

Photocatalytic Electrospun Nanofiber Membranes

Subjects: Materials Science, Coatings & Films

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Photocatalytic nanofiber membranes are nanofiber membranes infused with photocatalytic nanoparticles. The performance of photocatalytic membranes is attributed to the photogenerated reactive oxygen species such as hydroxyl radicals, singlet oxygen, and superoxide anion radicals produced from reactions with photogenerated electrons and holes introduced by catalytic nanoparticles such as TiO₂ and ZnO upon light irradiation. Hydroxyl radicals are the most reactive species responsible for most of the unselective photodegradation of unwanted pollutants.

Keywords: nanofiber membranes ; photocatalysis ; antimicrobial properties ; micropollutants ; wastewater treatment

1. Introduction

Micropollutants found in water systems continue to pose a threat to humans, aquatic biota, other living organisms as well as the environment. These micropollutants are classified as organic (e.g., herbicides, pesticides, dyes, pharmaceuticals, phenols, polyaromatic hydrocarbons, endocrine-disrupting chemicals and natural organic matter) ^{[1][2][3]}, inorganic (such as heavy metals, mineral acids, metal compounds, and cyanides) ^{[4][5][6]}, and biological pollutants (such as parasites, bacteria, pathogens, and viruses) ^{[7][8]}. Pollution of water by micropollutants can occur naturally and/or through the release of contaminants either intentionally or accidentally due to human activities such as mining, manufacturing, and agriculture. Recently, research has given much attention to the treatment and removal of strong recalcitrant pollutants such as phenols, alcohols, nitrogenous compounds, sulphur compounds and dyes that are mostly hydrophobic and resistant to biodegradation other methods of water treatment ^{[9][10]}.

The fabrication and application of electrospun nanofiber membranes embedded with photocatalytic and antimicrobial nanomaterials have been at the forefront of research in recent times ^[11]. Polymers such as polystyrene, polysulfone, polyethersulfone, polyester and polyacrylonitrile (**Figure 1**) have often been used in the production of nanofiber membranes with desired properties for various applications via an electrospinning process or other desired methods such as polymer blending and sea/island cross-section conjugation ^{[12][13][14]}. These polymers can be electrospun on their own or co-polymerised with other polymers depending on the required application. Polymers are often coupled with other materials to produce polymer products with superior properties compared to their mono-polymer counterparts ^[14].

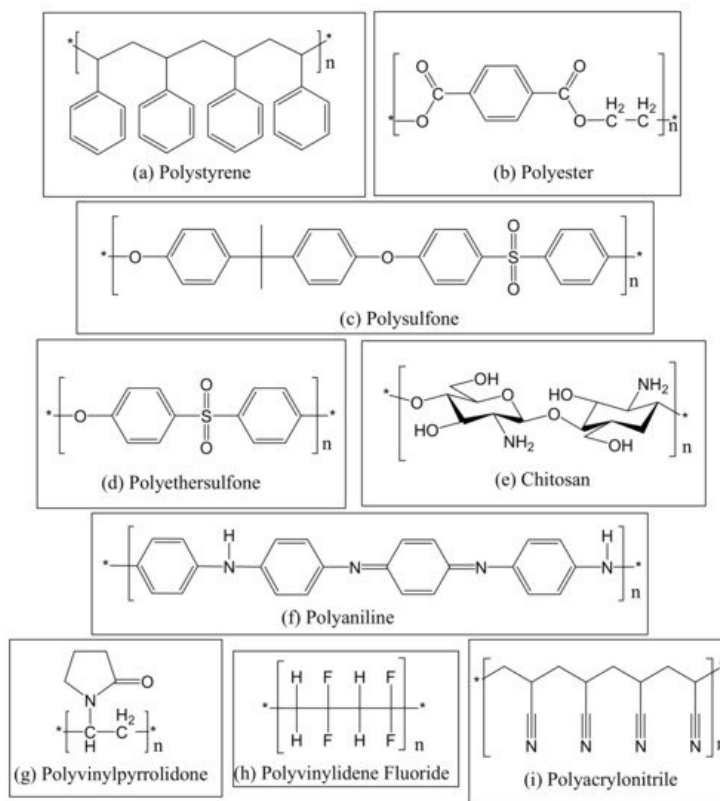


Figure 1. Chemical structures of different types of polymers that are often used in the fabrication of nanofiber membranes. The asterisk (*) indicates that the structure is continuous.

Polystyrene (PS) is one of the widely used polymers in nanofiber production. It exudes high electrical resistance and low dielectric loss. It is stiff and brittle [12], cheap, easy to handle, and displays a good balance of electrical, mechanical and chemical properties [15]. Polystyrene also finds application in heavy-duty polymer materials such as in containers and packaging of electronic goods, ion-exchange materials, membranes, sensors and filtration due to its ease of fabrication, dimensional stability and contact efficiency [16]. However, it is hydrophobic and this limits its full use in water treatment applications [17]. On the other hand, polyester (PET) is used to synthesise nanofibers, membranes and nanotubes for various applications [13]. Natural PETs have advantages such as low cost, ease of separation, low density, CO₂ sequestration, biodegradability and enhanced energy recovery compared to synthetic PET [18][19][20]. Beside nanofibers for water treatment, PET resins have been reinforced with natural fiber to make materials such as engine covers [20]. Commercially available bio-PET include poly(lactic acid) (PLA), polycaprolactone (PCL) and poly(ester amide) (PEA), among others [18].

In the class of thermoplastics, polysulfone (PSf) has been extensively studied in membrane technology and nanofiber fabrication. PSf materials have good heat-ageing resistance, high mechanical property, thermal and chemical stability [14] [21]. PSf based materials have been widely applied in food processing, biotechnology, and water treatment [14]. Polyethersulfone (PES) is another thermoplastic used in various material preparation processes as a modifier or as the main polymer. PES is a synthetic polymer that is non-degradable and biocompatible, oxidative, thermally stable, and exhibits hydrolytic stability, good film-forming and excellent mechanical properties [22]. It has found tremendous application in the fields of filtration, tissue engineering, bioreactors and haemodialysis [22][23][24].

Polyacrylonitrile (PAN) is one of the most widely used polymers for fabricating different types of membranes due to its excellent properties, which include ease of electrospinning, high solvent resistance, high mechanical strength, enhanced thermal and chemical stability, good membrane forming ability, biocompatibility and ease of modification [25][26][27][28][29]. PAN is also the predominant precursor to produce nano- to microscale carbon fibers due to its high fiber yield, high mechanical strength and elastic modulus tailoring [30][31]. The PAN polymer fibers are subjected to thermal treatment where they undergo carbonisation and graphitisation at the desired temperature, and are subsequently transformed into carbon fibers [31][32]. Other polymeric materials that are used for the fabrication of different types of membranes, including nanofiber membranes include chitosan, polyaniline, polyvinylpyrrolidone, and polyvinylidene fluoride [33][34][35][36] as shown in **Figure 1**.

Polymeric membranes are however susceptible to drawbacks such as fouling, poor flux, poor rejection, and short lifespan. As a result, efforts have been made to eliminate or reduce the occurrence of these setbacks and produce composites with superior properties. Methods of modification include additive blending, chemical treatment and surface grafting [37][38]. The

commonly practiced methods include the blending of two or more polymers, incorporation of nanoparticles and blending with photocatalysts, depending on the desired application and properties. Blending polymers and/or incorporation of nanoparticles may enhance or suppress the intrinsic properties or even add new/novel properties to the bare polymer material [39][40][41].

Figure 2 shows an example of a nanofiber membrane produced via electrospinning using fine and coarse polyacrylonitrile polymer coated with chitosan [42][43]. The nanocomposite membranes were fabricated with three layers: (I) nanofiber polyacrylonitrile coarse layer which was coated with (II) fine nanofiber polyacrylonitrile and finally with (III) chitosan [42][43]. It is demonstrated that traditional flat-sheet membranes can be coupled with nanofiber membranes to produce composite membranes with enhanced adsorption capacity, increased surface area to volume ratio, and ease of modification properties.

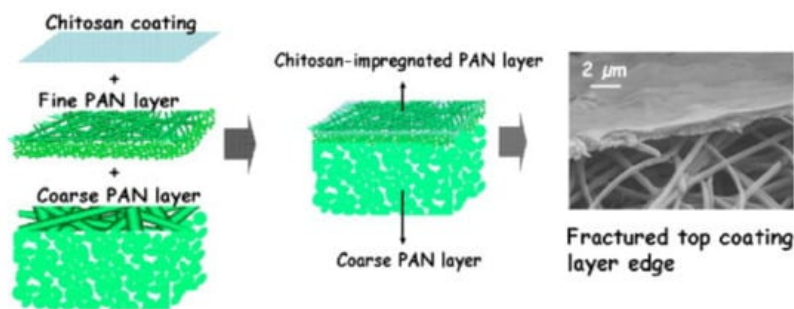


Figure 2. An example of a composite nanofiber membrane consisting of an electrospun polyacrylonitrile (PAN) layer coated with a chitosan layer. Reprinted from [42] with permission from Elsevier.

On the other hand, **Figure 3** demonstrates the electrospinning of PES nanofiber membranes infused with TiO_2 nanoparticles for simultaneous adsorption and photodegradation of water pollutants (organic dyes) as reported by Xu et al. [44]. The TiO_2 -PES nanofiber composite membrane was prepared via a combination of blending modification and electrospinning technology. Adsorption activity was reported to be via electrostatic attraction. Photodegradation studies resulted in the elimination of residual toxins completely and adsorption active sites were regenerated by continuous UV irradiation without any other treatments. Recyclability enhancement of over 95% after 5 cycles was obtained [44]. The incorporation of TiO_2 nanoparticles to the adsorption membrane introduced photocatalytic and self-cleaning properties, rendering the membrane more efficient and highly recyclable. In latter sections of this review, various other types of polymer-photocatalyst nanofiber membranes with specific examples are discussed in comprehensive detail.

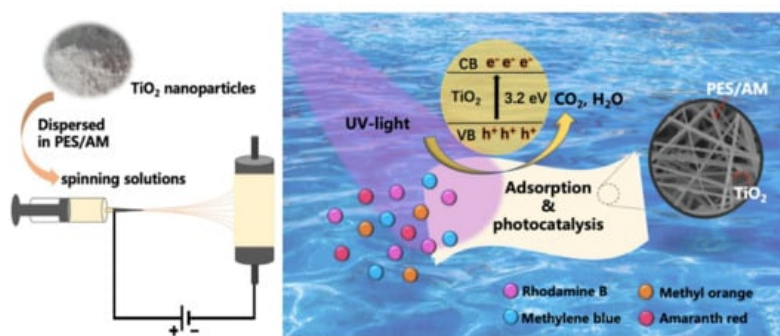


Figure 3. Fabrication of Polyethersulfone (PES)- TiO_2 nanofiber composite membrane via electrospinning as well as simultaneous adsorption and photodegradation of micropollutants. Reprinted from [44] with permission from Elsevier.

While this review is focused on nanofiber membranes infused with photocatalytic and antimicrobial nanoparticles, Nasreen et al. previously reviewed the general advancement of modification and application of electrospun nanofiber membranes in water treatment. The review emphasizes the importance of nanofiber membrane modification for enhanced efficiencies. Modifications discussed include surface modification (improved selectivity and hydrophilicity) and interfacial polymerization (improved strength, chemical/thermal stability and introduction of selective barrier layer, porous support and/or maintaining strength and configuration). The specific application of these nanofiber membranes covers the removal of heavy metals and microbes as well as desalination application [45].

2. Photocatalytic Electrospun Nanofiber Membranes

Photocatalysis is one of the most efficient treatment methods for wastewater containing different types of pollutants. Photocatalysts in their powder form produce tremendous results, often up to complete mineralisation; however, they have drawbacks such as poor recovery and secondary contamination due to leaching [46]. As a result, blending photocatalysts with support materials such as polymers and electrodes has been implemented and well-investigated [47]. Supporting or blending photocatalysts with other materials does not only enhance recovery and reduce secondary contamination but also enables recyclability, reusability and increased photocatalyst life span. In addition, hybrid processes involving photocatalysis and membrane technology demonstrate enhanced membrane properties for filtration, adsorption, and other related applications [48]. Trace organic contaminants which include pharmaceuticals and endocrine-disrupting compounds found in raw water, wastewater and sometimes in drinking water can also be effectively treated using photocatalytic membranes [49]. Such contaminants are found in low concentrations of microgram to nanogram per litre. **Figure 4** illustrates how a photocatalytic membrane operates during a water treatment process [50]. It is demonstrated that when wastewater comes into contact with the membrane, the water is filtered and clean water is collected on the other side of the membrane. At the same time, the pollutants that come with the wastewater are deposited on the surface of the membrane where they undergo photodegradation upon UV irradiation resulting in self-cleaning properties that eliminate membrane fouling.

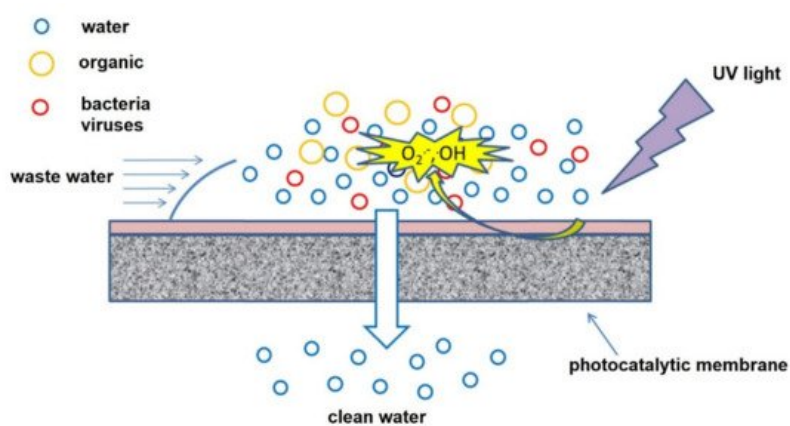


Figure 4. Schematic illustration of how a photocatalytic membrane operates with the photocatalytic layer on top degrading pollutants and membrane filtering the remaining pollutants. Reprinted from [50] with permission from Elsevier.

3. Antimicrobial Membranes

Microorganisms such as bacteria, fungi, archaea, algae, protozoa and viruses [51] form part of the water pollution system and are the cause of various types of waterborne diseases such as polio, malaria, cholera, hepatitis, diarrhea, ascariasis, malnutrition, ringworm and lymphatic filariasis, among others [52][53]. Due to the high levels of water pollution, the high ratio of water demand to water availability, as well as inefficient water treatment facilities; human beings, animals and aquatic biota have to bear the burden of waterborne diseases and infections [54][55][56].

Researchers are introducing the use of antimicrobial material in current water treatment methods. Nanocomposites are being produced with the addition of materials that have antimicrobial properties to use in hybrid water treatment processes [57]. Photocatalysis is one of the processes that has been reported to have the ability to “kill” a wide range of bacteria, viruses, algae, endospores, protozoa and fungi, and has also demonstrated the ability to inactivate prions and to destroy microbial toxins [51]. Membrane technology is another process that has such properties depending on the materials used to fabricate the membrane; however, they are well known to be prone to biofouling. Membrane biofouling is a challenging aspect to control using chemical, biological, or physical methods due to its compact nature, strong adaptive resistant microbes, and the cost of post-treatment, hence the need for modification [56]. **Figure 5** shows two different ways of fabricating a thin film composite membrane with antimicrobial properties that was used for water treatment, as reported by Zhu et al. In the first method, the thin film composite (TFC) membrane is chemically modified with Ag nanoparticles followed by coating with SBMA. In the second method, the TFC membrane is co-polymerized with SBMA followed by coating with Ag nanoparticles [57].

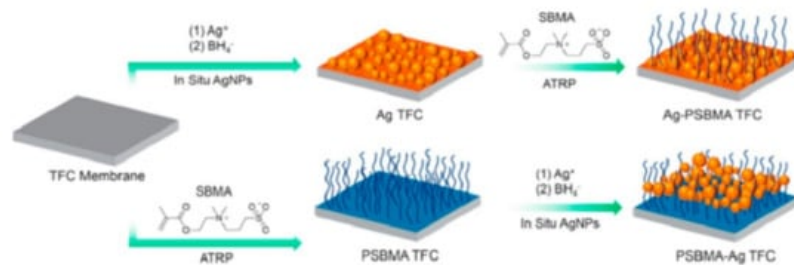


Figure 5. The fabrication process of antimicrobial TFC membranes used for water treatment. Reprinted from [57] with permission from Elsevier.

The modification of photocatalytic materials and membrane materials with Ag, Cu or graphene material enhances the antimicrobial properties of the materials which also suggests that such modified materials have self-cleaning/sterilising properties. **Figure 6** illustrates how an antimicrobial membrane with cleaning properties operates [57]. The use of photocatalytic and/or antimicrobial membranes in water treatment results in improved water quality, enhanced efficiency, and prolonged life span of the membrane by reducing/eliminating fouling [57][58][59]. The properties induced by antimicrobial agents have paved a way for the successful production of antimicrobial photocatalysts, antimicrobial membranes, and antimicrobial-photocatalytic membranes as evidenced by numerous research studies.

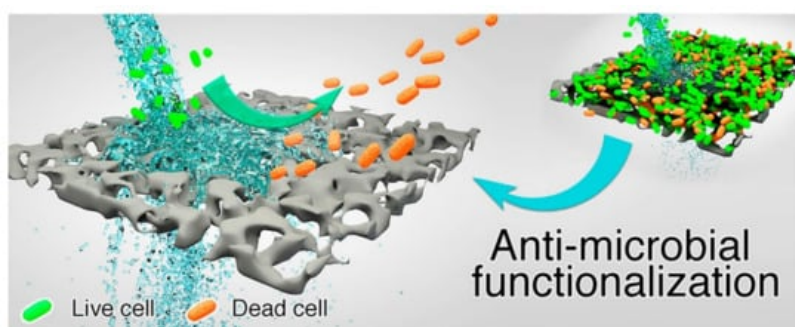


Figure 6. Illustration of antimicrobial activity on a surface of an antimicrobial membrane during water filtration. Reprinted from [57] with permission from Elsevier.

Zhang et al. fabricated thin-film composite membranes with enhanced antifouling and antimicrobial properties by the incorporation of palygorskite/TiO₂ hybrid material. Palygorskite and palygorskite/TiO₂ were embedded on a reverse osmosis polyamide membrane through interfacial polymerisation. The tubular structure of palygorskite played a role in the facilitation of water molecules through the thin-film membrane. The palygorskite-incorporated membranes had a 1.6-fold increase (up to 40 L/m² h) in water flux compared to the bare membranes. The palygorskite/TiO₂-containing membranes showed a 1.4-fold increase compared to the bare membranes. Antifouling properties were observed with increasing flux against humic acid and bovine serum albumin while antimicrobial properties were also successful against *Escherichia coli* [60]. Hee et al. studied the photocatalytic and antimicrobial activity of ZnO-incorporated electrospun nanofibrous membranes. Polyurethane nanofibers were fabricated by electrospinning followed by coating with polydopamine using the dip-coating method. For the incorporation of ZnO nanoparticles, the polydopamine-coated nanofibers were soaked in a ZnO aqueous solution followed by hydrothermal treatment to grow ZnO nanorods on the surface of the nanofibers. Characterization confirmed that the ZnO nanorods were grown and adhered to the polydopamine-coated polyurethane nanofibers as shown on **Figure 7**. The resulting material successfully degraded methylene blue within 180 min and showed positive antimicrobial properties against *Escherichia coli* [61].

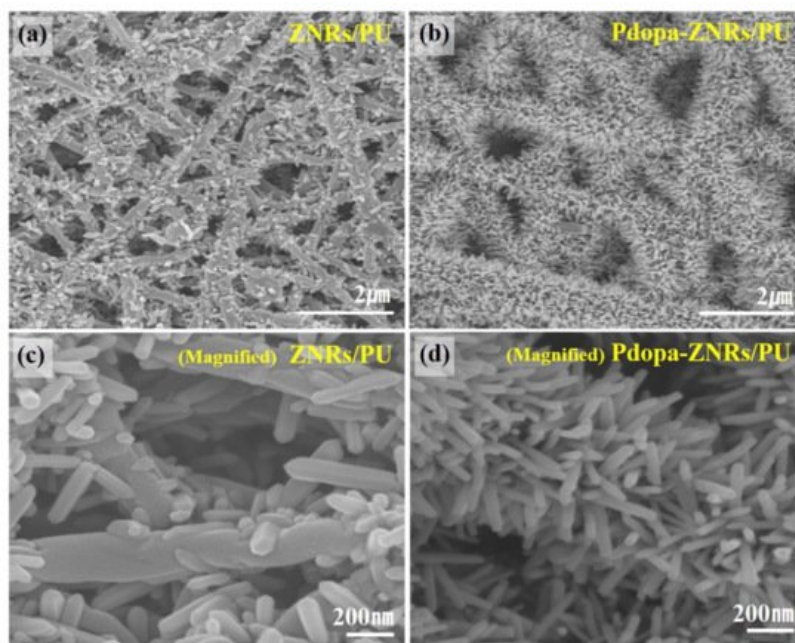


Figure 7. Field emission–scanning electron microscope (FE-SEM) images of (a,c) ZnO-nanorods/polyurethane, (b,d) Polydopamine-ZnO-nanorods/polyurethane. Reprinted from [61] with permission from Elsevier.

Panthi et al. produced a photocatalytic and antimicrobial bifunctional composite membrane immobilised with Ag_3PO_4 nanoparticles (**Figure 8**). PAN nanofibers were produced by electrospinning and then modified with amidoxime to use as anchoring sites for Ag^+ ions; AgNO_3 was used as the source of Ag. The composite was then reacted with Na_2HPO_4 to produce the final bifunctional composite membrane $\text{Ag}_3\text{PO}_4/\text{PAN}$. Antimicrobial activity was confirmed by testing against Gram-negative *Escherichia coli* and Gram-positive *Staphylococcus aureus*, respectively. The composite (150 mg) degraded up to 90% of methylene blue in a solution containing 50 mL of the dye (10 mg/L) within 60 min using a 200 W mercury lamp [62]. Xu et al. fabricated a hybrid antimicrobial nanofiltration membrane. The Ag- Cu_2O nanowires were prepared by grafting L-dopa on the surface of Cu_2O nanowires via in situ polymerisation which resulted in a zwitterionic surface suitable for the attachment of Ag^+ ions. The final Ag- Cu_2O -PSF composite was fabricated using the in-situ phase inversion method. Antimicrobial studies against *Escherichia coli* and *Staphylococcus aureus* using the composite revealed enhanced antimicrobial activity compared to that of the bare PSF membrane. Bovine serum albumin was used for protein rejection studies whereby the modified and pure PSF membranes rejected up to 94.70 and 86.81%, respectively. The modified membrane also achieved higher water flux (up to $164.1 \text{ L/m}^2\cdot\text{h}$) compared to the bare membrane ($40.4 \text{ L/m}^2\cdot\text{h}$). The modified membranes also demonstrated better flux recovery than the pure PSF membrane [63].

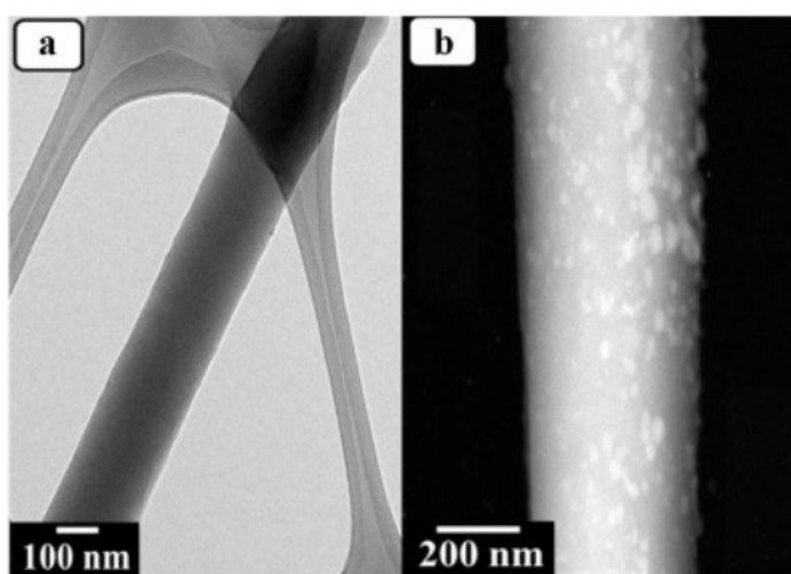


Figure 8. Transmission electron microscopy (TEM) images of (a) polyacrylonitrile (PAN) and (b) $\text{Ag}_3\text{PO}_4/\text{PAN}$ composite membranes used for photodegradation and antimicrobial studies by Panthi et al. Reprinted from [62] with permission from Elsevier.

Damodar et al. studied the self-cleaning, antibacterial, and photocatalytic properties of polyvinylidene fluoride (PVDF) membranes embedded with TiO₂ (**Figure 9**). The PVDF/TiO₂ membranes were prepared via the phase inversion method where the TiO₂ loading was varied from 0–4%. Generally, PVDF/TiO₂ membranes showed higher antimicrobial properties compared to the pristine PVDF membrane with 4% TiO₂ having the highest activity against *Escherichia coli*. Over 95% degradation of Reactive Black 5 was achieved within 60 min using the 2% PVDF/TiO₂ membrane while the pristine membrane showed no photocatalytic activity. The PVDF/TiO₂ membranes showed good antifouling and self-cleaning properties under UV irradiation with increased water flux and excellent flux recovery compared to pristine PVDF membranes. The photodegradation and self-cleaning processes on the membrane are as demonstrated in **Figure 9** [64].

Jalvo et al. studied the antimicrobial and anti-biofilm efficacy of a TiO₂ coated glass surface with self-cleaning properties. The material was prepared by coating a glass slide with the TiO₂ suspension. The coated glass slide showed cell reduction viability of over 99% during antimicrobial studies against biofilm-forming bacteria *Staphylococcus aureus* and *Pseudomonas putida*. Self-cleaning properties were tested against the degradation of adsorbed methylene blue. The material achieved 85% degradation confirming that the material has both self-cleaning properties and anti-biofilm efficacy added to their photocatalytic and antimicrobial properties [65].

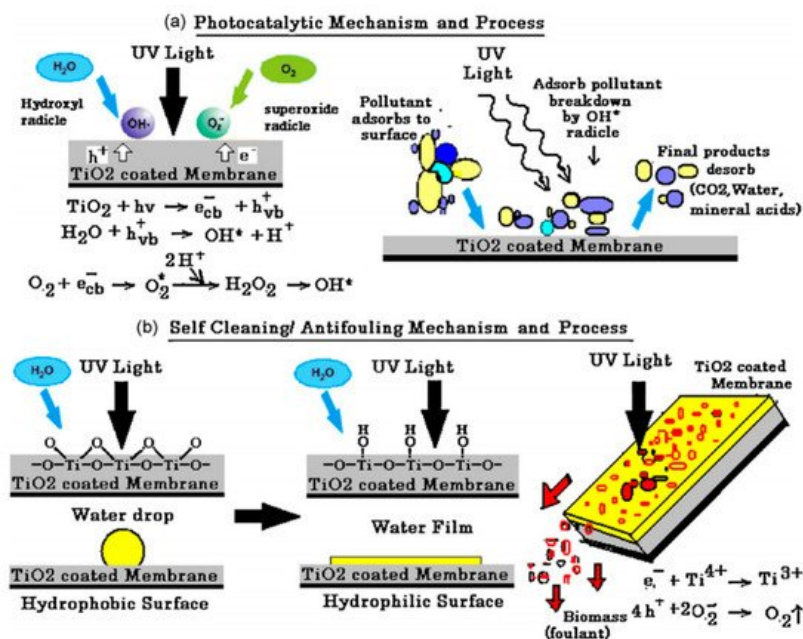


Figure 9. Demonstration of the (a) photocatalysis mechanism and process as well as (b) Self-cleaning/antifouling mechanism and process of polyvinylidene fluoride (PVDF)/TiO₂ membrane. Reprinted from [64] with permission from Elsevier.

Zhang et al. prepared a chitosan-based antimicrobial film against foodborne pathogens to use in food packaging under visible light. The material was prepared by coating a plastic film-covered glass plate with chitosan-TiO₂ emulsion crosslinked by epichlorohydrin, followed by drying naturally overnight. The obtained composite film was tested against *Escherichia coli*, *Staphylococcus aureus*, *Candida albicans*, and *Aspergillus niger*, and achieved 100% sterilisation within 12 h. Positive results were also obtained in terms of the prevention of microbial growth in packaged red grapes with an extended life span as shown in **Figure 10** [66].

Bodaghi et al. studied the photocatalytic antimicrobial effects of TiO₂ coated packaging film by conducting in vivo and in vitro tests. The TiO₂-polyethylene film was prepared using the melt blending method whereby modified TiO₂ powder, polyethylene granules, and glycerol were mixed and blended for an hour. The resulting nanocomposite film was used for all studies. Under in vitro studies, the film reduced the number of surviving cells for *Pseudomonas* spp. and *Rhodotorula mucilaginosa* by 4 and 2 logs compared to 1.35 and 0.64 log reduction of polyethylene, respectively. In vivo studies were conducted on packaged fresh pears under fluorescent light irradiation for 17 days where a significant decrease in mesophilic bacteria and yeast cells was observed for TiO₂-polyethylene [67].



Figure 10. Preservation of red grape packed in different materials at 37 °C for 6 days: (a) plastic wrap; (b) pure chitosan film; (c) chitosan-TiO₂ film. Reprinted from [66] with permission from Elsevier.

Antimicrobial activity is one of the most important properties in membrane technology. As indicated, antimicrobial activity prevents microbial membrane fouling which prolongs the lifespan of the membrane [68]. **Table 1** shows some of the nanomaterials with antimicrobial activity that can be blended with polymer nanofibers for various applications. Silver nanoparticles are well documented for their excellent antimicrobial activity and it is no surprise that the table shows that pristine and doped-Ag nanoparticles are mostly used for the preparation of antimicrobial nanofiber composite membranes [62][69][70][71][72][73][74][75]. **Table 1** further shows that there are other antimicrobial materials such as ZnO, CuO, poly(hexamethylene biguanide) hydrochloride, octadecyldimethyl[3-(trimethoxysilyl)propyl]ammonium chloride, Fe₃O₄-COOH, nisin, and metronidazole which can be further explored to reduce the demand of Ag nanoparticles for antimicrobial applications [61][76][77][78][79][80]. The application section of **Table 1** indicates that the application of antimicrobial membranes is not limited to water treatment but extends to areas such as cytotoxicity [81][82], drug release [76][79][82], and wound dressing [72][78][81][83][84].

Table 1. Antimicrobial nanofiber membranes and their various applications.

Polymer	Antimicrobial Agent	Method	Application	Antimicrobial Activity	Ref.
Polyurethane	Polydopamine-ZnO	Electrospinning	Antimicrobial (<i>E. coli</i>) Photodegradation (Methylene blue)	Active	[61]
Polyacrylonitrile	Ag ₃ PO ₄	Electrospinning	Antimicrobial (<i>E. coli</i> and <i>S. aureus</i>) Photodegradation (Methylene blue)	Active	[62]
Polyacrylonitrile	Ag nanoparticles	Electrospinning	Antimicrobial (<i>E. coli</i> and <i>S. aureus</i>)	Active	[69]
Polyacrylonitrile	Ag nanoparticles	Electrospinning	Antimicrobial (<i>E. coli</i> and <i>S. aureus</i>)	Active	[70]
Polyacrylonitrile	Ag nanoparticles	Electrospinning	Antimicrobial (<i>E. coli</i> and <i>S. aureus</i>) Forward osmosis	Active	[71]
Chitosan	Ag nanoparticles	Centrifugal spinning	Antimicrobial (<i>S. aureus</i>) Wound healing	Active	[72]

Polymer	Antimicrobial Agent	Method	Application	Antimicrobial Activity	Ref.
Polysulfone	CNT/Ag	Radical solution polymerization and wet-phase inversion	Antimicrobial (<i>E. coli</i> and <i>B. subtilis</i>)	Active	[73]
3D woven fabric filters	Ag nanoparticles	Electrospinning	Antimicrobial (<i>S. aureus</i>) Water treatment	Active	[74]
Polyvinyl alcohol	Polyimide-Ag	Electrospinning	Antimicrobial (<i>E. coli</i> and <i>S. aureus</i>) Oily wastewater treatment	Active	[75]
Polyacrylonitrile	CuO	Electrospinning	Antimicrobial (<i>E. coli</i> and <i>B. subtilis</i>) for breath masks Drug release	Active	[76]
Chitosan/poly(ethylene oxide)	Poly(hexamethylene biguanide) hydrochloride	Electrospinning	Antimicrobial (<i>E. coli</i> and <i>S. aureus</i>)	Active	[77]
Poly(ϵ -caprolactone) and gelatine	Octadecyldimethyl[3 - (trimethoxysilyl)propyl]ammonium chloride	Electrospinning	Antimicrobial (<i>S. aureus</i> and <i>P. aeruginosa</i>) Wound dressing	Active	[78]
Polylactic acid	Fe ₃ O ₄ -COOH	Electrospinning	Antimicrobial (<i>E. coli</i> and <i>S. aureus</i>) Drug delivery	Active	[79]
Triaxial	Nisin	Electrospinning	Antimicrobial (<i>S. aureus</i>)	Active	[80]
Cellulose acetate/polyester urethane	Polyhexamethylene biguanide	Electrospinning	Antimicrobial (<i>E. coli</i>) Cytotoxicity Wound healing	Active	[81]

Polymer	Antimicrobial Agent	Method	Application	Antimicrobial Activity	Ref.
Polycaprolactone/gelatine	Metronidazole	Electrospinning	Antimicrobial (<i>F. nucleatum</i>) Cytotoxicity (L929 Cells) Drug delivery	Active	[82]
Silk fibroin	Peptide motif	Electrospinning	Antimicrobial (<i>S. aureus</i> , <i>E. coli</i> , <i>S. epidermidis</i> and <i>P. aeruginosa</i>) Wound dressing	Active	[83]

4. Other Applications of Hybrid Photocatalytic Membrane Processes

Literature reports show that electrospun nanofiber membranes find application in a wide range of processes within and outside water treatment. It is worth noting that the applicability and efficiency of nanofiber membranes is highly dependent on polymer material intrinsic properties [11]. Therefore, it is imperative to study the properties of the nanofiber membranes prior to application to establish if the membrane is suitable for that specific application. Kaur et al. reviewed the various types of characterization techniques crucial for membranes analysis. These characterization techniques include: atomic force microscopy (surface roughness), scanning electron microscopy (morphology and cross-sectional internal structure), Fourier transform infrared spectroscopy (surface chemistry such as functional groups and bonding nature), tensile test (tensile/mechanical strength and durability), differential scanning calorimetry (thermal properties and rigidity), heat treatment and hot pressing (compactness, integrity and strength), contact angle (hydrophilicity and hydrophobicity) as well as Brunauer, Emmett and Teller (surface area, pore size and pore volume). The review also discusses the liquid intrusion-extrusion techniques for materials to be applied in aqueous solutions [84]. All these techniques form part of the basic analysis of membrane materials and are conventionally used. Other processes whereby photocatalytic and antimicrobial electrospun nanofiber membranes are used include filtration, adsorption and electrocatalysis.

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