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A Review on Photocatalytic Oil/Water Separation Membranes Based on Ti-MOFs

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Abstract

The escalating global issue of oil-water pollution demands advanced separation technologies that combine efficiency, sustainability, and reusability. Titani-um-based metal-organic frameworks (Ti-MOFs) have emerged as promising candidates due to their unique structural and functional properties. This review systematically examines the design, fabrication, and application of Ti-MOFs for oil-water separation, highlighting their superior attributes, including exception-al chemical stability, photocatalytic self-cleaning capability, and tunable wetta-bility. Key fabrication strategies such as in situ growth, physical compositing, and interfacial crosslinking are discussed, with emphasis on optimizing mem-brane performance through hierarchical pore engineering and heterojunction coupling. Practical applications demonstrate their efficacy in industrial wastewater treatment and marine spill remediation. Despite these advances, challenges remain in scalable synthesis and long-term stability under harsh conditions. Future directions include multifunctional membrane design and ecological impact assessments to facilitate real-world deployment. Ti-MOFs represent a transformative approach to sustainable oil-water separation, bridg-ing the gap between laboratory innovation and industrial implementation.

Keywords

Ti-MOFs; oil-water separation; photocatalytic; membranes

1. Introduction

With the rapid development of human industry and the extensive use of oily substances, global water and oil pollution problems have become increasingly severe, posing threats to human health and causing significant resource waste. Industrial oil-water mixtures and marine oil spills are the most common forms of pollution. Oily wastewater seriously endangers human health and ecological balance. Oil-water separation technology can efficiently achieve complete separation of the oil and water phases, recovering oil from industrial oily wastewater and purifying water quality to prevent indiscriminate discharge. The purified water can be recycled, reducing water resource consumption. Additionally, this technology can successfully separate spilled oil from seawater, purify marine environments, enable the recovery and reuse of leaked crude oil, and minimize petroleum resource waste. Therefore, research on oil-water separation technology holds significant theoretical and practical value for mitigating water and oil pollution and reducing resource waste [1-2].

Metal-organic frameworks (MOFs) are porous crystalline materials with numerous potential applications. They are coordination compounds formed by metal ions or clusters and organic ligands containing potential voids. MOFs exhibit several superior properties: their pore structures can be precisely controlled by adjusting the size of the central metal or organic ligands (i.e., tunable porosity); they typically possess ultrahigh specific surface areas; their synthesis is relatively simple under mild reaction conditions; and their coordination bonding enhances chemical and thermal stability. MOFs have been widely applied in energy storage, chemical separation, chemical sensing, catalysis, biomedicine, and water treatment [3-7]. Semiconductor photocatalysis is an effective strategy for wastewater treatment, and the development of highly active catalyst systems has attracted significant attention from researchers. The performance of MOFs-based oil-water separation membranes primarily depends on their ligand structures and the characteristics of uncoordinated metal sites, which directly regulate the hydrophilic/hydrophobic balance[8]. The separation mechanisms of MOFs membranes include size exclusion and hydrophilic/hydrophobic interactions, which are governed by two types of specially wettable MOFs materials—hydrophobic/oleophilic MOFs and hydrophilic/oleophobic MOFs. Hydrophobic/oleophilic MOFs membranes repel water while allowing oil phases to diffuse, permeate, and separate from water. Compared with hydrophobic MOFs, hydrophilic MOFs membranes are less prone to oil fouling, facilitating underwater oleophobicity. Most hydrophilic MOFs membranes also exhibit oleophobicity, forming a protective water layer that effectively prevents oil penetration and membrane fouling[9-11]. Ti-based MOFs, such as MIL-125(Ti), have garnered considerable attention due to their superior stability, catalytic activity, larger specific surface area, higher porosity, and tunable surface energy compared with other common MOFs.

This review summarizes the applications of Ti-MOFs in oil-water separation, focusing on their structural characteristics, preparation methods (e.g., physical compositing, chemical in situ growth), and photocatalytic self-cleaning functionalities. The practical performance of these materials is evaluated, and future research directions are proposed to address existing challenges.

2. Overview of Metal-Organic Frameworks (MOFs)

2.1. Structural Features and Chemical Composition

Metal-organic frameworks (MOFs) are porous crystalline materials formed by the self-assembly of metal ions or clusters and multidentate organic ligands via coordination bonds. Their basic structural units consist of metal nodes (e.g., Zr^{4+}, Zn^{2+}, Fe^{3+}) and organic ligands (e.g., carboxylates, imidazolates, pyridines). The metal nodes serve as "connection hubs," linking with electron-donating groups (–COOH, $–NH_2$, etc.) on the organic ligands to construct three-dimensional periodic networks. Common synthesis methods for MOFs include solvothermal synthesis, microwave-assisted synthesis, electrochemical synthesis, and mechanochemical synthesis.

2.2. Functional Properties and Application Advantages

Metal-organic frameworks (MOFs) exhibit remarkable advantages for oil/water separation due to their distinctive structural characteristics. These materials possess ultrahigh specific surface areas (1000-7000 m^2/g) and tunable micro/mesoporous structures (0.5-3.0 nm), providing abundant adsorption sites and efficient mass transfer channels[12-13]. The surface wettability can be precisely modulated through ligand functionalization (e.g., hydrophobic alkyl chains or hydrophilic amino groups), with fluorinated MOFs demonstrating exceptional oil selectivity (>95%). Notably, Ti/Zr-based MOFs (e.g., MIL-125, UIO-66) exhibit outstanding chemical stability in aqueous and acidic environments owing to their robust metal-ligand bonds (Ti-O, Zr-O). More importantly, certain MOFs (e.g., Ti-MOFs) integrate both adsorption and photocatalytic degradation capabilities, enabling self-cleaning regeneration through emulsified oil decomposition under UV irradiation. These superior properties make MOFs significantly outperform conventional adsorbents (e.g., activated carbon, polymer membranes) in separation efficiency, environmental adaptability, and renewability, thereby establishing a theoretical foundation for developing high-performance Ti-MOFs-based oil/water separation membranes.

2.3. Preparation Methods for Ti-MOFs Oil-Water Separation Membranes

2.3.1. Physical Composite Membranes

Physical composite membranes are primarily fabricated via vacuum filtration, dip coating, or layer-by-layer (LBL) assembly. Representative methods include secondary growth and LBL deposition. The secondary growth method involves nucleation and growth of MOFs crystals on a substrate chemically bonded with seed layers, ensuring uniform nucleation and high-quality membrane formation. For example, Kasik et al. employed a reactive seeding method to grow ZIF-68 membranes on ZnO-modified α-Al_2O_3 substrates, demonstrating selective permeation fluxes for liquid separation[14]. The LBL method relies on hydrogen bonding, coordination interactions, or electrostatic forces to sequentially introduce MOFs building blocks, enabling precise control over film formation. As illustrated in Figure 1, Gao et al. utilized γ-ray irradiation to activate polymer substrates, followed by LBL assembly to prepare ultrathin MOFs coatings with tunable thickness and morphology[15].

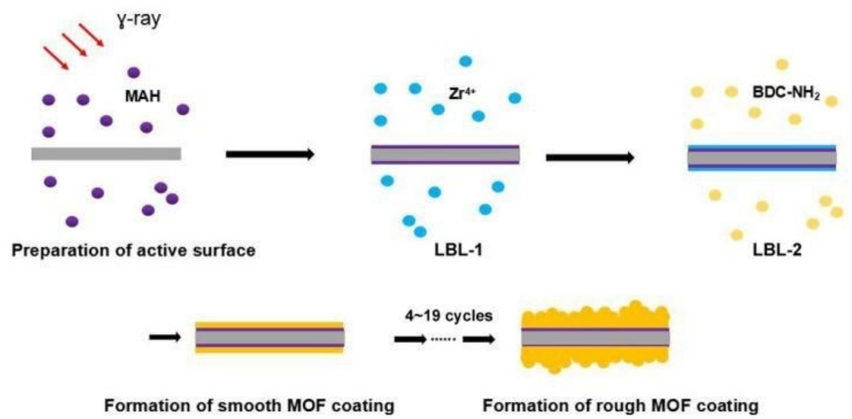

Figure 1. Schematic of the LBL assembly process for preparing ultrathin MOFs coatings via γ-ray irradiation-induced surface activation[15].

2.3.2. Chemical In Situ Growth

Direct in situ growth is a one-step process involving nucleation, growth, and crystallization of MOFs on substrates. This method entails immersing pretreated substrates in precursor solutions containing metal ions and organic ligands to facilitate gradual MOFs film formation[16]. Chemical modification of substrates can introduce heterogeneous nucleation sites, enhancing crystallinity and defect-free membrane formation. For instance, Zhou et al. employed inorganic modifiers (Zn^{2+}, Co^{2+}, Cu^{2+}) to electrodeposit ZnO nanorods, $Co(OH)_2$ nanosheets, and Cu_2O nanocubes on stainless steel mesh, serving as active sites for growing ZIF-8, ZIF-67, and HKUST-1 membranes (Figure 2)[17]. This approach strengthens mechanical stability through covalent bonding between Ti-MOFs and substrates.

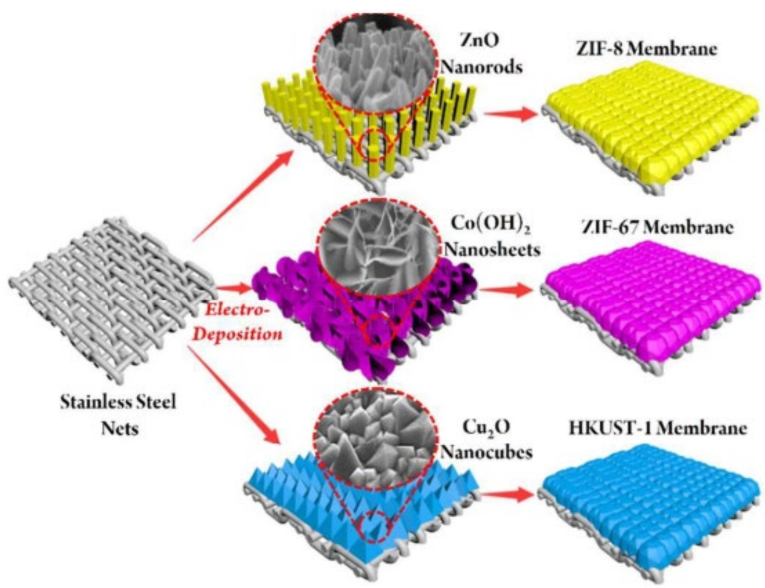

Figure 2. Schematic of electrodeposition-assisted in situ growth for three types of MOFs membranes[17].

2.3.3. MOFs-Derived Membranes

MOFs-derived membranes are obtained by calcining MOFs precursors to form porous TiO_2 or hybrid composite structures. For example, Campagnol et al. proposed a four-stage electrochemical deposition mechanism (nucleation, growth, coalescence, and detachment) for HKUST-1 films (Figure 3)[18]. Electrophoretic deposition can also drive charged MOFs nanoparticles to electrodes under an applied voltage, forming MOFs membranes.

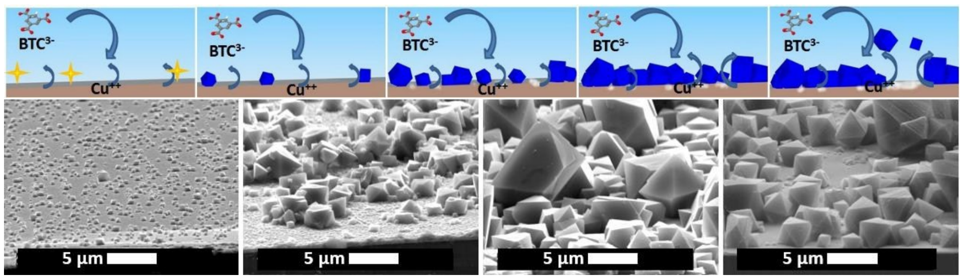

Figure 3. Proposed mechanism of MOFs anodic electrodeposition and SEM pictures, taken at a 75° angle with the normal, of the four phases: I Initial nucleation (a), II growth of islands (b), III intergrowth (c) III and detachment (d). Copper-coated wafer substrate, 2 V vs counter electrode, after 10 s, 10 min, 60 min and 125 min[18].

2.4. Unique Advantages of Ti-MOFs in Oil-Water Separation

Titanium-based metal-organic frameworks (Ti-MOFs), constructed from Ti-oxo clusters as secondary building units, demonstrate superior oil/water separation capabilities through their unique structural and functional attributes. The robust Ti-O coordination bonds (>500 kJ/mol) confer exceptional chemical and hydrothermal stability, enabling operation in acidic and humid environments where conventional MOFs (e.g., ZIF-8) degrade. Their inherent semiconductor properties facilitate photocatalytic self-cleaning via UV-induced ROS generation, enhancing recyclability by >300% compared to non-photoactive MOFs. The biocompatible Ti(IV) centers resist leaching, addressing environmental concerns associated with Zr/Cu-based analogues. Surface engineering through fluorinated ligands enables precise wettability control (superhydrophobicity $\theta>150°$), while multifunctional integration permits solar-thermal viscosity reduction of heavy oils. When composited with polymeric substrates (PU/PVDF)[19], these materials maintain structural integrity under dynamic conditions while preserving mass transport pathways, offering a sustainable platform for advanced separation technologies.

2.5. Composite Membrane Fabrication and Structural Optimization

Loading Ti-MOFs onto polymer substrates is critical for functional membrane fabrication[20]. Current strategies include:

2.5.1. Substrate Surface Modification

Plasma treatment, UV grafting, or chemical oxidation (e.g., HNO_3-treated PVDF) introduces active groups (–COOH, $–NH_2$) to strengthen MOFs adhesion[21-24]. Polydopamine (PDA) or polyethyleneimine (PEI) interlayers can serve as anchoring sites[25-26]. Figure 4 illustrates the fabrication process of PES-MWCNTs composite membranes via vacuum filtration. As demonstrated by Li et al[27]. Coating polydopamine-modified carbon nanotubes onto PES microfiltration membranes successfully yielded superhydrophilic membranes with exceptional separation capabilities.

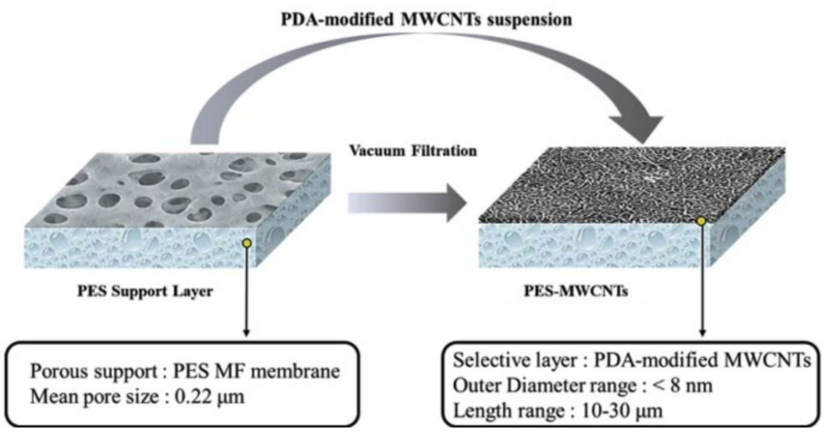

Figure 4. Schematic diagram of the facile preparation of PES-MWCNTs membranes through the deposition of PDA-modified MWCNTs onto the top surface of the PES MF membrane via a vacuum-filtration process[27].

2.5.2. In Situ MOFs Growth

When employing the solvothermal method, the polymer membrane is immersed in a MOFs precursor solution (e.g., containing a titanium alkoxide and organic ligands), allowing for the in situ crystallization and growth of MOFs on the membrane surface or within its pores. For instance, Li et al. demonstrated that by reacting a CZTS precursor solution with an FTO substrate under solvothermal conditions, a uniform nanocrystalline film could be directly grown on the conductive glass surface. This approach eliminates the need for secondary coating steps and significantly enhances interfacial adhesion . Similarly, MOFs precursor solutions can undergo in situ crystallization within polymer membranes via solvothermal treatment, enabling controlled growth within the pore channels. This strategy improves interfacial bonding and structural homogeneity

2.5.3. Hybrid Process Optimization

Blending MOFs with polymer solutions followed by non-solvent-induced phase separation (NIPS) forms composite membranes. Dispersants (e.g., PVP) prevent MOFs aggregation, and coagulant additives (e.g., ethanol) minimize MOFs loss[28-30].

2.5.4. Interfacial Crosslinking Reinforcement

Silane coupling agents (e.g., APTES) form covalent Si–O–Si bridges at MOFs-polymer interfaces, improving mechanical durability[31]. For example, APTES-grafted melamine sponges (MS) enhance SiO_2 nanoparticle adhesion (Figure 5). Dynamic bonds (e.g., metal-ligand coordination, Schiff bases) can further enable self-healing properties.

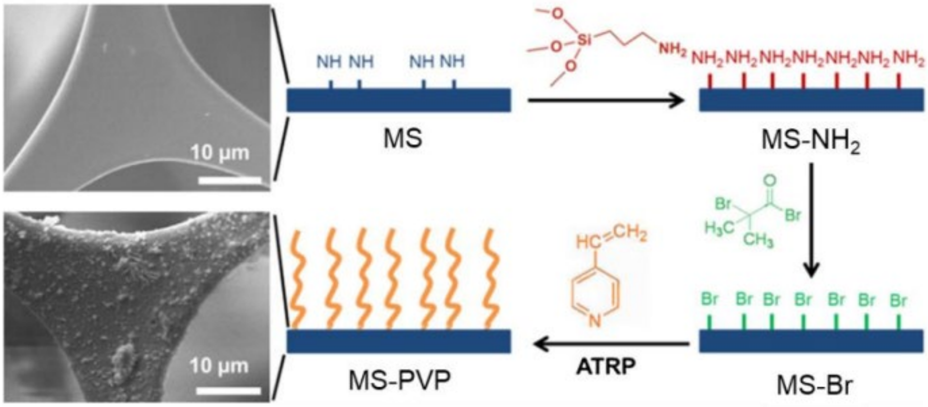

Figure 5. Schematic of preparation of MS-PVP sponges with SEM images of MS and MS-PVP sponges[31].

Ti-MOFs are ideal for separating complex oil-water emulsions, especially surfactant-stabilized systems and harsh environments.

3. Advances in Photocatalytic Ti-MOFs Oil-Water Separation Membranes

3.1. Photocatalytic Mechanism of Ti-MOFs

Ti-MOFs (e.g., MIL-125(Ti)) consist of Ti-oxo clusters (e.g., $Ti_8O_8(OH)_4$) and organic ligands (e.g., terephthalic acid, 2-aminoterephthalic acid). Their porous structures (800–1500 m^2/g) and tunable bandgaps (1.6–3.8 eV) confer semiconductor properties for photocatalysis. For instance, NH_2-MIL-125(Ti) extends light absorption to the visible region ($\lambda > 420$ nm) via $-NH_2$ functionalization, while Ti^{3+}/Ti^{4+} redox pairs activate H_2O to generate hydroxyl radicals for oil degradation[32].

3.2. Design Strategies for Photocatalytic-Separation Synergistic Membranes

3.2.1. Surface Wettability Control

Chemical modifications (e.g., fluorination, silanization) precisely tune surface energy. Liu et al. fabricated a superhydrophobic membrane (water contact angle: 162°) with 99.1% separation efficiency for emulsified oils (0.5–2 μm droplets) and a flux recovery rate (FRR) of 91% under UV light[33]. Its photocatalytic degradation rate (0.32 h^{-1}) surpassed that of TiO_2 membranes (0.15 h^{-1}).

3.2.2. Heterojunction Coupling

Z-scheme heterojunctions promote directional charge transfer. Chen et al. coupled NH_2-MIL-125(Ti) with S-dopedg-C_3N_4(SCN), forming Ti–N bonds that increased $\cdot O^{2-}$ yield by a factor of 3.2 and achieved 98.7% crude oil separation in saline water[34-36].

3.2.3. Hierarchical Pore Engineering

Electrospinning or NIPS constructs micro-/mesoporous gradients. Li et al. embedded MIL-125(Ti) in PAN nanofibers, achieving a water flux of 1520 $L\cdot m^{-2}\cdot h^{-1}\cdot bar^{-1}$ and 99.5% diesel/water emulsion rejection.

3.3. Practical Applications of Ti-MOFs Separation Membranes

3.3.1. Industrial Oily Wastewater Treatment

Membrane technology offers high energy efficiency and sustainability for oily wastewater remediation. Xu et al. developed spiral-wound MIL-125(Ti)/PVDF modules for a petrochemical plant (10 m^3/d capacity), maintaining 93% COD removal and <15% flux decline over 200 h[37]. SEM-EDS confirmed Ti-MOFs layer integrity (Ti uniformity > 95%).

3.3.2. Marine Oil Spill Remediation

Durability is critical for real-world applications. Zhang et al. designed floating Ti-MOFs/carbon fiber membranes for Bohai Bay oil spills, adsorbing 14 g/g of viscous crude (800 mPa·s) and degrading >85% hydrocarbons under sunlight[38-39]. Wave-driven self-cleaning enabled 50 reuse cycles with only 7.3% capacity loss.

4. Conclusions and Perspectives

Titanium-based metal-organic frameworks (Ti-MOFs) demonstrate remarkable potential for oil/water separation applications owing to their distinctive Ti-oxo clusters (bond energy >500 kJ/mol) and tunable surface properties. These materials exhibit exceptional chemical stability, photocatalytic self-cleaning capability (>3 times enhanced recyclability), and superior separation selectivity (>95% oil removal efficiency). When integrated with polymer substrates and functional modifications, Ti-MOFs membranes have achieved outstanding performance in industrial wastewater treatment (93% COD removal) and marine oil spill remediation (14 g/g adsorption capacity with >85% photocatalytic degradation). However, key challenges remain in: (1) developing cost-effective, scalable synthesis methods; (2) improving long-term stability under harsh operational conditions; (3) enabling multifunctional integration; and (4) conducting comprehensive environmental impact assessments. Addressing these limitations will facilitate the translation of Ti-MOFs technology from laboratory research to practical implementation, offering sustainable solutions for water purification and environmental remediation.

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