Chapter 1

Photocatalytic Membranes in Degradation of Organic Molecules

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Abstract

Heterogeneous photocatalysis is a technology widely applied to water purification and wastewater treatment under ultraviolet (UV) or even sunlight irradiation for the removal of a variety of environmental pollutants into harmless species. Application of membrane for immobilization of semiconductors and their suitability in photocatalytic degradation of dyes have recently been developed. Integration of photocatalysis with membrane processes significantly improve the membrane separation performance with reducing membrane fouling and improving permeate quality. This paper reviews recent progress in the photocatalytic membranes for wastewater treatment and water purification with an emphasis on the type of membranes, membrane fabrication, and applications in pollutant removal.

Keywords

Semiconductors, Organic Pollutants, Heterogeneous Photocatalysis, Ceramic Photocatalytic Membranes, Polymeric Photocatalytic Membranes

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1. Introduction

The introduction of industrial, pharmaceutical, and agricultural chemicals into the ecosystems, as a result of human activities, are now a considerable subject of environmental remediation. The complex mixture of these compounds and their non-biodegradable character make conventional wastewater treatment methods (physical, chemical, and biological methods) unable to completely remove them. Thus, the interest in these environmental effects leads to an increase of research activities for elimination of such pollutants.

Advanced oxidation processes (AOPs) have been proposed as alternative methods for the elimination of persistent organic compounds in wastewaters, air, and soil. The principle of AOPs is to generate hydroxyl radical and other radical species in the water, the very powerful oxidants are capable of oxidizing a wide range of organic compounds with one

or many double bonds. Nowadays, a photocatalytic process using a semiconductor as photocatalyst under light irradiation has been extensively applied for the oxidation of various organic pollutants.

Photocatalyst comes from the word “photo”, which means light, and “catalyst”, which refers to a substrate that accelerates chemical reactions by absorbing photons or light. It is noted that UV light works within the wavelength range of 280-400 nm (UVA, 315-400 nm, UVB, 280-315 nm, and UVC, 180-280 nm), while visible light works in the range between 400-700 nm. Since UV light activates most photocatalysts, a constant source is needed to ensure the continuity of the reaction. For doped photocatalyst that can function under visible light, direct exposure to sunlight is sufficient to initiate photocatalytic activity. The photocatalysis can be classified into homogeneous and heterogeneous categories [1]. In this review, we focus on heterogeneous photocatalysis using polymeric and ceramic membranes. A photocatalyst is a material that can induce reaction upon direct light absorption. Under an irradiation of light ($h\nu$) with equal or larger energy than the band-gap energy (the difference in energy between the valence band (VB) and the conduction band (CB)) of the semiconductor, electrons (e^-) migrate to the CB leaving holes (h^+) in the VB, thus the material is photoactivated due to the formation of e^-/h^+ pairs. The photogenerated e^- and h^+ are the basis of the heterogeneous photocatalysis using semiconductors (Figure 1).

Figure 1: General mechanism of photocatalysis.

2. Heterogeneous photocatalytic reaction

In the photocatalysis process, the photocatalyst produces electron-hole pairs (e^-/h^+) by absorbing radiation energy ($\geq$ band-gap energy), excites the electrons from valence band (VB) to conduction band (CB) and leaves holes in the VB. Afterwards the photogenerated electrons reduce the surface adsorbed oxygen or oxygen dissolved in water to superoxide anions ($O_2^{\bullet-}$). Similarly, holes oxidize the surface adsorbed H_2O or hydroxyl ($-OH$) groups to hydroxyl radicals ($OH^\bullet$). Subsequently, these reactive radicals react with the pollutant molecules, and degrade them into inorganic ions. Moreover, the holes in the VB possess strong oxidizing power, thus these could directly oxidize the pollutants adsorbed on the photocatalyst surface. In addition, electrons in the CB possess strong reducing power, which indirectly degrade the pollutants using $OH^\bullet$ radicals formed by photo-cleavage of hydrogen peroxide (H_2O_2), which is produced by reaction of $O_2^{\bullet-}$ with the proton (H^+). Moreover, in the case of dye degradation, the surface adsorbed dye also excites under light irradiation, migrates an electron into the CB of the photocatalyst and reduces the surface adsorbed O_2 to $O_2^{\bullet-}$, which reacts with H^+ to form a H_2O_2 and further photo-cleaved into $OH^\bullet$ radicals under light irradiation. The reaction can be expressed as follows:

The oxidizing agents can be semiconductor materials such as TiO_2 , SnO_2 , Sb_2O_3 , Cu_2O , CeO_2 , V_2O_5 , ZrO_2 , CdS , ZnO , MoS_2 , In_2S_3 , Bi_2WO_6 , $BiVO_4$, WO_3 , Semiconductors/Carbon Quantum Dots, and Semiconductors/g- C_3N_4 [2-5] (Table 1).

In the past years, a variety of semiconductors-based photocatalysts have been successfully prepared, such as TiO_2 and ZnO .

Table 1. Common semiconductors used in photocatalysis processes.

Name	Band gap	Valence band (V vs NHE)	Conduct band (V vs NHE)	Ref.
g-C ₃ N ₄	2.68	1.56	-1.12	[6]
Bi ₂₀ TiO ₃₂	2.36	2.59	0.23	[7]
CdS	2.4 5	1.5	-0.95	[8]
TiO ₂ (rutile)	3.0	2.5	-0.5	[9]
TiO ₂ (anatase)	3.2	2.5	-0.7	[9]
CdSe	1.749	1.520	-0.229	[10]
ZnS	3.6	1.8	-1.8	[9]
V ₂ O ₅	2.6	2.9	0.3	[11]
ZrO ₂	5	4.0	-1.0	[12]
ZnO	3.2	2.89	-0.31	[13]
CeO ₂	2.86	2.626	-0.234	[14]
In ₂ S ₃	1.96	1.18	-0.78	[15]
Bi ₂ WO ₆	3.10	3.38	0.28	[16]
WO ₃	2.8	3.2	0.4	[17]
BiVO ₄	2.59	2.83	0.24	[18]
SnO ₂	3.8	4.2	0.4	[19]
Sb ₂ O ₃	3.0	3.32	0.32	[20]
Cu ₂ O	2	0.4	-1.6	[21]

TiO₂ seemed to be a desired photocatalyst owing to its stability and low cost. However, its practical application is limited due to quick recombination of the photogenerated electron–hole pairs (e^-/h^+) and absorption of UV light (solar spectrum contains only 4-5% of UV light). Several tactics have been used to solve these problems, such as manipulate its structure and morphology, doping with metal elements to form TiO₂/narrow-band-gap-conductor in order to enhance the charge separation, and sensitization using organic dyes [22]. Band gap and efficient surface area increase with reducing the size of nanostructures. Therefore, it is possible to create TiO₂ with specific properties by tuning its size and shape. TiO₂ has three main crystal phases, Rutile, Anatase and Brookite which their band gap increases, respectively. Decorating TiO₂ with metal oxide (e.g. ZnO, Cu₂O and WO₃) and noble metals (e.g. Ag, Au and Pt) shift the fermi level to the more negative potentials, therefore the electron-hole pair recombination time decreases. Developing a green and low-cost photocatalyst with visible-light response still remains a significant challenge. Recently, various structures of TiO₂ such as nanotube [23], nanorod [24], nanoparticle [25] and flower shape [26] nanostructures were investigated as a photocatalyst.

Due to the promising applications in photocatalytic degradation of organic pollutants from waters and disinfection, heterogeneous photocatalysis has attracted a lot of attention

recently. An integrated water treatment process, known as a photocatalytic membrane reactors has been developed by combining photocatalytic oxidation and membrane filtration. Photocatalytic membrane reactors are prepared by the embedding of semiconductor particles such as titanium oxide into the membrane structure. Energy from light (i.e., visible or UV) is provided by photons and absorbed by catalytic particles to start the pollutant degradation reaction on the membrane surface. The high pollutant-degradation performance makes this technology a promising solution for the use in water- and wastewater-treatment processes. A variety of materials including organic, inorganic and metallic materials have been used as supports for the fabrication of photocatalytic membranes. Apart from immobilized semiconductor membranes, some pure photocatalytic membranes have been fabricated using TiO₂ nanofibers, nanowires or nanotubes. The next sections focus on elaborating and expanding these topics.

3. Photocatalytic membranes

Over the past few decades, membrane process has become one of the most effective technologies for water treatment due to its small footprint, superior separation efficiency and easy maintenance. However, in the conventional membrane processes, membrane fouling caused by the formation of a cake layer on the membrane surface usually results in pore blocking. This in turn results in a significant decrease in water flux and increased energy consumption and treatment costs. Moreover, membrane filtration can only concentrate pollutants into a high-concentration retentate, which needs further post-treatment before discharge. A hybrid photocatalysis-membrane system has been established to address these issues and shown to be a potentially effective treatment.

Currently, photocatalytic membrane reactors can be classified into four different configurations: (a) a slurry photocatalytic reactor followed by a membrane filtration unit, (b) inorganic or polymeric membrane submerged in a slurry photocatalytic reactor, (c) membrane placed inside a photoreactor whose internal walls are coated by a photocatalyst, and (d) photocatalytic membrane such as pure semiconductor or composite membrane (Figure 2). Among these four configurations, the photocatalytic membrane (d) has potential advantages over the other three configurations as it has the benefit having physical separation during membrane filtration as well as the organic degradation and anti-bacterial properties of the semiconductor achieved by photocatalysis in a single unit.

Figure 2. Different configurations of photocatalytic membrane reactors.

3.1 Polymeric photocatalytic membranes

Polymeric membranes are popular materials used as a support for photocatalysts in wastewater treatment. Various kinds of polymers such as polyamide (PA), polyvinylidene fluoride (PVDF), polyethersulfone (PES), polyurethane (PU), polyacrylonitrile (PAN), polytetrafluoroethylene (PTFE), cellulose acetate (CA), and sulfonated pentablock copolymer (s-PBC) have been modified by photocatalysts and applied in degradation of organic dyes. In preparation of photocatalytic membrane, photocatalyst particles can be either deposited onto the membrane surface or dispersed in the polymer solution for membrane casting.

A membrane process can be defined as splitting a feed stream by a membrane into a retentate (or concentrate) and a permeate fraction. Pressure-driven membrane processes use the pressure difference between the feed and permeate side as the driving force to transport the solvent (usually water) through the membrane. Particles and dissolved components are (partially) retained based on properties such as size, shape, and charge. Depending on their pore sizes, membranes can be classified as microfiltration (MF), ultrafiltration (UF), nanofiltration (NF) and reverse osmosis (RO) membranes Table 2.

Membranes applied in these processes can be divided, based on the material they are made of, into two types: polymeric and ceramic. Due to the lower price and simpler manufacturing, the polymeric membranes are more commonly used than the ceramic ones. However, polymeric membranes have some serious disadvantages when their application

in photocatalytic membrane reactors (PMRs) is considered, namely low resistance to the action of UV light or oxidizing conditions [27].

Table 2. Different types of membranes.

Membrane process	Material type^a	Pore size	Pressure range (bar)	Contaminant
Microfiltration (MF)	PTFE, PVDF, PP, PE, CE, PC, PS, PES), PI, PEI, PA, PEEK	0.1 μm to 10 μm	0.1-2	Bacteria
Ultrafiltration (UF)	PS, PES, S-PS, PVDF, PAN, CA, PI, PEI, PA, PEEK	2-100 nm	0.1-5	Macromolecules, bacteria, viruses
Nanofiltration (NF)	PA, PS, PES, S-PS, CA, P-PIP-A	~ 1 nm	5-20	Viruses, natural organic matter, multivalent ions
Reverse osmosis (RO)	CTA, Aramids ,PA, PEU	Non-porous	5-120	Viruses, natural organic matter, multivalent ions, monovalent ions (desalination,Ultr apure water)

^aRefer to abbreviations.

A wide variety of methods such as phase inversion, atomic layer deposition (ALD), chemical vapor deposition (CVD), sol–gel, electrospinning, hydrothermal, electrophoretic deposition technique, dip-coating, vacuum filtration, solvent-casting method have been applied to modification and enhance separation and anti-fouling performance of polymers [28-33]. Following we briefly described some of these photocatalytic membranes and their preparation methods.

3.1.1 Development of polyethersulfone and polysulfone membranes

Polymeric membranes are widely applied in water purification and wastewater treatment. Polyethersulfone (PES) and polysulfone (PS) are popular polymeric membranes due to their low cost, excellent chemical resistance, mechanical strength, high rigidity, thermal and biological stability[34]. Nevertheless, the conspicuous drawbacks of PS are its hydrophobicity and bad ultraviolet (UV) resistance. To overcome aforementioned problems in the line of membrane fouling alleviation, various approaches such as grafting with hydrophilic nanoparticles especially inorganic nanoparticles (e.g., ZrO_2 , Al_2O_3 , SiO_2 ,

TiO₂, Fe₃O₄, carbon nanotubes (CNTs) and etc.), blending with hydrophilic polymers, surface-coating and embedding hydrophilic nanoparticles have been done [35-39].

TiO₂ nanoparticles could remarkably affect the hydrophilicity of PS membrane, enhance mineralization of pollutants under ultraviolet (UV) light, and improve self-cleaning/antifouling property of the modified membranes. Ma et al. reported that the removal rate of model pollutant, dye Reactive Red ED-2B (RR ED-2B), was improved obviously using the inorganic membrane under UV irradiation in comparison with photocatalysis or membrane separation alone [40]. Yang et al. showed the water flux was nearly 30% improved and contact angle decreased from 84.7° to 41.4° with 2.0 wt.% TiO₂ blended [41]. Mozia et al. investigate the type of a TiO₂ photocatalyst (P25, ST-01, A700, and A800) on the stability of commercial polyethersulfone ultrafiltration membranes with different cut-off in a PMR. It was revealed that the shape and size of TiO₂ particles had a significant influence on the stability of the membranes during their operation in the PMR. It was concluded that the small and sharp-edged photocatalyst particles are especially detrimental to membrane stability [27].

The photocatalytic activity of fixed TiO₂ on the polymer structure decrease due to the loss of overall surface area associated with immobilizing the TiO₂. The design of smart TiO₂ morphologies (*e.g.* nanotubes, nanospheres, fibers) increases the photocatalytic activity. Schulze et al. showed that anodization of TiO₂ nanotubes on PES membrane has high photocatalytic activity in degrading methylene blue and diclofenac [42, 43].

Currently the stability of polymeric membranes under high energy UV irradiation is not fully documented, especially for extended exposure periods. Polymer degradation due to UV exposure or generation of free radicals during photocatalysis is in general an undesired process, often leading to a reduction in polymer molar mass and deterioration of the polymer properties.

One strategy of dealing with this disadvantage is using radical scavenger in the structure of polymeric membranes. It has been reported that PDA can act as free-radical scavenger as its chemical structure is similar to that of eumelanins, which are well known natural pigments for the protection of human body against UV by quenching reactive radicals generated by exposure to UV [44]. Wu. et al. and Feng et al. prepared doped polysulfone ultrafiltration membrane with TiO₂-polydopamine (PDA) nanohybrid for simultaneous self-cleaning and self-protection [45, 46]. In the TiO₂-PDA nanohybrid, PDA spheres acts as an adhesive substrate to hold densely covered photocatalytic TiO₂ nanoparticles, and also serves as a free-radical scavenger to protect PS membrane against the damage by free radicals produced by TiO₂ during UV exposure treatment. The spherical-shaped TiO₂-PDA can be easily doped into PS membrane via phase inversion method, which allows the

hydrophilic TiO₂-PDA spheres migrating to the hydrophobic PS surface and benefiting the antifouling ability.

In addition, impurity doping is other typical approach to extend the spectral response of the titanium dioxide to visible light region and decrease the negative effect by UV irradiation. Some metal elements (such as Fe, Cr, Co, Mo, Pd, Ag and V), non-metals (such as B, C, N, S, and F) and attachment to graphite and carbon nanotubes have been employed to tune the electronic structure and enhanced the photocatalytic activity of the titanium dioxide [47-52]. Incorporating the co-doped TiO₂ into PS membranes will likely modify the optical properties of the membrane, potentially enabling the photocatalytic activation of the embedded co-doped TiO₂ under less energetic visible light irradiation [49, 53]. Different proportions of N,Pd co-doped TiO₂ were embedded in a PS polymeric membrane through the phase inversion method and the membrane evaluated for the degradation of a dye (Eosin yellow) under visible light irradiation by Kuvarega et al [54]. Up to 92% dye degradation was realized with the 7% N, Pd TiO₂/PS nanocomposite membrane after 180 min of visible light irradiation.

Recently, Hir et al. synthesized reduced TiO₂ (r-TiO₂) by a simple reduction method using NaBH₄ and immobilized into PES matrix via phase inversion [55]. The occurrence of surface defects induced by Ti³⁺ species or oxygen vacancies led to the extension of the light absorption in the visible region. Results indicate that a complete MO removal was obtained when one piece of PES-13 wt% of reduced TiO₂ (PrT-13) film was used in a very acidic medium.

Mahlambi et al. prepared PS membrane containing nanosized carbon-covered alumina supported TiO₂ (CCA/TiO₂) as an inorganic-organic nanomembranes to photodegrade Rhodamine B dye under visible light illumination [56]. Table 2 summarized recent progress in modification and application of PES and PE in removal of organic compounds (Table 3).

Table 3. Application of photocatalytic polyethersulfone and polysulfone membranes in degradation of various types of organic pollutants.

Photocatalyst	Preparation method	Dye/Condition/Light region (source)	Ref.
PES–reduced TiO ₂	casting solution	-500 mL of MO (10 mg L ⁻¹), pH = 5.8 -300-Watt halogen lamp (PHILIPS 13096 ELH) irradiation for 540 min	[55]
PES/TiO ₂ film	Phase inversion	-1000 mL of MO (10 mg L ⁻¹), pH = 5.8 -UV (6 W UV-A lamp (HITACHI F6T5/BL))	[57]
PS/CCA/TiO ₂	Phase inversion	-100 mL of RhB (10 mg L ⁻¹) -Visible light (Newport 9600 Full Spectrum Solar Simulator equipped with a 150 W ozone free xenon lamp)	[56]
Fe-doped TiO ₂ /PS	Hydrothermal, phase inversion	-250 mL BPA (10 mg·L ⁻¹) -visible-light (500-W Xenon lamp)	[58]
PS/N,Pd co-doped TiO ₂ composite	Phase inversion	-100 mL of EY (100 mg L ⁻¹) -visible-light (solar simulator (Oriel, Newport), equipped with an Oriel 500W Xenon lamp)	[54]
TiO ₂ nanotube-PES	Anodization	-4 mL of MB (20 μmol L ⁻¹), Diclofenac (25 mg L ⁻¹) -UV-A sunlamp (Heraeus Original Hanau Suncare tanning tube 21/25 Slim, radiant flux density 7.6 mW cm ⁻²)	[42, 43]
PES/LSMM-OGCN	Phase inversion	-1 L of Phenol (10 ppm) -Ultraviolet lamp (Vilber Laurmat, λ = 312 nm, 30 W, light intensity 3.0 mW/cm ²) and a visible lamp (light-emitting diode (LED) lamp, λ = 420 nm, 30 W)	[59]

3.1.2 Fluoropolymeric membranes: PVDF and PTFE

Fluoropolymers like polyvinylidene fluoride (PVDF) has the advantages of high temperature resistance, chemical corrosion resistance, aging resistance and high strength due to stable –C–F bond in the main chain [60]. But the PVDF membrane application in

the field of water treatment and separation is limited because of the strong hydrophobicity and easy pollution. An easy way to address this issue is applying casting solution method. This method entraps the hydrophilic nanoparticles within the membrane matrix with good stability. However, the aggregation of nanoparticles could affect membrane morphology and alter membrane porosity, roughness, and mechanical strength. Meanwhile, the photodegradation rate of pollutants is suppressed because the entrapped photocatalysts are not easily accessible. Therefore, some researchers employed the self-assembly method, wherein semiconductor is bound on the surface of the membrane through non-covalent interactions [61]. The procedure is also straightforward, the fully formed membrane is dipped in a photocatalyst suspension for a certain period to allow coating [62-65]. In order to provide the necessary binding sites in the surface of PVDF, blending or grafting of hydrophilic materials like poly(styrene-alt-maleic anhydride) (SMA) [66] and PES [62, 67], poly(acrylic acid) (PAA) has been applied [68]. Plasma-induced graft polymerization is a simple technique that eliminates the need for chemical initiators or other components. The activated species of low energy plasma can trigger polymerization reaction on or near the surface of a wide variety of materials even with short bursts of plasma exposure [69]. You et al. grafted PAA on PVDF by plasma method to assembly of TiO₂ nanoparticles [68]. The membrane with 0.5% TiO₂ loading maintained the highest pure water flux and the best protein antifouling property. The TiO₂-modified membranes removed 30–42% of 50 mg L⁻¹ aqueous Reactive Black 5 (RB5) dye.

Damodar et al. prepared composite PVDF/TiO₂ membranes using general phase inversion method [70]. The modified PVDF membranes were prepared by adding different amounts of TiO₂ particles (0–4 wt.%) into the casting solution. The TiO₂ entrapped PVDF membranes were tested for its antibacterial property using *Escherichia Coliform* (*E. Coli*), photoactive property using Reactive Black 5 (RB5) dye and self-cleaning (antifouling) properties by fouling using 1% Bovine Serum Albumin (BSA) solution. Results showed that TiO₂ addition significantly affects the pore size and hydrophilicity of the PVDF/TiO₂ membrane. This also improves the flux and permeability of prepared membrane. Antibacterial study showed that the composite PVDF/TiO₂ membrane removes *E. Coli* at a very faster rate than neat PVDF membrane and membrane with 4% TiO₂ possess highest antibacterial property. The RB5 dye removal using PVDF/TiO₂ occurs under UV by photolysis.

Alaoui et al. prepared porous PVDF-based membranes filled with anatase (TiO₂) nanocrystalline particles using phase inversion technique for entrapment of TiO₂ [71]. The best photocatalytic membrane had a pore size of 0.96 μ m, with a maximum porosity of 86% at 0.5 TiO₂/PVDF weight ratio. The results indicate that after 7.5 h of irradiation for Brilliant Green (BG), the dye degradation ratio was about 81% for TiO₂/PVDF and 97%

for TiO_2 , and after 5h of irradiation for Indigo Carmin (IC), the dye degradation ratio was about 89% for TiO_2/PVDF and 97% for TiO_2 (Degussa P25).

Li et al. investigated Ag/TiO_2 embedment on the surface property of PVDF membrane [72]. Ag/TiO_2 improved the surface hydrophilicity of PVDF membrane in contrast with virgin PVDF membrane. Besides, Ag/TiO_2 nanoparticle provided PVDF membrane with an enhanced visible-light response activity. The absorption in the visible light range was derived from the localized surface plasmon resonance (LSPR) of Ag nanoparticles. $\text{Ag}/\text{TiO}_2/\text{PVDF}$ exhibits excellent activity on degradation of methylene blue (MB) and inactivation of bacteria [72, 73].

Nanocomposites of poly(vinylidene difluoride)-co-trifluoroethylene (PVDF-TrFE) with different concentrations of TiO_2 P25 nanoparticles (5, 10, and 15 wt.%) and ZnO nanoparticles (15 wt.%) were produced by Teixeira et al. and tested on the degradation of methylene blue (MB) [74]. It was found that increasing the photocatalyst concentration results in higher photocatalytic efficiencies; the degradation rates of 15% of TiO_2 and ZnO were similar; and the photocatalytic activity of composites in dye degradation with TiO_2 5%, TiO_2 10%, TiO_2 15%, and ZnO 15% decreased as 6%, 16%, 13%, and 11% after three utilizations [74].

Recently, many studies have shown that graphene-based membranes have great potential in filtration, separation and water desalination. Addition of GO (~ 1% rather than mass of polymer) can significantly improve the hydrophilicity, permeability and antifouling performance of the membranes [75-77].

One of the practical solutions for overcoming the photodegradation of the polymeric membrane is to protect the membrane surface with polydopamine (PDA) as a free-radical scavengers that is capable of absorbing and quenching the radicals produced by UV light [78]. Visible light photocatalysts have been highly developed owing to their efficient utilization of approximately 50% solar energy. In 2010, Ye team reported Ag_3PO_4 , with the band gap of 2.36 eV, and a valence band potential of 2.43 eV, it can absorb the UV-vis wavelength in the solar light less than 530 nm [79]. Zhang et al. prepared PDA/RGO/ Ag_3PO_4 /PVDF membrane, which can efficiently degrade and separate dye molecules (99% removal of MB) under visible-light irradiation. Polydopamine has good absorption ability to ultraviolet and visible light, and its good photoconductivity can enhance the generation of photogenerated e^-/h^+ under the visible light irradiation [60].

Bi_2WO_6 with perovskite structure has attracted tremendous attention due to its superior intrinsic properties such as narrow band gap, high physicochemical stability, low cost and non-toxicity. However, bare Bi_2WO_6 exhibits a short visible-light absorption limiting its photocatalytic efficiency. To address this problem, various efforts have been made, such

as ions doping, noble-metal deposition, nanostructured control, crystal facet engineering and heterostructure construction [80-86].

Heterostructure construction is one of the most effective means to strengthen the visible-light photocatalytic activity of Bi_2WO_6 . Recently, a few novel composites based on Bi_2WO_6 such as $\text{Bi}_2\text{WO}_6/\text{TiO}_2$ [87], $\text{g-C}_3\text{N}_4/\text{Bi}_2\text{WO}_6$ [84], $\text{WO}_3/\text{Bi}_2\text{WO}_6$ [88], $\text{BiOI}/\text{Bi}_2\text{WO}_6$ [89], $\text{Bi}_2\text{O}_2\text{CO}_3/\text{Bi}_2\text{WO}_6$ [90], $\alpha\text{-Fe}_2\text{O}_3/\text{Bi}_2\text{WO}_6$ [91], $\text{Ag}_2\text{O}/\text{Bi}_2\text{WO}_6$, $\text{Bi}_2\text{WO}_6/\text{GO}$ [92], $\text{Bi}_2\text{WO}_6/\text{SiO}_2$ [93], $\text{Bi}_2\text{WO}_6/\text{Fe}_3\text{O}_4$ [94] and $\text{Bi}_2\text{WO}_6/\text{BiPO}_4$ [95] has been developed. Li et al. obtained PVDF membranes modified with RGO/ Bi_2WO_6 with a double-layer coating method through non-solvent-induced phase separation [96]. The water contact angle was improved by about 30° by RGO/ Bi_2WO_6 ; this indicated the enhanced membrane hydrophobicity. The high desalination rate proved that all of the prepared membranes were appropriate for the membrane distillation (MD) process. The RGO/ Bi_2WO_6 /PVDF achieve 26.26%–59.95% removal rates in 10 mg/L aqueous ciprofloxacin (CIP) under visible light for 7.5 h. The best photocatalytic performance (59.95% removal rate) was obtained in 300 mL of 10 mg/L CIP under the irradiation of visible light for 7.5 h; the amount of RGO/ Bi_2WO_6 was influential in final result.

Metal-organic frameworks (MOFs), owing to their outstanding properties including large surface areas, well-ordered porosity and tunable molecular structure have been extensively researched in photocatalysis filed [97-99]. Guo et al. reported that by the hydrothermally prepared $\text{g-C}_3\text{N}_4/\text{NH}_2\text{-MIL-88B(Fe)}$ heterojunction (MIL-88B(Fe) = Iron-based MOFs); 100% of MB photodegradation was achieved in 120 min under visible light, much greater than the $\text{g-C}_3\text{N}_4$ and $\text{NH}_2\text{-MIL-88B(Fe)}$ individually [100]. Hu et al. fabricated composite film, $\text{Bi}_2\text{WO}_6/\text{MIL-53(Al)}/\text{PVDF}$, through a hydrothermal process combined with immersion phase inversion method [101]. The formation of heterojunction structure between Bi_2WO_6 and MIL-53(Al) , increased photocatalytic activity (95.3% degradation of RhB). The photocatalytic degradation mechanism by quenching tests revealed that the predominant reactive species were h^+ , $\cdot\text{O}_2^-$ and $\cdot\text{OH}$.

PVDF membrane was modified using atomic layer deposition (ALD) technique by coating three-dimensional (3D) TiO_2/ZnO photocatalyst on membrane surface and pore walls [102]. The hydrophilicity, permeability, photocatalysis and anti-fouling performances of modified membranes were investigated in detail. The composite modified membrane ($\text{TiO}_2:\text{ZnO}=1:3$) was more sensitive to visible light. The maximum flux of 0.017 cm/s was obtained for the composite modified membrane ($\text{TiO}_2:\text{ZnO}/1:3$ with ZnO content of 0.923 $\mu\text{mol}/\text{cm}^2$), due to enhanced hydrophilicity. Moreover, the 3D modified layer on membrane surface displayed excellent photocatalytic activity and stable reusability during methylene blue (MB) degradation.

Compared with TiO_2 , ZnO has rich micro-morphology, good biocompatibility and environmental safety, which make it quite popular in the field of photocatalysis. The electrons and holes generated on the surface of ZnO are easily recombined in the photocatalytic process, which reduces the photocatalytic efficiency. Therefore, considerable efforts including noble metal loading, semiconductor coupling, ion doping and special preparation process have been devoted to enhance the photocatalytic activity of ZnO [103].

Zhang et al. showed coupling ZnO with GO can effectively enhance the dispersibility and the photocatalytic efficiency [104]. They synthesized PVDF/GO/ ZnO photocatalytic membranes for degradation of organic dyes. The photocatalytic degradation rate of methylene blue (MB) could reach 86.84% , under Xenon irradiation (300 W). The radical trapping experiments with different active radical scavengers showed that the oxidizing species ($\cdot\text{O}_2^-$) plays an important role in the decolorization process of MB. The photocatalytic efficiency of the composite membrane for MB increase by addition of H_2O_2 , which may be attributed to the fact that the H_2O_2 , which is a strong oxidant, could suppress the e^-/h^+ pair recombination on the surface of ZnO by trapping electrons generated on the surface of ZnO [105].

Zinc sulfide (ZnS) with strong oxidization and fast generation of charges widely used as photocatalyst for toxic organic pollutant degradation [102]. To alleviate the quick recombination rate of the photoinduced e^-/h^+ pairs, GO exhibits excellent electron transfer performance and can reduce the recombination rate, which is helpful to enhance the photocatalytic activity. Du et al. prepared zinc sulfide/graphene oxide/polyvinylidene fluoride ($\text{ZnS}/\text{GO}/\text{PVDF}$) composite membrane by immersed phase inversion method. Uniform surface, high hydrophilicity (water contact angle = 62.2°), and enhanced permeability to $326.1 \text{ L}/(\text{m}^2 \text{ h})$ were characteristics of hybrid membrane [106].

$\text{BiVO}_4/\text{GO}/\text{PVDF}$ system showed enhanced photocatalytic activity degradation of methylene blue (MB), Rhodamine B (RhB) and Safranin-O (SO) in water under visible light irradiation as compared to the pure BiVO_4 catalyst, BiVO_4 and PTFE (polytetrafluoroethylene) decorated on the graphene sheet [107]. Graphene sheets in this composite enhances photocatalytic performance under visible light. Photoluminescence spectra proved the effective quenching of the photogenerated e^-/h^+ pairs is the main reason of enhanced photocatalytic activity.

The electrospun nanofiber mats of fluoropolymers with micro-sized porous structure can offer high specific area and good enrichment ability for organic compounds, leading to the improvement of the photocatalytic efficiency, stability and recycle ability [108-116].

Following we have summarized recent development of PVDF photocatalytic membranes in degradation of organic pollutants (Table 4).

Table 4. Application of photocatalytic fluoropolymeric membranes in degradation of various types of organic pollutants.

Photocatalyst name	Preparation method	Condition/ Light source	Ref.
Anatase/PVDF	Phase inversion	-400 mL of BG, IC (20 mg L ⁻¹) -UV Swig black lamp (Bioblock) at 365 nm	[71]
PDA/RGO/Ag ₃ PO ₄ /PVDF	Phase inversion	-100 mL of MB (20 mg L ⁻¹) -Visible light (common incandescent lamp, 200 W)	[60]
PVDF/GO/ZnO	Phase inversion	-60 mL of MB (10mg L ⁻¹) -Xenon illumination (300 W)	[104]
Au-TiO ₂ /PDA/PVDF	Vacuum filtration	-20 mL Tetracycline (10 mg L ⁻¹) -Visible light	[117]
TiO ₂ entrapped PVDF membranes	Phase inversion	-25 mL of RB5 (100 mg L ⁻¹) -UV light (UV-C, 15W)	[70]
Ag-TiO ₂ /PVDF film	Blending/photo reduction combined method	- 30 mL of MB (10 mg L ⁻¹) -Visible light (18W fluorescent Lamp)	[72]
ZnS/GO/PVDF	Phase inversion	-100 mL of MB (10 mg L ⁻¹) -photochemical reactor ³² (Beijing NBET Technology Co., Ltd) in sunlight range (250-800 nm)	[106]
PVDF-TrFE/TiO ₂	Solvent casting	-MB -17.6 μM /50 mL -UVA (365 nm ;6 Philips 8W mercurial fluorescent lamps–UMEX)	[74]
BiVO ₄ -GO-PVDF	Ultrasonication	-100 mL of MB, RhB, SO (20 mg L ⁻¹) -visible light (500 W xenon lamp through UV cut-off filters)	[107]

3D-TiO ₂ /ZnO/ PVDF	ALD	-100 mL of MB (10^{-5} mol L ⁻¹) -visible light (xenon light source, 200 W)	[102]
Bi ₂ WO ₆ /MIL-53(Al)/PVDF	Hydrothermal, immersion phase inversion	-250 mL of RhB (20 mg L ⁻¹) -visible light 500 W Xe lamp ($\lambda > 420$ nm)	[101]
PVDF/ Bi ₂ WO ₆ /rGO	Double-layer coating method through non-solvent-induced phase separation	-300 mL CIP (10 mg L ⁻¹) -visible light 500 W U-shaped xenon lamp	[96]
paper-like PVDF/TiO ₂	Electrospinning and electrospraying	-45 mL of BPA, 4-CP, and CMT (10 μ M) -UV (six 4-W blacklight blue lamps ($\lambda = 350\text{--}400$ nm, F10T8, Sankyo Denki, Japan)	[113]
ZnS/PVDF/MAA/TFA	Electrospinning	-15 mL of MB (5.3 mg L ⁻¹) -UV (two 15 W shortwave UV lamps (254 nm) (Kexing Company, Changsha China), distance between two lamps =15.0 cm)	[114]
TiO ₂ /PVDF–TrFE membrane	Solvent casting	-Tartrazine -10 mg L ⁻¹ -sunlight irradiation	[118]
TiO ₂ -PVDF-steel mesh	Coating, electrospraying, and thermal fixation	-45 mL of MO, RB4, SMX (10 μ M) - UV irradiation (six blacklight blue lamps (4 W, $\lambda = 350\text{--}400$ nm, Sankyo Denki F10T8))	[115]
PVDF/Ag ₂ NCN	Phase inversion	-100 mL Acid Blue 1 (10^{-5} M) -visible light (light irradiation density was 94.30 mW cm ⁻²)	[119]
TiO ₂ -PVDF ultrafiltration membranes	Phase inversion	-80 mL of RhB (2×10^{-5} M), pH = 6 -UV (250 W, high-pressure Hg lamp at 365 nm)	[120]

TiO ₂ /GO/P(VDF-TrFE)	Electrospinning	-13 mL MB (10^{-5} mol L ⁻¹) -UVA (high-power LED source (Thorlabs, 700 mA) with an excitation peak at 365 nm)	[121]
PVDF/ meso-TiO ₂	Dual-templated synthesis with solvent extraction	-20 mL of MO (10 mg L ⁻¹) -UV (ZF-1 three UV analyzer, Shanghai Jihui scientific analysis instrument Co., Ltd., China) (25 W, 365 nm))	[122]
PVDF/TiO ₂ nanofibers	Electrospinning	-200 mL of BPA (10 ppm) -UV radiation (UV lamp, Vilber Laurmat, France, λ = 312 nm, 30 W).	[123]
Er-Doped and Er/Pr-Codoped TiO ₂ / PVDF-TrFE	Solvent casting	-MB (1×10^{-5} M) pH 6.8 -UV (high-power light-emitting diode (LED) source (Thorlabs, 700 mA), at 365 nm (UV-A)	[124]
PVDF/anatase/silica nanocomposites	Thermal treatment	-MB (10 mg L ⁻¹) -UV (UV lamp, $\lambda_{\max}$ = 365 nm)	[125]
PVDF/plasma-grafted poly(acrylic acid) membrane with self-assembled TiO ₂	Plasma-induced graft polymerization	-RB5 (40 mg L ⁻¹) -UV (UV lamp (254 nm, 15W)	[68]
Naked-TiO ₂ capsulated in nanovoid microcapsule of PVDF	phase inversion	-100 mL of MO (15 mg L ⁻¹) -UV (200 W UV-lamp at 365 nm)	[126]
PTFE/Co ₃ O ₄	Dip coating	-Orange II (0.05 mM), pH = 7 -Suntest solar light simulator (90 mW/cm ²) .	[127]
TiO ₂ /PTFE Membrane	electrospinning, immersion, and calcination	-100 mL MB solution (2.5 mg L ⁻¹) -UV (lamp power 300 W)	[128]

Recently, there have been many reports of using modified poly(tetrafluoroethylene) (PTFE) fiber and membranes for the degradation of dyes [129, 130]. Modified PTFE fibers by acrylic acid and loaded Fe³⁺ were prepared and used as Fenton catalysts for the degradation of reactive Blue 222 in the pH range 3-9 under visible irradiation [131]. The study found that the loaded catalytic material had good performance in degradation of the azo dye, and the fibrous support could maintain good mechanical properties in the reaction

process, which avoided the strength decreasing of PTFE fiber carrier during catalytic degradation. However, modification and loading operations are difficult, low efficiency and high cost. Besides, fibers with large diameters are difficult to achieve with better catalytic properties. Kang et al., prepared a TiO₂/PTFE membrane catalyst by electrospinning, immersion, and calcination, and the catalyst was applied to MB degradation [128].

3.1.3 Development of photocatalytic polyaniline (PANI) membranes

A number of processing technologies such as template synthesis, self-assembly and electrospinning have been proposed for the preparation of polymer nanofibres. Among these, the electrospinning process seems to be a promising method which can be further developed for mass production of one-by-one continuous nanofibres from various polymers [132]. Electrospun fibers of ceramic or polymeric materials possess many extraordinary properties, such as small diameters and large specific surface areas. Additionally, the non-woven fibrous mats made of electrospun polymer fibers offer a unique capability of readily controlling the pore sizes. Till now, many synthetic and natural polymers have been electrospun into fibrous mats for various applications.

Various strategies have been employed to make TiO₂ photocatalysts highly efficient under visible light, including dye sensitization, polymer modification, non-metals doping, semiconductor coupling, and transition metal doping [133]. Conjugated polymers, such as polyaniline (PANI), poly(fluorine-co-thiophene) (PFT), polythiophene, polypyrrole and their derivatives can function as sensitizers to extend photoresponse of TiO₂ into the visible region effectively [133, 134]. PANI is one of the most fascinating conductive polymers and has received much attention for easy polymerization, high yield, relative high conductivities, low cost, and high stability [135]. Under visible-light irradiation, PANI generates π - π^* transition, injecting the excited electrons into the conduction band of TiO₂, and then the electrons transfer to an adsorbed electron acceptor to yield oxygenous radicals [136]. Meanwhile, synergetic effect of coupling photocatalysts with PANI proves to be effective to promote photoinduced charge separation and inhibit charge recombination [137, 138]. There are variously modified photocatalysts based on PANI such as PANI/TiO₂, PANI/BiVO₄, PANI/SnO₂, and PANI/MoO₃, PANI/PbS, and PANI/CoFe₂O₄/TiO₂ [139-142]. Lin et al. prepared a catalytic system of PANI/TiO₂ nanocomposites, which exhibited higher photocatalytic activity and stability for degradation of methyl orange than the bare TiO₂ under both UV and visible light irradiation [143]. Li et al. prepared PANI-doped TiO₂ and PLLA (poly(L-lactide)) fibers by a combination of coaxial-electrospinning and in-situ polymerization [144]. The aniline monomers were located in the core phase and in-situ polymerized by ammonium persulfate

(APS) after electrospinning. Photocatalytic degradation tests show that the membrane exhibit enhanced photocatalytic activity for degradation of methyl orange under visible light, likely due to the synergistic effect of PANI and TiO_2 .

Li et al. applied electrospinning method in preparation of bilayered composite fibrous membrane [145]. Polyaniline (PANI), Fe_3O_4 nanoparticles (NPs), and $\text{Bi}_2\text{WO}_6:\text{Yb}^{3+}, \text{Er}^{3+}$ were incorporated into polyacrylonitrile (PAN) and electrospun into the obtained fibrous membrane with $[\text{Bi}_2\text{WO}_6:\text{Yb}^{3+}, \text{Er}^{3+}/\text{PAN}]$ nanofibers as one layer and $[\text{PANI}/\text{Fe}_3\text{O}_4/\text{PAN}]$ nanofibers as the other layer. The degradation of Rhodamine B (RhB) was used to characterize the photocatalytic activity of the photocatalyst.

Table 5. Application of photocatalytic polyaniline membranes in degradation of various types of organic pollutants.

Photocatalytic name	Preparation method	Condition & light source	Ref.
$\text{TiO}_2/\text{PANI}/\text{PEO}$	Electrospinning & electrospaying	-10 mL CEPS solution in heptane at concentration of 0.05(v/v) -UV irradiation	[146]
PANI-doped TiO_2/PLLA	Coaxial-electrospinning and in-situ polymerization	-25 mL of MO solution (0.5 mg L^{-1}) -Visible light (250 W xenon lamp with a 420 nm cut-off glass filter was used as a visible-light source)	[144]
$\text{PANI}/\text{Fe}_3\text{O}_4/\text{PAN}/[\text{Bi}_2\text{WO}_6:\text{Yb}^{3+}, \text{Er}^{3+}/\text{PAN}]$ bilayered composite fibrous membrane	Electrospinning	-100 mL of RhB aqueous solution (0.05 mg L^{-1}) -UV-vis light irradiation (A 500 W mercury lamp)	[145]
$\text{PANI}/\text{TiO}_2/\text{SiO}_2$ nanofiber membrane	Electrospinning	- 3 mL of MO solution (1.5 mg L^{-1}) -Visible light (500 W xenon lamp with a 420 nm cut-off glass filter)	[133]

Neubert et al. prepared conductive electrospun fibrous membrane. by integration of electrospinning and electrospraying. The non-conductive polyethylene oxide (PEO) was blended with ($\pm$)-camphor-10-sulfonic acid (CSA) doped conductive polyaniline (PANI) for electrospinning. The conductive CSA/PANI/PEO composite fibers were produced upon electrospinning, then TiO₂ nanoparticles were sprayed and allowed to adsorb on the fibers. The photocatalytic activity of the prepared TiO₂/PANI/PEO was tested against the toxicant simulant 2-chloroethyl phenyl sulfide (CEPS) under UV irradiation. It was observed that the TiO₂ nanoparticles catalysts embedded PANI/PEO fibrous membrane decontaminated the toxicant CEPS significantly. Table 5 summarized reported photocatalytic polyamide membranes in degradation of organic pollutants.

3.1.4 Development of photocatalytic polyamide membranes

Kwak et al. prepared hybrid organic/inorganic reverse osmosis (RO) membranes composed of aromatic polyamide thin films underneath TiO₂ nano-particles by a self-assembly process, with photocatalytic destructive capability on microorganisms[64, 147]. Lombardi et al. prepared electrospun polyamide-6 membranes containing TiO₂ photocatalyst with self-cleaning, antibacterial and photochromic textiles, and high surface area [132]. By tailoring the electrospinning parameters, two different membranes (thicknesses 5 and 20 μ m, with inorganic/organic ratios of 10 and 20 wt%.) were successfully produced, The photocatalytic activity of both hybrid systems was evaluated by degradation of methylene blue under UV irradiation.

Zhang et al. applied immobilized TiO₂ nanoparticle on polyamide fabric in degradation of methylene blue [148]. It was found that when polyamide-6 (PA-6) fabric was treated in titanium sulfate and urea aqueous solution, anatase nanocrystalline titanium dioxide was synthesized and simultaneously adhered onto the fiber surface. The average crystal size of titanium dioxide nanoparticles was about 13.2 nm.

Daels et al. prepared TiO₂ functionalized nanofiber membranes via electrospinning on a single nozzle electrospin set-up by adding two types of TiO₂ nanoparticles (commercial Degussa P25 ~21 nm and synthetic colloids ~ 6 nm) at different concentrations to the PA-6 prior to the spinning solution or by post-functionalizing the electrospun membranes [149]. Both methods improved the degradation of methylene blue under UV irradiation although the post-functionalization with colloidal TiO₂ nanoparticles showed the best photocatalytic activity. Table 6 represents the recent progress in degradation of organic pollutants by photocatalytic polyamide membranes.

Table 6. Application of photocatalytic polyamide membranes in degradation of various types of organic pollutants.

Photocatalyst name	Preparation method	Condition & light source	Ref.
PA6 fabric/TiO ₂ nanoparticle	Hydrothermal method	-50 mL of MB solution (20 mg L ⁻¹) at natural pH -UV(20 W, λ = 254 nm, quartz ultraviolet lamp)	[148]
Electrospun PA 6/TiO ₂ nanoparticles	Electrospinning	-MB solution (10 mg L ⁻¹) -UV (300 W Osram Ultra-Vitalux lamp)	[149]
Electrospun PA6/TiO ₂	Electrospinning	-MB (5 ppm in ethanol) -UV (medium-pressure mercury arc lamp (Hamamatsu), average light intensity was ca 50 mW cm ⁻²)	[132]
TiO ₂ /PA 6 nanofibrous membranes	Dip coating	-MB (4.2 mg L ⁻¹) and isoproturon (5 and 10 mg L ⁻¹) solutions -UV-A, similar to sunlight (300 W Osram Ultra-Vitalux lamp, intensity 5 m W/cm ²)	[150]
TiO ₂ /PA 12 electrospun fiber mats	Electrospinning	-13 mL of MB solution (10 ⁻² mM) , pH = 6.8 -UV-A (high power LED source (Thorlabs, 700 mA, at 365 nm))	[151]

3.1.5 Development of cellulose-based photocatalytic membranes

Cellulose acetate (CA) is the most widely used cellulose ester, but its usefulness is limited by its low dimensional stability at high temperatures. Several strategies have been employed in an attempt to broaden the applications of this indirectly renewable polymer, since it is derived from cellulose, such as the formation of polymer blends, chemical modification through the introduction of functional groups, or grafting to the polymer chain. Preparation of organic/inorganic hybrid composites such as cellulose acetate with niobium, zirconium, titanium, aluminum, bismuth, and silicon oxides is a suitable route for its chemical modification [152].

Campos et al. prepared a cellulose acetate/TiO₂ composite by dissolving cellulose acetate in acetone:acetic acid (70:30 in volume) and adding quantities of titanium (IV) tert-

butoxide to obtain varying oxide content after phase inversion [152]. TiO_2 in the composite was a crystalline anatase phase. The composite showed effective photocatalytic activity in the degradation of MB under solar radiation. Yang et al. [153] fabricated the CA/ SiO_2 - TiO_2 hybrid via water vapor-induced phase inversion of CA solution and simultaneous hydrolysis/condensation of 3-aminopropyltrimethoxysilane (APTMS) and tetrabutyl titanate (TBT) at room temperature. Micro-nano hierarchical structure was constructed on the surface of the film. The flux of the film for the emulsion separation was up to $667 \text{ L m}^{-2} \text{ h}^{-1}$, while the separation efficiency was up to 99.99 wt%. Meanwhile, the film exhibited excellent stability during multiple cycles, performed excellent photo-degradation under UV light.

Bi_2O_3 is an attractive candidate semiconductor material because of its low cost, favorable direct bandgap of 2.5-2.8 eV, and good photostability in acidic conditions. Zhang et al. [154] prepared nanosized Bi_2O_3 to enhance the photocatalytic activity by a simple sonochemical route. In addition, Liu et al. [155] showed the crystalline phase evolution of Bi_2O_3 had an effect on photocatalytic performance. The effective utilization of visible light for photocatalytic membranes is main challenge. To extend the lifespan of photogenerated electrons and holes for higher photocatalytic activity, various strategies have been reported, such as doping with metal elements (Au-doped Bi_2O_3 [156]), nonmetal elements (S-doped Bi_2O_3 [157]) and coupling with other semiconductors to form heterojunction structures ($\text{Bi}_2\text{O}_3/\text{Bi}_2\text{MoO}_6$ [158], $\text{Bi}_2\text{O}_3/\text{g-C}_3\text{N}_4$ [158]). The heterojunction structure can not only combine at least two disparate functional materials in one system but also reduce the recombination rate of the electron-hole pairs. For instance, Juntrapirom et al. [159] found that the novel SnS/ BiOI heterostructures presented higher photocatalytic performance and better stability than a single semiconductor. Liu et al. [160] synthesized another $\text{BiVO}_4/\text{WO}_3$ heterojunction with an excellent visible-light response, a good photostability and high photoelectrochemical activity. However, it is difficult to separate it from treated water, which limits its practical application. The challenge is to develop an effective method to improve the recovery and reuse of the catalyst. CA-based membranes have been widely utilized as support membranes due to the advantages of biodegradability nontoxicity and low cost. Li et al. synthesized the $\text{Bi}_2\text{O}_3/\text{ZnS}$ heterojunction, by a one-step hydrothermal method and applied phase inversion method to immobilize $\text{Bi}_2\text{O}_3/\text{ZnS}$ nanocomposite in CA membrane. The prepared photocatalytic membrane was applied to degradation of RhB [1].

$\text{Bi}_{12}\text{O}_{17}\text{Cl}_2$, a new type of promising layered materials, has been demonstrated with excellent visible light photocatalytic activity for pollutant degradation [161]. However, the practical application of pure $\text{Bi}_{12}\text{O}_{17}\text{Cl}_2$ is still limited by the fast recombination electron-hole pairs and many methods have been used to modify and adjust the catalyst to visible

light region [162-164]. The visible light photoactivity of the obtained catalyst generally have a good effect on dye degradation. However, the visible light activity for the colorless organic contaminant still needs to be improved. Yu et al. [165] combined the $\text{Bi}_{12}\text{O}_{17}\text{Cl}_2$ with graphene oxide (GO) and dopamine together on surface of CA membrane. The $\text{Bi}_{12}\text{O}_{17}\text{Cl}_2$ can tune the interlayer spacing of GO to increase the flux [166]. During the reduction process, the hydrophilic interfaces of GO may change into hydrophobic because of the loss of oxygen functionalities [167]. Therefore, a proper reducing agent both achieving the reduction of graphene oxide and keeping the hydrophilicity of the GO interface is very important. Many researches indicated that dopamine with physicochemical properties of adhesion and self-polymerization is a milder and nontoxic reducing agent at weak alkaline pH. Therefore, dopamine has been introduced to reduce GO and obtain uniformed dispersion membrane solution. Results indicated that the as-prepared membrane can simultaneously degrade dye/colorless organic contaminant and separate oil/water emulsions. In addition, the use of dopamine has improved the adherence between $\text{Bi}_{12}\text{O}_{17}\text{Cl}_2$ with graphene matrix and made the RGO/PDA/ $\text{Bi}_{12}\text{O}_{17}\text{Cl}_2$ firmly together with the CA membrane. The photodegradation of MB solution and 4-CP solution under visible light was used to estimate the photocatalytic performance of the prepared membrane [165].

It was reported that electrospun CA nanofibrous membranes with large specific surface area, high porosity, and high permeability could be an effective support for photocatalysts such as ZnO, ZnS, TiO_2 [168-170]. This could create a synergistic effect that enhanced the catalytic performance of the individual materials.

Bacterial cellulose (BC)-hydrated membranes with nanosize superfine structure are good templates in the preparation of new organic-inorganic hybrids with specific morphology and size [171-173]. Among many possible nanoparticles to be incorporated in BC matrixes, TiO_2 nanoparticles have attracted considerable attention because of the relative ease of synthesis and their strong photodegradation activity. Table 7 summarizes the application of cellulose-based photocatalyst nanostructured materials for photocatalytic degradation of various organic pollutants.

Table 7: Application of cellulose-based hybrid photocatalysts in photocatalytic degradation of various types of organic pollutants.

Photocatalyst name	Preparation Method	Condition & light source	Ref.
RGO/PDA/Bi ₁₂ O ₁₇ Cl ₂ -CA	Vacuum filtration	-100 mL MB solution (15 mg L ⁻¹) and 4-CP solution (15 mg L ⁻¹) -visible light (long-arc Xe lamp, 500 W)	[165]
Cellulose (core)-TiO ₂ (sheath) fibers	Coaxial-lectrospinning	-Keyacid Blue dye (0.1 wt %) -A halogen light source (power density of ~27 mW/cm ²)	[174]
BC/titanium dioxide	Sol-Gel method	- MO solution (20 mg L ⁻¹) -ultraviolet lamp.	[175]
CA/TiO ₂ composite	Phase inversion	-25 mL of MB solution (12 × 10 ⁻⁶ mol L ⁻¹) -Uv radiation. (150W Hg lamp)	[176]
ZnS/BC nanocomposites	In Situ Precipitation using BC as template	-100 mL of MO solution(10 mg L ⁻¹) -UV light at a wavelength of 254 nm.	[177]
CA-TiO ₂ P90 (ratio: 10:1)	Casting soltion	-30 mL of MB or RhB solution (2 × 10 ⁻⁵ M) -UV radiation (two 15 W lamps, 365 nm)	[178]
CA/TiO ₂ composite ultrafine fiber	Electrospinning	-10 mL of MB solution (10 mg L ⁻¹) - UV source. (300 W mercury lamp)	[179]
SrTiO ₃ /TiO ₂ heterojunctioned nanofibers assembled on commercial CA	Electrospinning and hydrothermal treatment	-60 mL of AO 7 solution (50 mg L ⁻¹) -Upland UVP lamp (254 nm, 40 mW/cm ²)	[180]
TiO ₂ nanoparticles/cellulose fibers	Hydrothermal method	-4 mL of MO solution (0.25 mM) -UV-A lamp (50 cm long, 72 W, emission range 320–400 nm)	[181]
N-doped TiO ₂ nanorods in regenerated cellulose thin films	Phase inversion	- 150 mL of MB solution (40 mg L ⁻¹) - UV lamp (Vilber Laurmat, λ = 312 nm, 30 W) and white light-emitting diode (LED) flood light (CS-FL-30W, λ > 420 nm, 30 W).	[182]
C/SiO ₂ /TiO ₂	Dip-Coating or spin-coating	-MV dye (0.1%) - UV light (Xe 150-W lamp, the range of emission wavelength between 290-400 nm (UVA and UVB))	[183]
Dispersed-TiO ₂ on CA	Phase inversion	-25 mL of MB solution (12 × 10 ⁻⁶ M) -UV radiation (150W Hg lamp)	[152]

CA-polystyrene-ZnO	Solution dispersion blending method	-CR or RY-105 solution (100 mg L^{-1} , Neutral pH) -Sunlight irradiation	[184]
Bi ₂ O ₃ /ZnS Heterojunction Functionalized Porous CA Membranes	Phase-inversion	-50 mL of RhB solution (5.0 mg L^{-1}) -Visible-Light (xenon light source with the power of 200 W)	[185]
RGO/PDA/Bi ₁₂ O ₁₇ Cl ₂ /CA membrane.	Vacuum filtration method	-100 mL MB solution (15 mg L^{-1}) and 4-CP solution (15 mg L^{-1}) -visible light (long-arc Xe lamp, 500 W).	[165]
RGO-Ag-TiO ₂ -CA membrane	Hydrothermal and vacuum filtration method	-MB (20 mg L^{-1}) and RhB (30 mg L^{-1}) -visible light (500 W Xe lamp coupled with a UV-cutoff filter ($\lambda \geq 400 \text{ nm}$))	[186]
CA-PEG-PU/ZnO	Casting material and solution dispersion blending method	-50 mL of RR 11 (or RO 84) solution (100 mg L^{-1}) -Sunlight	[187]
Electrospun H ₄ SiW ₁₂ O ₄₀ /CA	Electrospinning	-100 mL of MO (or TC) solution (10 mg L^{-1}), pH = 2 -UV irradiation (300 W high pressure mercury lamp)	[188]
RGO/PDA/g-C ₃ N ₄ /CA composite	Vacuum Filtration	-100 mL of MB solution ($5 \text{ mg} \cdot \text{L}^{-1}$), 1 mL H ₂ O ₂ (wt% 30) -visible light irradiation (long-arc Xe lamp, 500 W)	[189]
CA-PU/ZnO	Casting material and solution dispersion blending method	-50 mL of RR 11 (or RO 84) ($100 \text{ mg} \cdot \text{L}^{-1}$) -UV-light	[190]
CA supported Ag@AgCl	Double diffusion technique	-10 mL of MO solution ($10 \text{ mg} \cdot \text{L}^{-1}$) -visible-light irradiation (500 W Xe arc lamp equipped with an ultraviolet cutoff filter ($\lambda > 420 \text{ nm}$)).	[168]

4. Inorganic membranes

Some studies found polymeric membranes damaged by UV irradiation, hydroxyl radicals and the penetration of the hard inorganic semiconductors particles. Further, organic solvents, high temperatures acidic or caustic solutions affect applications these polymeric membranes and restrict their utilization [191].

Since the pioneering work of Anderson et al. [192], synergistic coupling of ceramic membrane filtration and UV-assisted photocatalytic degradation has been receiving much attention [193]. Recently, ceramic membranes preferred over the conventional polymeric membranes due to their excellent thermal, chemical and mechanical stability and biological inertia for wide pH range adaptability, long service life and good recoverability [194, 195]. The cost of ceramic membranes has been continuously reduced during the last years.

Ceramic membranes, which can be used in microfiltration and ultrafiltration, have superior chemical, thermal, and mechanical stability compared to polymeric membranes, and the pore size can be more easily controlled [196]. Sintering and the sol-gel process are the most common techniques to prepare ceramic microfiltration membranes. Sintering is limited to pore sizes around 0.1 μm , practically all UF membranes are prepared with the sol-gel technique. The base materials for the preparation of ceramic membranes are alumina (Al_2O_3), titania (TiO_2), silica (SiO_2), and zirconia (ZrO_2). Ceramic nanofiltration membranes can also be produced, but, to date, the pore size of most of these membranes is still relatively high.

4.1 TiO_2 -based membranes

Since the pioneering work on the fabrication of TiO_2 ceramic membrane by Anderson and co-workers [192] many research studies have been carried out to fabricate TiO_2 membranes by coating TiO_2 films on various supports [197]. Titania membranes have received significant attention in recent years due to their unique characteristics such as high water flux, semiconducting properties, photocatalysis, and chemical resistances over other membrane materials such as alumina, silica, and zirconia [198]. Currently, TiO_2 membranes have been mainly prepared via conventional tape casting processes. Chemical vapor deposition (CVD), sputtering, and sol-gel methods are also applicable for the fabrication of homogeneous TiO_2 membranes. Ma and Quan modified the sol-gel dip-coating method to enhance the performance of TiO_2 membrane via doping with Ag and Si and HAP coupling [199]. Furthermore, a visible light responsive TiO_2 membrane was fabricated via co-doping C, N and Ce through a weak alkaline sol-gel process by Cao and co-workers [200]. Most recently, Liu deposited Ag nanoparticles on TiO_2 nanofiber and subsequently formed a Ag/ TiO_2 flat membrane on glass fiber substrate for disinfection under solar light irradiation [201].

In these methods, however, strict processing conditions are commonly required for high crystallinity of the ceramic membranes, which is very cumbersome and time consuming, and are often not economically viable. In addition, the TiO₂-based membranes studied so far are in the form of finite sized tubes with diameters of at least several millimeters or flat discs and consequently have low surface area to volume ratios. These low area to volume ratios compares unfavorably with polymeric hollow fiber modules with high values; this limits the application of current inorganic tubular and disc membranes. This limitation is most evident in catalytic membrane reactors, where it is desirable to maximize the area of the membrane to increase the permeation rate to remove the product species from the reaction zone. Recently various morphologies of one-dimensional (1D) nanostructured TiO₂, including nanowires, nanofibers, nanorods and nanotubes, have been synthesized by chemical or physical methods (Figure 3) [173, 202, 203].

Figure 3. Various morphologies of one-dimensional (1D) nanostructured TiO₂.

Regarding the geometry of the membrane, hollow fibers have the highest surface area per volume ratio, which makes them very appealing because of the possibility of obtaining small membrane modules with large surface areas [203]. There are a few methods available for preparing inorganic hollow fibers, including dry spinning a system of inorganic material and binder, wet spinning a suitable inorganic material-containing solution, depositing fibers from the gas phase on to a substrate, or pyrolyzing the polymers [204, 205]. Recently, the well-known phase inversion method, commonly employed for spinning polymeric hollow fiber membranes, have been successfully adopted in preparing the inorganic aluminum oxide (Al₂O₃) hollow fibers [198, 206, 207]. Because of the phase inversion characteristics, the prepared inorganic hollow fibers possess an asymmetric structure, which provides a better permeability for a given thickness. Thus, they can be used not only in many separation processes, but also to be served as a porous support for composite membrane formation.

Typically, TiO₂ nanowires are obtainable by hydrothermal method, through treating TiO₂ powders within steel pressure vessels autoclave under controlled temperature and/or pressure in strong basic NaOH solution. It found during hydrothermal reaction, some of the Ti–O–Ti bonds were broken and Ti–O–Na and Ti–OH bonds were formed, which result in the formation of TiO₂ nanowires [208]. These nanowires exhibit superior photocatalytic efficiency relative to conventional bulk materials as a result of its larger surface area and presence of quantum size effect.

Imai, et al. [197] synthesized TiO₂ nanotube membrane, by deposition of anatase films on the inner walls of alumina porous membranes. After dissolving the alumina membrane using an aqueous solution of ammonia, the deposited titania films were obtained as nanotubes. The channel diameter of the tubes was controllable over the range 50–150 nm by changing the deposition time. In this method the morphology of the anatase nanotubes was highly controllable because no annealing for crystallization was required.

Albu et al. [209] reported formation of a nanotubular layer of TiO₂, a thickness of >100 μm, electrochemically by controlled anodization of a Ti foil in a fluoride-containing ethylene glycol. This leads to a dense array of aligned TiO₂ nanotubes, attached to the Ti substrate with a diameter of 160 ± 30 nm and a wall thickness of 20 ± 5 nm. The titanium foil was removed by selective dissolution in water-free CH₃OH/Br₂ solution, followed by exposure to hydrofluoric acid (HF) vapor for 30 min to open the bottom of the tube.

More recently, immense efforts are devoted to the study of organizing of nanowire/fiber/tube into 2D nanomaterials, including membrane and sheet. These 2D nanomaterials exhibit new properties while retaining the properties of 1D nanomaterials.

Many researchers have endeavored to introduce TiO₂ on/into high-surface-area substrate. Among them, TiO₂–SiO₂ composites have been widely used in industrial applications and most extensively studied [133]. SiO₂ is an excellent catalyst support because of its chemical inertia, thermal stability and adsorption of reactants. Particularly, the nanofibrous SiO₂ supports have both high surface-area-to-volume ratio and favorable recycling properties. Electrospinning in combination with sol–gel processes has been proved a simple and effective method to produce polymer/metal oxide composite nanofibers. In many cases, the metal oxide nanofibers can be obtained by subsequent pyrolysis. Ding et al. fabricated the flexible and amphiphobic SiO₂ nanofibrous mats via electrospinning the blend solutions of poly(vinyl alcohol) (PVA) and SiO₂ gel, followed by calcination to remove the organic component [210]. Wang et al. prepared anatase mesoporous titanium nanofibers from calcination of the electrospun tetrabutyl titanate/poly(vinylpyrrolidone) (PVP)/pluronic123 (P123) composite nanofibers [211].

Table 8. Ceramic photocatalytic membranes for degradation of organic pollutants.

Photocatalyst name	Preparation method	Condition & light source	Ref.
TiO ₂ nanostructured hollow fiber membrane	Spinning-sintering technique	-20 mL of AO7 at a concentration of 20 mg L ⁻¹ -UV light (Four UV-A lamps (SYLVANIA Blacklite F8 W/BL350, emit at 330–370 nm))	[203]
TiO ₂ /Al ₂ O ₃ composite membranes	Dip coating	-8 mL of 30 µM MB solution, pH = 3 - UV radiation Two 15 W low pressure mercury UV tubes (Spectronics), at 365 nm ,light intensity of 3.48 mW/cm ²	[212]
ZnO quantum dots on hollow SiO ₂ nanofibers	Electrospinning and calcination	-UV (50 W high-pressure mercury lamp with main emission at 313 nm)) -100 mL of the RB solution 10 mg L ⁻¹	[213]
Mesoporous titania networks consisting of interconnected anatase nanowires	Templating by BC, Dip coating and calcination	-10 ⁻⁵ M RhB solution - 250 W high-pressure mercury lamp	[173]
TiO ₂ nanoparticles/Ag fibers	Electrophoretic deposition Method	- 5 mL of a 5 µM MB solution -UV (8 W low pressure mercury lamp, intensity 11 mW/cm ²)	[214]

Various strategies have been explored to improve the photocatalytic efficiency of TiO₂ NWs or nanotubes, such as tuning its crystallite size and structure [215, 216], doping with metal or nonmetal elements [217], sensitizing with other small bandgap semiconductor materials [218, 219], synthesizing branched structures [220], and postgrowth hydrogen annealing [221]. Doping materials with Sn or Sn⁴⁺ is an attractive approach [222], especially for TiO₂, as the small lattice mismatch between SnO₂ and TiO₂ leads to good structural compatibility and stability. Xu et al. [223], reported the one-pot hydrothermal with different ratios of SnCl₄ and tetrabutyl titanate, and a high acidity of the reactant solution synthesis and controlled Sn-doped TiO₂ nanowire arrays. The obtained Sn/TiO₂ NWs are single crystalline with a rutile structure. The application of ceramic photocatalytic membranes for degradation of various organic pollutants has been summarized in Table 8.

Abbreviations

Acid Orange (AO 7)	Polyacrylonitrile (PAN)
Advanced oxidation processes (AOPs)	Polyamide 6 (PA6)
Aliphatic polyamide (PA)	Polycarbonate (PC)
3-Aminopropyltrimethoxysilane (APTMS)	Polydopamine (PDA)
Ammonium persulfate (APS)	Polyetheretherketone (PEEK)
Aromatic polyamide (Aramids)	Polyether imide (PEI)
Atomic layer deposition (ALD)	Polyether sulfone (PES)
Bisphenol A (BPA)	Poly(ether urethane) (PEU)
Brilliant Green (BG)	Polyethylene (PE),
Bovine Serum Albumin (BSA)	polyethylene glycol(PEG)
Camphor-10-sulfonic acid (CSA)	Polyethylene oxide (PEO)
Carbon covered alumina (CCA)	Polyimide (PI)
Cellulose acetate (CA)	Poly(L-lactide) (PLLA)
Cellulose esters (CE)	Poly(piperazine amide) (P-PIP-A)
Cellulose triacetate (CTA)	Polypropylene (PP)
Chemical vapour deposition (CVD)	Polysulfone (PS)
4-Chlorophenol (4-CP)	Polytetrafluoroethylene (PTFE)
2-Chloroethyl phenyl sulfide (CEPS)	Polyurethane (PU)
Cimetidine (CMT)	Poly(vinylidene fluoride) (PVDF),
Ciprofloxacin (CIP)	Praseodymium (Pr)
Congo red (CR)	Reactive Black 5 (RB5)
Eosin Yellow (EY)	Reactive Orange 84 (RO 84)
Erbium (Er)	Reactive Red ED-2B (RR ED-2B)
Hydrophilic surface modifying	Reactive Red 11 (RR 11)
macromolecules (LSMM)	Reactive yellow 105 (RY-105)
Indigo Carmin (IC)	Reverse osmosis (RO)
Graphene oxide (GO)	Rhodamine B (RhB)
Malachite Green (MG)	Safranin-O (SO)

Materials from the Lavoisier Institute (MIL)	Silver cyanamide (Ag_2NCN)
Membrane distillation (MD)	Sulfamethoxazole (SMX)
Metal organic frameworks (MOFs)	Sulfonated polysulfone (S-PS)
Methacrylic acid (MAA)	Tetrabutyl titanate (TBT)
Methylene blue (MB)	Tetracycline (TC)
Methyl orange (MO)	tetraethoxysilane (TEOS)
Methyl violet 2B (MV)	Trifluoroethyl acrylate (TFA)
Microfiltration (MF)	Trifluoroethylene (TrFE)
Nanofiltration (NF)	Ultrafiltration (UF)
Oxygenated graphitic carbon nitride (OGCN)	Ultraviolet (UV)
Photocatalytic membrane reactor (PMR)	

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