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폐수처리용 박막나노복합체 기반 나노여과막: 제조 및 염료제거

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Thin Film Nanocomposite Based Nanofiltration Membrane for Wastewater Treatment: Fabrication and Dyes Removal

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요 약: 해당 연구는 산업 폐수에서 염료를 효율적으로 제거하기 위한 고급 박막 나노복합체(TFN) 기반 나노여과막을 개발하여 효과적인 폐수 처리 방법을 제시합니다. 최근 연구의 동향을 보면, 나노카본, 실리카 나노스피어, 금속-유기 프레임워크(MOF) 및 MoS_2와 같은 혁신적인 재료를 포함하는 TFN 막의 제조에 중점을 둡니다. 주요 목표는 염료 제거 효율을 향상시키고 오염 방지 특성을 개선하며 염료/염 분리에 대한 높은 선택성을 유지하는 것입니다. 이 논문은 넓은 표면적, 기계적 견고성 및 특정 오염 물질 상호 작용 능력을 포함하여 이러한 나노 재료의 뚜렷한 이점을 활용하여 현재 나노여과 기술의 제한을 극복하고 물 처리 문제에 대한 지속 가능한 솔루션을 제공하는 것을 목표로 합니다.

Abstract: This review addresses the pressing need for effective wastewater treatment methodologies by exploring advanced thin-film nanocomposite (TFN) nanofiltration membranes aimed at efficient dye removal from industrial effluents. Utilizing insights from recent research, the review focuses on the fabrication of TFN membranes incorporating innovative materials such as nanocarbons, silica nanospheres, metal-organic frameworks (MOFs), and MoS_2. The primary goals are to enhance dye removal efficiency, improve antifouling properties, and maintain high selectivity for dye/salt separation. By leveraging the distinct advantages of these nanomaterials—including large surface areas, mechanical robustness, and specific pollutant interaction capabilities—this review aims to overcome the limitations of current nanofiltration technologies and provide sustainable solutions for water treatment challenges.

Keywords: *dye, thin film composite membrane, nanofiltration, MOFs*

1. Introduction

In recent years, the escalating demand for advanced water filtration technologies has prompted extensive research into novel membrane materials aimed at addressing both environmental concerns and performance requirements[1]. Polymeric membranes have garnered considerable attention for water filtration applications but concerns over their environmental impact and performance limitations have underscored the need for

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sustainable alternatives. This review specifically focuses on thin-film nanocomposite (TFN) membranes, which integrate various nanomaterials such as nanocarbons, silica nanospheres, metal-organic frameworks (MOFs), and MoS_2 to enhance dye removal efficiency from wastewater.

The development of advanced TFN membranes is crucial for sustainable wastewater treatment. Traditional polymeric membranes often face challenges such as fouling, limited dye rejection, and low permeability. By integrating nanomaterials, TFN membranes offer a promising solution to these issues. The unique properties of nanomaterials, such as high surface area, tunable surface chemistry, and robust mechanical strength, make them ideal candidates for enhancing membrane performance. The research discussed in this review highlights the potential of TFN membranes to provide efficient, cost-effective, and environmentally friendly solutions for dye removal in wastewater treatment.

Additionally, polybenzimidazole (PBI)-based membranes have emerged as a robust solution for numerous filtration challenges, owing to their exceptional thermal and mechanical properties[2]. These membranes find an effective way to implement in high-temperature proton exchange membrane fuel cells, gas separation, and organic solvent nanofiltration. Moreover, the integration of functional nanomaterials, such as Ag_3PO_4/GO nanocomposites, into polyethersulfone (PES) matrices has demonstrated significant advancements in antibacterial and antifouling properties, along with improved dye separation efficiency[3]. Further innovations include the fabrication of nanocomposite membranes utilizing PVDF and HDTMA-modified clinoptilolite nanoparticles, exhibiting enhanced performance of the removal in reactive dyes from aqueous solutions[4].

Hydrophilic additives have also been employed to develop high-flux reverse osmosis membranes, showcasing improved water flux and salt rejection properties for brackish water desalination[5]. Furthermore, bio-inspired approaches, such as the utilization of tannic acid functionalized halloysite nanotubes in polysulfone membranes, have demonstrated remarkable improvements in hydrophilicity, antifouling properties, and dye separation efficiency[6]. The integration of eco-friendly nanofillers, such as $Fe_3O_4@SiO_2–NH_2$, into polyethersulfone membranes has shown promising results in salts, heavy metal ion, and dye removal, coupled with excellent long-term stability and reusability[7]. Graphene quantum dots (GQDs) have been effectively incorporated into polyvinyl chloride (PVC) membranes, enhancing permeability and anti-fouling properties for dye wastewater treatment[8]. Thin-film composite nanofiltration membranes have been scrutinized for their efficacy in dye desalination and concentration, offering high rejection rates for anionic dyes and salts [9-12]. Ceramic-based nanofiltration membranes have emerged as a robust alternative to polymeric membranes, exhibiting higher stability and effectiveness in wastewater treatment applications[13]. This review discussed about nanofiltration membrane for dye separation.

2. Dye Removal

Dye removal by physical methods, such as adsorption, membrane filtration, ion exchange, and coagulation, is an efficient approach involving mass transfer mechanisms. Adsorption, in particular, is a widely studied method due to its effectiveness in eliminating contaminants from wastewater using economical and readily available adsorbents. Various adsorbents, including silica gel, peat, fly ash, and carbon-based structures like activated carbon (AC), graphene (GR), and carbon nanotubes (CNTs), have shown significant potential. Silica gel is effective for removing basic dyes but faces industrial limitations due to fouling. Peat is a cost-effective alternative capable of adsorbing polar organic complexes and transition metals. AC, with its high surface area and porosity, is a benchmark adsorbent despite its poor regeneration and high cost. Fly ash, particularly from sugar industry byproducts like bagasse fly ash, offers economic viability and effectiveness in dye adsorption. Carbon-based structures exhibit high adsorption capacities for various dyes but face challenges due to filter clogging with suspended solids. Membrane filtration, another prominent method, offers high degradation efficiency, continuous operation, and lower energy consumption compared to traditional methods. These systems use materials such as polymers, zeolites, and ceramics to effectively separate high-molecular-weight solutes.

When looking into the treatment of dye-infused wastewater, the handling of membrane fouling is important for the effectiveness of nanofiltration (NF) techniques[14]. A new NF membrane, named TFNi, has been developed with a catalytically active PDA/ MnO_2 interlayer consolidated directly into the structure to improve its ability to clean by itself. The active involvement of MnO_2 nanoparticles in this interlayer not only enhances the membrane's surface hydrophilicity but also better water transport pathways, as a result, contributed to significant decrease in fouling and increasing water flow to 24 ± 2 L/m^2 h bar, surpassing conventional membranes. Experiments on fouling and subsequent chemical cleaning under numerous water chemistry conditions reveal the self-cleaning mechanisms of the TFNi membrane, which was made possible by the generation of free radicals by the MnO_2–H_2O_2 system within the interlayer, helping with the breakdown of contaminants in wastewater. This great approach, utilizing in-situ radical production from H_2O_2, shows promise for efficient dye wastewater treatment. The TFNi nanofiltration membrane with a PDA/MnO_2 interlayer offers improved self-cleaning capabilities, reduced fouling, and enhanced water flow, making it effective for dye wastewater treatment.

When it comes to the purification of water contaminated with dyes and antibiotics, the scientific findings of high-performance nanofiltration (NF) membranes with fabulous pollutant rejection and fouling resistance is crucial[15]. In response to this case, zwitterionic polyester NF membrane (ZNF) has been proposed, fabricated by utilizing a triethanolamine-based (Z-TEOA) monomer synthesized through the ring-opening reaction of TEOA with 1,3-propane sultone. These ZNF membranes, created via interfacial polymerization under alkaline conditions, exhibit accerlated hydrophilicity and permeability because of the zwitterionic nature of the thin-film layer. The most efficient membrane variant (ZNF1.5-5) demonstrates low water contact angle values (20.2°) and a great number of rejection rates for myriad pollutants like Congo red (99.9%), methyl blue (99.7%), orange G (95.9%), and tetracycline (96.7%). Its outstanding resistance to chlorine exposure, as proven by a maintained high rejection rate for methyl blue after exposure to chlorine, along with a notable flux recovery ratio (FRR) for methyl blue (97.8%), represents its superior fouling resistance. These results suggest the ZNF membrane's suitability for treating wastewater in industries such as textiles and pharmaceuticals. Zwitterionic polyester NF membranes (ZNF) exhibit high pollutant rejection, fouling resistance, and chlorine tolerance, making them suitable for treating wastewater in textile and pharmaceutical industries.

2.1. Nanocarbon meterials

To address the issue of wastewater treatment, numerous work has been done on the locating a better way to solve this problem. As a result, a number of approaches has been proposed with the evolvement of advanced thin film nanocomposite (TFN) membranes using graphene oxide quantum dots (GOQDs) and β-Cyclodextrin (β-CD) as innovative nanofillers, aiming at the effective removal of both cationic and anionic dyes from water[16]. The membranes were manufactured through a low-pressure interfacial polymerization technique on a polysulfone base, integrating a mix of β-CD, GOQDs, and tetraethylenepentamine (TEPA). This in fact has resulted in membranes with dramatically enhanced hydrophilic properties compared to traditional polymeric membranes. The optimal membrane variant, M_{PG2}, demonstrated doubling in pure water flow rate and a dye rejection efficiency of 99.6% for Congo red and 96% for Methyl blue, due to negative surface charges fostering Donnan exclusion. Moreover, the M_{PG2} membrane showed eminent salt rejection rates of 96% and 99.1% for Cl^- and NO_3^- ions, respectively.

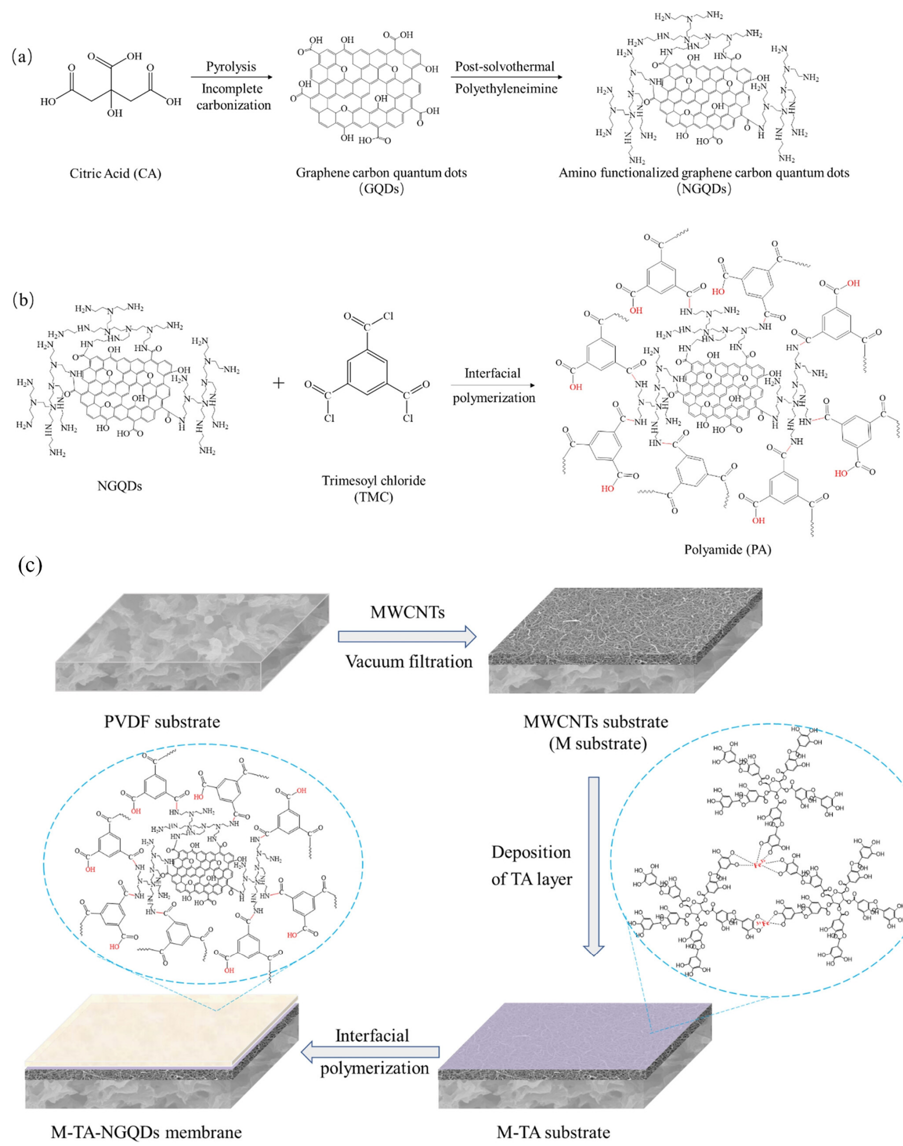

Fig. 1. Fabrication process of NGQDs (a), schematic of interfacial polymerization (b), and the general procedure diagram of the preparation of an M-TA-NGQDs membrane (c) (Reproduced with permission from Li *et al.*[17], Copyright 2023, American Chemical Society).

Advanced thin film nanocomposite membranes with GOQDs and β-Cyclodextrin exhibit improved hydrophilicity, high dye and salt rejection rates, and enhanced water flow.

In regards to the pursuit of effective dye/salt separation, especially under high salinity conditions, this research mentions about a thin-film composite membrane enhanced by a tannic acid (TA)-modified carboxylic multiwalled carbon nanotube (MWCNT) interlayer[17].

This design, linked with amino-functionalized graphene quantum dots (NGQDs) as monomers, yields a membrane that is thinner, more hydrophilic, and smoother than alternatives. The membrane showcases superior performance, with significantly higher pure water permeability and dye rejection rates (e.g., 97.79% for Methyl Orange) compared to membranes without the MWCNT interlayer. Notably, even at high salt concentrations (50,000 mg/L NaCl), it achieves excellent dye rejection (> 99%) with minimal salt rejection, alongside impressive chemical stability and

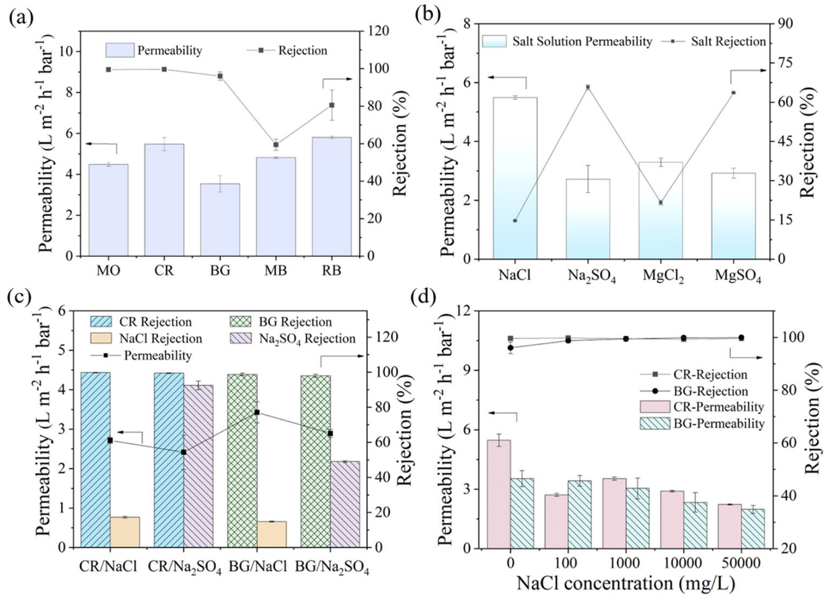

Fig. 2. Rejection rate and permeability of different dyes (a) and salts (b) by the M-TA-NGQDs membrane; the dye/salt separation performance of the M-TA-NGQDs membrane (c); and the effect of the concentration of NaCl on the separation CR and BG (d) (Reproduced with permission from Li *et al.*[17], Copyright 2023, American Chemical Society).

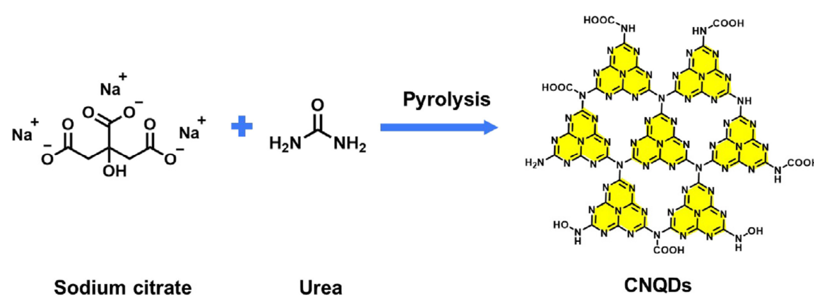

Fig. 3. Synthesis reaction of CNQDs (Reproduced with permission from Liu *et al.*[18], Copyright 2023, American Chemical Society).

fouling resistance, marking it as a promising results for treating high-salinity textile wastewaters. NGQDs-enhanced membranes demonstrate higher water permeability, excellent dye rejection, and fouling resistance, even under high salt concentrations, making them suitable for high-salinity textile wastewater treatment.

Next research concentrates on a novel approach to nanofiltration (NF) by assembling graphitic carbon nitride quantum dots (CNQDs) into thin-film nanocomposite (TFN) membranes[18].

The CNQDs contribute to a more permeable and loosely structured polyamide (PA) active layer, leads to a 210% expansion in water flux and 96% rejection of Methyl Blue, significantly outperforming standard composite membranes. These membranes also present remarkable resistance to common fouling agents, contributed to the enhanced surface hydrophilicity and smoother surface achieved through CNQD integration, displaying the valid possibility of CNQDs in creating efficient, antifouling NF membranes for dye wastewater treatment. NGQDs-enhanced membranes demonstrate higher water permeability, excellent dye rejection, and fouling resistance, even under high salt concentrations, making them suitable for high-salinity textile wastewater treatment.

Taking a vantage of plasma technology, scientists

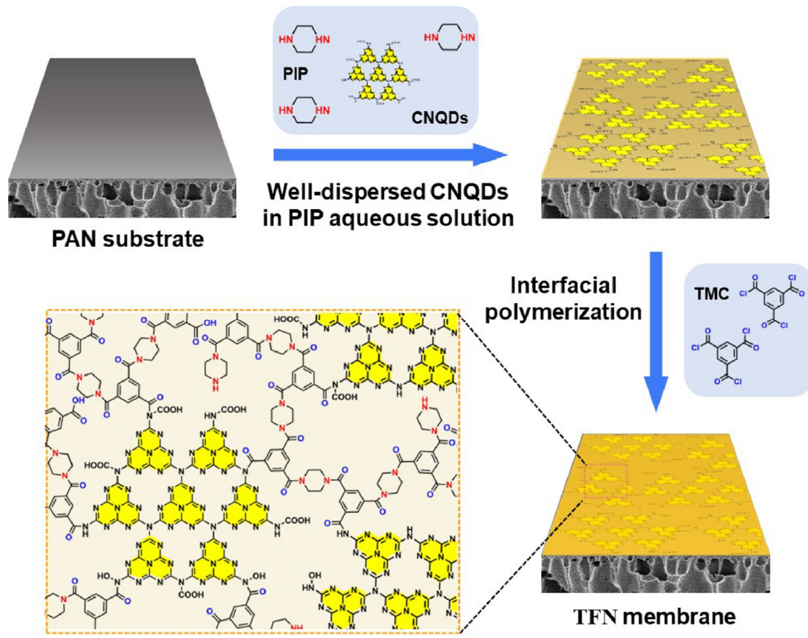

Fig. 4. Schematic illustration of the preparation of the CNQD-based TFN membranes (Reproduced with permission from Liu *et al.*[18], Copyright 2023, American Chemical Society).

have developed a nitrogen-doped graphene quantum dots (NGQDs)-based thin-film nanocomposite (TFN) membrane characterized by exceptional water transport and purification abilities[19]. The membrane's design, introduced by comprehensive studies, allows for an ultrahigh water permeability of 289 L/m^2 h bar and a dye separation efficiency of 99.96%. In addition to this, the membrane's stable photoluminescence enables real-time monitoring of dye fouling, offering a sustainable, high-performance solution for water purification and recycling. NGQDs-based TFN membranes achieve ultrahigh water permeability, excellent dye separation, and real-time monitoring of dye fouling, providing a sustainable solution for water purification.

When it comes to separating salt or dye mixtures in textile wastewater, it always accompanies the challenging part of it process. Therefore, a novel nanofiltration (NF) membrane is used in a loose thin film nanofibrous composite structure[20]. The membrane incorporates g-C_3N_4 nanosheets modified with polyethylenimine (PEI), achieving high water permeability (40.9 L/m^2 h bar) and exceptional dye rejection rates (>94%) while maintaining low salt rejection. Its peculiar photocatalytic properties under visible light secures easier level of cleaning and recyclability, demonstrating outstanding performance and sustainability for salt/dye separation tasks. NF membranes with g-C_3N_4 nanosheets modified with PEI exhibit high water permeability, exceptional dye rejection, and photocatalytic properties, enhancing cleaning and recyclability for salt/dye separation.

Dealing with another frameworks, metal-organic frameworks(MOFs) with carbon quantum dots(CQDs), also comes in place for creating self-cleaning, high-performance thin-film nano composite(TFN) membranes for dye removal[21]. The UiO-66- NH_2/CQD hybrid exhibits improved compatibility and photocatalytic activity within the polyamide matrix, significantly enhancing water permeability and dye selectivity. The membranes demonstrate the ability to recover water flux lost to dye fouling through photocatalytic degradation, offering a new avenue for designing advanced, self-cleaning nanofiltration systems for wastewater treatment. UiO-66-NH_2/CQD hybrid TFN membranes exhibit enhanced water permeability, dye selectivity, and self-cleaning properties through photocatalytic degradation.

2.2. Silica nanoshperes

The adventure of developing sustainable and cost-effective materials for constructing high-performance loose nanofiltration (LNF) membranes in relation to superior permeability and dye/salt selectivity is ongoing area of research. The proposal of utilizing silica-nanosphere was introduced to show its mechanism to make it work. By taking advantage of aminated lignin as the main resources, trimesoyl chloride for cross-linking, and AHS nanospheres as nano-enhancers, researchers have developed a lignin-based LNF membrane through interfacial polymerization[22]. This membrane demonstrates outstanding water flow rates, excellent efficiency in dye filtration, and assistance in salt passage. Improvement with L-arginine further boosts its water permeability (to 49.9 L/m^2 h bar) and salt flow, while keeping high dye filtration efficiency. The AHS nanospheres enrich the lignin-based barrier layer with numerous pathways for water and salt, with their hollow design lowering resistance to these flows. Additionally, the membrane hangs onto its structural integrity and filtration capability after exposure to acidic or basic solutions and maintains its performance even after a chlorination treatment of 5500 ppm h, showcasing excellent dye/salt separation stability and a slight increase in permeability. The membrane also portrays strong resistance to fouling, effective bactericidal (92.5%) properties, and hinders bacterial adhesion, maintaining persistent performance through an extended 80-hour dye/salt filtration process. Lignin-based LNF membranes with AHS nanospheres exhibit high water permeability, dye/salt filtration efficiency, fouling resistance, and bactericidal properties.

2.3. MOF

A flagship of nanofiltration(NF) membrane known as thin film nanocomposite (TFN) has been developed, incorporating photocatalytic materials within its selective layer for direct decomposition of contaminants on its surface[23]. This innovation utilizes a mixture of carboxymethyl cellulose (CMC) and MIL-53 (Fe) to create the selective layer, dramatically boosting the membrane's self-cleaning capabilities. The addition of MIL-53 (Fe) works as both a nanofiller and a photocatalyst. The optimal membrane, containing 0.5 mg of CMC and 0.075 mg of MIL-53 (Fe), achieved impressive water permeability rates of 39.83 L/m^2 h bar and proven superior dye rejection rates exceeding 99.00% for CR, G250, and MB dyes. Additionally, it showed notable salt rejection capabilities (61.49% for Na_2SO_4 and 17.22% for NaCl). This fabrication method presents a significant advancement in enhancing TFN NF membranes' separation and self-cleaning performance for organic separation processes. TFN membranes with CMC and MIL-53 (Fe) exhibit high water permeability, superior dye rejection, and self-cleaning capabilities, enhancing separation performance.

Another innovative approach has been developed for creating thin film composite (TFC) polyamide (PA) membranes enhanced with zeolitic imidazolate frameworks (ZIFs) like ZIF-8 and ZIF-93, designed for the nanofiltration (NF) removal of dyes from water, without using organic solvents[24]. This method, termed vapor phase interfacial polymerization (VIP), constructs the PA layer and incorporates the ZIFs in a manner that refuses conventional solvent use. Both ZIF-8 and ZIF-93 were prepared through standard methods and integrated into the PA layer in following two comparable configurations: first one described as a nanocomposite with ZIF intruded in the PA, and as a bi-layer with a ZIF layer within the helping layer and PA. Specifically, ZIF-93 was synthesized utilizing a water-based method for the bilayer approach, making it possible for a complete organic solvent-free process. These membranes exhibited water permeances of 2.6, 3.4, and 2.2 L/m^2 h bar for the dyes Rose Bengal, Sunset Yellow, and Acridine Orange, respectively, representing permeance increases of 69~117% in comparison to the standard VIP-TFC membrane, while still rejecting over 99% of the dyes. The enhanced performance of the bilayer PA/ZIF-93 membranes are attributed to their increased hydrophilicity, greater surface roughness, and thinner selective layers. Additionally, these membranes extensively showed superior stability, upholding performance levels over 34 hours of operation and across a broad pH range (3 to 9), showing no performance degradation. TFC PA membranes with ZIFs enhance water permeance, dye rejection, and stability in organic solvent-free processes.

2.4. MoS_2

In pursuit of sustainable water treatment solutions, there is growing interest in developing loose nanofiltration (LNF) membranes that can efficiently separate dye/salt mixtures with high water flow rates[25]. Traditional LNF membranes often face complicated challenges in balancing permeability with selectivity because of the limitations of their polymer-based selective layers. This experiment introduces a tannic acid (TA)-MoS_2 nanosheets (NSs) incorporated interlayer within a polyester (PE) based LNF membrane (termed iLNF-3), which demonstrates significantly improved water permeance of 55.8 L/m^2 h bar, exceeding that of a standard LNF membrane, which stands at 27.3 L/m^2 h bar. The iLNF-3 membrane also shows enhanced selectivity in dye/salt separation, with rejection rates of 99.6% for Congo red and 98.0% for Methyl Blue, while only rejecting 5.6% of Na_2SO_4. The membrane's superior performance in terms of ionic interference resistance, long-term stability, and antifouling capabilities is attributed to the TA-MoS_2 NSs interlayer. The existence of this interlayer improves water permeance through a “gutter” effect and the creation of nanochannels, and results in a thinner PE selective layer, improving overall filtration efficiency. Moreover, the selectivity benefits from the looser porous structure of the PE layer, a consequence of the slowed diffusion rate of the aqueous monomer during formation. The following empirical finding lays the groundwork for future development of iLNF membranes targeted at the treatment of dye-laden wastewater. TA-MoS_2 NSs interlayer in LNF membranes significantly improves water permeance and dye/salt separation, offering long-term stability and antifouling capabilities.

3. Conclusions

In conclusion the field of membrane technology for water treatment has witnessed significant advancements driven by the imperative for sustainable and efficient solutions. Through a comprehensive review and synthesis of recent research this review has illuminated the diverse strategies employed in the development of environmentally-friendly nanocomposite membranes. These nanocomposite membranes exhibit enhanced performance regarding water permeance dye rejection and antifouling properties thus offering a sustainable solution for water purification challenges. Moreover, advancements in thin-film composite nanofiltration membranes have enabled the desalination and concentration of dyes with high rejection rates for both anionic dyes and salts.

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