Review

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Nanofibers for Oil-Water/Water-Oil Separation

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Highlights

1. Members advancements in oil/water separation technologies utilizing electrospun nanofiber membranes have shown promising results.
2. The performance of Janus nanofiber membranes specifically designed for oil-water separations has yielded significant findings.
3. Membranes developed through innovative techniques for oil/water separation present a hopeful outlook for their application in promoting a sustainable environment.

Abstract

Water pollution poses a significant environmental challenge that has garnered considerable attention, primarily due to its role in depleting vital resources. The separation of oil from water presents a complex issue for industries, particularly when large quantities of stable oil/water emulsions are released. This article reviews recent advancements, particularly over the past seven years, in the membrane separation of oil-water mixtures utilizing nanofiber membranes through filtration and absorption methods. Among the various techniques for fabricating membranes, electrospinning has emerged as a favored approach due to its ease of mass production and the potential for integrating other functional materials at the nanoscale. This method has gained significant interest in developing innovative nanofibrous membranes characterized by selective wettability, optimized pore structures, and high specific surface areas. Electrospinning is recognized as the most versatile technique for producing nanofibers embedded with diverse active agents. Several strategies have been explored, including the electrospinning of polymer blends rather than single polymeric materials, surface modifications through coating or grafting, and the incorporation of nanofillers to create mixed matrix membranes. Notably, numerous efforts have been made to manipulate surface hydrophobicity and oleophobicity by designing hierarchical and Janus structures. The future of electrospinning technology appears promising for the design and fabrication of next-generation materials for oil-water separation.

Keywords: membranes; oil/water emulsion; polymers.; electrospinning; polluted water; Janus membranes

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1. Introduction

Water is essential for life, making conservation critical for environmental health. Water pollution, caused by harmful substances, threatens human safety and aquatic habitats, leading to more deaths annually than war. Major pollution sources include oil spills from offshore drilling and significant oil from factories and urban areas, which endanger drinking water and marine life by reducing oxygen levels. The existence of water-in-crude oil emulsions raises production and transportation expenses and may cause equipment malfunctions, pipeline corrosion, and operational challenges such as catalyst poisoning in downstream processes. Presence of water-in-crude oil emulsions increases production and transportation costs and can lead to equipment failures, pipeline corrosion, and operational issues such as catalyst poisoning in downstream processes.

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Membrane technologies are vital for oil/water separation, with nanofiber membranes offering advantages due to their superwetting properties. Research focuses on advancements in nanofiber production for treating oily wastewater, emphasizing the importance of material properties in enhancing separation efficiency. Fiber-based membranes exhibit unique characteristics that improve oil-water separation, presenting a promising solution for environmental challenges. Nanofiber membrane materials manufactured via electrostatic spinning for oil/water separation have become one of the emerging technologies to treat oil/water emulsions.

2. Fabrication Methods of Nanofibers

Numerous techniques are available for the fabrication of nanofibers, with electrospinning emerging as the preferred method due to its capacity to produce fibers characterized by a high surface area-to-volume ratio and a substantial number of inter/intra pores. Various electronic techniques are employed throughout the fabrication process. Electrospinning primarily encompasses two methodologies: solution electrospinning and melt electrospinning. The solution electrospinning technique often necessitates the use of potentially hazardous solvents, along with an additional solvent extraction process, which may result in diminished productivity. To enhance productivity, several advanced devices have been developed, including multijet systems that utilize a single needle, multijet systems with multiple needles, and needleless configurations. For commercial applications of nanofibers, mass production is crucial, typically achieved through multineedle electrospinning (MNE) or needleless electrospinning (NLE). The MNE system represents a straightforward approach that improves productivity and facilitates the fabrication of composite fibers. Kamin et al. [1] investigated the production of polymeric fibers at the nanoscale using a melt-blowing technique and their applications in marine oil spill remediation. They also examined the manipulation of process conditions in the melt-blowing process to create nanofibers suitable for oil/water separation. The high productivity and porous structure of polystyrene (PS) fibers indicate that this method is a promising strategy for fabricating porous fibers for oil spill remediation. However, significant time and effort must be invested before large-scale production can be achieved with adequate reliability.

3. Membrane Design

The design of nanofiber membranes for oil-water separation often involves surface modification or the creation of hierarchical or Janus structures. These methods enhance the membranes' separation efficiency by providing special wettability and morphological characteristics. Surface modification is often performed by polymer blending, surface coating, surface grafting, incorporation of nanofillers (mixed matrix membranes) and others. Hierarchical structures in membranes enhance their separation capabilities by utilizing capillary forces for fluid transport.

3.1. ENMs (Electrospun Nanofiber Membranes) Without Modification

Lin and Rutledge [2] studied the separation of oil-in-water emulsions stabilized by various surfactants using electrospun polyamide nanofiber membranes. They focused on dodecane emulsions stabilized by anionic, cationic, non-ionic, and zwitterionic surfactants, tested under dead-end and crossflow filtration at 2 psi. The membrane achieved a high oil rejection rate of 92.7 ± 1.5%, meeting EPA standards, especially for anionic surfactants in crossflow filtration. Barani et al. [3] explored polymer nanofibers as coalescing filters for separating secondary oil emulsions from oily wastewater at a semi-industrial scale, fabricating nanofibrous membranes from electrospun polystyrene (PS), polyacrylonitrile (PAN), and polyamide 6 (PA6) on polyester (PET) substrates.

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3.2. Chemical and Physical Surface Modification Techniques

3.2.1. Polymer Blending

Wang et al. [4] enhanced the mechanical strength of a composite membrane using a simple blend-electrospinning method with minimal carbon nanotubes (CNTs) in polyvinylidene fluoride (PVDF). The resulting CNTs@PVDF composite membrane exhibited significant improvements, including a water contact angle of 152°, porosity over 86%, and oleophilic characteristics, with an oil permeation time of 91.63 ms. Du et al. [5] explored electrospun membranes of polyethersulfone (PES) and sulfonated poly(ether ketone) (SPEEK) for oil/water separation, finding that the membranes' hydrophilicity could be adjusted by varying the PES to SPEEK mass ratio. Jiang et al. [6] developed nanofiber membranes with multi-stage roughness from a PVDF-silica blend solution, achieving exceptional separation efficiency (99 ± 0.1%) under gravity-driven conditions and a high separation flux of 1857 ± 101 L·m−2·h−1. These PVDF-SiO2 membranes demonstrated excellent multi-cycle performance and stable chemical resistance, making them advantageous for oil-water separation. Shakiba et al. [7] modified polyacrylonitrile (PAN) electrospun nanofibers with polyaniline (PANI) to improve oil/water emulsion separation, utilizing the hydrophilic properties of PANI's amino groups. PAN/PANI membranes were fabricated through two methods: blending PANI with PAN in the pre-electrospinning solution and coating PANI onto PAN nanofibers via in situ polymerization of aniline.

3.2.2. Surface Grafting

Surface grafting is a sophisticated technique used to immobilize hydrophilic polymer chains on membrane surfaces, creating a stable fouling resistance layer. This process requires the prior introduction of reactive groups on the membrane, achievable through initiator sites or exposure to low-temperature plasma, ultraviolet, γ-ray, or electron beam radiation. The surface-grafted polymer can be synthesized using either the 'grafting to' or 'grafting from' methodologies.

Research by Ma et al. [8] shows that modified polyimide (PI)-based membranes exhibit exceptional flux, high separation efficiency, and commendable reusability in oil/water separation applications. The fabrication of superhydrophobic/superoleophilic membranes involves immersing a polyimide (PI)-based nanofibrous membrane in a mixture of water, ethanol, ammonia, and dopamine, followed by modification with 1H, 1H, 2H, 2H-perfluorodecanethiol (PFDT). The resulting PFDT/PDA/PI membrane demonstrates remarkable performance in oil/water separation, achieving a flux of 8018.5 ± 100 L m−2 h−1 and separation efficiencies exceeding 99%. Additionally, Li et al. [9] successfully prepared cellulose nanofibers (CNFs) aerogels by grafting poly(N, N-dimethylamino-2-ethyl methacrylate) (PDMAEMA) polymer brushes, enhancing surface hydrophobicity. Furthermore, nanofibrous membranes with superhydrophilic and underwater superoleophobic characteristics have been created by grafting acrylic acid onto plasma-treated electrospun polystyrene/polyacrylonitrile (PS/PAN) membranes, as reported by Yi et al. [10].

3.2.3. Surface Coating

Surface coating enhances properties like hydrophobicity and oleophobicity. Naseem et al. [11] used recycled cellulose triacetate (TAC) film to create nanofiber membranes via electrospinning, incorporating graphene oxide (GO) and titanium dioxide (TiO2) for oil/water separation, achieving superhydrophilicity and improved water permeability. Eang and Opaprakasit [12] produced superhydrophobic PLA nanofibers treated with alkyl ketene dimer (AKD), demonstrating high oil absorption rates, particularly for diesel and cooking oils, with capacities exceeding 10 g/g fibers and over 10 cycles of adsorption-desorption. Zhang et al. [13] developed a superhydrophobic polyacrylonitrile (PAN) nanofibrous membrane with embedded Au nanoparticles, achieving 95%-97% separation efficiency for low viscosity oils and effective catalytic degradation of organic dyes. Zhang et al. [14] coated biodegradable electrospun stereocomplex polylactide (sc-PLA) nanofibers with gallic acid (GA)-TiO2, resulting in superhydrophilicity and efficient separation of various

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oil/water mixtures. Moghaddasi et al. [15] investigated water/vegetable oil emulsion separation using copolyamide 6,10 (coPA) mats coated with MXene, enhancing permeability, hydrophilicity, and separation efficiency.

3.2.4. Mixed Matrix Membranes (MMM)

Mixed matrix membranes have been studied for oil-water separation, with significant advancements reported by various researchers. Makaremi et al. [16] enhanced electrospun polyacrylonitrile (PAN) membranes with halloysite nanotubes (HNTs), achieving a 99.5% rejection ratio and a 760% increase in heavy metal ion adsorption with 3% HNT reinforcement. Khalaf et al. [17] demonstrated effective oil/water separation using poly(acrylonitrile-co-vinyl acetate) (P(AN-co-VA))/single-wall carbon nanotubes (SWCNTs) membranes. Ye et al. [18] fabricated PLA-based nanofiber membranes modified with metal oxides, significantly improving hydrophobicity and superoleophilicity for oily wastewater treatment. Xu et al. [19] developed a 3D carbon aerogel from cellulose nanofibers, poly(vinyl alcohol), and graphene oxide, enhancing oil/water separation through an eco-friendly carbonization process. Additionally, Xu et al. [20] introduced tubular PVC hybrid nanofiber membranes with high reusability for water-in-oil emulsions. Duan et al. [21] deposited Cu_2O nanoparticles on polypropylene membranes, achieving high separation efficiency. Huo et al. [22] created a superhydrophobic nanofiber composite membrane (PU/TiO_2/PDMS) that effectively separates oil from water, demonstrating durability and recyclability. Eng et al. [12] reviewed the status of MOF membranes in oil-water separation, covering synthesis strategies for both pure and mixed matrix membranes.

3.2.5. Janus Membranes

Janus membranes have an asymmetric structure with hydrophobic and hydrophilic layers, making them effective for oil-water separation. The capillary force drives fluid transport through these membranes. They feature two distinct surfaces, allowing water to be drawn through when the hydrophobic side contacts water in oil emulsions. Innovations in Janus membranes include the development of a thin selective layer for oil/water separation, optimized surface microstructure, and chemistry through silicification. Research has shown that these membranes can achieve high separation efficiency and impressive flux rates. Various methods, including electrospinning and plasma-etching, have been employed to enhance their performance.

3.2.6. Nanofibers Membranes Developed for Oil/Water Separation

Electrospun fiber membranes, particularly polyvinylidene fluoride (PVDF) nanofibers, are noted for their mechanical strength and corrosion resistance but face challenges with oil fouling, reducing flux and reusability. Developing biodegradable membranes from low-cost, hydrophilic materials offers a promising, sustainable solution, though reports on such advancements are rare. The work of Jiang et al. [23] suggested that the hybrid membrane has attractive potential application in wastewater purification as the membrane has excellent recyclability due to its outstanding anti-fouling and self-cleaning properties. Despite progress, scaling up electrospun nanofiber production in industry faces challenges that need to be addressed

Cheng et al. [24] developed superhydrophilic PAN nanofiber membranes via electrospinning for rapid oil-in-water separation, enhancing efficiency and reducing mass transfer resistance. Incorporating nanomaterials like TiO2 and SiO2 in polymeric nanofibers is common for oil/water separation. Thota et al. [25] created a composite membrane by integrating polystyrene with TiO_2 nanoparticles, achieving a high-water contact angle of ~155° for effective water-in-oil emulsion separation. Zhang et al. [26] produced a superhydrophobic PAN membrane with gold nanoparticles, demonstrating efficient water/oil separation and catalytic degradation of organic dyes. The Au@ZIF-8@PAN-TD membrane's hierarchical structure offers potential for purifying polluted oily water.

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A dip-coating strategy was developed to prepare two different Schiff base COFs [2,4,6-triformylphloroglucinol–4,4′-diaminobiphenyl (Tp-BD) and 3,5-tris(4-aminophenyl)benzene–terephthalaldehyde (TAPB-TPA)] on polyacrylonitrile (PAN) nanofibers at room temperature. The separation efficiencies of different samples with different oil and water concentrations were all greater than 95% and have great potential for separating water-in-oil mixtures [27].

Li et al. [28] developed NiCo-LDH@PANI/PAN membranes with a caterpillar-like structure for wastewater treatment, achieving superhydrophilicity (≈ 0°) and underwater superoleophobicity (≈ 161°). Fu et al. [29,46] created a hierarchical Al2O_3/TiO_2 membrane via electrospinning for gravity-driven oil/water separation, achieving 97.7% separation efficiency and 98% dye capture.

Graphene oxide (GO) is highly researched for membrane applications due to its thinness and layered structure. Its derivatives, particularly GO, offer cost-effective, energy-efficient, and simple water purification solutions. Fan et al. [30] developed a graphene-wrapped polyphenylene sulfide membrane, exhibiting chemical resistance and hydrophobicity for efficient oil/water separation and crude oil adsorption. Junaidi et al. [31] reviewed advancements in GO-based membranes, including free-standing, supported, and polymer nanocomposites for oil-water separation, utilizing filtration and adsorption techniques. Naseem et al. [11] fabricated nanofiber membranes from recycled industrial cellulose triacetate, combined with GO and titanium dioxide, for oil/water separation.

Li et al. [32] developed spherical-beaded nanofibers (nanofibers fabricated from solutions with different PVDF concentrations,) that create a strong microelectric field, aiding phase separation to form nano-nets with small pores. These membranes exhibit superhydrophobicity, broad pressure applicability, antifouling properties, and reusability, offering potential advancements in fibrous materials for separation applications. Guo et al. [33] developed flexible, superhydrophobic foam composites using carbon nanofibers on a PDMSfoam framework for enhanced oil/water separation. Liu et al. [34] created porous SiO_2 micro/nanofibrous membranes through a camphene sublimation process. The membranes showed high oil absorption PDMS significantly outperforming traditional SiO_2 membranes.

Li et al. [35] developed electrospun biodegradable polylactic acid (PLA) nanofibers combined with zeolitic imidazolate framework (ZIF-8) using seed-assisted in situ growth. These PLA@ZIF-8 nanofiber composites demonstrated efficient oil-water separation and self-cleaning capabilities due to high wettability and photocatalytic activity, providing an eco-friendly solution for industrial processes. Cheng et al. [36] created biodegradable superhydrophilic membranes using polylactic acid nanofibers and polyethylene oxide hydrogels, achieving a 61.9-fold increase in oil-in-water emulsion permeance (2.1 × 104 L m^2 h^{-1} bar^{-1}) with over 99.6% separation efficiency. Xu et al. [37] constructed a tubular PVC hybrid nanofiber membrane characterized by a three-dimensional structure exhibited excellent hydrophobic stability and superhydrophobicity under oil. Fang et al. [38] developed composite nanofiber membranes by electrospinning polyacrylonitrile (PAN) and polyurethane (PU), incorporating a porous nano-zeolite imidazolium skeleton (ZIF-8) to enhance hydrophobicity. The PAN/PU@ZIF-8 composite nanofiber membranes demonstrated excellent separation performance for various oil/water mixtures and water-in-oil (W/O) emulsions, achieving oil fluxes of up to 6391.01 $L \cdot m^{-2} \cdot h^{-1}$ and separation efficiencies exceeding 99.15%.

Li et al. [39] developed NiCo-Layered Double Hydroxide (LDH)@Polyaniline (PANI)/PAN membranes featuring a caterpillar-like structure for wastewater treatment, achieving superhydrophilicity (≈ 0°) and underwater superoleophobicity (≈ 161°). Fu et al. [40] created a hierarchical Al2O3/TiO2 membrane via electrospinning for gravity-driven oil/water separation, achieving a separation efficiency of 97.7% and a dye capture rate of 98% The presence of oil-in-water (O/W) emulsions significantly threatens aquatic ecosystems, contaminates potable water resources, and disrupts soil structure. There is an urgent requirement for effective and economically feasible methods to remove emulsified oil droplets from water. The coalescence technique has been identified as a promising approach for separating O/W emulsions. ZIF-8 can be utilized independently or integrated with other materials such as membranes and foams and can be modified to develop derivative materials suitable for oil/water separation applications [41]. Yue and Mukai [42] developed

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an in-situ growth strategy to decorate zeolitic imidazolate framework-8 (ZIF-8) on electrospun polyacrylonitrile (PAN) nanofibers for coalescence separation. The process began with pretreatment to activate the PAN membrane, creating anchor sites for zinc ions, followed by immersion in precursors of ZIF-8 particles. The incorporation of ZIF-8 enhanced the membrane's amphiphilicity, surface roughness, and imparted a positive charge, facilitating the demulsification and coalescence of negatively charged oil droplets. The ZIF-8/PAN nanofiber membrane achieved separation efficiencies exceeding 99.9% for surfactant-free emulsions and 97.1% for surfactant-stabilized emulsions. Additionally, it demonstrated an ultra-high permeation flux of 21,177 L·m−2·h−1 and 14,118 L·m−2·h−1, respectively, in long-term continuous separation processes. Li et al. [35] presented a biodegradable PLA@ZIF-8 nanofiber membrane created via electrospinning and seed-assisted growth, combining high wettability, photocatalytic activity, and efficient oil–water separation (>99.8% efficiency, flux 6280 $L/m^2/h$). The membrane exhibits self-cleaning, UV-driven diesel degradation (92.8%), and methylene blue removal (99.7%), with biodegradability confirmed after 120 days in soil, offering an eco-friendly solution for industrial oil–water separation.

A biodegradable superhydrophilic membrane was developed using polylactic acid nanofibers and polyethylene oxide hydrogels via electrospinning [43]. This design increased hydrogen bonding by 357.6%, transforming hydrophobic membranes into superhydrophilic ones, reducing fouling, and enhancing emulsion permeance by 61.9 times with over 99.6% separation efficiency. Additionally, hydrogen bonds accelerated polylactic acid biodegradation by over 30%, aiding waste treatment.

Ultrafiltration membranes effectively separate oil-water emulsions but face fouling issues due to oil droplet deposition. Li et al. [44] developed a hydrogel nanofiber membrane using PEO/AM/NaAlg on a PHB nanofibrous matrix, further enhanced with superhydrophilic PEO/PAM/CaAlg hydrogel via polymerization and Ca^{2+} cross-linking. The membrane demonstrated good anti-fouling properties, with a tensile strength of 1.95 MPa, stable flux, and high rejection rates after 3 hours of operation—2925.2 $L/m^2/h$ and 98.66%. The simple, cost-effective, and eco-friendly preparation method shows potential for oil-water emulsion separation.

The utilization of electrospun fiber membranes, particularly polyvinylidene fluoride (PVDF) nanofibers, is recognized for their mechanical strength and corrosion resistance. However, these membranes encounter significant challenges related to oil fouling, which adversely affects flux and reusability. Though electrospun PVDF-based composites showed a good future for oil/water mixtures, a lot still needs to be done before the large-scale treatment of oily wastewater. The main is the poor mechanical properties of the composites which are still a serious setback for the treatment of large quantities of oily wastewater. Besides this, most of the PVDF-based composites research need on design, preparation, and application. However, not much has been done as regards the extensive study on understanding the separation process, the mechanism and the likely theories governing the process [45].

The development of biodegradable membranes from low-cost, hydrophilic materials presents a promising and sustainable solution, although reports on such advancements remain scarce. The research conducted by Jiang et al. [23] indicates that hybrid membranes possess considerable potential for application in wastewater purification, attributed to their excellent recyclability and remarkable anti-fouling and self-cleaning properties. Despite advancements, the scaling up of electrospun nanofiber production in industrial settings continues to face challenges that require resolution.

Efficient separation materials for oil–water emulsions are essential for environmental protection and resource recovery. However, many current fibrous separation materials are characterized by large pore sizes and low porosity, which limit their separation effectiveness [46]. Despite the numerous advantages, fouling is an inevitable challenge in membrane separation, particularly in oil separation technologies where oil droplets adhere to membranes, reducing functionality. Innovations in adsorption technologies, such as porous foams, offer promising and cost-effective oil recovery alternatives. Overall, these advancements in nanofiber membrane technology have significantly

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improved oil/water separation efficiency and capabilities, employing various materials and innovative techniques to address diverse challenges in this field.

4. Summary

The article discusses the significant environmental challenge posed by water pollution, particularly due to oil spills and industrial discharges. It highlights advancements in membrane technologies, specifically nanofiber membranes, for effective oil-water separation. Nanofiber membranes can efficiently separate the free-floating oil, dispersed oil and emulsified oil droplets. Electrospinning is identified as a preferred method for fabricating these membranes due to its ability to produce fibers with high surface area and porosity. Various techniques, including polymer blending, surface modification, and the creation of hierarchical structures, are explored to enhance membrane performance. The article reviews several studies demonstrating the effectiveness of modified membranes in achieving high separation efficiencies and flux rates for oil-water emulsions. Innovations such as Janus membranes, which feature asymmetric structures for improved separation, are also discussed. Overall, the advancements in nanofibers membrane technology are presented as promising solutions for addressing the challenges of oil-water separation in polluted environments.

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