spin coating: In the dip coating, a substrate is immersed into the coating solution at an optimized immersion speed; due to the capillary effects, the coating solution adheres to the substrate surface. A drainage stage is followed to drain excess coating films. Finally, upon solvent evaporation, a gel layer forms on the substrate surface [114,218,219]. The dip coating method can be applied on diverse surface morphologies, even on complex heterogeneous surfaces. The type of surface functional groups, withdrawal rate, number of dipping cycles, humidity, temperature and the physical properties of the coating solution (e.g., density, viscosity, surface tension, and pH) affect the coating quality [114]. Typically, in dip coating the coating thickness is between 0.1 and 100 μm [114].

Spray coating is a promising technique to fabricate thin-film layers of organic materials. It starts by an atomization stage where the coating solution is sprayed onto a substrate surface. To provide a uniform coating composition, the surface temperature should be constant during the spraying. Deposition process gradually produces a thin film of the coating material upon spraying. Spray coating is an effective and low-cost approach that produces high-quality thin films at industrial scales [220]. Spray coating is also been to produce different SHSO surfaces [75,85].

Spin coating also produces uniform thin films on a rotating substrate. This method allows for a controlled coating thickness through repeated cycles. The ultimate film thickness depends on the concentration of the polymer solution, solvent evaporation rate, surface tension, and spinning speed [221,222]. The spin coating method is extensively employed for manufacturing superhydrophobic surfaces [181–184].

Layer-by-layer assembly: The LbL technique is a simple and cheap deposition approach that constructs thin film on surfaces by electrostatic interactions between different layers [39,187,223]. The process allows for a coating with controlled thickness and functionality [39]. Surface roughness can also be provided using NPs in the layers that can be obtained at room temperature [187]. An in-depth review of the LbL technique is given by Ariga et al. [224]. The LbL surface modification can be conducted through immersion, spin, spray, and electrochemical techniques [225]. In general, the LbL approach can be combined with other surface modification methods to attain the desired surface structure and chemistry.

Chemical and physical vapor deposition: Physical vapor deposition (PVD) and CVD methods can effectively deposit thin films onto the surface of a substrate, through vaporization, condensation, and deposition steps that are performed at atomistic scales. In the PVD, the vaporized atoms or molecules from a liquid or solid phase are transferred through a vacuum or low-pressure system to be deposited and condensed onto the surface of a substrate. This method includes various

operations, such as plasma sputter bombardment and high-temperature vacuum evaporation. The ultimate thickness of the thin film varies between 1 and 1000 nm [226]. In the CVD method, solid materials from a gaseous phase deposit onto the surface of the heated substrate through chemical reactions, forming thin films, powders, or crystals. CVD provides a uniform coating with desired physical and chemical properties [114]. Apart from expensive raw materials, the gaseous phase in this method can be toxic, flammable, explosive, and corrosive that triggers hazardous effects.

Electrodeposition: is a versatile conventional surface modification technique, containing an electrochemical cell with a reference electrode (cathode) and a counter electrode (anode) to generate a controlled current at a given voltage. Darmanin et al. [227] provided a systematic review on electrochemical methods for making hydrophobic surfaces. Electrodeposition can be used to produce a variety of nanostructures and morphologies for different applications, including oil-water separation. It is an energy-efficient and cost-benefit method that is usually conducted at ambient temperature. The main challenge in electrodeposition techniques is related to the fabrication of the template, since the shape and size of coated film strongly depend on the electrode substrate characteristics [228].

Electrospinning: is an efficient strategy to construct micro-nano fibrous with controlled features, such as fiber diameter and structure [229]. This technique uses a high-voltage (5 to 50 kV) and a syringe pump to emit a polymer or sol-gel from a spinneret on the surface of the substrate at a constant injection rate [230]. Superhydrophobic surfaces can be fabricated through surface modification of electrospun membranes/fibers; alternatively, electrospun deposition of superhydrophobic fibers onto various materials can also be employed to functionalize them. The typical limitations of electrospinning for surface modification are low mechanical integrity and separation efficiency for gravity-based oil-water separation [231]. Applications of electrospinning to manufacture surfaces with a special wettability are widely reported for effective oil-water separation [105,232,233].

Grafting: is a method in which, either polymer is added to the surface (grafting-on) or monomers are polymerized to the surface through an initiation (grafting-from). In grafting, a polymer can be attached to another polymer surface [209,234]; the grafting can be proceed in repeated cycles.

Sol-gel: is a mature and relatively inexpensive strategy for modifying the surface morphology and surface chemistry; this method is particularly suitable to prepare functionalized metal, and metal oxide NPs and composites. The sol-gel technique includes five steps [235], namely, 1) precursor preparation, 2) hydrolysis, 3) condensation, 4) growth of particles, and 5) agglomeration.

Thermal approach: is a process in which fine molten or semi-molten particles are sprayed onto the surface [236,237]. The source of energy for this method can be electrical arc and combustion. This technique offers coating thicknesses from several μm to over 100 μm [236]. Various techniques, such as wire-arc, high-velocity oxy-fuel, and plasma spraying are used in this method. Different materials, such as ceramics, plastics, alloys, and composites can also be used with thermal method. Residual stress can, however, negatively affect the stability (and life) of the coating layer and can be considered as a major disadvantage of the thermal coating methods [238].

Plasma irradiation: is one of the most widely used method for the modification of surfaces [234]. Plasma is a partially ionized gas, containing free electrons, ions, and neutral species like molecules, atoms, and radicals that are formed by subjecting a gas to energy for electron generation. The electrons can be accelerated in high electric fields by removing them from neutral molecules, causing the generation of free radicals, atoms, and ions. The ion bombardment of surface with high energy levels can trigger a random fragmentation on the surface, further etching or depositing chemicals onto the adsorbent surfaces [239]. One of the advantages of plasma treatment is its flexibility to create different surfaces [240].

Table 2
Features of different coating methods used for surface modification.

| Coating Application Method | Process | | | | | | Coating | | | |
|---------------------------------|---------|-------|------|-----------|-------------|------------|---------|---------------------------|--------------|---------|
| | Simple | Cheap | Fast | Flexible* | Large scale | Repeatable | Uniform | Controllable [†] | Hierarchical | Precise |
| Chemical Vapor Deposition (CVD) | • | | • | • | | • | • | | | • |
| Dip Coating | • | • | | • | | | | | • | |
| Electrodeposition | | • | | | • | • | | • | • | |
| Electrospinning | • | • | | | | | | | • | |
| Femtosecond Laser Irradiation | | | | | | • | | | • | • |
| Grafting | | | | • | | | | • | • | |
| Ion Beam Irradiation | | | • | | | • | | • | | • |
| Layer-by-Layer Assembly | • | | | • | | | • | • | • | |
| Plasma Irradiation | | | • | • | • | • | | | | • |
| Sol-Gel | • | | | • | | | • | | • | |
| Spin Coating | • | • | | | | • | • | • | • | |
| Spray Coating | • | | • | | • | | | | • | |
| Thermal | | • | • | • | • | • | | | • | |

* Flexibility in terms of handling complex geometries or multiple materials.

[†] In terms of thickness.

Ion beam irradiation: shoots a high-energy level ion onto the surface of a substrate to generate hydrophobicity features. This method is controllable, fast, and environmentally friendly in which the type of ion beams and energy can be changed to achieve desirable surface wetting. For example, high energy ions collisions with one-layer carbon atoms of graphene can induce a graphene nanopores [241].

Femtosecond laser irradiation: Since the invention of lasers in 1960, they have found a wide range of applications, including oil-water separation [242,243]. A femtosecond laser emits ultrashort optical pulses to generate hierarchical micro- and nano-structures onto the surface of SS [98], polymers [244,245], silicon [246], titanium [247], and aluminum [248]. This method has an advantage of not using chemicals for surface modification. It is also more stable than chemically-treated surfaces [166].

A summary of the advantages of different coating methods used in the literature of SHSO surfaces is provided in Table 2. The coating process and coated layer features are screened (see Table 2). For each process, the features such as simplicity, cost-effectiveness, processing time, flexibility in handling complex geometries and various materials, large-scale application potential, and repeatability of the coating process are included. For the coated layer characteristics, features such as uniformity of the coating layer, controllability of the coating thickness and functional groups, potentials in creating hierarchical micro- and nano-roughness, and the precision of coating are listed.

5. Fabrication of superhydrophobic and superoleophilic membranes

Superhydrophobic and superoleophilic have found great interest in oil-water separation application. The key features of the surface, such as energy, roughness, charge, and functional groups can be engineered to promote simultaneous hydrophobicity and oleophilicity [249]. As it was discussed earlier, by solely changing the surface chemistry of smooth surfaces, a superhydrophobic surface with a WCA > 150° cannot be achieved. Experimental works have shown that even hexagonal close-pack of aligned -CF₃ functional groups (that have very low surface energy) on smooth glass surface results in a maximum WCA = 119° [250]. Hierarchical micro- and nano surface roughness are required to produce superhydrophobic surfaces; without surface roughness, the

superoleophilic condition cannot be achieved. A schematic of the process to produce simultaneous superhydrophobic and superoleophilic surface as well as their classification with application to oil-water separation are given in Fig. 5. As it is clear from Fig. 5, the wettability modification process is usually conducted in three stages, including pretreatment, morphology modification, and surface chemistry modification. The pretreatment includes steps, namely; cleaning (physical and chemical) and activation where contaminations and weak hydrophilic oxidized films are removed, and new and reactive hydroxyl groups are attached to allow for a better surface chemistry modification. To prepare hierarchical micro- and nano surface roughness, top-down and bottom-up methods are used to create roughness by either removing or adding rough features, respectively. In surface chemistry modification, new chemicals are bonded to the surface. These chemicals can be inorganic, organic, and/or hybrid inorganic-organic materials.

5.1. Pretreatment

The pretreatment process prepares the surface for a better bonding of low surface energy materials, which is usually required to achieve a superhydrophobic and superoleophilic surface. The pretreatment stage generally includes physical and chemical cleaning, and activation. The physical cleaning removes weak boundary layers (loose material) through methods such as abrasion and sandblasting. Similarly, the chemical cleaning stage removes organic surface contaminations as well as old oxide layers [33,52]. For chemically cleaning of the surface, usually successive cycles of detergent, ethanol, and acetone are implemented to remove the organic contaminations [34]. Utilizing ultrasonic cleaning helps to scrub the surface with ultrasonic energy, leading to a high-quality cleaning [33]. 5–15 min ultrasonic solvent cleaning cycles are usually used in the pretreatment stage [251,252]. A diluted acid solution is used to remove the old surface oxides [77]. After the sample is cleaned, it is usually oven-dried at 80 °C for about 1 h [64] or dried using N₂ gas [76].

The physical and chemical cleaning methods commonly follow an activation stage in which the old oxidized surfaces are removed and replaced by new and reactive oxide layers. A schematic of the activation process mechanism is depicted in Fig. 6. The fresh and reactive

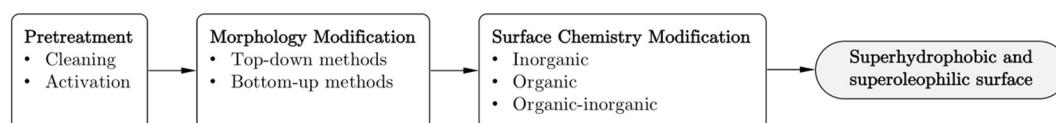


Fig. 5. A typical process to fabricate superhydrophobic and superoleophilic surfaces.

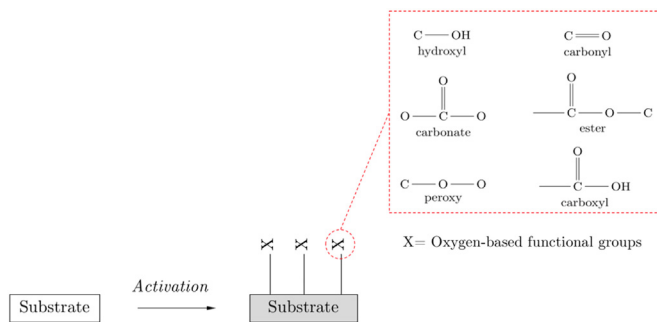


Fig. 6. A schematic of activation of oxygen based functional groups.

functional groups will be of critical importance in the surface energy control by chemicals such as silanes. In general, the activation of polymers results in the polar oxygen-based functional groups, as shown in Fig. 6 [167,253].

In general, strong oxidizers, such as a mixture of concentrated H_2SO_4 (98 wt%) with concentrated H_2O_2 (30 wt%) or that with CrO_4 (or H_2CrO_4) are used. The mixture of concentrated sulfuric acid and hydrogen peroxide (2:1–7:1) is called piranha solution [33], which is highly reactive and should be handled with extra care [33]. Oxygen or air plasma can be alternatively employed in the activation process. Other methods, such as the use of UV radiation, UV radiation with ozone, and corona method (mainly for plastics) are also utilized for the activation process. The use of air or oxygen plasma has found to be a promising approach that not only gives a superior cleaning and activation, but also provides advantages over wet chemical and UV/ozone activation in terms of the energy input, safety, hazardous waste, corrosion, thermal load, processing time, and versatility in handling a broader range of material surfaces [35]. However, in comparison, the O_2 (or air) plasma activation technique contains a higher number of variables to be optimized.

A comparison between the pretreatment methods was conducted by Lukose [254], where the surfaces of Au and Ag films were treated by different methods, including UV irradiation, piranha solution, oxygen plasma, and air plasma. The results for Au film are presented in Fig. 7. The contact angle of water on Au exposed film after seven days is 106° , as shown in Fig. 7(a). Upon 10- and 20-min UV irradiation, the contact angle decreases to 78° and 70° , respectively. Pretreatment with piranha solution and oxygen plasma makes the surface more hydrophilic, as seen in Fig. 7(d)–(f). The WCAs on surfaces treated with piranha and oxygen plasma are similar. However, air plasma is found superior to all methods, giving a contact angle of 15° (see panel (f) of

Fig. 7) [254]. Similarly, for the case of Ag film, the minimum WCA of 18° was obtained in the case of oxygen plasma [254].

5.2. Modifying surface morphology

Surface roughness is found to exhibit a significant role in the wetting characteristics. The effect of hierarchical surface roughness on wetting behavior is studied in several research and review papers [255–261]. The hierarchical surface roughness helps to achieve the superhydrophobicity condition, as explained by the Cassie-Baxter wetting condition. Inspired by lotus leaf, researchers employed the biomimetic hierarchical surfaces to create materials with super-wetting or non-wetting characteristics for different applications, as reported in the literature [41,177,262–264].

In general, methods of surface morphology modification can be divided to top-down and bottom-up methods. In the top-down category, lithography, etching (using chemicals, laser or plasma), annealing, and sandblasting can be included [37–45]. Bottom-up methods of creating hierarchical structures include various approaches, such as LbL assembly, anodizing, hydrothermal, electrodeposition, electrospinning, colloidal assembly, rough polymer films (with micro- and nano roughness features), templating, replication, casting, and 3D printing [38,39,41–43,46,47]. Samples of modified surface morphology obtained by top-down methods (panels (a)–(d) of Fig. 8) and bottom-up methods (panels (e)–(h) of Fig. 8) are given, which demonstrate hierarchical micro- and nano-roughness morphology, as required for the superhydrophobic condition.

5.3. Surface chemistry modification

After developing micro- and nano- surface roughness, the surface energy of the building block of the hierarchical material should be controlled to meet the condition of superhydrophobic and superoleophilic; to achieve this criterion, the surface energy should be between the surface energy of oil and water, as explained earlier [28]. If the surface energy of the building block already satisfies this range, further modification is not required. Otherwise, an additional process will be conducted through different methods, such as CVD, spray coating, spin coating, dip coating, LbL assembly, sol-gel, anodizing, hydrothermal, electrospinning, and plasma (laser and UV) irradiation. In general, coating chemicals can be divided into inorganic (metals and non-metals), organic coatings, and hybrid organic-inorganic materials [39,42].

In the class of inorganic coatings, the silicone-based chemicals are the most popular choice. This list includes different types of silanes [42] such as PDMS, methyltrichlorosilane (MTS), trimethyltrichlorosilane,

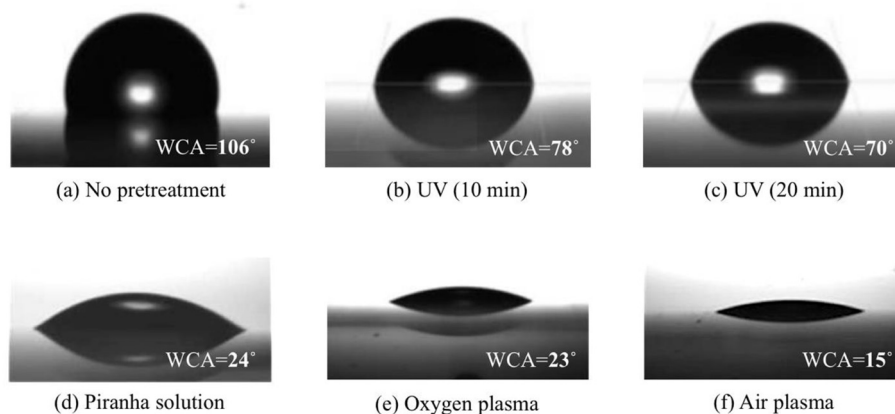
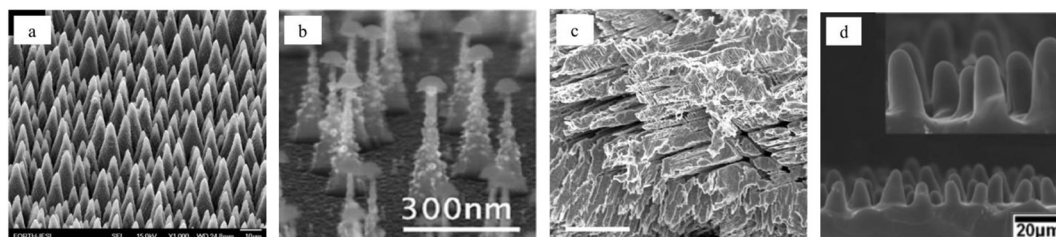


Fig. 7. Effect of pretreatment method on contact angle of water on Au coated film: (a) no pretreatment, (b) 10 min UV irradiation, (c) 20 min UV irradiation, (d) piranha solution, (e) oxygen plasma, and (f) air plasma [254].

(Top-down methods)



(Bottom-up methods)

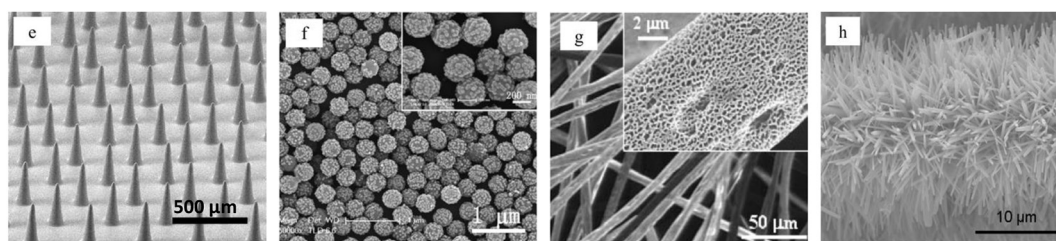


Fig. 8. Different methods of surface morphology change with application to oil-water separation. Top-down methods: (a) femtosecond laser irradiation on the platinum surface [265], (b) oxygen and bromite plasma etching and reactive ion synthesis [266], (c) SS HF acid etched [267], and (d) lotus-like papillary structure using soft lithography of polydimethylsiloxane (PDMS) [268]. Bottom-up methods: (e) template technique to create PDMS cone array [269], (f) raspberry-like colloidal system of PAA-functionalized PS core with silica NPs [270], (g) PS in tetrahydrofuran (THF)/dimethylformamide (DMF) solvent through electrospun [271], and (h) hydrothermal approach for creating ZnO nanorods [272].

octadecyltrichlorosilane (OTS), MTES, TEOS, perfluoroalkylsilane (PFAS), perfluorooctyltriethoxysilane (PFTOS), hexadecyltrimethoxysilane (HDTMS), HMDS, mercaptopropyltrimethoxysilane (MPTMS), aminoethylaminopropyl polydimethylsiloxane (AEAPS), 1H,1H,2H,2H-perfluorooctyl trimethoxysilane (PFOTMS), and vinyltriethoxysilane (VTES). Other inorganic chemical coatings include graphene, graphene oxide, CNTs, and metallic and metal oxides chemicals (Ag, Al, TiO₂, and CuO). In the category of organic polymer coatings, thiols are the mostly used coatings, for example, *n*-dodecanethiol (DDT), dodecanethiol, *n*-octadecylthiol, and 1H, 1H, 2H, and 2H-perfluorodecanethiol (PFDT). Other important organic polymers include PE, PTFE, Teflon AF® or amorphous fluoropolymer, polyvinyl (PV), polyvinylchloride (PVC), polyvinylidene fluoride (PVDF), polystyrene (PS), polybenzoxazine (PBZ), polyimide (PI), and polyethylenimine (PEI).

As described in Fig. 5, three stages of pretreatment, morphology modification, and surface chemistry modification are required to achieve a superhydrophobic and superoleophilic surface. A summary of different methods for wettability alteration to superhydrophobic and superoleophilic with application to oil-water separation is provided in Table 3.

6. Superhydrophobic and superoleophilic membranes for oil-water separation

Superhydrophobic and superoleophilic membranes were first proposed in 2004 to be employed for oil-water separation [174]; since then, there are extensive studies on different superhydrophobic and superoleophilic membranes and sorbents. In this section, we only focus on the membranes. First, we classify three different types of superhydrophobic and superoleophilic membranes based on the pore structure, namely, mesh, porous, and film (see Fig. 9). Each category is divided into sub-categories based on different attributes, found in the literature. This classification is by no means unambiguous. For example, when functionalizing a metal mesh by colloidal assembly, the surface roughness created by the micro- and NPs can grow a porous structure onto a 2D metal mesh; however, we classify it as a mesh-type superhydrophobic and superoleophilic membrane. In the same fashion,

we consider a functionalized fabric, single-layer graphene membrane as a mesh-type membrane.

6.1. Mesh-based superhydrophobic and superoleophilic membranes

Over the last decade, functionalized meshes with special wettability have gained exceptional interest for oil-water separation purposes. The metallic mesh material provides good mechanical strength, flexibility and thermal resistance with a low-cost, featuring an excellent substrate to fabricate superhydrophobic and superoleophilic filters [273]. In this section, we classify the mesh-based superhydrophobic and superoleophilic membranes based on the type of substrate (e.g., SS, Cu, and others). For each substrate, the chemicals and coating methods used for surface modification are discussed. A summary of the mesh-based superhydrophobic and superoleophilic membranes with application to oil-water separation is presented in Table 4.

Stainless steel: Recently, there has been considerable interest to fabricate superhydrophobic and superoleophilic coatings on SS mesh for obtaining a high separation efficiency for water-oil mixtures [68,274]. The idea of using superhydrophobic surfaces to fabricate superoleophilic SS mesh-based membranes was proposed in 2004 by Feng et al. [29]. They coated the SS mesh with a suspension of PTFE particles (30 wt%), PVAC as an adhesive (10 wt%), PVA as a dispersant (8 wt%), SDBS as a surfactant (2 wt%), and water as a thinner (50 wt%) [29]. They cleaned a SS mesh (50–200 μm) and then sprayed the coating solution onto the mesh and cured it at 350 °C. The coated mesh featured a WCA = 156.2 ± 2.8°, and a sliding angle of 4°. Furthermore, the OCA using diesel oil showed a zero value. However, the proposed technique has been criticized due to its low thermal and mechanical stability [249]. The SS mesh-based superhydrophobic and superoleophilic membranes are extensively cited in the literature [34,50,54,65–68,72,75,82,84–88,103,185,275–277]. The PTFE-coated SS meshes are found to promote superhydrophobicity and superoleophilicity, leading to a reasonable oil-water separation efficiency [82,87,89]. By default, PTFE exhibits hydrophobicity (with a WCA in the range of 98° to 112°). Also, it has excellent thermal, chemical, and abrasion resistances; thus, PTFE-coated SS mesh can be used under

Table 3

A summary of available pathways for wettability alteration to superhydrophobic and superoleophilic with implication of oil-water separation.

| Process | Common materials and methods | Objectives |
|---------------------------------|---|---|
| Pretreatment | <p>Physical cleaning [33]</p> <ul style="list-style-type: none"> abrasion and sandblasting <p>Chemical cleaning [34]</p> <ul style="list-style-type: none"> solvents (deionized water, acetone, ethanol, chloroform, and detergent) ultrasonic-aided cleaning <p>Activation [35,36]</p> <ul style="list-style-type: none"> piranha solution (H_2SO_4/H_2O_2) H_2SO_4/H_2CrO_4 (or CrO_3/H_2SO_4 solution) air or O_2 plasma UV/ ozone Corona treatment (for plastics) | <p>Removing weak bonds and loose material</p> <p>Removing organic surface contaminations</p> <p>Eliminating the old oxidized layers (or chlorinated, and fluorinated surfaces) and growing fresh and reactive oxide layers.</p> |
| Surface morphology modification | <p>Top-down methods [37–45]</p> <ul style="list-style-type: none"> lithography etching (chemical, plasma or laser) annealing sandblasting <p>Bottom-up methods [38,39,41–43,46,47]</p> <ul style="list-style-type: none"> layer by layer assembly hydrothermal (crystal growth) anodizing and electrodeposition electrospinning colloidal assembly (micro/nano particles) rough polymer film templating, replication, casting, and 3D printing | <p>Creating surface roughness, or hierarchical micro- and nano structure to enhance wetting or non-wetting.</p> |
| Surface energy modification | <p>Inorganic chemical coatings [42]</p> <ul style="list-style-type: none"> Si-based (silanes: PDMS, MTES, TEOS, PFAS, HMDS, PFOTMS, and; applied through dip coating, spray coating, spin coating, and LbL assembly) C-based (graphene, graphene oxide, and CNTs; applied through CVD, phase separation, and solution immersion) metallic and metallic oxide (Ag, ZnO, Al, TiO_2, and CuO; usually applied through electrodeposition, plasma deposition, anodizing, hydrothermal, and solution immersion) <p>Organic coatings [42]</p> <p>Usually applied through the template, dip coating, spin coating, spray coating, electrospinning, and LbL assembly:</p> <ul style="list-style-type: none"> thiols (hexadecane, <i>n</i>-octadecyl, dodecane, and perfluorodecane) PE, PTFE, and Teflon AF® fluorinated methacrylates PV, PVC, and PVDF PS, F-PBZ, PI, and PEI fatty acids such as stearic acid <p>Hybrid inorganic-organic coatings [39,42]</p> <p>Usually combined through hydrothermal, dip coating, spray coating, sol-gel, CVD, and LbL assembly.</p> | <p>Reducing surface energy to promote the superhydrophobic and superoleophilic condition.</p> |

harsh conditions (e.g., acidic-basic solutions, corrosive compounds, abrasive mixtures, and high temperatures). The PTFE robustness makes it a promising organic-based coating for superhydrophobic and superoleophilic surfaces. However, this excellent chemical resistance can be a drawback when it is used to dissolve PTFE into solvent for being used in electrospinning technique [104]. Qin et al. [89] modified Feng's experimental method of using PTFE suspension by adding polypropylene sulfide (PPS) and achieved a similar WCA of 156° . Some researchers used stearic acid ($CH_3(CH_2)_{16}COOH$) as a surface chemistry modifier to induce superhydrophobic and superoleophilic properties on the metal mesh via immersion method [34,67,68] and spray coating [65,66]. Stearic acid is an organic-based coating material that can also be applied for enhancing the corrosion resistance [278]. In other studies, stearic acid used along with various NPs, such as $Mg(OH)_2$ [65], Cu crystals [67], and ZnO [68,278] to create superhydrophobic and superoleophilic SS mesh-based membranes. Li et al. [34] and Liu et al. [67] coated a SS mesh by mixture of stearic acid and Al/ZnO and Cu NPs via immersion approach and created superhydrophobic and superoleophilic membranes with a WCA of 156° and 153° , respectively. Zhang et al. [68] grew a hierarchical ZnO micro-nano structure and applied immersion coating in stearic acid to obtain a superhydrophobic

and superoleophilic SS mesh for oil-water separation application. The functionalized mesh featured a WCA of 156° and a separation efficiency of 95% where up to 10 separation cycles were performed. The prepared mesh was also stable under harsh operating conditions, such as acidic-basic conditions, and corrosive and saline solutions [68]. Wu et al. [90] applied ZnO NPs on an SS mesh through the spin coating method with the aid of Teflon® AF as a surface chemistry modifier, where a WCA of 157° was achieved for the superhydrophobic and superoleophilic membrane.

Several inorganic materials have been applied to functionalize SS meshes. For example, CNTs with a low density (1.4 g/cm^3) and tubular network exhibit a high strength (46 M.Nm/kg) which is 300 times higher than SS metals. Furthermore, CNTs have been widely recommended for synthesis of superhydrophobic coated surfaces due to their high thermal conductivity, stability, and nanoscale dimensions. Given these properties and superhydrophobic and superoleophilic characteristics, CNT coating on SS mesh can be used to facilitate oil-water separation [83]. CNT has been applied to modify both the surface roughness [82] and chemistry [83,96,275] in the fabrication of superhydrophobic and superoleophilic mesh. For instance, Hsieh et al. [275] fabricated a fluorinated CNT onto carbon fabric using spin coating

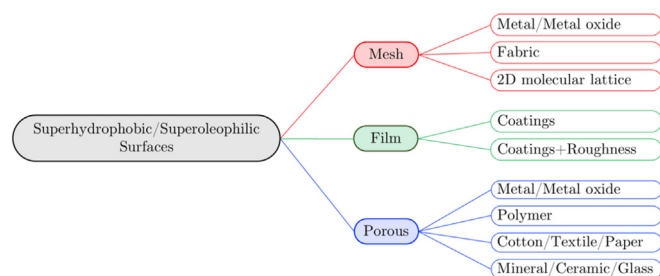


Fig. 9. Classification of superhydrophobic and superoleophilic membranes with application to oil-water separation.

method, and obtained 99.7% separation efficiency, and 165° WCA. The fluorination of CNT contributes to the high separation efficiency. In a similar study, Lee et al. [82] grew CNT on the SS surface via CVD technique and fabricated a superhydrophobic and superoleophilic mesh with a WCA of 163° . Cerium oxide (CeO_2) is a rare earth chemical oxide that is widely applied in glass polishing, optical devices, humidity sensors, and solid oxide fuel cells. Due to its inherent hydrophobicity, it also has gained attention in oil-water separation. Matin et al. [86] spray-coated a suspension of CeO_2 to fabricate an SS mesh with a WCA of 153° and an OCA of 0° .

Moreover, a variety of coating techniques are used to fabricate superhydrophobic and superoleophilic SS meshes with application to oil-water separation; the techniques include rough polymer and colloidal assembly [49,52,53,55,56,59–61,64,72,75,85–87,89,91,103–111], spray coating [29,53,65,66,75,82,86,276], electrodeposition [50,84], CVD [82,83], and immersion [67,73]. Colloidal assembly is a common method of fabricating superhydrophobic and superoleophilic SS mesh-based membranes. Polymeric materials are suitable to bond NPs, such as CNT [82], silica [50], ZnO [68], particle clusters of Cu, Ni, and Fe [72], CeO_2 [86], attapulgite [85], and $\text{Mg}(\text{OH})_2$ [65]; the NPs are used to modify micro-nano hierarchical structures on the mesh surface. Researchers have also used other polymeric materials such as PTFE [82,87,89], Teflon® AF [90], PFTOS [50], PU [91], polydopamine (PDA), and *n*-dodecylmercaptan [72]. Although various superhydrophobic and superoleophilic coatings have been successfully fabricated using the colloidal assembly, significant limitations remain. For instance, materials with a low-surface-energy are required in the coating composite; these components are expensive and have a short life. Alternatively, high-temperature curing and UV post-treatments are needed to improve the stability and mechanical strength of the coating film [279]. Spray coating has shown a high separation efficiency for oil-water mixtures in the literature. For example, Baig et al. [276] manufactured an SS membrane using spray coating that exhibited a high separation efficiency of 99% and a WCA of around 150° . Moreover, their coated membrane had a high potential to remove organic pollutants due to its high photocatalytic performance under UV irradiation. Cao et al. [65] applied spray coating of a mixture of phenol formaldehyde and $\text{Mg}(\text{OH})_2$ NPs in stearic acid to obtain a superhydrophobic and superoleophilic SS mesh-based membrane with a WCA of about 151.4° ; the mesh was used to remove soybean oil from water with a separation efficiency of 94.6% for up to 10 cycles. They concluded that the separation efficiency of an oil depends on its viscosity and volatility. They examined different oils such as trichloromethane, petroleum ether, *n*-hexane, toluene, and soybean oil. Trichloromethane resulted in a relatively lower separation efficiency (92.1%), which was attributed to its high volatility. Soybean oil featured the highest separation efficiency (94.6%) due to its high viscosity that tends to stick to the tubes wall and mesh [65]. Yang et al. [85] used different types of oils, such as lubrication oil, hexadecane, and proline to estimate the potential separation efficiency of their fabricated SS mesh. They spray-coated epoxy/attapulgite (44.4 wt%) on the SS mesh surface; the superhydrophobic and superoleophilic membrane was used for oil-water separation purposes. The WCA was consistently at

$160^\circ \pm 1$, even after 30 separation cycles, with a separation efficiency of 98%; the technique led to excellent superhydrophobic and superoleophilic properties. For the stability tests, they exposed the mesh to harsh conditions (95% relative humidity, 150°C for 48 h, washing, and drying), after which no significant decrease in the WCA of the coated mesh was observed [85]. Xiang et al. [72] used one-step electrodeposition approach to deposit particle clusters of nickel (Ni), Cu, and iron (Fe) on the surface of SS. Simultaneously, *n*-dodecyl mercaptan (NDM) was grafted on the substrate surface, using PDA, to modify the surface chemistry. The prepared mesh demonstrated a WCA of 162° and 98.6% separation efficiency for oil-water mixtures. After ten separation cycles, the obtained efficiency and WCA slightly reduced to 97.8% and 155° , respectively. Also, the mesh showed a high mechanical stability upon abrading tests, and immersion in solutions with different magnitudes of pH and salinity content. Dip coating method is commonly employed as a facile strategy to create superhydrophobic and superoleophilic properties on SS mesh [50,84,278,280,281]. For instance, Wang et al. [280] immersed SS meshes into a sol-gel based solution containing perfluoroalkyltriethoxysilane; they obtained a superhydrophobic and superoleophilic surface with a WCA 148° . The sol-gel featured room temperature condition, and provided a homogeneous coating [282]. Du et al. [66] employed the dip coating technique to fabricate a superhydrophobic and superoleophilic SS mesh. The coated mesh exhibited a WCA of 152° with an excellent stability under acidic and basic conditions, and/or hot water. It also showed at least 97% separation efficiency for kerosene-water mixtures after 40 cycles.

Copper meshes: Cu is another material that is widely used to create superhydrophobic and superoleophilic surfaces [52,53,69,74,76–79,91–95] owing to its excellent chemical and physical properties [53], malleability [94], extensibility, thermal conductivity, and adjustable pore sizes [69]. Some researchers used Cu oxide or Cu hydroxide to create micro-nano structures on the Cu-based surfaces along with chemicals to obtain superhydrophobic and superoleophilic membranes [94]. La et al. [283] fabricated a superhydrophobic and superoleophilic Cu mesh with Cu $(\text{OH})_2$ nanoneedle arrays via the electrochemical method, followed by 1H,1H, 2H, and 2H-PFTOS surface chemistry modification. Later, Cao and Cheng [78] developed flower-like clusters composed of nano-sized ginkgo-leaf-like lamellas on the surface of the Cu mesh after modification with DDT. The prepared mesh had a WCA of 155° and an OCA of 0° ; the superhydrophobic and superoleophilic mesh was used to separate oil/water emulsions. The designed mesh demonstrated a separation efficiency of 98% that was stable after 10 cycles. Pi et al. [53] developed a superhydrophobic and superoleophilic Cu mesh-based membrane where the Cu_2S and Cu_2O micro- and nano-roughness structures were modified with PDMS. The prepared mesh showed an oil-water separation efficiency of over 99.2% for free oil (light and heavy). Their mesh was stable under harsh conditions, such as exposure to hot water, hyper-saline solutions, strongly acidic systems, and basic solutions [53]. Cao et al. [91] investigated hot water repellency using superhydrophobic Cu mesh. They used hydrophobized SiO_2 NPs and a water-borne PU modified with AEAPS to improve the mechanical stability of the SiO_2 NPs deposited onto the surface of Cu mesh. Although the methodology to spray composite silica-PU solution was simple, the coated mesh did not resist against basic, acidic and hypersaline solutions for 24 h. The superhydrophobic and superoleophilic coated mesh provided a WCA of 162.5° and a high recyclability with 95.5% separation efficiency even after 40 separation cycles.

In terms of coating strategy, electrochemical deposition is a widely used technique in the literature that can be combined with other methods, such as dip coating [52], grafting [80], and vapor deposition [93]. For instance, Cao et al. [93] constructed a superhydrophobic mesh via electrodeposition and vapor deposition techniques on a candle soot (carbon NPs). In this electrode-based system under DC voltage (-0.5 V), Cu mesh and platinum sheet acted as the cathode and anode, respectively. Chain-like structures of agglomerated soot

Table 4
Superhydrophobic and superoleophilic mesh-based membranes for oil-water separation.

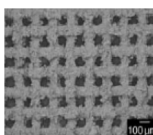
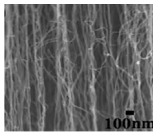
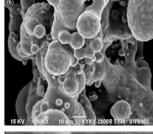
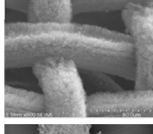
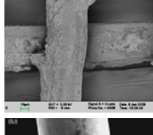
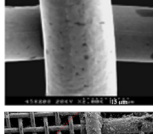
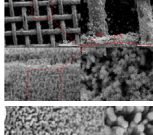
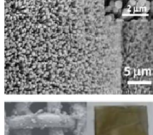
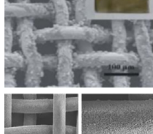
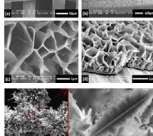
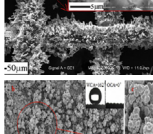
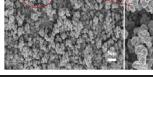
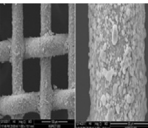
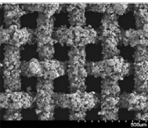
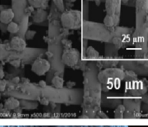
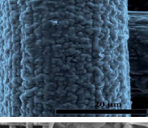
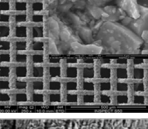
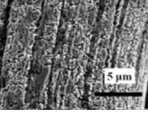

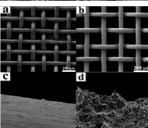
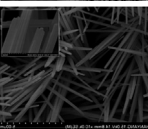
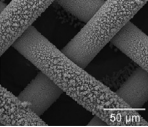
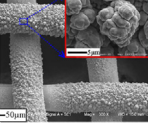
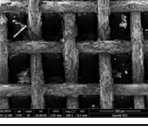
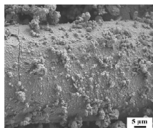
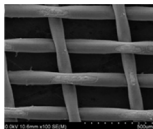
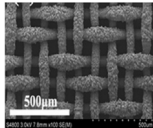
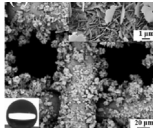
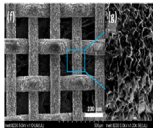
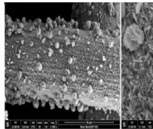
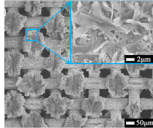
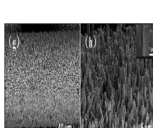
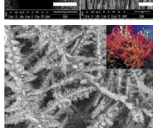
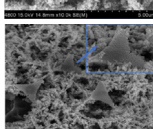
| Chemistry | Surface modification | | Structure | Pore opening | WCA (OCA) | Oil state (Separation tests) | Ref. |
|--------------------------|---|---|--|--------------------------------|-----------------|------------------------------|------|
| | Roughness | Method | | | | | |
| PTFE suspension (30% wt) | PS spheres (2–5 μm) | SS mesh spray coating |  | 30–420 μm | 156 (0) | free | [87] |
| PTFE 35 | CNT (100 μm average, 0–970 μm) | spray coating, CVD (CNT) |  | 100 nm (spacing CNT) | 163 (0) | free, o/w emulsion | [82] |
| PTFE-PPS | composite (nm/ μm) suspension film | spray coating |  | 26–440 μm (mesh) | 156.3 (0) | free | [89] |
| CNTs | CVD of CNT on SS with Al_2O_3 barrier (OD = 10–30 nm, length > 5 μm) | CVD |  | 44 μm (325 mesh) | 145–150 (0) | free, w/o emulsion | [83] |
| PFTOS | silica NPs (220 nm), agglomerates (1–5 μm) | dip coating, sol-gel |  | 75 μm | 154.8 (n/a) | o:w | [50] |
| PFAS | acid erosion | dip coating, sol-gel |  | 40–1500 mesh | 148 (0) | o:w | [54] |
| Teflon® AF | ZnO rod or flower-like micron and nano structure (1–2 μm) | spin coating |  | 200 μm | 157 (0) | o:w | [90] |
| Stearic acid | ZnO crystal growth on SS mesh | dip coating, crystal growth |  | 38–600 μm | 160 (0) | o:w | [84] |
| ZnO, stearic acid | clustered structure consisting of nanosheets | immersion |  | 50 μm | 156 (0) | o:w (1:1 vol) | [68] |
| Stearic acid | Al film deposition and ZnO 2D nano/micro flakes (width = 1.5 μm , height = 2 μm) | immersion |  | 25–600 μm | 156.3 (0) | o:w | [34] |
| Stearic acid | hierarchical micro-nano scale structure of Cu crystals | immersion |  | 100 μm | 153 \pm 3 (0) | o:w (1:1 vol) | [67] |
| PDA, NDM | particle clusters of Cu and Ni ions | simultaneous electrodeposition, polymerization |  | 200 μm | 162 \pm 1 (0) | oil/water mixtures | [72] |

Table 4 (continued)

| Chemistry | Surface modification | | Structure | Pore opening | WCA (OCA) | Oil state (Separation tests) | Ref. |
|--|---|---|--|--------------|---------------|------------------------------|------|
| | Roughness | Method | | | | | |
| CeO ₂ NPs | deposited CeO ₂ particles (100–500 nm) | spray coating |  | 50, 75 μm | ~153 (n/a) | o:w (1:1 vol) | [86] |
| Epoxy/attapulgite nanocomposite | attapulgite particles (320 mesh) | spray coating |  | 150 μm | 160 ± 1 (0) | oil/water | [85] |
| Stearic acid/phenol formaldehyde/Mg(OH) ₂ | Mg(OH) ₂ particles | spray coating, calcination |  | 150–500 mesh | 155.2 (n/a) | o:w (1:1 vol) | [65] |
| Stearoyl chloride (through acylation reaction) | polyaniline and polypyrrole | electrodeposition, polymerization |  | 30 μm | 154 ± 1.5 (0) | o:w (1:3 vol) | [88] |
| Hexadecanethiol | ytterbium oxide (Yb ₂ O ₃) | spray coating, annealing |  | Micron-sized | ~ 150 (0) | oil-water mixture | [75] |
| Stearic acid | HF acid etching, Ag micro/nano hierarchical structures | spray coating |  | 500 mesh | 152 (0) | o:w (1:1 vol) | [66] |
| <i>n</i> -DDT | Cu(OH) ₂ nano needles (7 μm long, 150–300 nm diameter) | Cu mesh dip coating, solution immersion |  | 50–1000 μm | 151 (0) | o:w (1:1 vol) | [77] |
| Dexadecanethiol | acid corrosion (HNO ₃ 4 M) | dip coating, solution immersion |  | 44–490 mesh | 153 (0) | o:w mixture | [76] |
| PFAS | Cu(OH) ₂ nano needles (200 nm from anodizing) | electrochemical anodizing, dip coating |  | 389 μm | 170 (0) | free | [52] |
| <i>n</i> -octadecylthiol | Cu NPs (2 μm film thickness) | grafting, electrodeposition |  | n/a | 154.1 (0) | o:w mixture | [80] |
| Lauric acid | Cu clusters | electrodeposition with lauric acid |  | 80–124 μm | 155.5 ± 3 (0) | o:w mixture | [92] |
| Dodecanethiol | CuO through annealing (400 °C) | immersion coating |  | 400 μm | 162 (n/a) | free | [79] |

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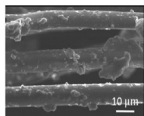
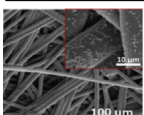
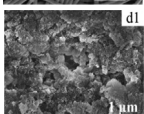
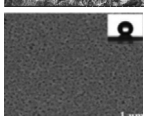
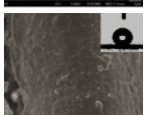
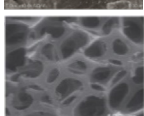
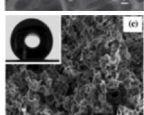
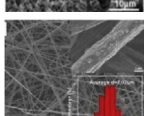
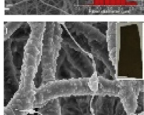


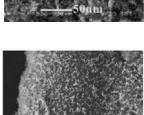
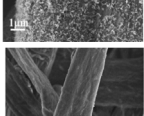
Table 4 (continued)

| Chemistry | Surface modification | | Structure | Pore opening | WCA (OCA) | Oil state (Separation tests) | Ref. |
|---|--|---|--|--------------|-----------------|------------------------------|-------|
| | Roughness | Method | | | | | |
| PDMS | micro/nano binary structure by Cu ₂ S and Cu ₂ O composite | dip coating |  | n/a | 153.3 ± 0.7 (0) | o:w (1:1 vol) | [53] |
| Carbon NPs (candle soot) | electrodeposition, chain-like structures of agglomerated soot (C) NPs formed | electrodeposition, vapor deposition |  | n/a | 153 (<5) | o:w (1:1 vol) | [93] |
| Modified PU, AEAPS, hydrophobic silica NPs | silica NPs | spray coating |  | 85 μm | 162.5 (0) | o:w (1:1 vol) | [91] |
| Cu@Ag modification with DDT | flower-like clusters composed of nano-sized ginkgo-leaf-like lamellas | acid etching |  | 60–200 mesh | 155 ± 1 (0) | w/o emulsions | [78] |
| Stearic acid | etched wire, and dense polymer sheets (width = 3–10 μm, thickness = 300 nm) | immersion |  | 120 μm | 155.8 ± 1.1 (0) | o:w (1:1 vol) | [69] |
| Alkaline solution of NaOH and (NH ₄) ₂ S ₂ O ₈ | peony flower-like structures by Cu(OH) ₂ | immersion |  | ~58 μm | 154.39 (0) | o:w (1:1 vol) | [94] |
| Cupric myristate Cu(CH ₃ (CH ₂) ₁₂ COO) ₂ | coral shape micro-nano-binary hierarchical structure after anodization | one-step anodization process |  | 70 μm | 153 (0) | w/o mixture | [95] |
| Ni mesh 1-octadecanethiol | Ni ₃ S ₂ nanorods | Other metal meshes hydrothermal |  | 75 μm | 151 (n/a) | o:w (1:1 vol) | [74] |
| Fe mesh Bismuth coating | coral-like bismuth oxide structures and irregular petal folds | chemical etching, immersion, anneal |  | n/a | 163 (0) | o:w (1:1 vol) | [284] |
| Ti mesh (Cu-coated) CuCl ₂ aqueous solution | micro/nanostructures of TiO ₂ and CuO, Cu ₂ O | HF etching, immersion, annealing |  | n/a | 158 (0) | o:w mixture | [107] |

(C) NPs were grown on the Cu mesh. The fabricated mesh with a WCA 150° separated oil from water with an efficiency of around 90% after at least 30 separation cycles. Dip coating was employed with Cu-based superhydrophobic and superoleophilic meshes [53,76,77]. Pan et al. [77] immersed a Cu mesh into an aqueous solution of NaOH and K₂S₂O₈ and subsequently modified it with DDT. Over 97% of the oil was separated from water at experimental conditions. Yanlonga et al.

[79] prepared a superhydrophobic and superoleophilic Cu mesh membrane through annealing at 400 °C and subsequently, immersion coating in DDT. The WCA was approximately 162°, and the separation efficiency of more than 95% for oil-water mixtures was achieved. Yang et al. [69], employed the immersion technique to coat a Cu mesh with stearic acid and ethanol solution. The separation efficiency of more than 97% with at least 20 times repeatability for the coated mesh was

Table 5
Superhydrophobic and superoleophilic porous membranes for oil-water separation.

| Type | Surface modification | | | Structure | Pore size | WCA (OCA) | Oil state | Ref. |
|-----------------------------|---------------------------------|--|--|---|------------------|---------------|-------------------------|-------|
| | Chemical | Roughness | Method | | | | | |
| Polyester textile (fabric) | TEOS/VTES | TiO ₂ NPs | dip coating |  | 5–10 (μm) | 152.4 (n/a) | o:w mixture | [55] |
| Polyester non-woven fabrics | PBZ | TiO ₂ NPs | dip coating, thermal curing |  | 18.9 (μm) | 155 ± 1 (0) | o:w mixture | [106] |
| PVDF | PVDF/DMF | hydrophobic silica NPs (7–40 nm) | immersion polymerization (delayed phase inversion) |  | n/a | 157 ± 2 (0) | w/o emulsion | [108] |
| PVDF-MF | hexamethyldisilazane | hydrophobic silica NPs | dip coating |  | 0.45 (μm) | >150 (0) | w/o emulsion | [49] |
| PET textile membrane | TEOS, HMDS | hydrophobic silica NPs (10 nm) | sol gel, dip coating |  | n/a | 150 (0) | o:w mixture | [56] |
| PU foam | FAS/ethanol | acid etching | dip coating |  | n/a | 155 (0) | o:w mixture | [48] |
| PAR-b-PDMS (polyarylester) | fluorinated PAR-b-PDMS | hierarchical micro/nano (PAR-b-PDMS) | spray coating |  | 0.1–0.8 (μm) | 163 ± 2.3 (0) | o:w (3:7 vol) | [57] |
| PVDF membrane | PVDF powders, N,N-DMF/acetone | fibrous PVDF | electrospinning |  | 2.02 ± 0.31 (μm) | 153 (0) | w/o emulsion | [195] |
| PVA/PTFE nanofiber | PVA/PTFE | PTFE | electrospinning, sintering |  | n/a | 155 (0) | o:w (1:1) | [104] |
| PI nanofiber | 1H, 1H, 2H, 2H- PFDT | micro/nano-scale hierarchical (300 nm fiber) | electrospinning, dip coating |  | n/a | 153 (0) | o:w (1:1), w/o emulsion | [73] |
| PVC membrane | PVC in THF, glacial acetic acid | n/a | dripping |  | 10–50 (nm) | 151.5 (8) | n/a | [310] |
| Filter paper | PS/toluene | hydrophobic silica NPs (14 nm) | dip coating |  | n/a | 157 (0) | o:w mixture | [103] |
| Filter paper | Dodecafluoroheptyl methacrylate | n/a | dip coating |  | 40 (nm) | 152 (0) | w/o emulsion | [178] |

(continued on next page)

Table 5 (continued)

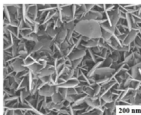
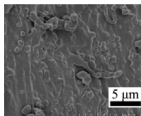
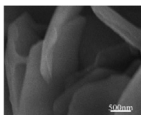
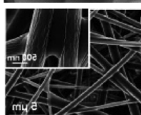
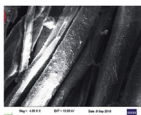
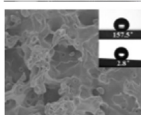
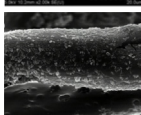
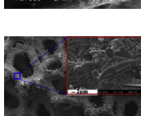
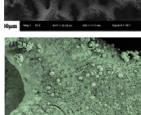
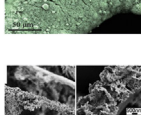
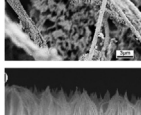
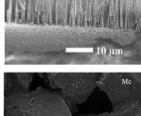
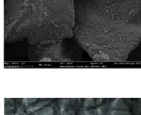
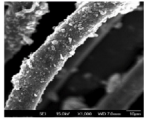
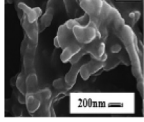
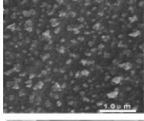
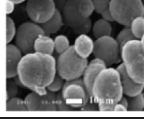
| Type | Surface modification | | | Structure | Pore size | WCA (OCA) | Oil state | Ref. |
|-----------------------|---|--|--|---|--------------------------|---------------|---------------|-------|
| | Chemical | Roughness | Method | | | | | |
| Filter paper | stearic acid | Cu(OH) ₂ micro/nano structure | adsorption, immersion |  | n/a | 153 (0) | w/o emulsion | [70] |
| Filter paper | PFOEMA | PFOEMA grafted chains | grafting, polymerization |  | n/a | 157.5 (n/a) | o:w mixture | [301] |
| Cellulose-based | stearic acid | layered double hydroxide uniform crystals | immersion, crystal growth |  | 150 (nm) | 154 ± 1.8 (0) | o:w (1:1 vol) | [23] |
| cellulose acetate | fluorinated poly benzoxazine | silica NPs (7–40 nm) | electrospinning, in-situ polymerization |  | 40 (nm) | 161 (3) | o:w (1:1 vol) | [105] |
| Cotton fabric | non-fluorinated hydrophobic reagents (HDTMS and stearic acid) | zirconia particles subsequently AgBr | immersion |  | n/a | 153 (0) | o:w (1:1 vol) | [58] |
| Cotton fabric | PDMS, PMMA, THF | n/a | spray coating |  | n/a | 157.5 (0) | o:w emulsion | [60] |
| Cotton and PS fabrics | PS, MPTMS | hydrophobic silica NPs | solution immersion |  | n/a | 154–156 (0) | o:w mixture | [59] |
| Fe foam | stearic acid | micron-sized leafy crystals and nano-scale particles | metals chemical etching, annealing |  | 300–500 (μm) | 157 (0) | o:w (2:1 vol) | [71] |
| Cu foam | NDM | Cu(OH) ₂ | electrodeposition, immersion |  | <150 | 154 (0) | o:w emulsion | [305] |
| Silica sponge | Co NPs/ PDMS | hierarchical Co microstructures | minerals/ceramic/glass self-assembly electrospinning and calcination |  | fiber diameter of 2.5 μm | 156.9 (n/a) | o:w mixture | [109] |
| Alumina membrane | n/a | alumina nano pyramids (2–3 μ high, 1 μ spacing) | high field anodizing |  | 180 (nm) | 152.4 (0) | n/a | [102] |
| Alumina membranes | PTFE | decomposed PTFE particles | thermal decomposition, sintering |  | 0.10 (μm) | 155 (0) | o:w mixture | [308] |
| Single-wall CNT | n/a | CNTs | C-based membrane filtration/release of SWCNT suspension |  | n/a | 94 (0) | w/o emulsion | [96] |

Table 5 (continued)

| Type | Surface modification | | | Structure | Pore size | WCA (OCA) | Oil state | Ref. |
|-----------------------------|--|------------------------------|----------------------------|---|-----------|-----------|---------------|-------|
| | Chemical | Roughness | Method | | | | | |
| CNTs | perfluoroalkyl methacrylate (Zonyl® 8740, DuPont™) | CNTs micro/nano | spin coating |  | n/a | 165 (0) | o:w (1:1 vol) | [309] |
| Composites | | | | | | | | |
| ZnO/PDMS nano composite | PDMS | hydrophobic ZnO NPs (14 nm) | spray coating, combustion |  | n/a | 155 (<2) | n/a | [61] |
| ZnO/PS cotton textile | PS, stearic acid | hydrophobic ZnO NPs (150 nm) | drop coating, hydrothermal |  | 11 (μm) | 155 (0) | o:w mixture | [64] |
| PP/methyl-silicone membrane | PP/methyl-silicone (in tetrachloroethane) | PP granules (15–20 μ) | casting |  | n/a | 152 (0) | o:w (1:1 vol) | [110] |

obtained, where the WCA was 155.8°. The fabricated mesh demonstrated high stability under acidic-basic and salty solution conditions. Zhang et al. [94] used the immersion techniques to coat a Cu mesh using a solution of NaOH and (NH₄)₂S₂O₈, which resulted in peony flower-like Cu(OH)₂ on the surface of the Cu mesh. The superhydrophobic and superoleophilic Cu mesh exhibited a separation efficiency of 95% after 10 cycles.

Other metal meshes: The SS- and Cu-based meshes dominate superhydrophobic and superoleophilic membranes that are used for oil-water separation applications. Other metal-based meshes, such as Ni [74], Fe [284], and Ti [107] have also been employed to prepare special wettable materials for oil-water separation. For example, Ni mesh is used due to its malleability, durability, air permeability, anticorrosion, and thermal tolerance. The base material is prepared through hydrothermal method where Ni₃S₂ nano-rods are created; later, the hierarchical rough structure is coated with 1-octadecanethiol [81]. Also, hydrothermal and chemical etching techniques are used to create hierarchical micro- and nanostructures, including nanorods, nanoneedles, and nanowires on the Ni mesh surfaces. For example, Jian et al. [74] utilized Ni meshes that were modified with 1-octadecanethiol and used for oil-water separation. Their superhydrophobic and superoleophilic mesh led to an efficiency more than 94% after ten cycles even under immersion test in 3.5% NaCl solution for two days. Fe mesh is a cheap, available, and highly durable candidate for the SS meshes. Yu et al. [284] used an Fe-based mesh that was coated with bismuth. The process created coral-like bismuth oxide structures and irregular petal folds. The Fe mesh was also etched in the acid to create additional surface roughness. The final coated mesh exhibited excellent wettability and durability in cyclic oil-water separations. Ti is light, flexible, and thermally stable; hence, it has been used as a base material to create superhydrophobic and superoleophilic membranes with application to oil-water separation [292]. Yu et al. [107] coated Ti with Cu oxides to fabricate a superhydrophobic and superoleophilic metal mesh. The superhydrophobic and superoleophilic Ti mesh resulted in a separation efficiency of more than 96% after at least 20 cycles. The Ti mesh exhibited outstanding stability and durability after subjecting to water and air atmosphere for the lab-based corrosion test.

6.2. Superhydrophobic and superoleophilic porous membranes

Utilization of porous materials (filters) for oil-water separation has been a conventional practice in some chemical and energy industries [285]. Porous materials have attracted considerable interest to be used as raw materials for the fabrication of superhydrophobic and superoleophilic surfaces [286]. In this part, we review superhydrophobic and superoleophilic porous materials that are categorized based on their material, including polymers, cotton/textile, filter paper, metals, minerals, ceramics, glasses, carbon-based, and composites. A summary of the superhydrophobic and superoleophilic porous membranes is provided in Table 5.

Porous polymers: Membrane surface modification by incorporating numerous types of NPs into the polymeric membrane is also used in fabrication of superhydrophobic and superoleophilic membranes. One of common polymers that is used for oil-water separation is PVDF due to its favorable properties, such as low surface energy, high mechanical strength, and high physical and chemical stabilities [287–289]. These membranes may have limitations for separation of surfactant-stabilized emulsions when the membrane pore size is large [290,291]. The properties of the modified membrane are affected by incorporating polymeric materials as well as NPs. Commonly NPs such as Ti (oxide) [55,106] and silica [49] are used in the matrix of porous polymer membrane. Ti is a light nanoparticle with high thermal stability and superhydrophobic properties, which has been widely utilized in oil-water separation [55,106]; it also features a self-cleaning character [48]. However, in some cases, a superhydrophobic TiO₂ surface can become hydrophilic, for example, under UV exposure [292]. Zhang et al. [106] used TiO₂ NPs with PBZ on polyester non-woven fabrics using dip coating and thermal curing with application to oil-water separation. PBZ is a cost-effective and low surface energy material with high thermal stability and good resistance to harsh environment and UV irradiation. Moreover, it generates a surface with hydrophobic and oleophilic properties [106]. Similarly, Yu et al. [55] utilized TiO₂ NPs and dip coating technique with TEOS, and VTES. The coated surfaces provided an oil-water separation efficiency of 98% and 95%, respectively [55]. Using low-cost silane coupling agents (TEOS/VTES) resulted in a stable membrane even after 24 separation cycles [55].

Silica NPs are also promising in fabrication of superhydrophobic and superoleophilic porous membranes. They are common NPs that are relatively inexpensive and can be produced with controlled particle size and surface energy to be integrated in modifying superhydrophobic and superoleophilic porous PVDF membranes [49,108] and PET textile [56]. As the impermeable and dense adhesive layers can reduce the permeate flux [293], Wei et al. [108] used delayed phase inversion method to immobilize the SiO₂ NPs on the PVDF membrane without using adhesive; they achieved a high separation efficiency (99.95%). A functional PU foam is another modified superhydrophobic and superoleophilic porous membrane that can be fabricated through dip coating; these functionalized foams can float on the water due to low density and light weight; it may be used for the capture of oil spills. The PU foam modified with PFAS is chemically stable and reusable that can have a separation efficiency over 95% after 10 cycles [48]. Spray coating as an alternative surface modification strategy is employed to a much less extent with superhydrophobic and superoleophilic polymeric porous membranes. Li et al. [57] fabricated a porous polymer membrane using a fluorinated polyarylester polydimethylsiloxane block copolymer (PAR-b-PDMS) through spray coating. The modified membrane exhibited a stable superhydrophobicity that can effectively treat oil-water mixtures (99%) with at least 50 cycles reusability.

Electrospinning is becoming a widely used technique in the category of superhydrophobic and superoleophilic porous membranes [73,104,195]. For instance, a WCA of 153° was achieved using electrospinning method and applying N, N-DMF/acetone on the ultrathin electrospun fibrous PVDF membranes [195] and PFDT/PI nanofibers [73]. Both research studies reported an excellent separation efficiency of above 99%. Also, the PFDT/PI nanofiber showed excellent recyclability (at least 20 cycles) with consistent superhydrophobicity and good durability under harsh operating conditions. In another study, a hybrid PVA/PTFE nanofibrous membrane was prepared through electrospinning technique [104]. The developed PTFE membrane showed superhydrophobic behavior with a WCA around 155° and efficient gravity-driven oil-water separation. The robust membrane mechanical strength was a result of the sintering process that caused the stability of nanofiber in the membrane under a high vibration environment [104].

Cotton and paper: Cotton textiles have attracted great interest in oil-water separation due to particular characteristics such as easy handling, flexibility, biodegradability, environmentally-friendly, low cost, and high efficiency [58]. The cotton fabrics can be wetted by water and oil simultaneously due to their hydroxyl functional groups on the surfaces. Typically, superhydrophobic cotton textiles are designed for water-repelling or self-cleaning purposes [43,294]. Only a few studies in the literature employed superhydrophobic textiles for oil-water separation [295,296]. As the cotton-based materials lose their superhydrophobicity due to the lack of a strong attachment between the cotton fibers and low surface energy materials, it is important to find a robust coating with high mechanical stability for large-scale and long-term applications [297]. Silane, as an inorganic chemical, has been commonly utilized for coating surfaces through covalent attachments consisting of one or more silicons [298]. Singh et al. [58] generated superhydrophobic and superoleophilic cotton fabric via immersion and drying methods. They used HDTMS and stearic acid with zirconia particles, followed by AgBr modification (for constructing surface roughness). The zirconia particles have a high bond dissociation energy and strong covalent characteristic that can enhance durability. The AgBr can be used when a visible light photocatalyst is needed to degrade the organic compounds. The prepared coated fabric could effectively separate a wide range of oil-water mixtures with high efficiency (>99%) even after 10 cycles [58]. It was noticeable that the modified cotton retained its properties under harsh environmental conditions, such as acidic, alkaline, salty, and UV irradiation. Zhang et al. [59] used solution immersion to coat cotton and PS fabrics with MPTMS and SiO₂ NPs. The superhydrophobic and superoleophilic fabric was used for oil-water separation. The coated surface exhibited mechanical durability, easy

repairability, and anti-fouling behavior with the ability of self-cleaning of the organic solvents. Moreover, the prepared surface illustrated a significant performance to separate liquids with different surface tensions and temperatures where the separation efficiency was above 95%. Zhou et al. [299] applied fluorinated alkylsilane onto the cotton fabric. The modified superhydrophobic and superoleophilic cotton demonstrated high separation efficiency of the oil-water mixture. PDMS is a silicon rubber with a high flexibility and mechanical, which is used to coat cotton fabrics without using any adhesives [175]. Liu et al. [60] fabricated a superhydrophobic and superoleophilic cotton fabric through spray coating PDMS, and PMMA in THF solution. The modified cotton fabric displayed a WCA of 157.5° with excellent stability in the harsh environment. The as-prepared cotton provided a high performance in oil-water emulsion separation, anti-fouling, and self-cleaning [60]. Self-polymerization of dopamine under alkaline conditions leads to form PDA, which has a strong adhesive force [72]. Xu et al. [300] used PDA and Ag NPs to fabricate a superhydrophobic cotton fabric for separation of oil and water mixtures with self-cleaning properties. Cellulose is an abundant natural organic polymer that is known as an environmentally friendly and biocompatible material. The cellulose-based filter papers are used in the fabrication of superhydrophobic and superoleophilic surfaces [23,70,301]. For instance, modification of filter paper with a PS solution in toluene [103] created a superhydrophobic and superoleophilic membrane with a WCA around 157°. Fluorinated PBZ [105], coated with silica NPs, formed nanoscale roughness and increased the WCA up to 161°. Both fabricated surfaces demonstrated a good performance with the separation efficiency above 96% for different oil/water volume ratios. Huang et al. [178] used dodecafluoroheptyl methacrylate to fabricate a superhydrophobic and superoleophilic filter paper, using dip coating. Wu et al. [301] employed F-based materials, such as polyperfluorooctylethylmethacrylate (PFOEMA) through grafting onto filter papers. The covalent chemical bond between the low surface energy fluorinated materials and filter paper provides a high chemical resistance with a promising performance in oil-water separation. The cellulose-perfluorooctyl ethyl methacrylate porous membrane showed over 95% oil-water separation efficiency with excellent reusability (10 times) [301]. However, as fluorinated compounds are nonbiodegradable, they are considered as environmentally undesirable materials [58]. Stearic acid, as a low-surface-energy material with self-assembly capability, can be alternatively deposited onto the filter paper/cellulose-based surface to exhibit superhydrophobic and superoleophilic feature [23,70].

Porous metals: The three-dimensional porous metal foams provide a large specific area with a well-developed porous structure, high strength, and low cost in comparison with the traditional two-dimensional materials; they have been alternatively used to fabricate superhydrophobic and superoleophilic membranes for treatment of oily-water systems [302–304]. Liu et al. [71] designed a superhydrophobic and superoleophilic Fe foam using annealing and chemical etching to create micro-nano hierarchical structures on the substrate surface. The surface energy was reduced through coating with stearic acid. The modified superhydrophobic and superoleophilic Fe foam exhibited a high separation efficiency (more than 95%) with exceptional physical and chemical stability [71]. Also, a porous Cu foam was fabricated, through growing Cu(OH)₂ nanowires on the surface of a Cu mesh via electrodeposition, followed by chemical modification with NDM through immersion approach [305]. The as-prepared foam exhibited a high durability, and a high performance in continuous separation of oil-water systems at a high flux. The modified foam showed the ability of demulsification due to having the cage-like structure; this membrane pore structure resulted in the collision and coalescence of micron-size water droplets [305].

Porous minerals, ceramic, and glass: These materials feature high density, fragility, and incompressibility (their volume is not affected by changing temperature and pressure), compared to the other materials. However, they can be applied in the harsh environment due to

their excellent thermal stability and erosion resistance [306]. Sponges as the three-dimensional superhydrophobic and superoleophilic materials are frequently used due to low weight, low price, high mechanical stability, high flexibility, and high separation capacity [285]. However, superhydrophobic and superoleophilic sponges are usually used as a sorbent rather than a filter in oil-water separation. Mi et al. [109] fabricated a superhydrophobic and superoleophilic silica sponge, using cobalt (Co) NPs and PDMS as a surface modifier; the sponge was used in oil-water separation both as a sorbent and a filter. The Co NPs provided hierarchical microstructures and added remote controllability of modified sponge by imparting magnetic properties. The modified silica sponge showed excellent superhydrophobicity and superoleophilicity with high surface area, good thermal resistance, good flexibility, and reasonable durability. The as-prepared sponge exhibited a separation efficiency up to 99.9%, which decreased to 97% after ten cycles of separation test [109]. Ceramic membranes feature excellent mechanical strength, high chemical resistance, and exceptional thermal stability [307]. Indeed, the alumina membrane with excellent resistance in the harsh chemical cleaning can be applied in the different ranges of pH and temperature for oil-water separation applications. Yao et al. [102] fabricated nanostructured alumina membrane through electrochemical anodization in the oxalic-acid electrolyte. In this morphology dependent technique, a superhydrophobic and superoleophilic alumina film is created without using low energy chemicals. Tang et al. [308] fabricated a superhydrophobic and superoleophilic alumina membrane with PTFE through thermal decomposition (sintering). The modified alumina membrane led to higher than 97% water rejection over four hours.

Carbon-based porous membranes: Shi et al. [96] used free-standing, single-wall CNTs to fabricate a thin membrane (70–120 nm thick) for oil-water separation application. The membrane was superoleophilic, and hydrophobic with a WCA of 94° . One of the features of this thin membrane is its exceptionally high permeate flux up to $100,000 \text{ L}/(\text{m}^2 \cdot \text{h} \cdot \text{bar})$, which is up to three orders of magnitude higher than that for the commercial filters. They used the filter to separate emulsions with and without surfactant; it was possible to separate the emulsified oil with 99.95% efficiency, even after 20 cycles [96]. The membrane also had a high chemical resistance to acid and base (except for strong oxidizing acids). Hsieh et al. [309] used carbon fabrics with fabric diameter of 0.3–0.9 mm in the presence and absence of multi-wall CNTs for oil-water separation purposes. The CNTs were used to add nano-roughness, with an average diameter 30–50 nm that were synthesized through catalytic CVD method. The prepared membrane

was spin coated with perfluoroalkyl methacrylic copolymer (Zonyl® 8740, DuPont™) and exhibited superhydrophobic and superoleophilic properties with a WCA of 165° [309]. The membrane was capable of separating oil from water up to 99.7% efficiency. Their results showed a dramatic decrease in the separation efficiency when the diameter of carbon fibers was increased from 0.3 mm to 0.9 mm, as shown in Fig. 10. This reduction in separation efficiency was more pronounced for the fabric without CNTs, compared to the carbon fiber with CNTs. For example, the separation efficiencies of 0.9 mm fabric with and without CNTs were decreased to 90%, and 70%, respectively [309]. The permeate flux was also significantly affected by the diameter of the carbon fibers. However, permeability reduction due to an increase in the fiber thickness was more pronounced in the membrane decorated with CNTs, as observed in Fig. 10. This study suggests opposite effects of the (hierarchical) surface roughness on separation efficiency and permeate flux; a process optimization is thus required to find an optimal roughness on superhydrophobic and superoleophilic membranes, especially for high throughput applications. Also, this study highlights the importance of membrane thickness on oil-water separation efficiency and membrane flux. Both the separation efficiency and the permeate flux will increase as the membrane thickness decreases. For this reason, ultrathin mesh-based membranes and 2D molecular lattice (such as graphene) seem to be promising.

Porous composite membranes: Another practical alternative to fabricate superhydrophobic and superoleophilic surfaces is nanocomposites. Chakradhar et al. [61] prepared a ZnO-PDMS nanocomposite through a simple spray coating and combustion. The ZnO provided micro and nanostructure that is necessary to achieve superhydrophobic condition. The WCA of the ZnO coating was around 108° , which increased to 155° after modification with PDMS. ZnO/PS cotton textile modified with stearic acid is also utilized for fabricating superhydrophobic and superoleophilic composite membranes [64]. Moreover, using the casting method, PP/methyl silicone membrane was constructed for oil-water separation purposes [110].

6.3. Superhydrophobic and superoleophilic films

In this section, we review superhydrophobic and superoleophilic films that have applications in oil-water separation. Various substrates, such as polymers [62,111,311], metals [63,201,312], NPs [313], and silanes [51] can be employed to prepare superhydrophobic and superoleophilic films. Several methods, including electrodeposition [111], solution immersion [314], CVD [193], dip coating [51], and

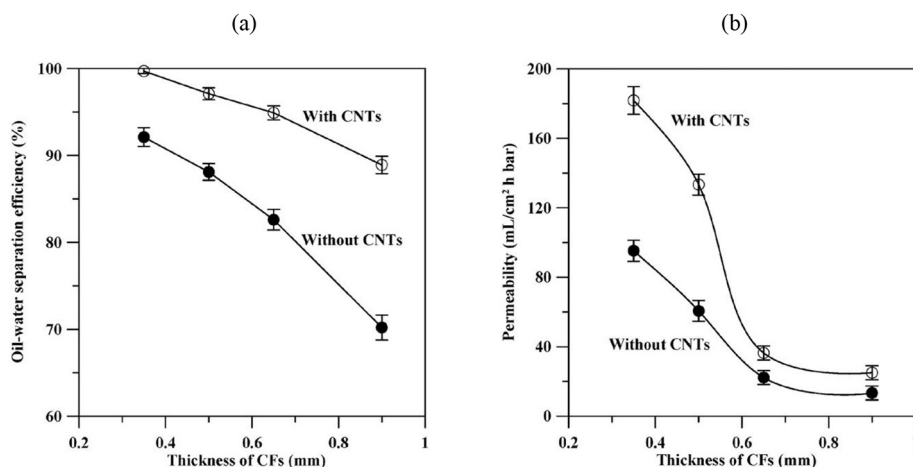
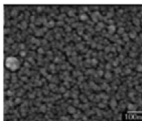
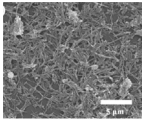
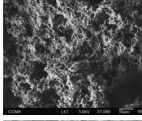
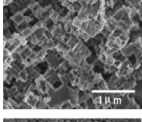
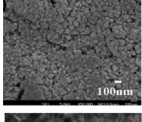
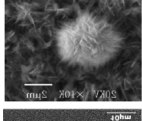
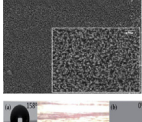

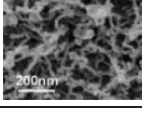


Fig. 10. Impact of carbon fiber thickness (membrane thickness) and multi-wall CNTs roughness on: (a) separation efficiency and (b) membrane flux, for a superhydrophobic and superoleophilic membrane [309].

Table 6
Superhydrophobic and superoleophilic surfaces for oil-water separation application (Film).

| Type | Surface modification | | | Structure | WCA (OCA) | Ref. |
|---|--|--|-------------------------------|---|-----------------|-------|
| | Chemical | Roughness | Technique | | | |
| PFA film | PFA | plasma-etched channels (3 μm , 6 μm spacing) | plasma etch, CVD |  | 156 (0) | [193] |
| Surface TiO ₂ nanowires and PDMS | TiO ₂ nanowires (Width: 20–40 nm Length: 5– >10 μm) | | Solution immersion |  | 158 (0) | [314] |
| Polymer film | polyehtylendioxythiophene | rough film | electrodeposition |  | 156 (<5) | [111] |
| Modified Al Surface | MTS | etched/corroded Al | acid etch, solution immersion |  | 169.7 (0) | [63] |
| Polymer film | TMS, DMS | hydrophobic silica NPs | spray coating |  | 168 (0) | [62] |
| Oxidized Cu | n/a | Cu petal like (1–4 μm thick, 20–70 nm slices) | chemical oxidation of Cu |  | 158 (<5) | [312] |
| Boehmite surface | n/a | crystal growth (2–3 μm) | thermal, crystal growth |  | 152.8 (0) | [201] |
| Multiwall-CNT | alkyl-pyrene | CNTs | noncovalent functionalization |  | 158 \pm 2 (0) | [313] |
| Silicone nanofilament | OTS, PFOTS | film | plasma treatment, dip coating |  | 165 (2) | [51] |

spray coating [62] are usually used to make superhydrophobic and superoleophilic films. In an interesting study, Wang et al. [193] fabricated PTFE hierarchical network film and polytetrafluoroethylene-perfluoropropylvinylethers (PFA) particle rough structure; the first film was oleophobic with an OCA of 138°, while the second film was superoleophilic with an OCA near zero [193]. Both films were superhydrophobic with a WCA > 150°. They justified this difference due to different conformation of the $-\text{CF}_2$ functional groups on the film surface. The surface was pretreated by plasma etching, followed by CVD. The result showed 156° WCA. Tang et al. [201] successfully prepared a superhydrophobic and superoleophilic film on a boehmite substrate by a thermal pretreatment and crystal growth (2–3 μm roughness). They obtained a WCA around 152.8°. Pei et al. [312] reported a higher WCA when oxidized Cu was employed as the base substrate. To manipulate the film surface wettability, the surface can be textured with nanomaterials, such as CNTs [313] or TiO₂ [314]. Two forces play a significant role in constructing superhydrophobic and

superoleophilic CNT films: 1) van der Waals force to cover the CNT surface with low surface energy chemicals via attachment, and 2) covalent forces to attach the hydrophobic groups onto the CNT surface [315]. Darmanin et al. [111] used electrodeposition to create a rough film of polyehtylendioxythiophene; the film featured a WCA of 156° and an OCA < 5°. Li et al. [63] etched the Al surface with acid immersion and reduced the surface energy, using MTS [63]; their results revealed promising advantages of organosilane to create stable superhydrophobic film with a high WCA of 169.7°. Zimmermann et al. [51] used plasma treatment and applied dip coating with OTS, and perfluorooctyltrichlorosilane (PFOTS); they observed excellent water repellency with a WCA of 165°. Zhang et al. [62] spray coated trimethylchlorosilane (TMS) and dimethylsiloxane (DMS) on the surface of the polymer film, using NPs for creating roughness on the film, resulting in a WCA of 168°. Table 6: summarizes important information (e.g., type and chemical nature/structure) on the superhydrophobic and superoleophilic films for oil/water separation.

7. Challenges and future perspective of superhydrophobic and superoleophilic membranes for oil-water separation

Superhydrophobic and superoleophilic membranes have found great applications in oil-water separation, such as oil spills and oily wastewater treatment. However, they have limited capabilities to separate volatile components as well as systems with potential fouling [209]. Also, the membrane stability under harsh operating conditions such as strong acids, bases, oxidizers, and saline solutions is uncertain; the hydrophobicity can decrease at high temperature conditions. The effects of operating parameters, including temperature, cross-flow velocity, pH, trans-membrane pressure, and the molecular size of solute in separation membranes should not be underestimated, especially in the presence of solid particles that cause rapid fouling. Furthermore, pH influence on membrane material and larger molecular size of solute augments rejection is still complicated, and contradictory results have been reported in the open sources [209]. Systematic investigation of important aspects (e.g., wettability impact, separation mechanisms, and fouling phenomenon) in superhydrophobic and superoleophilic membranes is overlooked in the literature.

For a given oil-water mixture, the separation efficiency is related to the membrane thickness, pore size, wetting state, and surface roughness. A decrease in the membrane thickness increases the separation efficiency, permeate flux, and energy consumption. Therefore, the use of an ultra-thin membrane with a good mechanical strength can be accepted as a promising future trend in membrane filtration. The use of 2D molecular lattice (atom thick membrane) of graphene and single-wall CNTs, and/or other meshes with ultra-thin fibers is expected to be a part of future studies in this area.

Utilization of controlled hierarchical surface roughness is favored in separating dispersed and emulsified oil contaminations from an oil-water mixture. The surface roughness improves the oil separation efficiency; however, it reduces the maximum permeate flux. Therefore, an optimal design of surface roughness is required. Such an optimal design should include the effect of hierarchical surface roughness on membrane fouling for a realistic and effective operation. Using lithography technique and micromachining, it is possible to fabricate a desired surface roughness structure; however, with the current technology, large-scale production is expensive, which would be a limiting factor. Perhaps the advancement of 3D printers with high resolutions can improve the scalability of hierarchical roughness that is imperative in fabricating superhydrophobic and superoleophilic membranes.

8. Conclusions

This paper systematically reviews the application of the SHSO porous membranes, mesh-based membranes, and films in oil-water separation. We do not cover the literature review related to SHSO sorbents (for oil spill application) in this study; this can make an excellent subject for another review paper. This work is summarized with the following important points:

- Considering techno-economic and safety aspects, the suitability of the pretreatment methods is in the following order: air plasma > oxygen plasma > piranha solution > UV irradiation.
- Among the widely used surface modification chemicals (silanes, thiols, stearic acid, oleic acid, lauric acid, carbon nanotubes, and polyethylene-derives (co)polymers), silanes with long functional groups have shown better SHSO performances; however, the fluorine atoms on the functional groups can impose environmental issues.
- Layer-by-layer assembly and grafting are the coating methods that are able to control coating thickness and functional groups. Simpler coating techniques, such as dip coating, spray coating, electrospinning, and sol-gel are cheaper alternatives that can produce uniform and high-quality SHSO coatings. Hierarchical micro- and nano-scale roughness structures can be created precisely by proper methods

such as CVD, femtosecond laser irradiation, lithography, and templating; however, they are not economically viable for large-scale applications with the current technology. Facile coating techniques that can simultaneously modify surface roughness and chemistry are promising in the fabrication of SHSO surfaces.

- Micro- and nano-surface structures, which account for hydrophobicity of membranes, can be damaged under external mechanical forces and harsh conditions (hot water, brine, acidic solutions, and alkaline solutions). Therefore, a guideline is immediately needed to assess the long-term stability and durability of the SHSO membranes under harsh operating conditions.
- Despite extensive studies on characterization and oil separation efficiency of SHSO membranes, impairment of the superhydrophobicity with exposure to high temperatures and adsorption of polar components of crude oil (such as asphaltenes and resins) is not adequately studied and can be considered in the future research. Furthermore, the potential benefits of the SHSO membranes in mitigating fouling effect, originated from water-based solids, are completely overlooked in the literature. This can be an interesting topic for future research investigations.
- Further technology development in large-scale femtosecond laser ablation, high-resolution 3D printing, and the use of 2D materials such as graphene are expected to lead to a breakthrough in the use of SHSO membranes with application to oil-water separation.

Nomenclatures

Acronyms and chemical formulas

| | |
|------------------|--|
| AEAPS | Aminoethylaminopropyl polydimethylsiloxane |
| API | American petroleum institute |
| APP | Ammonium polyphosphate |
| CeO ₂ | Cerium oxide |
| CNT | Carbon nanotube |
| CVD | Chemical Vapor deposition |
| DDT | Dodecanethiol |
| DMF | <i>N, N</i> -dimethylformamide |
| DMS | Dimethylsiloxane |
| Fe | Iron |
| FL | Functional layer |
| HDMS | Hexamethyldisilazine |
| HDTMS | Hexadecyltrimethoxy silane |
| LbL | Layer-by-Layer |
| MF | Microfiltration |
| MPTMS | Mercaptopropyltrimethoxysilane |
| MTES | Methyltrimethoxysilane |
| MTS | Methyltrichlorosilane |
| NPs | Nanoparticles |
| o/w | Oil-in-water |
| OCA | Oil contact angle |
| OTS | Octadecyltrichlorosilane |
| PAR-b-PDMS | Polyarylester polydimethylsiloxane block copolymer |
| PBZ | Polybenzoxazine |
| PDA | Polydopamine |
| PDDA | Polydiallyldimethyl ammonium chloride |
| PDMS | Polydimethylsiloxane |
| PE | Polyethylene |
| PEI | Polyethylenimine |
| PFA | Polytetrafluoroethylene-perfluoropropylvinylethers |
| PFAS | Perfluoroalkylsilane |
| PFDT | Perfluorododecanethiol |
| PFOEMA | Polyperfluorooctylethylmethacrylate |
| PFOTS | Perfluorooctyltrichlorosilane |

| | |
|-------|--------------------------------|
| PFTOS | Perfluorooctyltriethoxysilane |
| PI | Polyimide |
| PMMA | Polymethylmethacrylate |
| PPS | Polypropylene sulfide |
| PS | Polystyrene |
| PTFE | Polytetrafluoroethylene |
| PU | Polyurethane |
| PV | Polyvinyl |
| PVA | Polyvinyl alcohol |
| PVAC | Polyvinyl acetate |
| PVC | Polyvinylchloride |
| PVD | Physical Vapor Deposition |
| PVDF | Polyvinylidene fluoride |
| RO | Reverse osmosis |
| SDBS | Sodium dodecylbenzenesulfonate |
| SS | Stainless steel |
| TEOS | Tetraethylorthosilicate |
| THF | Tetrahydrofuran |
| TMCS | Trimethyltrichlorosilane |
| UF | Ultrafiltration |
| UV | Ultraviolet |
| VTES | Vinyltriethoxysilane |
| w/o | Water-in-oil |
| WCA | Water contact angle |

Variables/symbols

| | |
|-------|--|
| f_1 | The total area of the solid under the liquid drop per unit projected area below the drop (m^2) |
| f_2 | The total area of the air under the liquid drop per unit projected area below the drop (m^2) |
| r | The ratio of the actual rough surface area to that of the smooth area (—) |

Greek letters

| | |
|----------|-----------------------------------|
| γ | Surface energy (mN/m) |
| θ | Static contact angle ($^\circ$) |

Subscripts

| | |
|-----|---------------|
| App | Apparent |
| CB | Cassie-Baxter |
| LG | Liquid-gas |
| LS | Liquid-solid |
| OA | Oil-air |
| SG | Solid-gas |
| W | Wenzel |
| WA | Water-air |
| Y | Young |

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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