Surface Engineering of Nanofiber Membranes via Electrospinning-Embedded Nanoparticles for Wastewater Treatment

Jagdeep Singh, Sourbh Thakur, Rakesh Sehgal, A. S. Dhaliwal, and Vijay Kumar

Abstract Nanofibers (NFs) are fibers with diameters in the nanometer range and have found numerous applications due to their unique properties. Researchers are still trying to improve the properties of electrospun-based fibers by using unique nanomaterials for solving environmental problems especially the treatment of wastewater. The modification of NFs has been carried out by decorating and embedding the various types of nanoparticles, such as noble metals, carbon nanomaterials, and metal oxide nanoparticles onto the surface of the membrane. The decorated surface of the NFs membrane possesses high surface area, surface energy, additional functionality, and anti-fouling properties that make them a suitable candidate for wastewater treatment application. This chapter highlights the modern trends in the surface engineering of NFs via electrospinning embedded nanoparticles (NPs) for wastewater treatment. The shape and size of Ag and Au NPs prepared under different reducing and stabilizing agents are also reviewed. The electrospun polymer NFs embedded with different NPs and surface modifications of NF membranes are discussed. The critical issues related to the use of electrospun polymer NFs embedded with different NPs for wastewater treatment along with a concluding note on possible future directions on this have also been included.

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1 Introduction

Water is an elixir of life and it is important to mankind for drinking as well as for many other daily activities and we cannot sustain our life without it. Due to the rapid growth of the global population, modernization, and industrialization, there is tremendous depletion in the quality and quantity of water. Out of total water available on the earth, only 2% is fresh and potable, another 98% is saline. Moreover, out of the small portion of freshwater, only 0.77% of water is easily accessible because a major part of the world's population has limited access to pure drinking water. Wastewater or environmental pollution is mostly initiated from household products, industrial wastage and medical waste, municipals, which contaminate the water. The various pollutants from different origins, such as heavy metal ions, synthetic dyes, and other organic and inorganic materials caused a serious deterioration in the water quality [1–3]. This contaminated/unhealthy water causes various perilous diseases among people. Hence, to lessen its drawbacks for the protection of the environment, the technology to retrieve water from wastewater is one of the major concerns for the scientific community. A number of techniques (chemical, physical, and biological) such as chemical precipitation [4], adsorption [5], sonochemical [6], and electrochemical degradation [7] are available to tackle such types of pollutants and retrieve water from wastewater efficiently. The various advantages and disadvantages of these technologies are presented in Table 1. These methods provide a slow rate of purification, require accessories and expensive equipment, and therefore encourage the scientific community to go for an alternative method. Due to simplicity, high efficiency, and cost-effectiveness, membrane filtration technology has been demonstrated to be an effective and viable technique for the exclusion of several pollutants [8–10].

In the modern era, nanotechnology, an emerging science; has fascinated scientists due to its novel size-dependent properties [14, 15]. Researchers are trying to improve the quality of current technology by inventing unique materials. Nanofiber is one of the great inventions for its unique characteristics. Nanofibers can be considered as two separate words "nano" and "fibers". Historically, "nano" is used to describe the reference unit, nanometer, and the word fiber is defined as a slender, elongated, thread-like object or structure. Polymer nanofiber is the 1D nanomaterials having a highly porous structure and large surface-to-volume ratio. Polymeric nanofibers are better to use these days because of their certain properties like enormous specific surface area, high tensile strength, huge stiffness, comprehensive flexibility, sustainability, and unique dynamic mechanical, electrical and thermal properties [16, 17]. Polymeric nanofibers have found applications in the field of decontamination, catalysis, aerospace, fuel cell, solar energy, filtration, superabsorbents, energy storage, or as scaffolds for tissue engineering and wound dressings [18–22]. A lot of methods

Table 1 Various techniques for the wastewater treatment with advantages and disadvantages

Techniques	Methods	Advantages	Disadvantages	References
Chemical techniques	Chemical precipitation Neutralization Redox method Electrolytic Coagulation	Simple technology Economic Very efficient for metal and fluoride elimination	Chemical consumption Physiochemical monitoring of the effluent (pH) Unproductive in exclusion of the metal ions at low concentration High cost Cause secondary pollutants	[11]
Physical techniques	Ion exchange Air flotation Membrane separation Adsorption	Stable treatment methods, Simple operation and management, Wide variety of target contaminants Highly effective process (adsorption) with fast kinetics	Expensive Non-destructive processes Non-elective methods, Performance depends upon the type of material Rapid saturation and clogging of the reactors Not effective with certain types of dyestuffs and some metals	[12]
Biological techniques	Bioreactors Biological activated sludge Biofilm methods Anaerobic biological treatment Natural biological treatment Enzymatic decomposition	Efficiently eliminates Biodegradable organic matter, Microorganism degrade organic pollutants into harmless substances Low cost and stable effect	Essential to create a feasible environment Needs organization and care of the microorganism and physicochemical parameters Slow process, little biodegradability of certain molecules, Poor decolorization	[13]

are available in the literature for the production of nanofibers such as melt fibrillation [23], template synthesis [24], island-in-sea [25], magneto spinning [26], and gas jet techniques [27]. Electrospinning is the most embraced technique to produce polymer nanofibers. It has a low cost, a large selection of materials, strong versatility, and simple modification methods [28]. The concept of nanotechnology has led to a new membrane at the nanoscale with enhanced performance standards and with new functionality, such as large surface area and surface energy, high permeability, catalytic reactivity, and fouling resistance. Nanostructure membranes or mats based on nanofibers are amazing materials that could be synthesized via in-situ and ex-situ incorporation of nanoparticles/nanofiller into the polymer. The embedding and decoration of these nanoparticles/nanofiller add new functionalities to the resulting nanocomposite as well as change the physical, chemical, optical and electronic properties of the solution. This chapter highlights all the strategies investigated by the various researchers in the surface engineering of nanofiber membranes via electrospinning embedded nanoparticles as nanofiller such as noble metal (Ag and Au), TiO_2, SiO_2, carbon nanotubes, graphene, and graphene oxide. The change in the inherent properties of the polymer by the incorporation of embedded nanoparticles as nanofiller is also explored. Furthermore, the utilization of the nanofibers membrane for wastewater treatment is also explored. The various techniques and methods to minimize the problem of membrane fouling are also discussed in detail.

2 Basics of Electrospinning

The electrospinning (ES) phenomenon was reported for the first time by an astronomer, William Gilbert. In the 1960s, Sir Geoffrey Taylor analyzed the phenomenon comprehensively, studied mathematical aspects, and afterward, he put forward the modeled cone of fluid formed by an electric field. This cone has a certain specific shape now known as "Taylor cone" [29, 30]. He detected that when brought near an electric field, the drop of water turned into conical in shape and the leftover water of this drop get ejected in the form of smaller droplets. ES is a multipurpose technique to produce nanofibers via an electrically charged jet of the polymer solution. This process has four important aspects; a direct current power supply with high voltage (10–50 kV), a metallic needle having a blunt tip, a grounded conductive collector, and a syringe that holds polymer solution, as demonstrated in Fig. 1. First, the polymeric material whose nanofiber one wants to develop is dissolved and taken in a syringe as displayed in Fig. 1.

The polymeric solution contained in the syringe will be impelled out through the metallic tip at a controlled feeding rate, and there is the formation of polymer droplet at the tip of the pipette. The applied high potential creates a high electric field, so, there is the production of charges on the surface of polymer droplet and the Taylor cone is also formed. When the repulsive force is enough to chock the surface tension of polymer droplets then the polymer droplets get extended and its conical shape is obtained. Hence, the nanofiber or mat is formed on the grounded conductive collector.

The advantages of this technique are that by changing the experimental parameters such as polymer concentration, needle diameter, molecular weight, solution conductivity, applied voltage, solution viscosity, air humidity, flow rate, and the working distance between the tip of the needle and the collector, the surface morphology of the electrospun nanofibers can be easily controlled [31–34]. The different distances

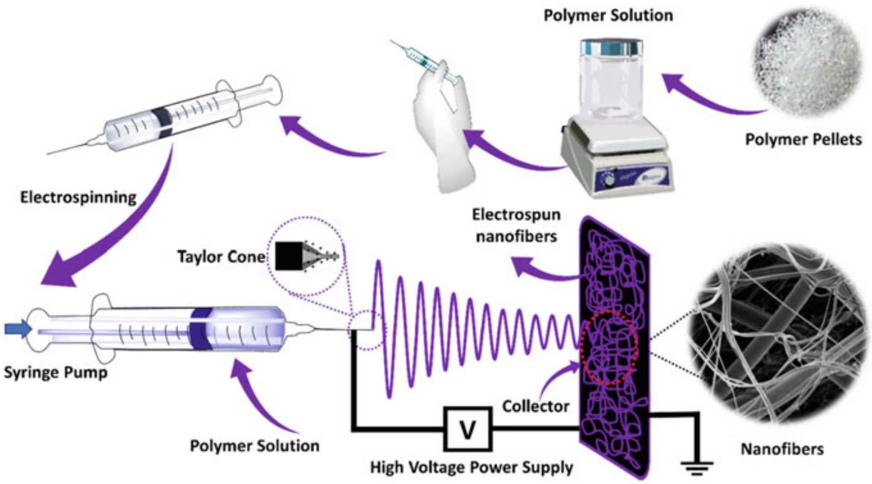

Fig. 1 The graphical representation of electrospinning process

among the needle and collector corresponding to the varying morphology of fibers are produced by different deposition times and evaporation rates. It was shown that the fiber diameter reduced as the distance increased [35]. The implemented voltage is another vital factor in the electrospinning techniques, which ends in the production of nanofibers. Sufficient voltage is required to overcome the surface tension of the polymer solution. Usually, 10–50 kV DC voltage is applied in this method. The decrease in the Taylor cone and an increase in jet speed at the same feed rate are obtained by increasing the applied voltage. These effects result in a fall in the fiber diameter and a rise in evaporation of the solvent [36]. The concentration of polymer solution additionally determines the morphology of nanofibers, breaking of polymer chains into pieces brought about by low weight concentration of the polymeric solution, which, in turn, was due to applied high voltage and surface tension of the solution. It prompts the development of beads or chain-type fibers. Alternatively, an increasing weight concentration of the polymeric solution tends to rise in the viscosity and resulted in an increase in the chain structure of fibers [37]. Flow and feed rate also regulate the surface morphology and pore size of nanofibers. Various studies represent that with the increase in the flow rate, the fiber diameter and pore size also rise up [38]. However, a flow rate above the critical value leads to the formation of fibers that lack the ability to dry. Moreover, the conductivity of polymer solution also affects the quality of nanofiber, an increment in the conductivity of the solution, charge on the surface of droplet additionally expands, which produces Taylor cone yet the fiber breadth diminishes [39]. Also, the selection of polymer prompts diverse webs of nanofibers. The determination of solvents for the electrospinning process is another significant reason for acquiring smooth and beadles electrospun NF [40]. The polymer ought to be totally soluble in a solution that has a reasonable boiling point for simpler evaporation like distilled water. The molecular weight

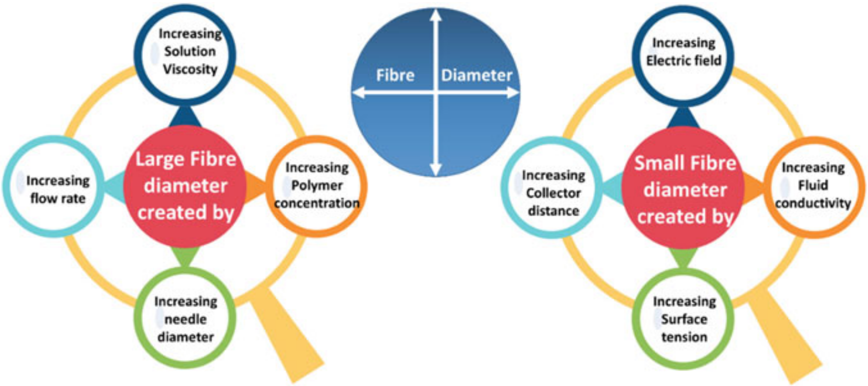

Fig. 2 Effect of various synthesis parameters in the electrospinning process

of the selected polymer has a significant influence on properties like conductivity, viscosity, or surface tension [41]. Low molecular weight polymer will, in general, form globules rather than fibers, and the expanding value of the molecular weight of polymer yields fibers with bigger diameters. The rise in temperature leads to falling in fiber diameter and high humidity, which causes pores on the surface of the fibers. Figure 2 sums up the impact of different ES limits on the NF formation, structure, and morphology.

3 Synthesis of Au or Ag Nanoparticles

During the last decades, the synthesis of Ag and Au NPs has been carried out with different methods for getting different shapes and sizes. Surface plasmon resonance (SPR) is the most important optical property of metallic NPs, which comprises collective oscillations of conduction electrons excited by the EM wave. This property is directly related to shape, size, and the adjacent medium. The nanostructure having different shapes and morphologies possesses different electronic, optical, chemical, and magnetic properties, which may be appropriate for various applications. The reductive procedure can be categorized into physical and chemical methods. Initially, silver/gold salts and capping compounds need to be dissolved in solvents. Then, the solution needs to be reduced with reducing agents (sodium borohydride, sodium citrate, alcohols, and poly (vinyl pyrrolidone)) to get stable colloidal NPs with different sizes and shapes. After the completion of the reaction, these precipitates can be collected through centrifugation. Several materials have been employed for the synthesis and stabilization of the silver and gold nanoparticles (Table 2).

Table 2 Shape and size of Ag and Au nanoparticles prepared under different reducing and stabilizing agents

Precursor	Reducing agent	Stabilizing agent	Shape/size (nm)	References
$AgNO_3$	Trisodium citrate	Trisodium citrate	Spherical/30–60	[42]
$AgNO_3$	$NaBH_4$	Dodecanoic acid (DDA)	Spherical/7	[43]
$AgNO_3$	Ethylene glycol	–	Nanowires/diameters of 30–40 nm	[44]
$AgNO_3$	Ascorbic acid	–	Nanowires/diameters of 30–40 nm	[45]
$AgNO_3$	Ethylene glycol monoalkyl ethers	PVP	Nanoprisms	[46]
$HAuCl_4$	$NaBH_4$	Citrate	Spherical/13 nm	[47]
$HAuCl_4$	CTAB	Ascorbic acid	Nanoflower/45 nm	[48]
$HAuCl_4$	Chitosan	–	Spherical/10–15 nm	[49]
$HAuCl_4$	Tannic acid	–	9 nm	[50]

Ag and Au NPs have different properties such as higher surface area, surface energy, SPR, and stability, which make them versatile materials for many applications in plasmonics, biomedical research, sensing, and catalysis, etc. NFs show distinct properties for example extremely high surface-to-weight ratio, low density, highly porous structure, and tight pore size for various advanced applications. The combination of Au or Ag NPs with polymer NFs has abundant potential to improve its properties. The addition of Au and Ag NPs with nanofiber can change its surface morphology and increase the surface area and energy. This addition and decoration of Ag and Au NPs into the NF can be done in two different methods, ex-situ and in-situ. The systematic representation of in-situ and ex-situ methods for the synthesis of NFs is displayed in Fig. 3. In the ex-situ, i.e., the first method, Ag and Au NPs with unlike morphologies are synthesized separately and the prepared NPs can be added into polymer precursor solution. In the second method, stabilized NPs are added to the polymeric solution and there is no need to add any precursor and stabilizing agent. Moreover, the in-situ method comprises the presence of precursors in the polymeric solution. For this, the silver/gold salt precursor is dissolved into the polymeric solution with a reducing and stabilizing agent. The reduction process transfers the Ag/Au ions into Ag and Au NPs. Normally, to complete the reduction of Ag/Au ions into Ag/Au NPs, self-reducing polymers such as chitosan and PVP are used here. The biomolecules present in these polymers are the cause of the reduction of ions. Moreover, other methods such as heating, UV irradiation, microwave irradiation, gamma irradiation are also employed for the reduction of ions into NPs [51–53].

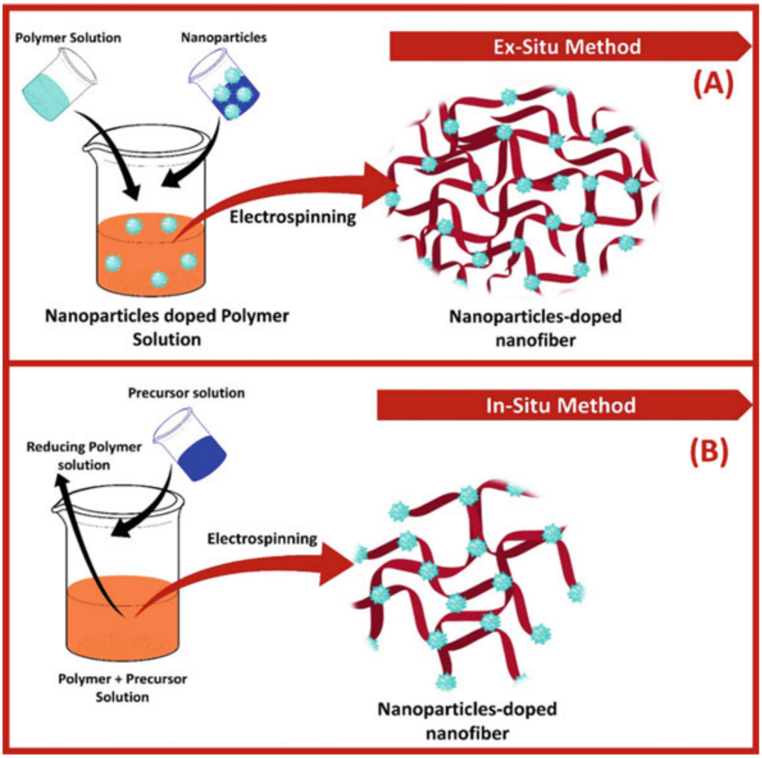

Fig. 3 The systematic representation of in-situ and ex-situ methods

4 Electrospun Polymer Nanofibers Embedded with Different Materials

4.1 Electrospun Polymer Nanofibers Embedded with Ag and Au Nanoparticles

The electrospun polymer embedded in noble metal nanoparticles or decorated with it could be synthesized using the above-stated in-situ and ex-situ methods. In the ex-situ method, the metal NPs are synthesized using a reducing and stabilizing agent and then dispersed into the polymer precursor solution [54]. Jin et al. [55] reported a one-step method to synthesize Ag NPs in a poly(vinylpyrrolidone) matrix. Wang et al. [56] carried out the synthesis of polyvinyl pyrrolidone/ silver nanocomposite by PVP ethanol solution without any reducing agent. Ethanol is used as a solvent for electrospinning and it reduces the silver ions. The characterization performed in this study approves that the average diameter of the nanofiber has been about 80 nm with the average diameter of the silver nanoparticles of about 8 nm. Aadil et al. [57]

synthesized poly (vinyl alcohol)-lignin nanofiber mats loaded with Ag NPs. The in-situ synthesis of Ag NPs has been carried out using alkali lignin extracted from Acacia wood as a reducing agent and then the synthesis of ultrafine nanofiber mats of Acacia lignin combined with Ag NPs is done by using the electrospinning technique. The size of nanofiber synthesized using electrospinning is in the range of 100–1000 nm and this size does not fall in the nano range. Hence, different designs in electrospinning are formed through phase separation splitting by exposure to the high electric field. In this method, nanonets are formed, which is also called a spider web or spider net form. Pant et al. [58] synthesized Ag NP-embedded polyurethane nanofiber/nanonet structured membrane. The uniform distribution of Ag nanoparticles on the surface of PU nanofiber is achieved by the in-situ method. Figure 4 shows the FE-SEM and TEM micrographs of synthesized nanofiber and from it is clear that the presence of spiderweb-like nanonet structure is accompanied by the main fiber, which offers a large surface area.

Nguyen et al. [59] fabricated Ag NPs loaded nanowire mat based on PVA using the electrospinning method. The synthesis has been optimized by changing the microwave irradiation time and electro-spinning parameters. The shape and size are manipulated by altering the microwave energy. By increasing the irradiation time, the size of particles is optimized for suitable applications. The mechanical strength

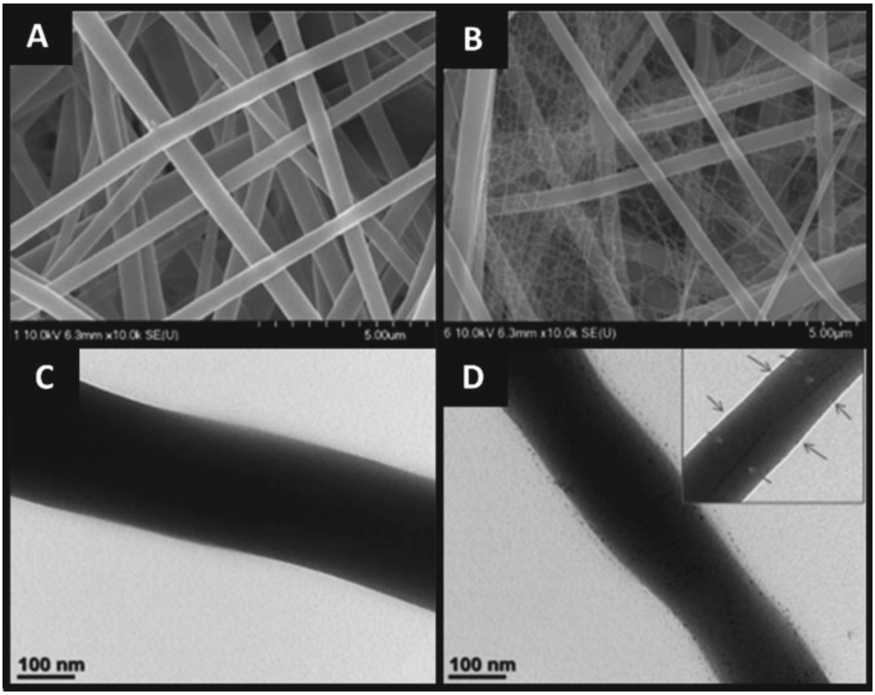

Fig. 4 FE-SEM images of pristine PU NFs (**A**) and Ag/PU composite nanofibers. **B**–**D** are their respective Bio-TEM images. The inset in **D** shows the Ag NPs embedded in the nanofiber [58]

is also examined by the stress–strain curve of the PVA nanofibrous mat and after the introduction of Ag NPs. The result shows that the strength of the pure PVA fibrous mat is improved by the loading of Ag NPs. Hence, the nanocomposite fibrous mats were more brittle and stronger than the pure PVA fibrous mat. Dong and co-worker [60] fabricated Ag porous films by the heat treatment of $AgNO_3$ doped poly(vinyl alcohol) (PVA) electrospun nanofibers with smooth and super-long nanofibers with an average diameter of about 500 nm. Moreover, after 2 h of heat treatment at 600 °C in air, the morphologies are completely degraded and the porous film is formed having a pore size of about microns to 10 mm. Tijing et al. [61] conducted the in-situ synthesis of Ag NPs inside the polymer. For this, $AgNO_3$ precursor is dissolved in the polymer solution and afterward electrospinning, the synthesized mat is exposed to UV light to synthesize the Ag NPs. Various other authors have also reported the production of Ag NP-embedded nanofiber membrane by electrospinning method [62–67].

Similar to the above-mentioned approaches to embedding the NPs into a polymer matrix, various researchers reported several nanofibers embedded with Au NPs. Au NPs are synthesized using various methods and then dispersed into the polymer precursor solution [68–76]. In order to functionalize or decorate the nanoparticles on the surface of nanofiber, different techniques such as dipping, spraying, and layer-by-layer deposition are used [77–79]. By these methods, different NPs are located on the surface of the polymer rather than inside the polymer matrix. For this, strong bonding between NPs and polymer surfaces is required. Otherwise, the NPs released from the surface of the polymer and can cause secondary pollution. Another method to functionalize nanofibers is the coaxial electrospinning technique. In this technique, two concentric nozzles are used instead of a single nozzle. The inner nozzle provides the polymeric solution while the outer nozzle functionalizes the outer surface by spraying the NPs on it. Hence using this method, nanoparticles decorated nanofiber can be easily fabricated simultaneously. Various researchers have developed a nanofiber decorated with NPs [80–85]. Yu et al. [86] studied the development of polyacrylonitrile (PAN) nanofibers coated with Ag NPs with a modified coaxial electrospinning process. The result confirms the uniform distribution of Ag NPs on the surface of PAN nanofibers. Wang et al. [87] decorated Au NPs on the surface of PVA electrospun nanofiber. The synthesized nanofiber of PVA possesses sulfur as a functional group through it and the Au NPs are attached to the surface by gold–sulfur bonding interactions. Figure 5, shows the steps involved in the procedure to decorate the nanoparticles on the surface of PVA. First, PVA electrospun fibrous is prepared by in situ cross-linking and dipped into the acidic solution to stimulate the cross-linking between hydroxyl groups of the PVA and the aldehyde groups of GA. The MPTES (3-mercaptopropyltrimethoxysilane) introduces the thiol groups on the PVA/GA mat. Therefore, Au NPs are homogenously immobilized on the mat through an Au NPs–sulfur bonding interaction. Figure 6 displays a graphic demonstration of the hydrogen peroxide sensor based on the horseradish peroxidase/Au NPs−PVA/glassy carbon electrode.

Dong et al. [88] reported the decoration of Ag, Au, and Pt NPs on the surface of nylon 6 fiber. Ag, Au, and Pt NPs are synthesized by means of sodium citrate and

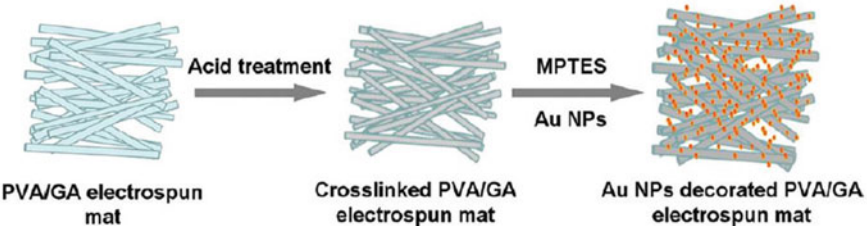

Fig. 5 Schematic fabrication of water-stable functional Au NPs–PVA/GA electrospinning nanofibrous mats. Reprinted with permission from [87]. Copyright (2012) American Chemical Society

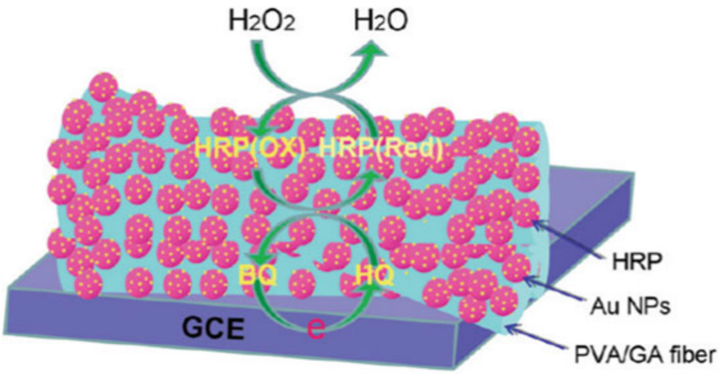

Fig. 6 Schematic presentation of the hydrogen peroxide biosensor. Reprinted with permission from [87]. Copyright (2012) American Chemical Society

nylon 6 nanofiber mats, formed by ES. The decoration of metal nanoparticles is determined by the hydrogen bond interactions between the amide groups in the nylon 6 backbone and the –COOH groups capped on the surface of the metal NPs. Son et al. [89] fabricated assemble noble metal nanostructures by means of electrospun catechol-rafted PVA nanofibers. The –OH groups of PVA can be used to functionalize its surfaces. The synthesized PVA-g-catechol NFs have been employed as a chemically reactive template for the reduction of metal ions into solid metal nanostructures. The SEM and elemental mapping confirm the uniform distribution of Ag, Au, and Pt NPs on the surface of PVA-g-catechol nanofibers.

4.2 Electrospun Polymer Nanofibers Embedded with Carbon Nanoparticles

The synthesis and characterization of electrospun nanofiber embedded or decorated with carbon nanoparticles have received significant attention from many researchers. Carbon nanotubes (CNTs) possess remarkable thermal, mechanical, and electrical properties, and combining them inside the polymer NF has shown improved properties with relatively low percolation thresholds. The most difficult challenge in the path to developing nanofiber embedded with carbon nanomaterial is to prevent bundling and agglomeration. This problem is explored by synthesis via electrospinning method, by uniform dispersion of CNT onto the surface of the nanofiber. Chen et al. [90] synthesized high strength polyimide nanofiber membrane containing multiwall carbon nanotubes (MWNTs) by using the electrospinning method. The thermal and mechanical properties of the polyimide matrix have been expressively enhanced with the addition of MWNTs. Xiao et al. [91] also synthesized mechanical strong nanofiber by mixing multiwalled carbon nanotubes (MWCNTs) with PAA/PVA polymer solution. The MW-CNTs amalgamated PAA/PVA nanofibers are first cross-linked and then employed as a reducing reactor to reduce Fe(III) ions into zero-valent iron nanoparticles. Weng et al. [92] functionalized MW-CNTs and strengthened the poly(methyl methacrylate) (PMMA) nanofiber mats with average diameters range of 370–800 nm. Moreover, the electrical and mechanical properties of the synthesized NF mat are better as compared with native PMMA nanofibers.

4.3 Electrospun Polymer Nanofibers Embedded with Metal Oxide Nanoparticles

Metal oxide NPs played a vital role in the formation of new materials for a range of applications. The metal oxide NPs have a large surface area, which is required in various advanced applications. A large number of synthesis methods are reported for the synthesis of metal oxide NPs such as co-precipitation [93], thermal decomposition [94], hydrothermal [95], sol–gel method [96], ultrasound irradiation [97], and biological method [98]. Co-precipitation and thermal decomposition methods are the most important methods for the synthesis of metal oxide NPs [99]. Various metal oxide NPs embedded/decorated in NFs such as ZrOlasma/wet chemistry, coating, nanofibers/activated carbon composite [100], polyacrylonitrile nanofiber webs comprising titanium dioxide (TiO_2) [101], cellulose acetate/silica composite nanofibrous membrane [102], TiO_2 NFs doped-activated carbon [103], polyethylene terephthalate (PET) with CuO nanoparticles [104], metal oxide/silica NFs [105], metal oxide-coated polymer NFs [106], Fe_2O_3 doped on ZnO NFs [107], ultrafine metal oxide-decorated hybrid carbon NFs [108] are also available in the literature. Therefore, various nanofibers embedded with noble metal (Ag, Au, and Pt), carbon nanomaterial (CNTs and MW-CNTs), and metal oxide nanoparticles (TiO_2, Fe_2O_3, CuO, etc.) are available in the literature and have been used as a promising material for various advanced applications including wastewater treatment. This review highlights the various electrospun nanofibers. Various researchers fabricated the electrospun nanofibers embedded with nanoparticles. But still, more research work is needed to develop modified nanofibers. Hence, the various modification techniques to develop nanofibrous membranes with better properties are also explored in the next section. Furthermore, the applications of various electrospun nanofibers membrane in the field of wastewater treatment are described in the next sections.

5 Surface Modification of Nanofiber Membranes

The surface modification is employed to obtain enough performance for a particular application. The surface modification has been carried out either chemically or physically by varying the atoms/molecules in the existing surface, changing the surface morphology, or coating over the surface with materials. The physical modification includes methods such as coating, adsorption, and blending. Moreover, the chemical techniques, for example, the introduction of functional groups by plasma treatment or wet-chemistry reactions, or yet grafted polymers covalently bonded to the surface and layer by layer electrostatic interaction. The surface modification techniques are divided into two categories: (i) post-treatment technique and (ii) one-step treatment during ES. In post-treatment methods, the synthesized membrane has been treated after ES using plasma/wet chemistry, coating, and grafting method. Moreover, in one-step treatment, the NPs are added to the precursor before the synthesis. The embedding of NPs to the polymer solution before ES gives them significant properties. The embedded NPs offer a large surface area, surface energy and help to increase the surface roughness of the fibers and thus amplify the wettability of electrospun nanocomposites. The improved properties of electrospun membranes with various types of NPs are studied by various authors [109–115]. There are many other studies related to the improvement of the properties of nanofibrous membranes, reported by various authors, available in the literature [116–119]. In these studies, researchers have tried to increase the mechanical properties as well as to increase inter-fiber adhesion. Membrane fouling is an exceptionally complex phenomenon that has linked with the accumulation of unwanted materials/particles such as colloids, macromolecules, and salts on the surface of the membrane or inside the membrane pores. Various types of fouling such as organic fouling, inorganic fouling or scaling, colloidal fouling, and biofouling have been accommodated on the membrane surface as shown in Fig. 7.

With the purpose to cut the tissue of membrane fouling, several techniques are invented in the last few decades. The surface modification by the decoration of NPs onto the membrane surface and by the coating of the hydrophilic polymer layer is one of the suitable methods to prevent the problem of fouling. But the coating method involves high cost, complexity, and pollutant production. Hence, the embedding and decoration of nanoparticles onto the surface of the membrane is an emerging technique, and several investigations on the preparation of mixed matrix membranes (MMMs) with nanofiller are available in the literature. The nanoparticles based on

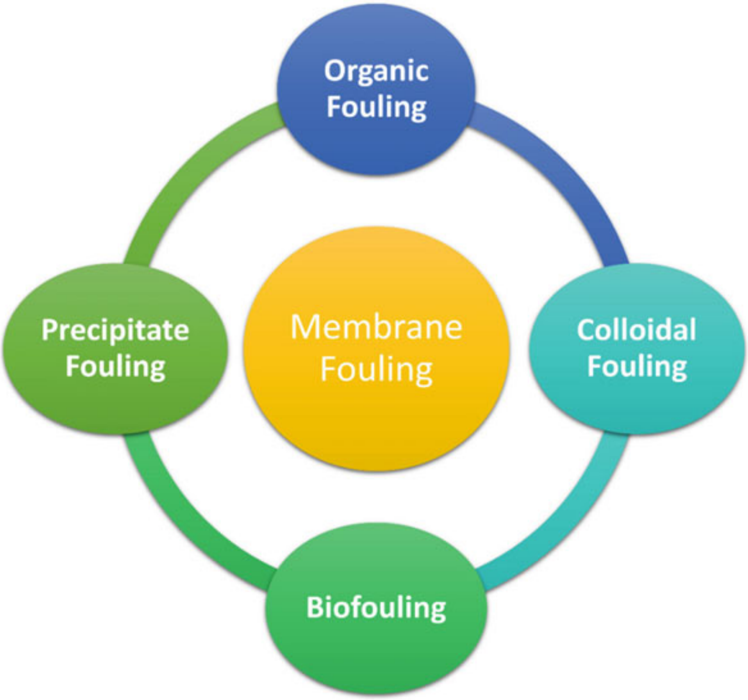

Fig. 7 Various types of fouling accommodated on the membrane surface

noble metals, carbon, and metal oxides have also overcome the fouling problems. The decorated and embedded nanoparticles into the nanofiber membrane can increase the hydrophilicity, contact angle, surface roughness, or imposing bactericidal agents and charge on the surface of the membrane, which may cut the membrane biofouling.

6 Applications of Nanofiber Membranes for Wastewater Treatment

Membrane technology has become the most significant method for the purification of wastewater. This technique is utilized for the elimination of suspended solids, microorganisms, organic, inorganic pollutants, and heavy metal ions dissolved in the aqueous solution [120]. Several membranes processes based on the different types of membranes such as microfiltration (MF), ultrafiltration (UF), nanofiltration (NF), and reverse osmosis (RO) are available in the literature for the treatment of water and desalination and Table 3 shows the properties and purposes of these membrane types and the capability of the membranes for the elimination of certain pollutants from the wastewater as described in Fig. 8 [121, 122].

Table 3 Various membranes techniques with all specifications

Membrane type	Pore sizes (nm)	Material passed	Material retained	References
Nanofiltration (NF)	0.1–1	Water, sugar, and monovalent ion	Solutes, MW > 500, di- and multivalent ion	[123, 124]
Reverse osmosis (RO)	Not relevant	Water	All dissolved and suspended solute	[125]
Microfiltration (MF)	>50	Water, salt, and macromolecules	Particles, bacterial, yeast, etc	[126]
Ultrafiltration (UF)	1–10	Water, salt, and sugar	Macromolecules, colloids, lattices, and solute >10,000	[127]
Forward osmosis (FO)	Not relevant	Water, salt, and sugar	Organics, minerals, and other solids	[128]

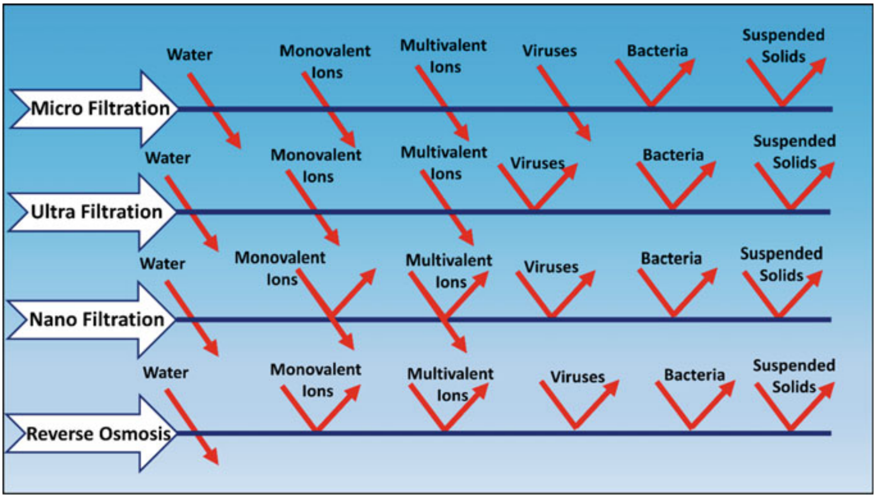

Fig. 8 Capability of the various membranes for the removal of certain pollutant from wastewater

6.1 *Nanofibers in Microfiltration*

The utilization of electrospun nanofibrous membrane has been taken place for wastewater treatment at the microfiltration level. For this, cellulose nanofiber membranes with a diameter ranging between 200 nm and 1 µm have been synthesized and treated in sodium hydroxide solution in H_2O/C_2H_5OH to get regenerated cellulose NF mesh. The filtration result confirms the reduction in pressure drop and an increment in flux as compared with the viable microfiltration membranes and after regeneration using elution *buffer*, it presented reusability [129]. Gopal et al. [130] designed an electrospun nanofibrous membrane, i.e., polysulfone to eliminate microparticles from the solution. The synthesized membrane was capable of removing 99% of 10, 8, and 7 μm particles. In another study, the formation of an electrospun polyvinylidene fluoride membrane having a fiber diameter of about 380 nm has been done. This membrane is used to isolate 1, 5, and 10 μm polystyrene particles. The results point out the rejection of about a maximum of 90% particles from feed solutions to the membranes to decrease the possibility of fouling [131]. Kaur et al. [132] fabricated an electrospun membrane of polysulfone and polyvinylidene fluoride with a high angle of contact by using diverse surface transforming macromolecules. The angle of contact of the blended EM gets reduced from 140° to 54°. Moreover, in comparison to non-blended EM, the water flux is increased to 20% in the case of blended EM. Moradi and Zinadini [133] reported a large value of flux and anti-fouling graphene oxide (GO) NP set in PAN nanofiber microfiltration membranes. The incorporation of GO NPs into the PAN polymeric matrix pointedly amplified the permeation flux of the resulting membrane in both cross-flow and dead-end filtration systems. Moreover, the anti-fouling ability and high flux make them a good membrane for water treatment. Various other reports are available in the literature that developed membrane for micro-filtration purposes [134–136]. The results conclude that the electrospun nanofibers play a significant role in microfiltration due to the superior physical features with high filtration efficiency.

6.2 Nanofibers in Ultrafiltration

Ultrafiltration is another important method due to the low fouling effect and high flux for the filtration of proteins, emulsions, and virus colloids of the size range of about 1–100 nm. Wang et al. [137] synthesized the electrospun nanofibrous based on poly (vinyl alcohol) (PVA). It is found that the nanofiber exhibited high molecular weight and mechanical performance. The recorded flux rate (130 L/m^2) is significantly higher as compared with the commercial UF membranes. Yoon and Lee [138] reported membrane based on polyacrylonitrile coated with the thin top layer of chitosan. The average diameter of the synthesized membrane ranges from 124 to 720 nm. It also exhibits a higher flux rate and filtration efficiency as compared with the commercial filtration system. Dobosz et al. [127] reported a high-porous nanofiber based on cellulose and polysulfone with improved flux and fouling resistance. The fouling resistance is improved by about 90% as compared with the control membranes. This is possibly due to the decrease in the contact time. Polyaniline/polysulfone membranes have been fabricated and the performance of filtration has been tasted using cross-flow equipment by taking PEG aqueous solution, water, and bovine serum albium as feed solution. The results revealed that the synthesized membrane has higher hydrophilicity and permeability. Moreover, the antifouling characteristics of PANI/PS membranes displayed a better performance than PS substrate during the filtration [139].

Reverse osmosis (RO) is an important technique for the production of freshwater by distillation of seawater using a membrane having pore sizes of 0.1–1 nm [140]. Tian et al. [141] and Bui et al. [142] reported electrospun nanofiber as forward osmosis membrane based on polyethersulfone (PES) and polysulfone (PSf). The MPD and TMC monomers have been utilized to formulate an active barrier layer onto the fibrous supporting layer. High water flux and low salt flux are the major possessions of the membrane.

6.3 Nanofiltration

Nanofiltration techniques have been extensively applied to effectively remove heavy metals, dyes, and other impurities present in wastewater due to its high efficiency, low cost, small pore sizes, and ease [143, 144]. Ritcharoen et al. [145] reported membrane for nanofiltration based on cellulose acetate fiber mat coated with chitosan/sodium alginate and poly(styrene sulfonate). Yoon et al. [137] reported polyacrylonitrile support with high flux thin film nanofibrous composite (TFNC) membranes in the nanofiltration process. The active layer onto the membrane has been produced by IP of trimesoylchloride, piperazine, and some additives and utilized in the nanofiltration of divalent salts ($MgSO_4$). The TFNC membrane has shown higher rejection rates (2–22%) as well as higher permeate fluxes (21–42%) than those of TFC membranes. It is also directed that the concentration of piperazine has played an important part in controlled flux and rejection capacity. The effect of nanofibers structure and film composition in the nanofiltration process is examined by Kaur et al. [132]. In this study, they explored the impact of fiber diameters on filtration and study the separation performance with the help of dead-end filtration cells of 2000 ppm concentration of $MgSO_4$ as feed solution. The consequences revealed that with a reduction in the diameter of the fiber, the pore size also decreased, which amplified flux with high salt rejection. The results revealed that nanofiltration with an increment in the applied hot pressure membrane showed lower flux and higher rejection than those of TFNC membranes [146]. Another study related to the improvement in the performance of membrane based on PET nonwoven by spun used the DMF and DMF/NMP mixture onto the membrane as a support layer. The supporting layer upsurges the adhesion between the forming PET nonwoven scaffold and PES nanofibrous layer. The produced membrane showed almost double flux compared with that of NF-90 with an equal salt rejection ratio [147]. Nanostructure membrane can also be modified using the nanomaterials such as noble metal oxide NPs, carbon nanomaterials, and metal NPs. Nanotechnology provides advanced performance nanofibers material with amazing properties such as enhanced catalytic reactivity, fouling resistance, high permeability, and water treatment [114]. The high efficiency, low capital need, quality of treated water, and effective disinfection are other features of this technique [123]. These modified membranes have been used for removing various particles, bacteria, and viruses, natural organic matter, water hardness metals, organic and inorganic substances. The detailed discussion of the nanofiber membrane decorated or embedded with different nanomaterials for wastewater treatment is discussed in the following sections.

6.4 Electrospun Noble Metal (Ag, Au, and Pt) Nanofiber Composites for Wastewater Treatment

Nanoparticles based on noble metals, i.e., Ag, Au, Pt, and Pd, are essential for modern nanotechnology because of their outstanding optical, physical, and chemical properties [148, 149]. The modification of nanofibers by decorating onto the surface or by embedding inside it is important for improving its properties. Taurozzi et al. [150] synthesized a membrane based on polysulfone with incorporated Ag particles in order to decrease biofouling. The availability of the Ag NPs implanted in the membranes has been quantitatively evaluated by the extent of growth inhibition of biofilm because of ionic Ag released by the nanocomposites. Gunawan et al. [151] fabricated a novel membrane using Ag/MWNTs coated on PAN hollow fiber membrane for biofouling control and water disinfection. Ag NPs of optimized sizes have been deposited on the surface of polyethylene glycol-grafted MWNTs. The Ag/MWNTs layer coated on the outside surface acts as a disinfection barrier. The nonstop filtration test against E. coli confirms the much-boosted antifouling properties and antimicrobial activities of the membrane against E. coli. Most of the E. coli cells were observed on the composite membrane with dented cell walls. This is caused by the direct contact of the Ag NPs and cells. Furthermore, Ag NPs amalgamated into PSf ultrafiltration membranes possess antimicrobial properties toward a large number of bacteria, comprising Escherichia coli, Pseudomonas mendocina, and bacteriophage. Interestingly, the utilization of nanoparticles with various polymeric phases yields similar properties [152]. Dasari et al. [153] also revealed that the existence of Ag NPs in NFs leads in lesser fouling of the NFs. Sui et al. [154] deposited the Ag NPs onto the PAA/PVA/PW12 fibrous by layer-by-layer (LBL) self-assembly technique for the degradation of MB solution. Also, the effects of charge, the number, and components of the LBL film on the photodegradation of MB dye have been optimized. It is found that the negatively charged surface has higher activity as compared with the positively charged surface. The resultant NF has excellent photocatalytic activity, stability, and recyclability. Pant et al. [155] fabricated Ag-impregnated TiO_2/nylon-6 nanocomposite mats with good photocatalytic and antibacterial properties. TiO_2 NPs present in the nylon-6 solution are able to form a spider-wave-like structure while ES enabled the UV photoreduction of $AgNO_3$ to Ag. The antibacterial and photocatalytic activity of the resultant NF mat revealed that the mats containing NPs are more effective as compared with the mats without Ag NPs. Ag NP decorated cellulose nanofibrous membrane has decent antibacterial functions and separation efficiency is a promising candidate for drinking water as reported by Chen and

Peng [156]. In another study, a nano-enabled membrane was fabricated from two components, i.e., a silver nanoparticle, poly (4-vinyl-N-hexylpyridinium bromide) and silver bromide embedded nanoparticles. The cationic polymer/silver bromide nanoparticle composite membranes showed potent long-lasting antibacterial activity toward gram-positive and gram-negative bacteria [157]. Lv et al. [158] synthesized Ag NP-decorated porous ceramic complex for water treatment. The synthesized composite can be kept for long durations and it is enduring under washing, even with ultrasonic irradiation, without losing NPs from the surface of the membrane with strong sterilization and antibacterial property. Moreover, the complete reduction of 4-nitrophenol has been carried out by using nanomembrane made up of alumina and polymers by LBL adsorption of citrate-stabilized Au NPs and polyelectrolytes [159].

6.5 Electrospun Carbon Nanofiber (CNF) Composites for Wastewater Treatment

The first carbon nanofiber (CNF) reported by Thomas Edison was contrived by bamboo and carbonizing cotton and used as a filament of an electric bulb in 1879 [160, 161]. The fabrication of carbon nanofibers is done by electrospinning method by the use of the precursors of the CNFs. By changing the kind of polymer solution and processing parameters, the properties of synthesized CNFs are regulated. PAN is often employed in the synthesis of electrospun CNF materials [162]. Moreover, polybenzimidazole (PBI), PVA, poly (vinylidene fluoride) (PVDF) polyimides (PIs), lignin, and phenolic resin have also been used. CNFs membranes have been used for the elimination of many organic pollutants such as organic dyes [163, 164], organic solvents [164, 165], and volatile organic compounds [166, 167]. The potential material based on carbon is electrospun CNTs, which have been utilized for the treatment of wastewater due to their properties [168, 169]. Singh et al. [170] reported unmodified electrospun CNF for the exclusion of the disinfection by-products from water by regulating carbonization temperatures during synthesis to <500 °C. The extent of graphitization of the polymeric precursor may be prevented by low carbonization temperatures. Thus, sorption capacity is decreased. Moreover, unmodified CNTs are relatively weak (brittle material). Hence modification of CNFs for this application is required. In order to improve the material flexibility, silica (SiO_2) nanoparticles have been embedded in the CNFs matrix or by the introduction of macrospores via insertion of detachable parts in the precursor solution [171–173]. It is seen that the composite membrane is harder than the pristine CNF membrane when the embedded SiO_2 concentration is retained at 2.7wt.%, beyond which the membrane toughness decreases [171]. The modified nanofiber embedded with Fe_2O_3, SiO_2, $CaCO_3$, ZnCl, tetra ethoxy orthosilicate [173, 174] nanoparticles based on the polymer such as poly(methyl methacrylate) [175], terephthalic acid, and poly(styrene-co-acrylonitrile) [176] are used for the removal of organic dyes, oil [151, 177, 178].

CNTs are widely used 1D nanomaterials because of the unique mechanical, magnetic, and electrical properties of CNTs [179]. Moreover, the high surface area and high adsorption ability make CNTs very good candidates [180]. By considering the properties of CNTs, several researchers have studied the influence of embedding CNTs within the nanofibers [90, 92]. The embedded CNTs inside nanofibers can improve their mechanical strength, electrical and thermal properties [181, 182]. Ji et al. [183] fabricated PAN NFs with enhanced mechanical properties by embedding the MWCNTs inside them. Srivastava et al. [184] reported nanocomposites incorporating aligned CNT walls. The study showed applications for the membrane to eliminate heavy hydrocarbons from petroleum and to filter the bacterial contaminants such as Escherichia coli or poliovirus of nanometer-size (25 nm) from water. In vitro testing has shown substantial antimicrobial activity of carbon nanotube composite films against Staphylococcus warneri, Staphylococcus aureus, and Staphylococcus warneri colonization. Xiao et al. [91] fabricated MWCNT-reinforced electrospun polymer NFs comprising zero-valent iron NPs for environmental remediation. The synthesized nanofibrous mat shows the outstanding proficiency for decolorization of model dyes such as methyl blue, acid fuchsine, and acridine orange with a more than 90% decolorization percentage. Moreover, the synthesized nanofibrous mat is found to be effective for the degradation of trichloroethylene with a degradation efficiency is about 93%. Singh et al. [170] testified the confiscation of carcinogens disinfection by-products (DBPs) present in water by using MWCNTs assimilated in the carbonized nanofibrous membranes (CNMs). The adsorption capacity for chloroform and monochloroacetic acid is about 554 mg/g and between 287 and 504 mg/g, respectively. Moreover, Salipira et al. [185] have also reported that cyclodextrin polyurethanes comprising of CNTs is effective for the removal of trichloroethylene as compared with the granulated activated carbon because of its higher surface area. Wang et al. [186] reported a novel GO-based nanofiltration membrane on a highly porous GO@PAN mat for water treatment application. A high rejection performance is shown by the membrane (nearly 100% rejection of Congo red, and 56.7% for Na_2SO_4).

6.6 Electrospun Metal Oxide Polymer Composites for Wastewater Treatment

Various types of metal oxide nanoparticles such as ferric oxide, titanium oxides, manganese oxides, magnesium oxides, cerium oxides, and aluminum oxide-embedded nanofibers are used as a promising material for the removal of various pollutants from wastewater because of its nontoxic nature, high photosensitivity, and large bandgap [187–194]. Mu et al. [195] fabricated one-dimensional ZnO-carbon nanofiber (CNF) heteroarchitectures via ES for the degradation of Rhodamine B. They reported that the synthesized nanofiber has higher photocatalytic activities as compared with the pure ZnO. Furthermore, it could be easily recycled without the reduction of photocatalytic activity because of its one-dimensional nanostructure property. PA6@FexOy nanofibrous membrane has been synthesized via electrospinning technique combined with a hydrothermal strategy for the removal of chromium Cr (VI) ions from wastewater [196] with an adsorption capacity of 150 mg Cr/g nanofibrous membrane. Furthermore, the Freundlich adsorption isotherm indicates the multi-layer adsorption onto the surface of the nanofiber membrane. Wang et al. [197] prepared a novel PAN/TiO_2 electrospun system for the degradation of dyes. The removal of the dye occurs by the interaction of methylene blue with SiO_2 in montmorillonite. The rate of adsorption upsurges with an increase in montmorillonite, and the rate of degradation increases with an increment in spin-coating layers of TiO_2. Liu et al. [198] prepared polyaniline (PANI)-coated TiO_2/SiO_2 nanofiber membranes by a combination of ES, calcination, and in situ polymerization with enhanced visible light photocatalytic degradation activity. The figure depicts the time-dependent UV–vis spectra of the MO (methyl orange) solution. It is clearly indicating that the efficient degradation of MO dye has taken place. Furthermore, the digital photos (inset figure) also visually confirm the degradation of MO dye. Joo Kim et al. [199] also testified multifunctional TiO_2-fly ash/polyurethane nanocomposite membrane for the effective removal of heavy metals (Hg, Pb) as well as the removal of MB dyes with good antibacterial activity and enhanced water flux.

Moreover, various researchers modified the silica with amino or thiol groups earlier to ES via hydrolysis poly-condensation. Then the functionalized nanofibers have been used for the removal of Cr(III) or Cu, respectively [200, 201]. Similarly, Taha et al. reported the amino-functionalized cellulose acetate/silica composite for the effective removal of Cr(VI). Here, the silica component served both to support the surface functional groups and to improve material stability [102]. Dastbaz and Keshtkar [202] verified an alternate approach to the co-electrospinning of silica by integrating SiO_2 nanoparticles surface-functionalized with aminopropyltriethoxysilane (APTES) to merge amine functional groups into electrospun PAN. The loading and functionalization of nanoparticles inside the nanofiber have been optimized for improving the adsorption capacity for Cd^{2+} , U^{6+} , Ni^{2+} , and Th^{2+} ions from water. Also, the regeneration study confirms that the nanofiber could be used for industrial applications repetitively without any notable decline in its adsorption capacity. Teng et al. [203] reported mesoporous polyvinyl alcohol (PVA)/SiO_2 composite nanofiber membrane functionalized with cyclodextrin groups for water purification purposes. The produced nanofiber membranes have a decent performance in adsorption of indigo carmine dye with the most adsorption capacity is about 495 mg/g. Moreover, the membranes have worthy recycling properties for practical usage. Hu et al. [204] reported thermally stable and flexible CuO nanocrystal-decorated SiO_2 nanofibers for the removal of Rhodamine B. The nitrogen adsorption–desorption isotherm has been carried out for surface properties and results confirm that both the samples, SiO_2 and SC fibers, exhibited a type-IV isotherm, and total pore volume and the specific surface area of the SiO_2 fibers are recorded to be 0.012 cc g^{-1} and 12.02 m^2 g^{-1}, respectively. While the SC (0.25) nanofiber is extra porous and its total pore volume and specific surface area increased to 0.073 cc g^{-1} and 55.59 m^2 g^{-1}, respectively. Taha et al. [102]

reported novel NH_2-functionalized cellulose acetate (CA)/silica composite nanofibrous membranes for the removal of Cr (VI) ions with maximum adsorption capacity is about 19.46 mg/g. Afkhami et al. [205] reported sodium dodecyl sulfate (SDS)-coated nano-alumina with polyvinylidene fluoride membrane for effective removal of metal cations Cd(II), Pb(II), Co(II), Cr(III), Mn(II), and Ni(II) from wastewater samples. The consequences showed that the composite membrane had high adsorption capacity for Cr(III), Cd(II), and Pb(II) and in mixed ion systems. Desorption experimentations by elution of the adsorbent with a mixture of menthol and nitric acid showed that the modified alumina NPs could be used again without significant property losses even after three adsorption–desorption cycles. Thus, modified nano-alumina with SDS is favorable and useful for the exclusion of these metal ions. The high adsorption capacity makes it a promising candidate material for Cr(III), Pb(II), and Cd(II) removal. Furthermore, the ES NFs with active Fe(II) and Fe(III) as nanofillers inside the PAN and PVA have been synthesized for the effective removal of Cr(VI) and Ar ions from wastewater [206, 207]. The main disadvantage of these NFs is the leaching of iron salts from the nanofibers over time. Thus, the NPs are a promising method to make the NF with an active site due to its durability. In this view, Patel et al. modified PAN composites with surface-deposited iron oxide NPs. The PAN nanofiber-embedded iron oxide nanoparticles possessed about half the capacity for the removal of Congo Red dye [208]. Xiao et al. [91] fabricated electrospun NFs embedded with zerovalent iron NPs for potential environmental applications. The NF mat containing iron NPs has a porous structure and capable of quick decolorization of an organic dye (acid fuchsine) with percentage degradation is about 95.8% within 40 min. Moreover, a resultant NF mat has been employed for the remediation of many other contaminants such as PCB, TCE, and toxic metal ions (e.g., arsenic). The resultant NF possesses high efficiency for the removal of Rhodamine B dye and Cu^{2+} ions from the aqueous solution [209]. Horzum et al. [210] synthesized chitosan fiber-supported zero-valent iron nanoparticles for the removal of inorganic arsenic from aqueous solutions. Zhu et al. [211] synthesized membrane based on chitosan with magnetic NPs (γ-Fe_2O_3). The SEM and TEM studies revealed that synthesized membranes have numerous pores and folds on the surface, which give active sites for dye entrapment. This membrane exhibited good adsorption ability and adsorbed up to 70% methyl orange at pH6.

7 Conclusions and Outlook

Electrospun nanofibrous membranes have many interesting and controllable properties that provide good separation efficiencies when it is used as filtration and adsorption membrane. Adsorption membranes have applications in the removal of various organic/inorganic and heavy metal ions from wastewater, while filtration membranes can be used for the separation of various bacteria, viruses, and oil–water emulsion. Due to the advancement of technology, the nanofibrous membranes can be easily modified by researchers in terms of precision for using the electrospinning method and efficiency for the removal of various pollutants. Amazing nanomaterials and surface modification techniques have been realized for developing novel nanofibrous membranes with high surface area, porous structure, high tensile strength, huge stiffness, comprehensive flexibility, and sustainability. The surface modification also reduces its fouling nature and overcomes the various limitations while used as a device for wastewater treatment application. In this review, an attempt has been made to review different polymeric electrospun nanofibrous membranes utilized in water purification applications. However, the surface modification of electrospun nanofibrous membranes by using pre and post-treatment techniques by embedding and decorating various nanostructures based on noble metals (Ag, Au, and Pt), carbon nanostructures (CNT, GO, MWCNT), and metal oxide nanoparticles onto the native membranes are also explored. The utilization of various modified nanofibrous membranes for the removal of different pollutants from wastewater is also highlighted in this chapter. It may be concluded that some details about various nanofibrous membranes with good removal efficiencies are available in the literature. However, systematic research work is required for the development of nanofibrous membranes with improved properties for wastewater treatment on an industrial scale with recyclability and reusability.

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