

Superhydrophilic mesh membrane coated with flower like TiO₂ nanosheets for oil/water mixtures separation

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Abstract

The development of novel membranes capable of efficiently and accurately separating oil and water is essential for effective oily wastewater treatment. In this study, we successfully fabricated a superhydrophilic stainless steel mesh membrane (SSM) by incorporating flower-like TiO₂ nanosheets (TiO₂-FLn) onto its surface via a hydrothermal process. The unique nanostructure of TiO₂-FLn imparts exceptional superhydrophilicity and underwater superoleophobicity to the SSM, enabling efficient oil-water separation. The SSM/TiO₂-FLn membrane demonstrates outstanding performance, exhibiting a separation efficiency of 98%, an ultrahigh flux of 75,228 L/m²·h, remarkable recyclability, and long-term durability. Furthermore, the SSM/TiO₂-FLn membrane maintained a separation efficiency consistently above 94% even after 15 cycles. Additionally, it exhibited exceptional thermal and chemical stability under various conditions, including different pH ranges and NaCl concentrations. The successful fabrication of this innovative membrane holds significant promise for advancing oily wastewater treatment practices.

Keywords: oil/water separation; SSM/TiO₂-FLn; superhydrophilicity; stainless steel mesh.

Introduction

The increasing demand for oil extraction, fueled by industrial expansion and rising living standards, has resulted in significant environmental challenges. Oily wastewater, a byproduct of extraction and industrial processes, poses a substantial threat to human health and the environment if not treated appropriately [1]. Oil spills into aquatic ecosystems further exacerbate these concerns, leading to substantial energy loss and the release of numerous pollutants into the ecosystem [2]. To mitigate these issues, the development of advanced, durable, and cost-effective technologies for separating water from oily wastewater is imperative for both environmental sustainability and resource recovery [3]. Common techniques employed in oily wastewater treatment include oil-water separators, gravity separation, ultrasonic treatment, electrocoagulation, and biological processes [4, 5]. However, limitations such as low treatment efficiency, the generation of secondary pollutants, and the large scale of these systems have consistently hindered their effective application. These challenges

have motivated researchers to explore novel and more efficient methods to enhance treatment processes and mitigate their adverse impacts [6]. Given the interfacial nature of oil-water mixtures, a promising strategy for their separation involves creating porous surfaces that exploit contrasting wettability properties. These porous materials, capitalizing on the differential wetting behavior of water and oil, can efficiently separate the two phases [7]. Owing to their unique characteristics, superwetable surfaces with tunable wettability have emerged as a highly effective and straightforward solution for oil-water separation [8, 9]. These materials can be broadly classified into two categories: superhydrophobic/superoleophilic and underwater superhydrophilic/superoleophobic surfaces [10, 11]. Porous surfaces exhibiting superhydrophobic/superoleophilic properties have attracted significant attention due to their ability to repel water while strongly adhering to oil, leading to extensive research in recent decades. A wide range of materials have been investigated as wetting surfaces, including sponge- and foam-based materials [12], fabrics [13], stainless steel mesh membranes (SSMs) [14], carbon-based materials and their derivatives [15], particles and powders [16], and other raw materials. Among these, SSMs have emerged as an innovative solution for oil-water separation due to their numerous advantages. These membranes, combining robust mechanical properties, tunable pore sizes, and the ability to integrate various functionalities, offer significant potential to overcome the challenges of traditional separation methods [17]. To fabricate SSMs with special wettability properties, researchers have focused on modifying the surface structure of the membranes using various techniques to achieve different wetting behaviors. Generally, fabrication methods for SSMs with specific wettability properties typically involve physical, chemical, and physicochemical approaches such as dip coating, spray deposition, electrochemical deposition [18], chemical vapor deposition [19], thermal methods [20], and solution-phase deposition [21]. A variety of materials are employed to create surfaces with distinct wettability properties. For example, Jinmei He et al. developed a superhydrophilic copper mesh coated with ZIF-67@Cu(OH)₂ nanowires for separating oil/water mixtures. Their membrane exhibited a permeation flux of 23,854 L. m⁻².h⁻¹ and a separation efficiency of 99%, with excellent recyclability and durability [22]. Moreover, Lingrui Zhang et al. used a superhydrophilic mesh membrane coated with tannic acid-ZIF-8@MXene composites to separate oil-water mixtures. The as-prepared mesh membrane demonstrated outstanding properties such as durability, antifouling, recyclability, a high flux permeation of 69,093 L. m⁻².h⁻¹, and an underwater oil contact angle of 154.2° [23]. Xiangying Yin et al. used the hydrothermal method to grow MOF-303 crystals on the surface of the mesh membrane, creating a surface with superhydrophilicity and underwater superoleophobicity properties. The prepared mesh membrane was able to separate oil-water mixtures and soluble dyes simultaneously, with a permeation flux of more than 12,308 L. m⁻².h⁻¹ and a separation efficiency of 99.35% [24]. Despite the promising performance of earlier SSMs, modified SSMs frequently encounter practical limitations, including high reagent costs, poor mechanical strength, and limited chemical stability, especially under harsh conditions [25]. Additionally, oily wastewater is often generated in complex environments with high salt concentrations or varying pH levels, making it challenging to treat. Therefore, materials with specific properties such as availability, cost-effectiveness, and high stability are crucial for developing SSMs with special wettability. This can play an integral role in overcoming the challenges associated with treating oily wastewater. TiO₂, a low-cost, nontoxic material with excellent thermal, chemical, and mechanical stability, has garnered

significant research attention as a coating for imparting hydrophilicity and oleophobicity to substrates. Various TiO_2 nanostructures, including nanoparticles, nanotubes, nanofibers, and nanowires, have been used to construct coatings on meshes and membranes for oil/water separation, and the underlying factors influencing the separation process have been extensively studied [26-30]. Among the mentioned TiO_2 structures, flower-like TiO_2 nanosheets are a relatively novel material and have been mentioned less frequently in other studies as a coating material on the surface of mesh membranes for separating oil-water mixtures. The hydroxyl functional groups in TiO_2 confer hydrophilicity to surfaces, making it a promising material for creating surfaces with tailored wettability properties [31].

This study investigates a novel mesh membrane coated with flower-like TiO_2 nanosheets (SSM/ TiO_2 -FLn) for efficient oil-water separation with superhydrophilic and underwater superoleophobic special wettability. We comprehensively characterized the membrane's surface morphology, wettability, and physicochemical properties. Additionally, we evaluated its performance in separating both light and heavy oil-water mixtures, focusing on separation efficiency, special wettability, stability, and recyclability.

Materials and methods

Materials

The stainless steel mesh membrane (steel 320, pore size 44 μm) was obtained from Behlor Company (Tehran, Iran). Nitric acid (HNO_3 , 65%), ammonia solution (25%), titanium butoxide (TBOT), isopropyl alcohol (IPA), chloroform (CHCl_3), toluene ($\text{C}_6\text{H}_5\text{CH}_3$), n-hexane (C_6H_{14}), dichloromethane (CH_2Cl_2), acetone ($\text{C}_6\text{H}_6\text{O}$), ethanol ($\text{C}_2\text{H}_6\text{O}$), and hydrochloric acid (HCl) were purchased from Merck Company and Sigma Aldrich. All chemicals were of analytical grade and used without further purification.

Fabrication of SSM/ TiO_2 -FLn

The SSMs underwent a multi-step preparation process. Initial cleaning involved ultrasonic treatment with acetone, ethanol, and water. Oxide layers were removed by immersing the membranes in a 3M HNO_3 solution. A 0.075 M TBOT solution was prepared and used to coat the cleaned membranes, followed by drying at 60 °C. This process was repeated three times. Hydrothermal treatment at 600 °C in a high-temperature tube furnace was then conducted to hydrolyze TBOT and form TiO_2 . Finally, the membranes were thoroughly washed with deionized water and ethanol [32].

Characteristics of prepared mesh membranes

To conduct a thorough characterization of the membranes, we employed a suite of advanced analytical techniques. Field emission scanning electron microscopy (FESEM- Mira 3-XMU) was utilized to visualize surface morphology, while energy-dispersive X-ray spectroscopy (EDS) provided insights into chemical composition. Contact angles of water in air (WCA) and underwater oil (UWOCA) were measured using a contact angle instrument (KRUSS GmbH, Germany). Furthermore, the crystalline structure of TiO_2 was analyzed through X-ray diffraction (XRD). Finally, infrared spectroscopy (FTIR) (model: TENSOR 27, Germany) was employed to investigate the structural characteristics of the chemical bonds formed within the coated mesh membrane.

Oil-water separation and wettability experiments

A simple experimental setup was employed to evaluate the oil/water separation capabilities of SSMs. Two tubular Plexiglas containers, joined by an aluminum flange,

held the SSMs. A mixture of various oils (n-hexane, toluene, chloroform, and dichloromethane) and water (1:1 ratio, total volume 100 mL) was introduced from the top and allowed to flow over the membrane surface. Separation occurred solely due to gravity, without external force, and the separated water was collected in a beaker placed below. Equations (1) and (2) were used to calculate separation efficiency and permeated flux.

$$R = \frac{m_1}{m_0} \times 100\% \quad (1)$$

$$Flux = \frac{V}{AT} \quad (2)$$

Where m_0 , m_1 denote the weights of deionized water before and after separation and T (h), A (m^2), and V (L) represent the filtration time, effective filtration membrane area, and filtration volume, respectively [33]. The superhydrophilicity and underwater superoleophobicity of the SSMs were assessed by measuring the contact angle of water droplets in air and oil droplets underwater.

Reusability and stability of prepared membranes

To investigate the reusability of SSM/TiO₂-FLn membranes, 15 repeated cycles of oil-water separation were conducted, monitoring permeation flux and separation efficiency in each cycle. To evaluate membrane stability, thermal stability was assessed by exposing the coated membranes to a range of temperatures (100-500 °C), and chemical stability was evaluated by exposing them to alkaline and acidic conditions (pH=4-12) as well as various salt concentrations (0.5-2 M), while measuring permeate flux and separation efficiency.

Results and discussion

Membranes characterization

The surface morphology of pristine and coated mesh membrane are shown in Fig. 1. As it can be seen in Fig. 1a-d pristine SSM exhibits an interwoven structure with arranged wires of approximately 34 μm diameter and pore sizes of 44 μm .

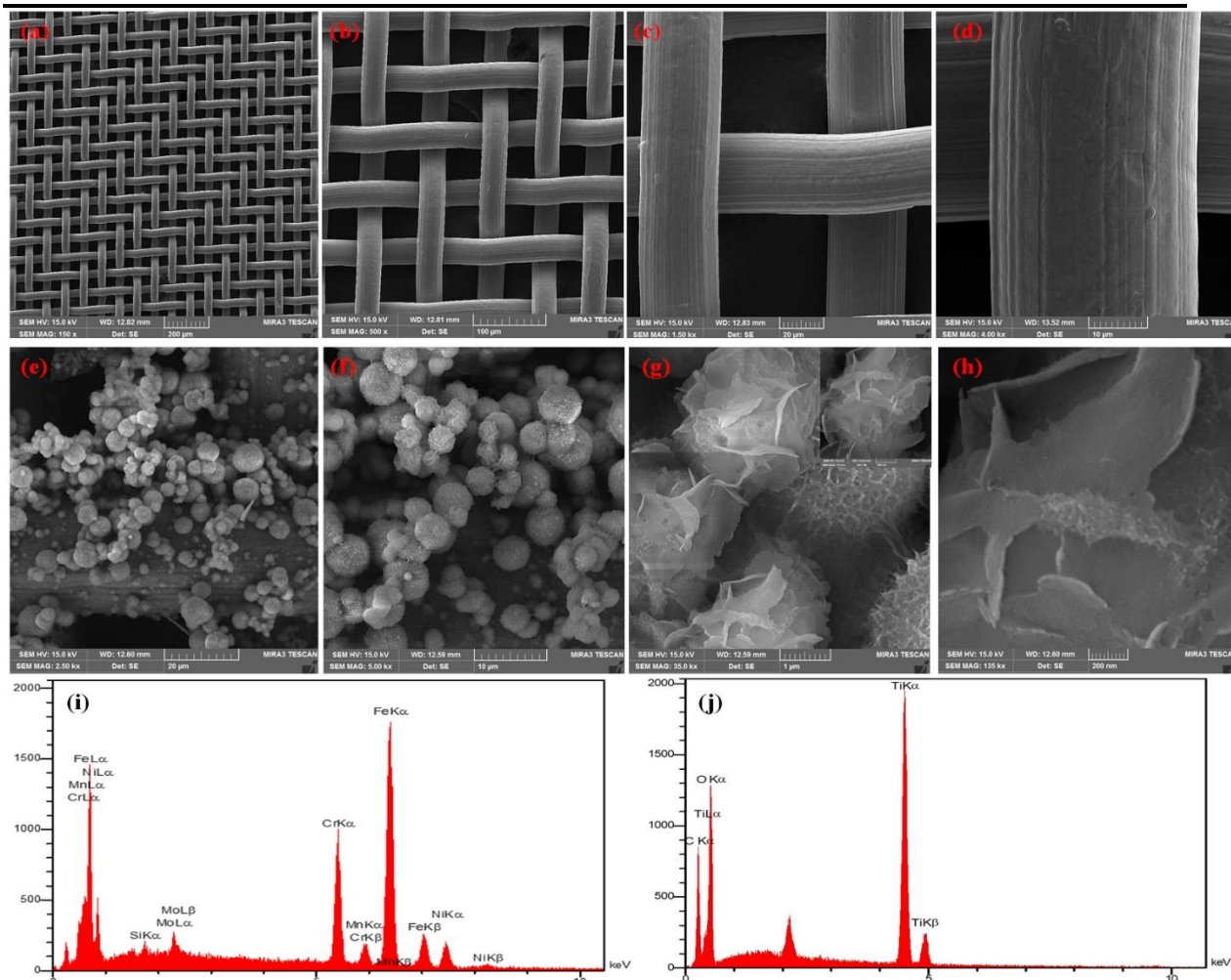


Fig. 1. SEM images of pristine mesh membrane (a-d), and coated mesh membrane with flower like TiO_2 nanosheet structure (e-h), the EDX images of pristine (i), and SSM/ TiO_2 -FLn (j).

The hydrothermal process effectively coated the pristine mesh membrane with TiO_2 particles, as illustrated in Fig. 1e-h. The resulting TiO_2 particles exhibit a unique flower-like morphology with a thin structure and clearly visible nanosheets, as shown in Fig. 1g-h. The hydrothermal process increases the roughness of the pristine SSM's smooth surface and decreases its pore size, making it suitable for oil/water separation [34]. Additionally, a comparison of EDX images of the pristine SSM and SSM/ TiO_2 -FLn reveals the successful coating of TiO_2 particles onto the surface of the SSM.

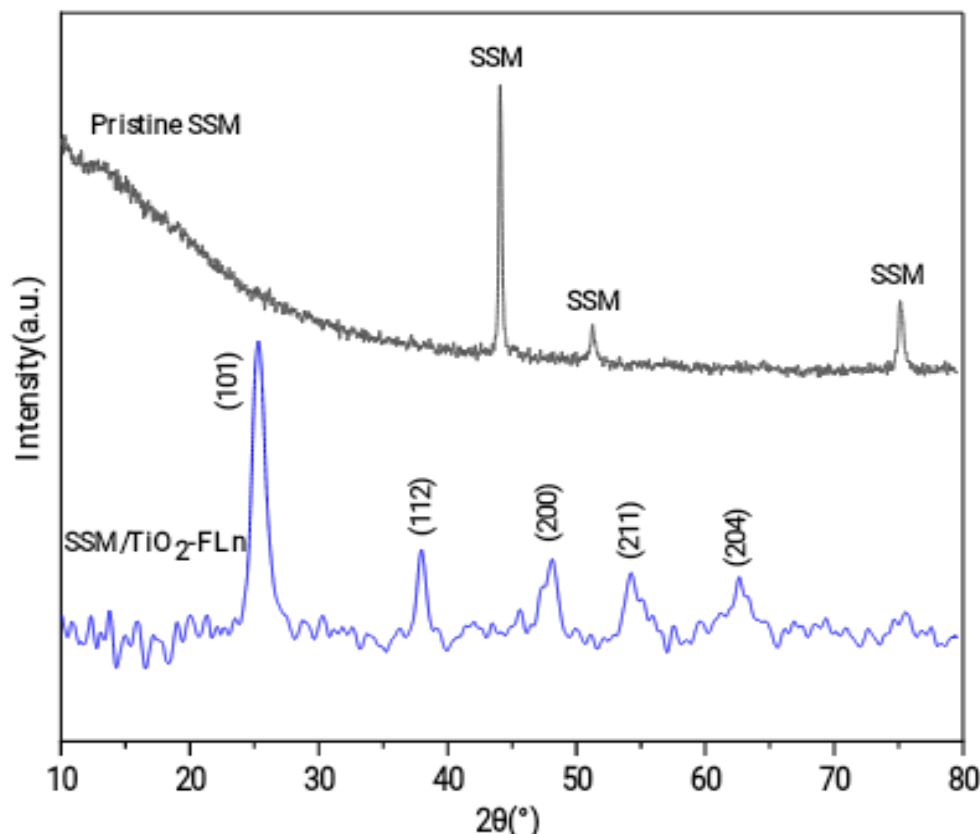


Fig 2. XRD patterns of pristine SSM and SSM/TiO₂-FLn

Fig. 2 illustrates the XRD patterns of the pristine SSM and the modified SSM/TiO₂-FLn. As shown in Fig. 2, the diffraction peaks observed at 43.7°, 50.9°, and 75.0° correspond to the characteristic peaks of the pristine mesh surface [35]. XRD analysis of SSM/TiO₂-FLn confirms the anatase-phase structure of the flower-like TiO₂ nanosheets, as evidenced by the characteristic peaks at 25.3°, 37.8°, 48.0°, 54.9°, and 62.7° in the spectrum, which correspond to the (101), (112), (200), (211), and (204) crystal planes (JCPDS 00-021-1272) [36].

ATR-FTIR spectroscopy was employed to analyze the composition of pristine SSM and SSM/TiO₂-FLn, as shown in Fig 3. The spectrum of pristine SSM reveals the presence of O–H and CO₂ stretching vibrations at 3570–3900 cm⁻¹ and 2100–2400 cm⁻¹, respectively [37]. A prominent peak at 3400–3500 cm⁻¹ indicates the presence of hydroxyl groups and physically adsorbed water on the TiO₂ surface [38]. The characteristic peak at 880 cm⁻¹ confirms the formation of TiO₂ through O–Ti–O bond stretching vibrations [39]. Additionally, a peak at 1604 cm⁻¹ suggests the chemisorption of water molecules due to H–O–H bending vibrations [40]. The presence of Ti–O bonds is confirmed by the peak at 1438 cm⁻¹ [41].

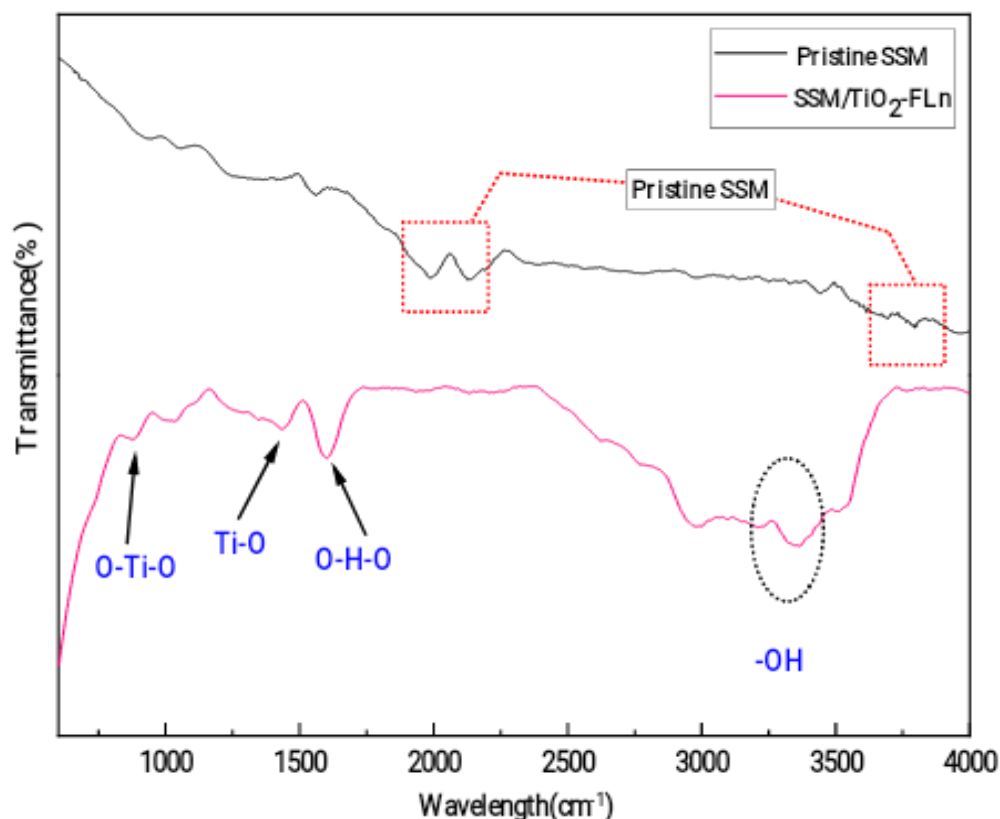


Fig. 3. ATR-FTIR absorbance spectra (b) of the pristine SSM and SSM/TiO₂-FLn.

Wettability and separation performance

The surface wettability of the mesh membranes was evaluated using WCA and UWOCA measurements. As shown in Fig. 3a, the pristine SSM exhibits a WCA of 112° and a UWOCA of 128°. After modification with flower-like TiO₂ nanosheets, the hydrophilicity of the membranes improved significantly, resulting in a decreased WCA of 0° and a considerable increase in UWOCA to 159° (Fig. 3b). Additionally, the underwater superoleophobicity of SSM/TiO₂-FLn was investigated by measuring the underwater contact angle of various oils. Fig. 3c shows that SSM/TiO₂-FLn exhibits UWOCAs of 159°, 160°, 156°, and 155° for hexane, toluene, chloroform, and dichloromethane, respectively. These results indicate the crucial role of flower-like TiO₂ nanosheets in imparting special wettability properties to the membranes. Furthermore, the anti-oil adhesion and superoleophobic nature of SSM/TiO₂-FLn were examined by observing the behavior of a hexane droplet attached to the membrane surface. As shown in Fig. 3d, the hexane droplet detached from the surface without adhering, demonstrating the anti-oil adhesion properties of the modified membrane.

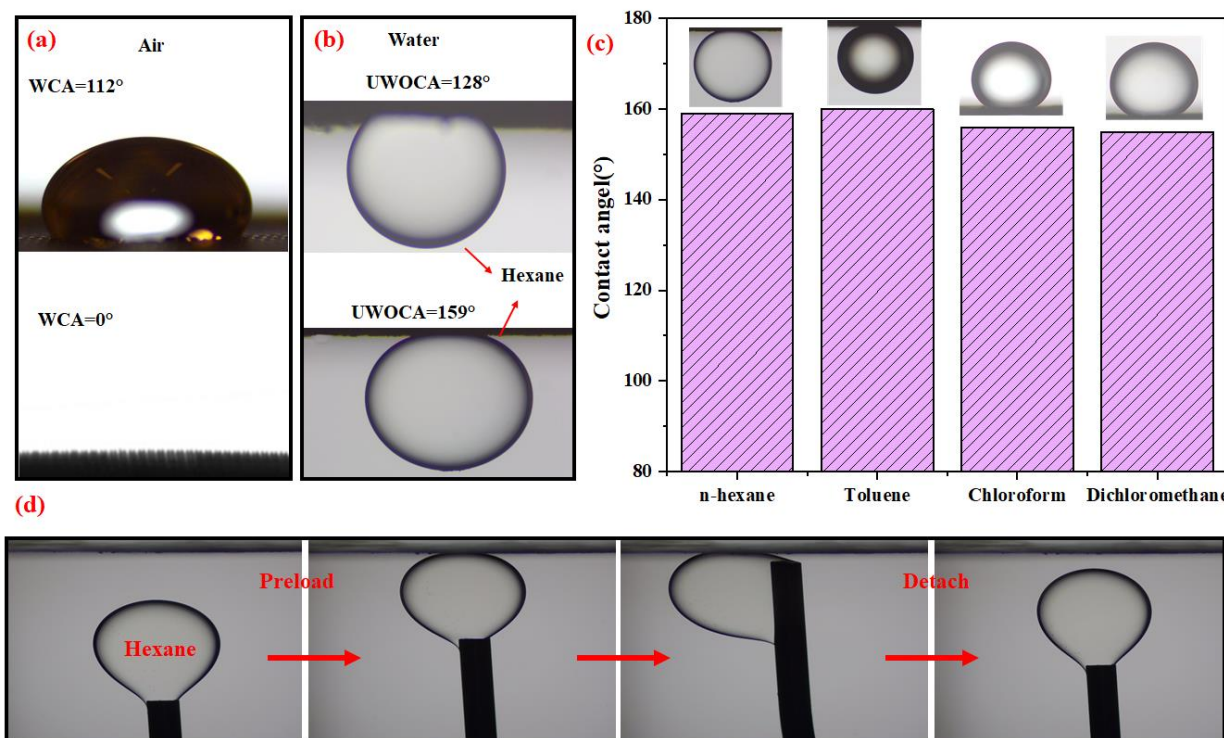


Fig. 4. WCA and UWOCA of pristine and modified SSM (a,b), UWOCA of different oils (c), and dynamic underwater oil resistance.

The separation efficiency and flux of various light and heavy oil-water mixtures were examined under gravity. The SSM/TiO₂-FLn membrane exhibited excellent oil-water separation performance, with water passing through readily while oil was retained above the mesh. All oil-water mixtures achieved separation efficiencies exceeding 96%, with n-hexane demonstrating the highest efficiency at 98% (Fig. 5). Additionally, all mixtures exhibited high flux permeability, with n-hexane and chloroform showing the highest (75,228 L/m²·h) and lowest (73,570 L/m²·h) values, respectively. Generally, the physicochemical properties of the membrane surface play a crucial role in its special wettability, which is a critical parameter for oil-water separation [42]. As previously discussed, the presence of hydrophilic functional groups like hydroxyl on the SSM/TiO₂ surface, as confirmed by ATR-FTIR analysis, is a primary factor influencing the membrane's wettability.

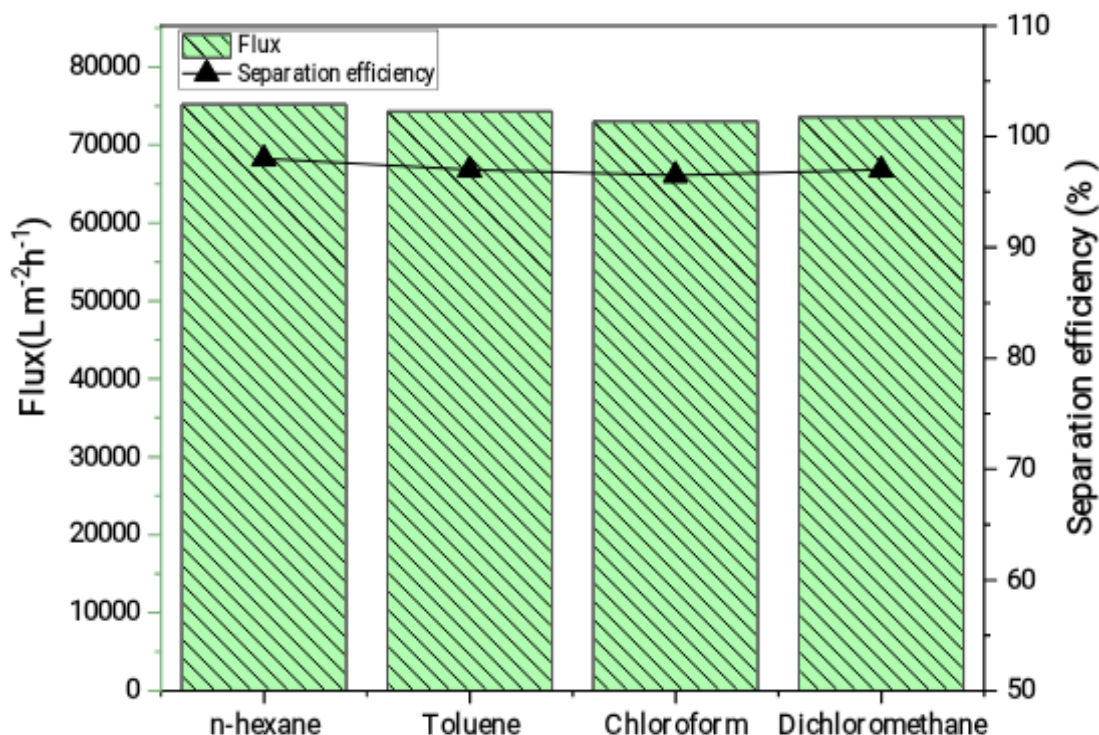


Fig. 5. Flux and separation efficiency of SSM/TiO₂-FLn

The durability properties of mesh membranes

To evaluate the reusability, thermal stability, and chemical stability of the SSM/TiO₂-FLn membrane, a series of experiments were conducted. For reusability assessment, 15 consecutive cycles of oil-water separation were performed, as depicted in Fig. 6a. Despite minor fluctuations, the permeation flux and separation efficiency remained consistently above 75,000 L/m²·h and 94%, respectively, demonstrating excellent reusability. To assess chemical stability, the membrane was immersed in solutions with varying pH values (1-12) for 12 hours. As shown in Fig. 6b, the permeation flux and separation efficiency exhibited only slight decreases under these conditions, indicating minimal impact on coating stability. To evaluate thermal stability, the membrane was exposed to different temperatures (100-600 °C). The results, presented in Fig. 6c, demonstrate that the membrane maintains its superhydrophilicity up to 300 °C. However, at higher temperatures (500 °C), the UWOCA decreased to 121°, suggesting limited thermal stability. Additionally, the separation efficiency decreased slightly at higher temperatures. The effect of NaCl concentration on UWOCA and oil-water separation efficiency is shown in Fig. 8d. As observed, increasing NaCl concentration resulted in a slight decrease in UWOCA from 157° to 154° and a minor reduction in oil-water separation efficiency to 97%.

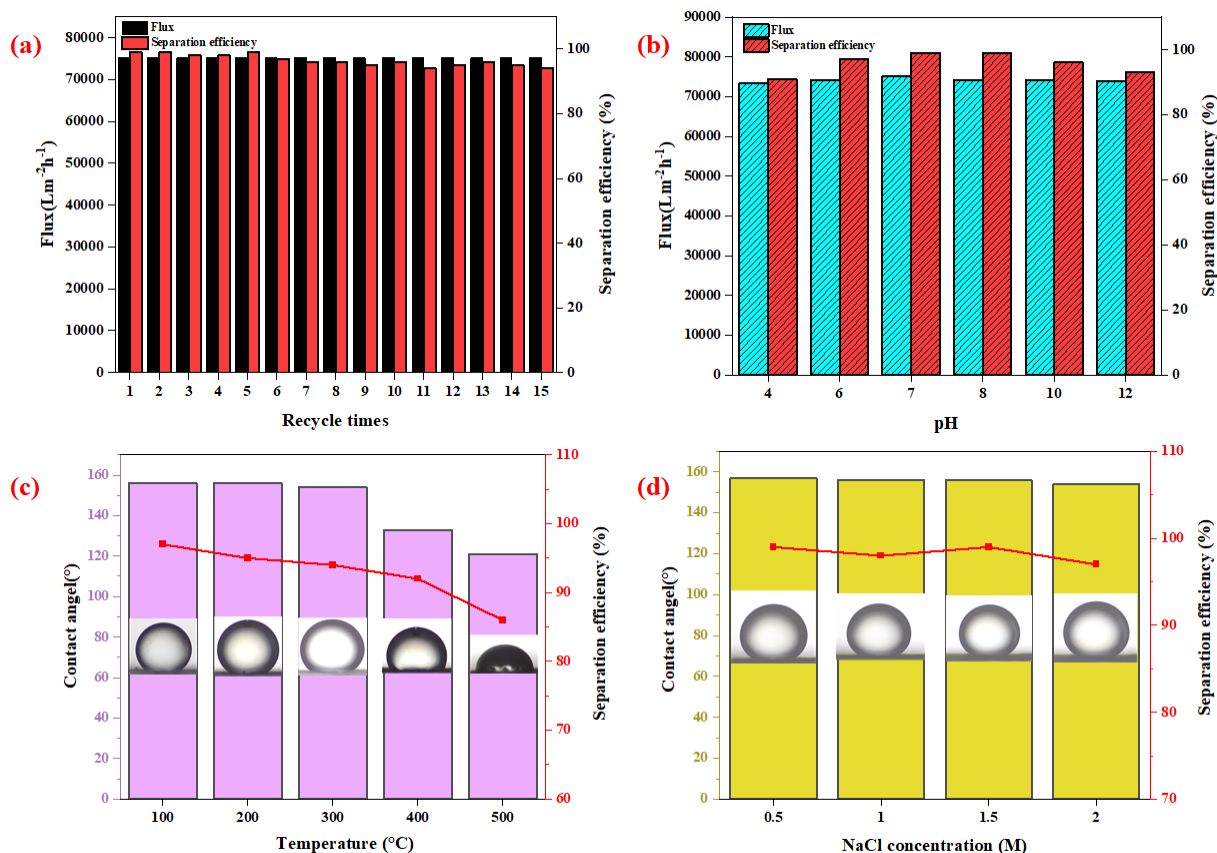


Fig. 6. Reusability properties (a), chemical tolerance at different pH values (b), thermal stability (c), and different NaCl concentrations stability (d) of SSM/TiO₂-FLn.

Conclusion

This study presents the development of a groundbreaking superhydrophilic and underwater superoleophobic mesh membrane with exceptional oil-water separation capabilities. The membrane demonstrated a separation efficiency of 99% and a flux permeation exceeding 75,000 L/m²·h. Characterization using FESEM, EDX, XRD, and ATR-FTIR confirmed the successful growth of flower-like TiO₂ nanosheets on the mesh membrane surface. The SSM/TiO₂-FLn exhibited remarkable special wettability, with underwater oil contact angles consistently exceeding 157°. Moreover, the membrane demonstrated exceptional recyclability, thermal and chemical stability, and maintained its performance under harsh acidic, alkaline, and salty conditions. These findings highlight the potential of this novel membrane for practical applications in oil-water separation.

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