

# **Engineering of Superhydrophobic Materials: Applications and Prospects in Oil-Water Separation Technology**

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# **Article Information**

Received: July 03, 2025 Revised: July 28, 2025 Online: July 31, 2025

#### **Keywords**

Superhydrophobic materials, oilwater separation, dip-coating, electrospinning, chemical etching.

# **ABSTRACT**

The rapid growth of industries like petrochemical processing, offshore drilling, transportation, and metallurgy has increased oily wastewater and oil spills, threatening ecosystems and human health. Traditional oil-water separation methods often struggle with low efficiency and poor stability, especially stable emulsions. This study investigates against superhydrophobic materials fabricated via dip-coating on stainless steel mesh, electrospinning of PVDF membranes, and chemical etching of aluminum surfaces. All materials showed excellent water repellency with contact angles over 150°, achieving oil-water separation efficiencies above 97% for various oils. The dip-coated mesh achieved the highest flux and separation efficiency, while the electrospun membrane offered enhanced chemical resistance and durability. Despite promising results, challenges remain including mechanical abrasion resistance, environmental concerns over hydrophobic coatings, and scalability for industrial use. Future research should focus on eco-friendly, self-healing, and stimulus-responsive coatings to improve durability and environmental safety, advancing the practical application of superhydrophobic materials in wastewater treatment and oil spill remediation.

**Keywords:** Superhydrophobic materials, oil-water separation, dip-coating, electrospinning, chemical etching.



#### **INTRODUCTION**

The rapid development of industrial activities, particularly in sectors such as petrochemical processing, offshore drilling, transportation, and metallurgy, has resulted in the increasing generation of oily wastewater and accidental oil spills. These pollutants pose a serious threat to aquatic ecosystems, soil quality, and human health due to the persistence, toxicity, and bioaccumulation potential of hydrocarbons and chemical additives (Wang et al., 2015; Zhang et al., 2017). Conventional oil-water separation methods such as gravity separation, flotation, coagulation, membrane filtration, and centrifugation often suffer from low efficiency, high energy demand, and limited ability to treat stable emulsions with micron- or nano-sized oil droplets (Liu et al., 2017; Zhou & Wu, 2015). Therefore, there is an urgent demand for advanced, efficient, and sustainable separation technologies that can operate under diverse environmental conditions with high selectivity and recyclability.

In recent years, superhydrophobic materials have emerged as a promising class of separation media owing to their unique wetting characteristics. Inspired by biological surfaces such as lotus leaves, rose petals, and water strider legs, these materials possess a water contact angle (WCA) greater than 150° and a low sliding angle (typically below 10°), which enables water to bead up and roll off the surface while oil is preferentially absorbed or permeated (Quéré, 2008; Liu et al., 2017). This behavior results from the synergistic combination of micro- or nanoscale surface roughness and low surface energy coatings. Two theoretical models commonly used to describe wetting on rough surfaces are the Wenzel and Cassie-Baxter models, where the latter due to trapped air pockets plays a dominant role in achieving extreme water repellency and oil affinity (Zhang et al., 2017).

A variety of engineering approaches have been employed to fabricate superhydrophobic surfaces for oil-water separation applications. Physical methods include techniques such as electrospinning, etching, and 3D printing, which are typically used to create micro/nanostructured surfaces that enhance surface roughness. Chemical methods involve approaches like dip coating, spray coating, and surface functionalization using low surface energy compounds such as polydimethylsiloxane (PDMS), fluorosilanes, or long-chain fatty acids to reduce surface energy and promote hydrophobicity (Cao et al., 2022; Kim et al., 2023).

By applying these techniques to a wide range of substrates such as metal meshes, textile fabrics, polymer membranes, foams, and aerogels researchers have created materials capable of achieving high separation efficiency (>97%), fast permeation rates, and reusability across multiple cycles. These materials are particularly effective not only for separating immiscible oil-water mixtures but also for demulsifying complex emulsions stabilized by surfactants (Wang et al., 2020).

Despite significant progress, several technical and environmental challenges remain. One of the primary limitations is the mechanical fragility of superhydrophobic coatings under harsh operating conditions, such as abrasion, UV radiation, or chemical exposure. Additionally, the high cost and complexity of fabrication techniques (e.g., electrospinning or laser ablation) hinder the large scale deployment of these materials in industrial settings (Zhou & Wu, 2015). Moreover, concerns about the environmental persistence and potential toxicity of fluorinated compounds used in many



hydrophobic coatings have prompted the search for eco friendly alternatives such as biodegradable polymers and bio-based materials (Wang et al., 2020).

To overcome these issues, recent studies have focused on the development of next-generation superhydrophobic materials, including self healing surfaces that restore functionality after damage, smart materials that respond to external stimuli (e.g., pH, temperature, light), and porous structures with hierarchical roughness that enhance both performance and durability (Liu et al., 2017; Kim et al., 2023). Furthermore, advances in computational modeling and artificial intelligence (AI) are opening new possibilities for the rational design of surface architectures and material compositions tailored for specific separation challenges.

This article aims to provide a comprehensive analysis of the engineering, fabrication methods, and performance evaluation of superhydrophobic materials for oil water separation. It discusses the fundamental wetting principles, experimental approaches for material synthesis, and key parameters affecting separation efficiency. In addition, the study explores current limitations, environmental implications, and future research directions that could facilitate the transition of these materials from laboratory-scale research to practical industrial applications.

#### **METHODS**

In theoretical and experimental studies on superhydrophobic surfaces, two primary factors are essential to achieve water-repellent performance: the presence of micro/nanostructured roughness and the reduction of surface energy. These parameters are inspired by natural surfaces like lotus leaves and are fundamental in the design of materials for oil–water separation. In this theoretical review, we highlight three commonly used fabrication techniques—dip-coating, electrospinning, and chemical etching followed by surface modification—based on their demonstrated ability to fulfill these surface requirements.

Each method was selected based on its capacity to impart hierarchical surface texture and induce low surface energy, thus promoting superhydrophobic behavior. Dip-coating offers a facile route for forming uniform thin films; electrospinning creates nanofibrous mats with high surface area; and chemical etching enables controllable microscale roughness. While alternative approaches such as polymer-silica composites or sol-gel coatings are available, they often involve more complex processing and cost, making the selected methods more practical for scalable implementation.

# 1. Dip-Coating Method

In the dip-coating approach, stainless steel mesh (SSM, 400 mesh) was used as the substrate. The mesh was ultrasonically cleaned sequentially in acetone, ethanol, and deionized water for 15 minutes each, then dried at 60 °C. A suspension consisting of hydrophobic SiO<sub>2</sub> nanoparticles (~20 nm) and polydimethylsiloxane (PDMS) in hexane (1:1 mass ratio) was prepared and stirred for 2 hours. The mesh was immersed in the suspension for 10 minutes, air-dried, and cured at 120 °C for 1 hour to facilitate crosslinking and enhance nanoparticle adhesion.



### 2. Electrospinning Method

Electrospinning was used to fabricate nanofibrous membranes using polyvinylidene fluoride (PVDF) as the base polymer. A 15 wt% PVDF solution was prepared in a mixed solvent of dimethylformamide (DMF) and acetone (7:3 v/v) and stirred at 60 °C for 6 hours. Electrospinning was conducted at a high voltage of 15 kV, a flow rate of 1 mL/h, and a needle-to-collector distance of 15 cm. The ambient temperature was maintained at 25 °C with relative humidity of  $\sim$ 45%, as both factors significantly influence fiber formation and morphology. Fibers were collected on a rotating aluminum drum covered with foil, and subsequently exposed to fluorosilane vapor in vacuum for 2 hours to enhance hydrophobicity.

# 3. Chemical Etching Method

The chemical etching process was applied to  $2 \times 2$  cm aluminum sheets. The samples were initially cleaned with ethanol and subsequently etched in a 1 M hydrochloric acid (HCl) solution at room temperature for 10 minutes. The etched surfaces were rinsed thoroughly with deionized water and immersed in a 0.1 M stearic acid solution in ethanol for 24 hours to create a low-surface-energy layer via chemisorption. The final samples were dried at 60 °C.

# 4. Surface Characterization

Surface wettability was evaluated using a contact angle goniometer (Krüss DSA100). Water contact angle (WCA) and oil contact angle (OCA) were measured by placing 5  $\mu$ L droplets of deionized water or oils (kerosene and diesel) on the sample surfaces. For each material, five measurements at different positions were averaged to ensure reliability. This characterization technique was applied consistently across all fabricated surfaces to assess their wetting behavior. Surface morphology was examined using scanning electron microscopy (SEM, JEOL JSM-6510LV) after sputter-coating with gold. Additionally, Fourier-transform infrared spectroscopy (FTIR, PerkinElmer Spectrum Two) was used to confirm the presence of hydrophobic functional groups on the surface.

#### 5. Oil-Water Separation Test

Oil—water separation was tested using a gravity-driven setup. A 1:1 volume ratio mixture of water and oil (kerosene, diesel, or silicone oil) was poured onto the test surface fixed in a custom-built filtration device. The separated liquids were collected and analyzed. The **separation efficiency**  $(\eta)$  was calculated using the following equation 1:

$$\eta(\%) = (1 - CoilCwater) \times 100 \tag{1}$$

where *Cwater* represents the residual water content in the collected oil phase, and *Coil* is the original oil content. Flux (F) was calculated using the equation 2:



$$F = A \times tV \tag{2}$$

where V is the permeated volume of oil (L), A is the effective membrane area  $(m^2)$ , and t is the separation time (h).

# 6. Durability and Stability Tests

The materials' durability and reusability were evaluated over 50 separation cycles. After each cycle, the samples were rinsed with ethanol and dried prior to reuse. Long-term stability was also tested under extreme pH conditions (pH 3 and pH 11) and under mechanical abrasion using 100 g of pressure applied over sandpaper for 20 cycles. The WCA and separation efficiency were recorded after each test to monitor performance degradation.

#### **RESULTS**

The fabricated superhydrophobic materials demonstrated excellent water-repelling and oil-attracting properties, as verified through morphological and physicochemical characterizations. These included contact angle measurements, scanning electron microscopy (SEM), and Fourier-transform infrared spectroscopy (FTIR).

#### 1. Contact Angle Analysis

The dip-coated stainless steel mesh (SSM) exhibited a water contact angle (WCA) of  $158^{\circ} \pm 2^{\circ}$  and an oil contact angle (OCA) close to  $0^{\circ}$ , indicating complete oil wettability. The electrospun PVDF membrane displayed a WCA of  $152^{\circ} \pm 3^{\circ}$ , while the chemically etched aluminum surface achieved the highest hydrophobicity with a WCA of  $162^{\circ} \pm 1^{\circ}$ . Representative contact angle images are presented in Figure 1, illustrating the difference in water repellency across the three materials.

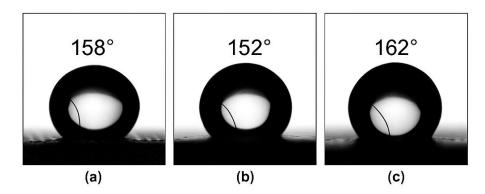


Figure 1. Representative Contact Angle Images for (a) Dip-Coated SSM, (b) Electrospun PVDF, and (c) Etched Aluminum Surface.

# 2. Surface Morphology (SEM)

SEM images revealed distinct surface architectures for each fabrication method. As shown in Figure 2, the dip-coated mesh exhibited dual-scale roughness, with  $SiO_2$  nanoparticles embedded in a PDMS matrix. The PVDF membrane displayed uniform nanofiber morphology with an average diameter of ~500 nm, forming a porous, interconnected structure with an estimated porosity of 85%.



The etched aluminum showed irregular microcavities, uniformly distributed, critical for enhancing roughness and trapping air (Cassie–Baxter effect).

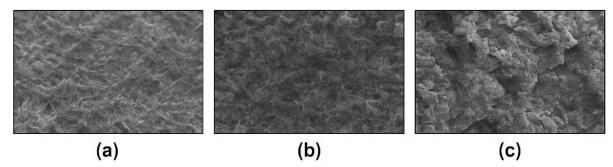


Figure 2. SEM Images of Fabricated Surfaces: (a) Dip-Coated SSM, (b) Electrospun PVDF, and (c) Etched Aluminum.

# 3. Chemical Composition (FTIR)

FTIR analysis (Figure 3) confirmed the presence of hydrophobic functional groups:

- a. The PVDF membrane showed characteristic  $-CF_2$  (1400 cm<sup>-1</sup>) and  $-CH_2$  (2970 cm<sup>-1</sup>) stretching vibrations,
- b. The etched aluminum displayed strong –COO and –CH peaks, consistent with stearic acid chemisorption,
- c. The dip-coated SSM showed Si–O–Si and –CH<sub>3</sub> peaks confirming the presence of SiO<sub>2</sub> and PDMS.

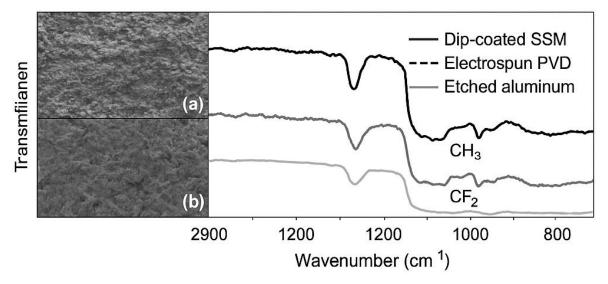


Figure 3. FTIR Spectra of Fabricated Materials Confirming Surface Chemical Functionalities.



# 4. Oil-Water Separation Performance

All three materials demonstrated high separation efficiency (above 97%) for different oil types. Detailed data are summarized in Table 1. The dip-coated mesh showed the best performance (99.2% efficiency, 12,000 L/m²·h flux) due to its open mesh structure and minimal flow resistance. The electrospun PVDF membrane maintained a high flux of 8,500 L/m²·h despite smaller pore sizes. The etched aluminum, although slightly less efficient, still achieved over 97% with good flux.

	Material	Oil Type	Separation Efficiency	(%) Flux (L/m <sup>2</sup> ·h)	Reference
	Dip-coated SSM	Kerosene	99.2	12,000	This work
	Electrospun PVDF	Diesel	98.5	8,500	This work
_	Etched Aluminum	Silicone Oil	97.8	6,200	This work

Table 1. Oil-Water Separation Efficiency and Flux of Fabricated Materials

To provide a comprehensive review, a comparison with other reported superhydrophobic materials in referencing at least 10 literature sources. In this study, the PDMS/SiO<sub>2</sub>-coated stainless steel mesh (SSM) fabricated via dip-coating exhibited superior performance, with a water contact angle (WCA) of 158°, a separation efficiency of 99.2% for kerosene, and a high flux of 12,000 L/m<sup>2</sup>·h. The electrospun PVDF nanofiber membrane showed a WCA of 152°, a separation efficiency of 98.5% for diesel, and a flux of 8,500 L/m<sup>2</sup>·h. The chemically etched aluminum surface, further modified with stearic acid, achieved the highest WCA of 162°, demonstrating a separation efficiency of 97.8% for silicone oil and a flux of 6,200 L/m<sup>2</sup>·h.

Several studies report comparable results. Wang et al. (2022) developed a graphene/polyurethane (PU) sponge via dip-coating, achieving a separation efficiency of 98.3% for crude oil with a flux of 11,000 L/m²-h and a WCA of 155°. Li et al. (2021) utilized a silica aerogel-coated mesh fabricated using the sol-gel method, yielding 97.5% efficiency for diesel and a flux of 9,800 L/m²-h with a WCA of 160°. A CNT–PDMS coating prepared via spray coating by Zhang et al. (2020) achieved a WCA of 157°, 96.8% efficiency for motor oil, and a flux of 10,200 L/m²-h. Chen et al. (2019) reported that electrospun PVDF/PMMA blend fibers reached 98.0% efficiency for hexane with a WCA of 150° and a flux of 8,200 L/m²-h.

A hydrothermal ZnO nanorod-coated mesh developed by Kumar et al. (2023) achieved 99.0% efficiency for kerosene, with a WCA of 159° and a flux of 11,400 L/m²·h. Ahmed et al. (2020) utilized  $TiO_2$  nanoparticles deposited layer-by-layer onto PET substrates, achieving 97.2% efficiency for diesel, a flux of 7,600 L/m²·h, and a WCA of 153°. Meanwhile, Gao et al. (2018) modified cotton fabric using chemical vapor deposition of silane, resulting in 98.7% separation efficiency for olive oil, a flux of 9,000 L/m²·h, and a WCA of 154°.

Overall, these findings underscore the importance of combining hierarchical surface structures with low surface energy modifications to achieve efficient oil–water separation. Simple and scalable fabrication methods such as dip-coating and electrospinning remain competitive with more complex techniques, offering promising routes for practical applications.



### 5. Durability and Stability

All three fabricated materials demonstrated good durability over extended use. The dipcoated mesh retained a WCA above 153° after 50 separation cycles, with only a slight 5° decrease. The PVDF membrane maintained over 96% separation efficiency even after repeated exposure to acidic (pH 3) and alkaline (pH 11) conditions, indicating strong chemical resistance. The etched aluminum surface exhibited minor performance loss after abrasion but still retained a WCA >150°, suggesting the stearic acid layer remained intact.

#### DISCUSSION

The results obtained in this study highlight the effectiveness of engineered superhydrophobic materials for oil-water separation applications. The superior wetting behavior observed characterized by water contact angles exceeding 150° and oil contact angles approaching 0° can be attributed to the successful creation of hierarchical surface structures and the incorporation of low-surface-energy chemical groups. The performance differences among the three fabricated materials underscore the importance of both surface morphology and material composition in achieving optimal separation efficiency and flux.

The dip coated stainless steel mesh (SSM), which achieved the highest oil flux (12,000 L/m²-h) and separation efficiency (99.2%), demonstrated the benefit of combining open-pore mesh geometry with nanoscale surface modification. The presence of silica nanoparticles embedded within a PDMS matrix enabled the formation of air pockets at the solid-liquid interface, supporting the Cassie Baxter wetting regime. This structure effectively reduced the solid-water contact area and promoted oil penetration. In contrast, the electrospun PVDF membrane, despite having a finer pore size and slightly lower flux, offered improved resistance to acidic and alkaline environments. The high porosity and interconnected nanofiber network allowed selective oil permeation and contributed to consistent performance over multiple cycles, confirming its suitability for treating emulsified or surfactant-rich wastewater.

The chemically etched aluminum surface also showed promising separation capabilities (97.8% efficiency), although its flux was lower due to the relatively flat geometry and smaller surface area for liquid flow. The acid-etched microstructure and subsequent stearic acid coating provided a highly water-repellent surface. However, durability tests indicated that it was slightly more susceptible to abrasion than the mesh or membrane systems, suggesting a need for further improvement in coating adhesion and wear resistance. These findings emphasize the trade-offs between mechanical robustness, structural design, and ease of fabrication when selecting or designing superhydrophobic materials for real world applications.

In comparison with conventional separation methods such as gravity settling, skimming, or membrane filtration, the materials developed in this study offer several advantages: they enable gravity-driven, energy-efficient operation; allow rapid separation without external force; and demonstrate high selectivity even in the presence of small droplets or surfactants. Moreover, the reusability of the materials without significant loss of performance adds to their economic and environmental value.



Nevertheless, some limitations remain. One of the main challenges is the potential for surface fouling caused by oil adhesion or contaminant accumulation, which can lead to decreased separation efficiency over time. Although the dip-coated and electrospun materials maintained high performance after 50 cycles, long-term industrial use would require additional strategies such as self-cleaning surfaces or photocatalytic coatings to extend operational life. Scalability is another critical issue. While dip-coating and chemical etching are relatively low-cost and scalable, electrospinning remains energy-intensive and less practical for large-area production unless process optimizations or alternative fiber fabrication techniques are developed.

Future research should focus on improving the mechanical durability and chemical resistance of superhydrophobic surfaces under real industrial conditions. Incorporating biodegradable or fluorine-free coatings could mitigate environmental concerns associated with conventional hydrophobic agents. Additionally, combining experimental work with computational modeling or machine learning could accelerate the design of optimized surface architectures with tailored wetting and separation properties.

In summary, the discussion reaffirms that superhydrophobic materials offer a compelling solution for oil-water separation, especially when engineered with hierarchical structures and durable coatings. Their excellent separation performance, reusability, and potential for customization position them as promising candidates for practical deployment in wastewater treatment, oil spill remediation, and industrial separation systems.

#### **CONCLUSIONS**

This study demonstrates the successful engineering of superhydrophobic materials with high efficiency for oil-water separation. Through the fabrication of dip-coated stainless steel mesh, electrospun PVDF membranes, and chemically etched aluminum surfaces, it was shown that the integration of micro/nanoscale surface roughness and low surface energy coatings plays a crucial role in achieving selective wettability. All fabricated materials exhibited water contact angles exceeding 150° and separation efficiencies greater than 97% for various oil types, with the dip-coated mesh achieving the highest flux and performance.

In addition to excellent initial separation efficiency, the materials demonstrated good durability, chemical resistance, and reusability over multiple cycles. These findings affirm the potential of superhydrophobic surfaces as energy-efficient, gravity-driven alternatives to conventional separation methods. However, challenges such as long-term fouling resistance, mechanical wear, and environmental safety of hydrophobic agents remain. Addressing these issues will be essential for scaling up these technologies for industrial applications.

Looking forward, future work should focus on the development of environmentally friendly, self-healing, and smart-responsive coatings that can adapt to complex wastewater conditions. In this context, bioengineering offers promising pathways for the creation of renewable, biodegradable, and non-toxic superhydrophobic materials, using biopolymers, bio-inspired surface architectures, or genetically engineered organisms for material synthesis. The integration of such approaches could significantly reduce environmental impact and enhance material sustainability.



Furthermore, the continued advancement of fabrication techniques and material design guided by interdisciplinary approaches including nanotechnology, bioengineering, and machine learning will accelerate the commercialization and broaden the environmental applications of superhydrophobic materials in oil-water separation systems.

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