

Carbon-Based Membranes for Desalination

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1 Introduction

Global water shortages across all continents have led to an explosive practice of desalination. Currently, thermal-based desalination is the most frequently applied technology in the Middle East. However, due to some inevitable restrictions of thermal distillation desalination technologies particularly in terms of their high power consumption, membrane-based desalination has been addressed as the key and mainstream desalination technology. Currently, desalination using reverse osmosis (RO) represents the desalination core process, which holds promise for constant growth in the future primarily owing to its lower energy consumption and process maturity. However, the trade-off relationship between water flux and rejection has restricted the further improvement of separation performance of commercial thin-film composite polyamide RO membrane. Therefore, the strategy to simultaneously elevate the permeability and salt rejection is becoming an attractive hot spot of the membrane field. Another main obstacle for the sustainable use of commercial RO desalination is membrane fouling, which reduces the efficiency of the RO processes and greatly increases their cost. Typically, membrane organic fouling is resulted from the adsorption and deposition of natural organic matter (NOM) on the membrane surface or into the pores. This in turn, leads to pore plugging phenomena as well as the formation of a thick cake layer on the membrane surface.

Different types of materials and modifying approaches have been explored for the fabrication of RO membranes so that the morphology and structure of the membrane can be modified to improve the separation performance in terms of permeability and selectivity as well as their applicability in respect to robustness as well as chemical and biological stabilities. Recently, the interplay between nanotechnology and membrane technology has prompted the revolutionary advances in RO desalination. The advancements made in this field have provided feasible solutions that are capable of solving many longstanding critical issues that correspond to conventional RO membranes. These nanomaterials possess desired structures as well as

surface charges and functional groups to enhance hydrophilicity to the resultant nanocomposite membranes, and hence facilitate greatly improved water flux. The highly permeable membrane is an essential factor in membrane design as it is crucial to reduce the energy consumption where the operating pressure for the RO processes can be lowered. Besides that, only smaller membrane area is needed to provide high water productivity. As such, the material cost, as well as the carbon footprint, can be reasonably reduced. Another issue that challenges the lifespan of the conventional membranes is the fouling phenomenon. Foulants such as organic substances and microorganisms have been well recognized as the main issues that deteriorate the durability of the polymeric membranes, which eventually imparts higher maintenance cost to replace the membranes as well as the chemical expenses to clean the membranes periodically. Most of the time, due to the seawater conditions, biological and organic fouling of the membrane are very prone to occur and have more influence on RO systems' efficiency. Hence, the presence of nanomaterials that are bestowed with inherent antifouling properties towards these fouls is deemed to enhance the sustainability of the membranes.

Over the past few decades, significant advances have been proposed for the fabrication of RO membranes using nanotechnology approaches. The nanotechnology approaches have sought to progress seawater desalination progress by making it a more economical and sustainable option. Particularly, carbon-based nanomaterials such as graphene, graphene oxide (GO), and carbon nanotubes (CNTs) have been addressed as the emerging candidates to advance the field of water treatment, particularly in desalination. It is expected and envisioned that the application of carbon-based membrane for RO seawater desalination will be an attractive approach to address the cost and sustainability issues of existing desalination technologies. In particular, highly permeable nanomaterials will favorably increase the water flux thereby providing an opportunity to reduce energy consumption through a lowered feed pressure while maintaining the same water. Additionally, membrane fouling is an important factor that controls maintenance and subsequently the cost of RO processing. Therefore, developing of the carbon-based membrane with inherent antifouling capability is urgently needed to reduce the energy and chemical expenses.

This chapter first covers a brief introduction to carbon-based nanomaterials, which include their classifications and characteristics desired for desalination. Next, the preparation and performance evaluation of the standalone carbon-based membranes and nanocomposite membranes for desalination are presented. Finally, the challenges and future outlook of the carbon-based membranes for commercial desalination applications are highlighted.

2 Carbon-Based Nanomaterials

2.1 Classification of Carbon-Based Nanomaterials

Emerging carbon-based nanomaterials such as graphene, GO, and CNTs have been extensively used in water treatment. These nanomaterials possess exceptional and unique

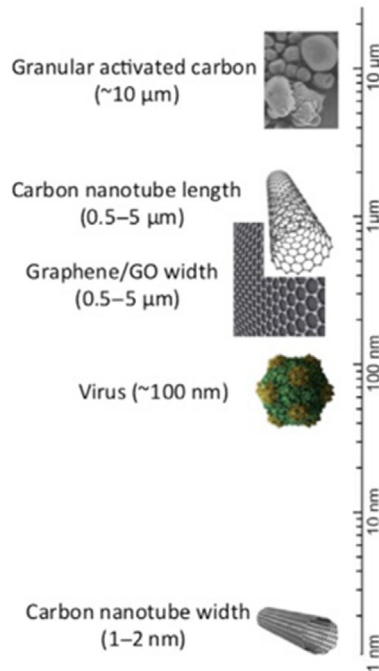


Fig. 1

The size of some emerging carbon-based nanomaterials for water treatment application [1].

properties to be used alone or as nanofiller in nanocomposites. For instance, the desired aspect ratios of these carbon-based nanomaterials promote the high surface area to volume ratio, which in turn imposes significant benefits compared with their macro- or microscale counterparts. Fig. 1 illustrates the size of the carbon-based nanomaterials relative to a virus [1].

CNT is an allotrope of carbon that is made up of rolled-up sheets of graphitic layers. Depending on the number of graphitic sheet layers, CNTs can be more specifically divided into multiwalled carbon nanotubes (MWCNTs) and single-walled carbon nanotubes (SWCNTs) in which the former have multiple graphitic layers, and the latter are characterized by a single cylindrical graphene shell. Recently, advances in the bottom-up synthesis approach have enabled precise control of the nanotube diameter of CNTs down to ~ 1 nm and lengths of a few centimeters [2].

Since its discovery, graphene has attracted global attention for a wide range of applications. The commonly known graphene family nanomaterials include single- or few-layered graphene, graphene nanosheets, graphene ribbons, GO, and reduced graphene oxide (rGO) [3]. Graphene is a 2D nanomaterial that is made up of covalently bonded carbon atoms arranged in a hexagonal honeycomb lattice. One of the most desired characteristics of graphene is its highly tunable carbon backbone, which can favorably lead to versatile

surface functionalization and modification. While the perfect graphene sheet is known to be impermeable to all gases, recent computational and experimental studies have proven that graphene can be used as a membrane through the controlled defects formed on the nanosheets [4]. Subnanometer pores have been successfully created via approaches such as electron–ion bombardment and oxidation [5]. Compared with nanosheet graphene, GO is made up of asymmetrical sheets due to the presence of functional groups on the basal planes and at the edges of the sheets. These oxygen-containing groups, such as hydroxyl, carboxyl, and carbonyl groups could, in turn, allow the dispersion of GO in aqueous medium without the aid of any stabilizing agents. Graphene oxide framework (GOF) consists of the layering of GO sheets that are covalently interconnected by linear boronic acid pillaring units that act as the linkers. GOF was first synthesized by Srinivas et al. [6] for gas adsorption. By taking the advantage of surface functionality of GO layers the accessible surface areas were enhanced by separating the GO layers apart using pillaring molecular structures. With this concept, the GOF was synthesized through the formation of boronate ester bond where B–O bondings were established via the boronic acids and oxygen functional groups on the GO layers. With the presence of these linkers, the interlayer spacing of the resultant layered GOF structure was greatly expanded. In fact, the chemical properties of the linkers are highly tunable to cater for desired pore size, volume, as well as the accessible surface area.

2.2 Characteristics of Carbon-Based Nanomaterials for Desalination

Advances in the synthesis and characterizations of nanomaterial offer new opportunities to address the challenges met in desalination by increasing the separation efficiency while reducing the energy and material costs [7]. Ion transport through nanopores is an emerging research area as it holds great potential to advance a wide range of applications particularly in the field of separation. Owing to their superhydrophobicity and smooth inner cores, CNTs have served as a unique nanofluid model system to study the water–ion interaction based on their frictionless hollow structure [8]. Interestingly, the electrostatic charge distributed on the CNT can also impact different water and ion separation effects. It has been observed that, by changing the charge patterns on CNTs, the CNTs can be controlled to facilitate the water intake while entirely hindering the passage of ions through the channels [9]. The separation behavior of nanoporous graphene (NPG) indicates that it can serve as an alternative candidate for RO membrane. In contrast with the typically used thin-film composite membranes, which face some underlying issues regarding the understanding towards the mechanisms of salt rejection and water permeation mainly due to the polyamide active layer with an amorphous structure, the uniqueness of graphene in terms of the atomic thickness presents a straightforward principle for desalination [10]. In general, the short-range interactions between water molecules or ions with the confined material may involve a combination of steric, dispersion, dipole, and electrostatic interactions. On

the other hand, when long-range interaction is considered, the electrostatic fields due to the electrically charged ions and neutral water molecules can selectively exert forces on ions, hence depleting, accumulating, or transporting the ions.

Being a 2D ultrathin planar material, graphene holds very promising application in water transport. Owing to its extremely high mechanical strength, it required considerably low pressure to allow the ultrafast molecule passage and serve as the next generation of desalination key materials [11]. The study that provided insights into the monovalent ion transport and dynamics in NPG pointed out that steric exclusion is the main contributor to the ion partitioning ability. On the other hand, dielectric exclusion is only responsible for the minor ion partitioning effect, but it caused a slightly higher concentration of larger Cl^- ions than K^+ ions [12]. More impressively, the water permeability of NPG has been predicted to be as high as $\sim 1000 \text{ L/m}^2\text{-h-bar}$ with the optimized porosity. Hence, this characteristic represents the promising design of ultrahigh permeable membrane for desalination [10]. On the other hand, as a hybrid material, GOF combines the advantages offered by both graphene and the linker in which it can simultaneously exhibit hydrophobicity and tunable linker concentration, hence contributing to excellent water permeation and separation [13].

3 Standalone Carbon-Based Membranes for Desalination

3.1 Carbon Nanotube Membranes

CNT membranes refer to either an open-ended single CNT or an array of vertically-aligned CNTs incorporated across a polymer film. The pioneering work by Hind et al. [14] showed that by matching the tube opening size and its tube entry functionalities to that of the targeted molecule, the nanoporous membrane design with improved chemical selectivity and ultrafast transport can be a significant breakthrough in the fields of molecular separations. During the typical growth process of CNT, the nanotube size is determined by the diameter of the metallic catalysts. This provides a practical opportunity to tailor the pore diameter and structure-controlled synthesis through the predefined nanosized catalyst [15]. Principally, the hollowed structures of CNT can allow precise control of pore dimension at the nanoscale, hence can constitute energy barriers at the tube entries and facilitate molecular size-oriented sieving [16]. Many computational works have evidenced that when passing through the nanotubes, fluid such as water molecules form ordered hydrogen bonds, which resulted in atomically smooth and frictionless hydrophobic interface.

As-synthesized CNTs tend to aggregate and normally contaminated with impurities such as metal catalysts. Therefore, functionalization of CNT is often performed prior to their application for desalination. More importantly, the modification of CNT pores is very

crucial to enhance the selectivity towards the targeted ions. Additionally, functionalization is also an attractive approach to decrease energy consumption by enhancing the permeability and physical adjustability. Fig. 2A illustrates the possible tip and core functionalization of CNT membrane. In brief, tip functionalization allows the attachment of selective functional groups at the nanotube entrance meanwhile the core functionalization bestows desired functionalities at the sidewall or interior core of the nanotubes [16]. Besides that, functional metallic particles such as Cu and Ag can also be used to decorate

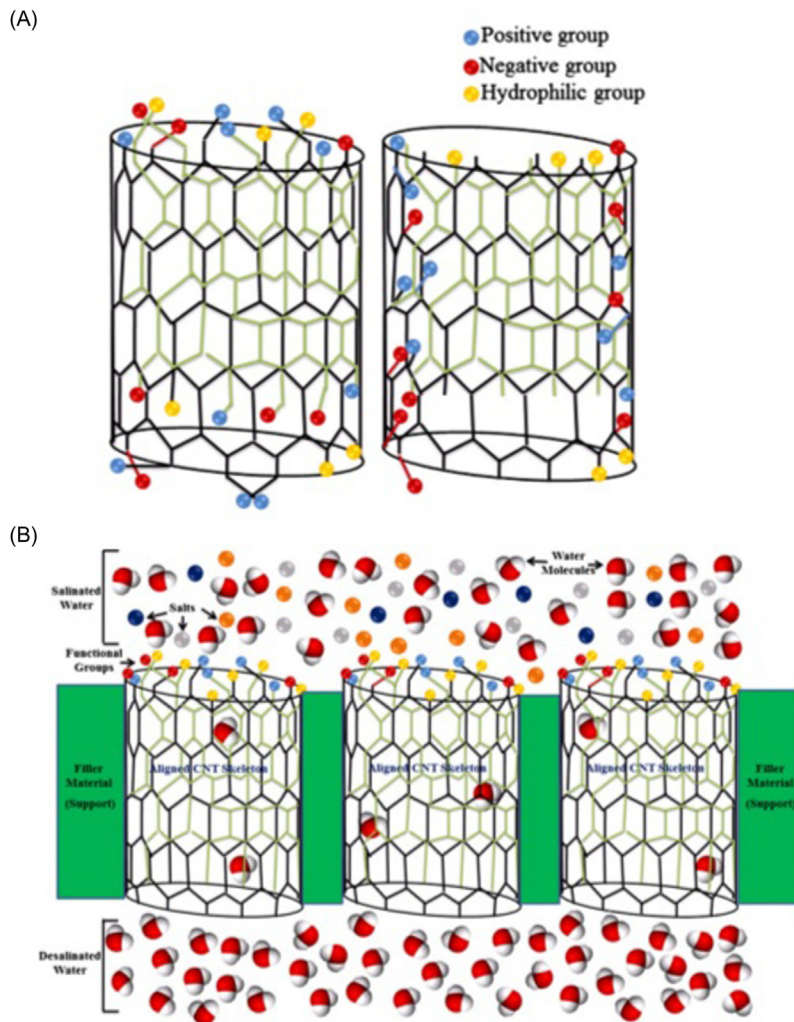


Fig. 2

(A) The tip and core functionalization of CNT and (B) the passage of water molecules through CNT membranes [16].

the CNT membranes and broaden the applications for desalination. For instance, Ag-decorated CNT membranes play an important role in minimizing the biofouling phenomenon, hence extending the membrane lifespan [17]. Fig. 2B schematically depicts the passage of water molecules through the CNT membranes, while the functionalized CNTs act as a “gatekeeper” to hinder the passage of salt ions, hence promoting promising salt rejection ability.

Liu et al. [18] proposed an ion-responsive membrane based on zwitterion functionalized CNTs (ZCNTs) at the entrances and internal walls of the membrane nanochannels to obtain ultrahigh multivalent ion rejection. With the integration of the ion-responsive building, the membrane rejection ability has been well tuned to efficiently perform desalination. Ce-initiating atom transfer radical polymerization was applied to fabricate the poly (sulfobetaine) (PSB) brushes grafted CNTs. In the resultant ZCNTs, the cylindrical CNTs act as the core while PSB layers grafted from both the outer layers and tips of porous CNTs served as the skin. With the unique coaxial cable structure of the resultant ZCNTs, the mean effective pore diameter of was drastically reduced from 1.24 to 0.54 nm. The gatekeeping effects rendered by the supermolecular assembly and super hydrophilicity PSB blocks were ascribed as the primary reason for the ultrahigh selective permeabilities of monovalent anions against divalent anions. This implies that the ZCNTs hold promising applications in desalination for water with NaCl concentration ranging from 100 to 600 mol/m³. Ultralong CNTs (UCNTs) modified with plasma have been attempted for salt adsorption [19]. The ultralong CNTs were first deposited onto a mixed cellulose ester (MCE) porous support through vacuum filtration technique prior to the plasma treatment. The desalination performance testing indicated that the UCNTs demonstrated the adsorption capacity that was about twice that of activated carbons. As a result, the amount of salt removal was much higher than that of the commercial porous carbon material used in the capacitive deionization. It was predicted that 250 g of UCNTs is sufficient to desalinate about 100 L of brackish water with a salinity of 10,000 ppm. It suggests the possibility of this material be applied as portable point-of-use desalination systems. More recently, Lee and Park developed a highly densified vertically aligned (VA) CNT membranes with pore density 3×10^{12} pore/cm² [20]. The mechanical strength was improved through the polymerized urethane that was infiltrated between CNTs during densification in ethanol stream. Owing to the CNT pores size of 4.1 nm that allow higher nanotube packing density, the VA-CNT membrane exhibited 938 times higher water permeability than the conventional ultrafiltration membrane.

3.2 Graphene and Graphene Oxide Membranes

In recent years, molecular dynamic (MD) simulations have been well applied to provide further understanding of the high water permeability and the mechanisms involved in the water passage through NPG, as well as the correlations between the specific size and

chemistry of nanoporous [10]. The findings from this computational work suggested that unlike CNT, the permeability or the flux of water molecules across NPG is not restricted in a single file, but rather controlled by the passage through the nanopore in a favorable geometric orientation and hydrogen-bonding configuration [12].

Owing to the high mechanical strength (up to 1 TPa), the thin layer of porous graphene can be applied for forward osmosis (FO) or pressure retarded osmosis (PRO) without the requirement of a support layer. The omission of the support substrate can serve as a promising strategy to suppress the interfacial concentration polarization and enhance the performance of the FO membranes [21]. Using MD simulation, Gai et al. [22] functionalized porous monolayer graphene membrane with fluorine and nitrogen for FO desalination that used 0.6–5.0 mol/L NaCl as the draw solution. The water transport study showed that the fluorinated graphene membrane demonstrated much higher flux than that of functionalized with nitrogen. During the adsorption of water molecules and Na^+ ions by the fluorine and nitrogen atoms in the graphene pores, it was found that the adsorption energy of fluorinated membrane is lower compared with the nitrogen counterpart. Due to the decrease in adsorption energy, the lower interactions between the graphene pore and solutions were weakened, hence facilitated the passage of both salt ions and water molecules. As a result, the fluorinated graphene membrane exhibited excellent water flux of $28.1 \text{ L cm}^2 \text{ h}^{-1}$, which was nearly 20,000 times higher than that of commercial cellulose triacetate membrane. The ultrahigh water flux also indicated that the fluorinated membrane may hold potential in PRO for power generation based on its high power density.

Nicolai et al. [13] applied MD to evaluate the performance GOF desalination as a function of linker concentration, membrane thickness, and applied pressure. Based on the orientation where the graphene sheets positioned at the yz plane whereas the linkers are oriented in the XY plane as illustrated in Fig. 3A, they found that water permeability of GOF membranes with the largest pore size was twice as large as that of the smallest pore size. The findings also showed that the water permeability of GOF membranes can be further improved by optimizing the linker concentration, which plays a critical role in tuning the pore sizes and pore spacing. The best performing GOF membrane obtained in this study could demonstrate 100% salt rejection and water permeability range from ~ 5 to $25 \text{ Lcm}^{-2} \text{ day}^{-1} \text{ MPa}^{-1}$. In addition, the comparison in Fig. 3B–C shows that GOF membranes were comparable with other commonly studied standalone membranes such as graphene membrane and CNT membranes.

4 Carbon-Based Nanocomposite Membranes for Desalination

The fabrication of nanocomposite membrane containing inorganic materials and polymer can provide membranes with some unique properties, as well as possibly induce new functions and characteristics based on their synergetic effects. Emerging nanocomposite membranes show the potential of high permeability and energy reduction, thus further studies at a larger

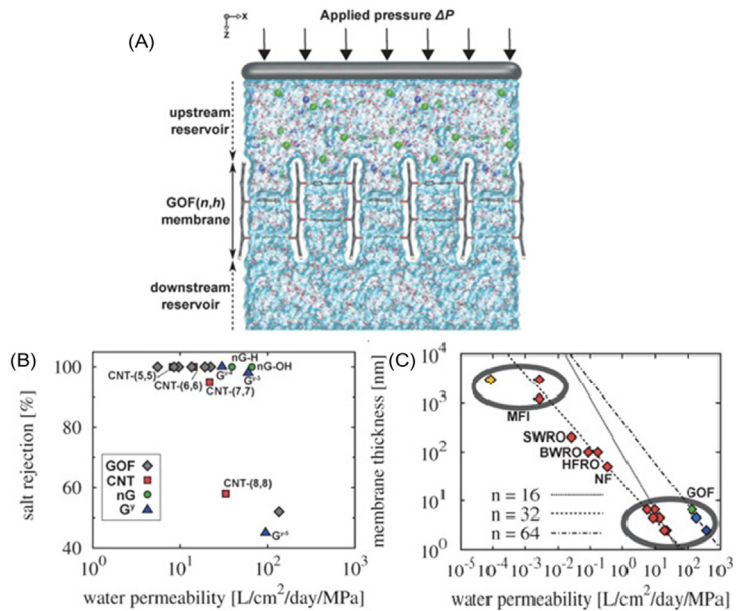


Fig. 3

(A) The orientation of GO and linkers during the desalination simulation, (B and C) The desalination performance comparison of GOF membranes with other contemporary membranes [13].

scale are required to determine their sustainable operation. Several hybrid configurations are currently being explored for desalination application. These configurations include carbon nanotube/polymer and graphene/polymer. A combination of two materials (hybrid) has shown to be more efficient than the utilization of a single material by its own. It is believed that these emerging materials will not only provide a means to produce pure water from brackish water or salt-containing wastewater, but also potentially treat seawater with higher salinity at low operational and maintenance costs, particularly in terms of electricity consumption and cleaning chemicals usage. In general, two strategies have been adopted to prepare nanocomposite membranes, which are thin-film nanocomposite (TFN) membranes, where carbon-based nanoparticles are deposited on the surface of a polymeric membrane, and mixed matrix membranes (MMMs), which are fabricated via direct entrapment of carbon-based nanoparticles within the polymeric matrix [23].

4.1 Carbon Nanotubes/Polymer Nanocomposite Membranes

CNTs/polymer nanocomposite membranes have gained considerable attention and are considered as the cutting edge generation of high-performance membranes for water desalination through the use of RO. For the preparation of this membrane, a more effective dispersion of CNTs in the polymer matrix needs to be achieved by functionalizing the

CNTs with desired functional groups. The most challenging step, that is, the opening of the CNTs tip by either acid treatment or oxidation, may cause disruption of tube integrity and thinning of the CNT wall and subsequent failure of membrane channels. The functionalization of CNTs could also open up promising avenues to gain a better anchoring of nanotubes in polymeric host materials and thus yield better reinforcement of composites.

Extensive work has been reported in the literature regarding the nature of fast transport of water through CNT channels by establishing strong hydrogen-bonding network between water molecules, slip length, the interfacial friction of water at graphitic interfaces and inherent smoothness of the interior of CNTs. Incorporation of modified MWCNTs can also alter the pore size and porosity of membranes, and subsequently, alter their water permeability and salt rejection. This finding is evident as shown by scanning electron microscopy (SEM) images in Fig. 4, by the embedding of polydopamine-coated MWCNTs into the polysulfone membrane matrix [24]. Yin et al. [25] stated that the surface pore size will increase with the increase of MWCNTs loading due to the accelerated solvent and nonsolvent exchange, thus encouraging the formation of a more porous structure. However, the surface pore size will decrease with further increase in MWCNTs loading for the MWCNTs/polysulfone (PSF) membranes. Subsequently, there will be low porosity and low degree of pore interconnectivity, leading to a diffusion delay. Another study found that oxidized MWCNTs can enhance the wettability of membrane materials and the electrostatic repulsion between the PES membranes and foulants [26].

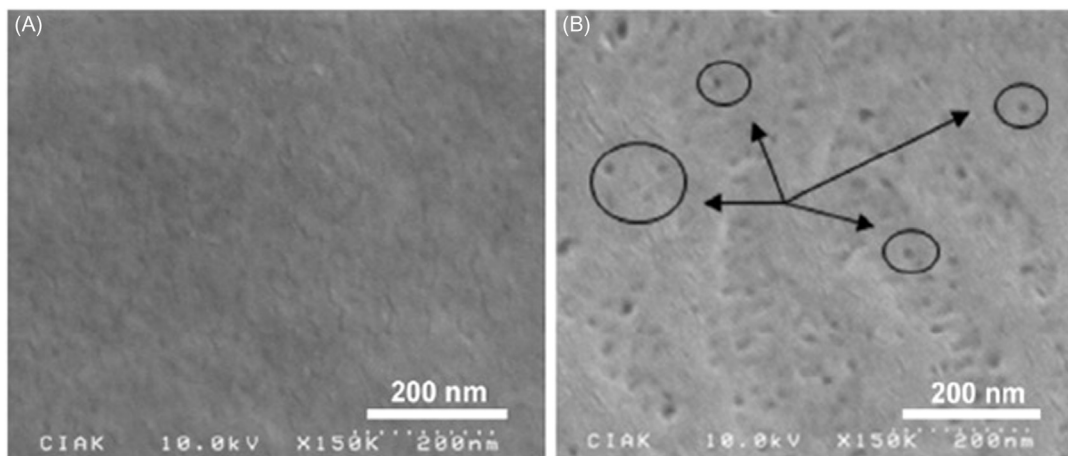


Fig. 4

SEM surface images of (A) polysulfone membrane and (B) 0.1 wt.% polydopamine-coated MWCNTs/polysulfone nanocomposite membrane [24].

According to Zhao et al. [27], a significant hydrophilicity enhancement was observed when nanocomposite membrane containing hyperbranched poly(amine-ester) functionalized MWCNTs and polyvinylidene fluoride (PVDF) was fabricated. This is because the increased surface coverage of hydrophilic –OH groups from the MWCNTs is able to create a water layer on the membrane surface when hydrogen bonding and water molecules come into contact. Thus, the adsorption of the foulant on the membrane surface could be prevented. In general, membrane fouling resistance can be enhanced as the surface hydrophilicity of a membrane has increased [28].

In another study, the effect of MWCNTs concentration on morphological structure, together with water permeability and a salt rejection of polycaprolactone (PCL) modified MWCNTs/polyethersulfone (PES) membranes, was investigated. The pure water flux was increased about 50% when 3 w/v% PCL-MWCNTs was incorporated into PES matrix. It was observed that the membrane porosity increased and sponge-like pores in the sublayer of the PES membrane were turned into finger-like pores in the PCL-MWCNT/PES blend membranes [29]. In addition, MWCNTs grafted by poly(methylmethacrylate) (PMMA) to synthesize polyamide thin-film composite (TFC) membrane via interfacial polymerization (IP) can enhance the water flux up to 62%, which in the study had demonstrated higher rejection for Na_2SO_4 of 99%. The fabrication route of these membranes is illustrated in Fig. 5. The formation of an ultrathin polyamide rejection layer on the PSF substrates was obtained by the reaction of piperazine and trimethyl chloride between the interface [30].

Furthermore, the loading amount of the MWCNTs has been found to have great influence on membrane performance. A previous study obtained a higher salt rejection and water flux for MMMs embedded with CNTs at 0.1 wt.% [31]. It was reported that the mechanical strength of the nanocomposite membrane was increased when 0.12 wt% of carboxyl functionalized MWCNTs was blended with PVDF/polyvinylalcohol (PVA) ultrafiltration (UF) membrane. The results showed that the break strength, elongation at break, and Young's modulus were enhanced by 60%, 215.5%, and 56.7%, respectively, in comparison with the original membrane [32]. The findings concluded that even a small amount of MWCNTs can benefit the final product and make it as the primary choice for composite reinforcement.

To date, the advancements in nanotechnology open the doors to the fabrication of TFN RO membrane for desalination. The incorporation of CNTs in the support membrane and/or dense thin polyamide (PA) layer can improve the performance of the RO membranes and consequently reduce the desalination cost and environmental impacts. The rate of water transport can be enhanced by the formation of ordered hydrogen bonds and additional water path for water molecules through the nanochannel, provided by the CNTs in the PA layer. Amini et al. [33], for instance, found that the TFN membrane embedded with 0.05 wt.% functionalized MWCNTs exhibited nearly 14% water permeability improvement ($3.6 \text{ L/m}^2 \text{ h}$) and 28% increase in salt rejection (89.3%) compared with that of neat TFC

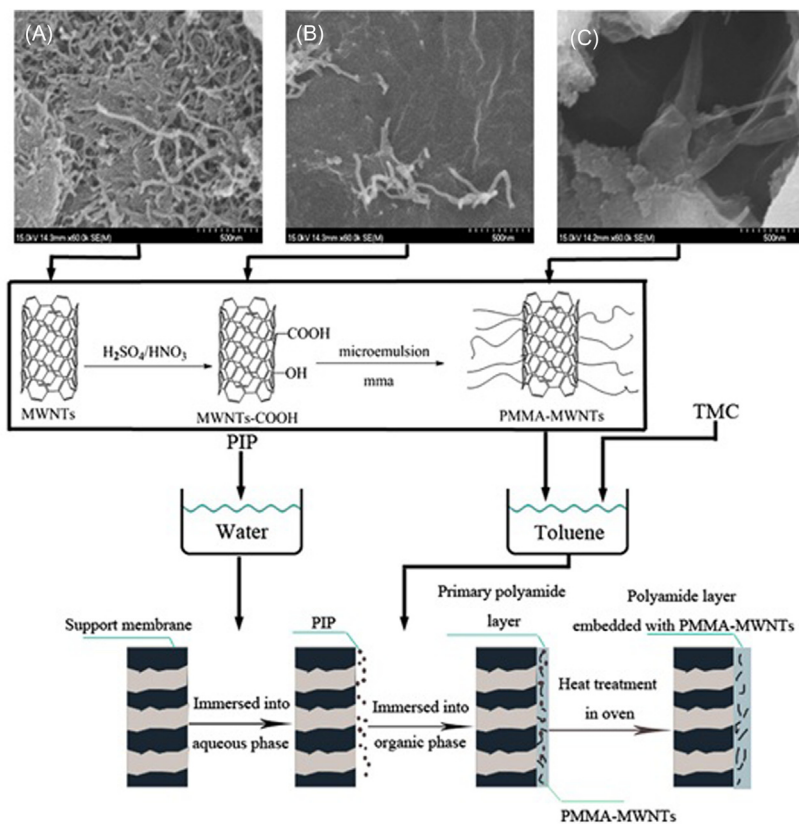


Fig. 5

The fabrication route of TFN membrane embedded with poly(methylmethacrylate) hydrophobic modified MWCNTs via IP [30].

membrane. Goh et al. [8] fabricated CNT immobilized polyamide-imide (PAI) and polyetherimide (PEI) hollow fiber membrane, which exhibited 44% enhancement in water permeability without significant compromise on the salt rejection. However, it was revealed that the amount of energy that could be minimized by usage of CNT/polymer nanocomposite membrane would not be significant unless the approach toward the design of membrane module configuration was redefined to reduce the concentration of polarization, and membrane fouling issues were addressed [34].

4.2 Graphene/Polymer Nanocomposite Membranes

High energy demand, membrane fouling, and the trade-off between salt rejection and water flux still remain as challenges in the creation of membranes for water desalination. Recently, researchers have found that the promising membrane properties in terms of water

permeability, antimicrobial properties, and mechanical strength could be achieved by adding GO into the polymer matrix. The desired physicochemical properties of graphene/polymer nanocomposite membranes depend on the interfacial bonding between the polymer matrix and graphene layers, in addition to the distribution level of graphene layers inside the polymer matrix. This is because, GO is a highly chemically modified form of graphene that consists of a variety of functional groups, such as hydroxyl and epoxide groups on the basal plane and carboxylic acid at the edges. These functional groups could control the van der Waals forces and compatibility between the GO and polymer. To overcome the incompatibility issues of GO/polymer, oxidation of graphene has been suggested to introduce highly hydrophilic and stable colloidal dispersions [35].

According to Zinadini et al. [36], GO demonstrates high hydrophilicity when incorporated within PES membrane. This is attributed to the migration of carboxylic acid, hydroxyl and other functional groups of GO to the surface of the membrane during the phase inversion process. In contrast, the hydrophobic nature of the PVDF membranes, which induces severe membrane fouling, will cause a decrease in flux during operation [37]. Many efforts have been devoted to enhancing the hydrophilicity, pure water flux, salt rejection, and antifouling properties of PVDF-based nanocomposite membrane in the past several years. Ayyaru and Ahn [38], for example, incorporated sulfonic acid groups functionalized GO into PVDF matrix to create a highly hydrophilic membrane. In this case, GO can be functionalized easily with sulfonic groups without affecting its morphological structure. The results demonstrated that the nanocomposite membranes exhibited higher water flux ($460 \text{ Lm}^{-2} \text{ h}^{-1}$) compared with pure PVDF membranes ($290 \text{ Lm}^{-2} \text{ h}^{-1}$). This was attributed to the presence of additional functional groups ($-\text{SO}_3\text{H}$) on GO, which possessed stronger hydrogen-bonding groups, compared with the $-\text{COOH}/-\text{OH}$ groups present in GO.

Moreover, introducing hydrophilic GO into PSF matrix through classical blending method has demonstrated another strategy to improve the properties of PSF membranes. Rezaee et al. [39] reported that the spongy structure and closed-end drop-like pores of the PSF membrane could transform into finger-like pores and have larger open-end channels in GO/PSF nanocomposite membrane. The previous study showed that employment of 1 wt.% GO in the PSF matrix exhibited pure water flux of $50 \text{ Lm}^{-2} \text{ h}^{-1}$ at 4 bars, and significant changes in microvoids in the sublayer of the GO/PSF membrane had been observed. The salt rejection can also be tailored by manipulating the pH of feed solution [40]. Recently, polymer-functionalized GO has been considered as the most preferred choice for making advanced GO/PSF nanocomposite membranes. The salt rejection has been improved by the addition of up to 3 wt.% amount of GO. This is attributed to increasing surface repulsive interaction by GO [41].

In relation to this, excellent antifouling and antibacterial properties of GO/polymer nanocomposite membranes can be achieved by doping the GO with zinc oxide (ZnO). GO

nanosheets could act as a versatile platform for better nanomaterials dispersion due to the presence of carboxylic and hydroxyl functional groups. The previous study showed optimum membrane performance as demonstrated by ZnO-GO/PSF nanocomposite membranes with ZnO-GO loading of 0.6 wt.%. The water permeability increased from 0.89 to 5.11 $\text{Lm}^{-2} \text{h}^{-1} \text{bar}^{-1}$ when ZnO-GO was added into the polymer matrix [42]. The positive effects of the concurrent use of GO and ZnO on PES membranes have also been further studied by using a double-casting phase inversion technique. The ZnO-GO/PES nanocomposite membranes have shown higher fluxes and more resistance to organic fouling compared with commercial NF-270 membranes [43]. In another study, hyperbranched polyethyleneimine modified-GO was found to have superior antibiofouling properties when incorporated within PES membrane. This is attributed to the electrostatic repulsion of GO, which acts as a barrier for foulant adsorption on the nanocomposite membrane surface [44]. Similarly, antifouling properties were also reported in manufacture of ultrafiltration membrane using isocyanate-treated GO/PSF nanocomposite membrane [45].

Titanium dioxide (TiO_2) nanoparticles have also been extensively investigated due to their high hydrophilicity, low toxicity, lower material cost, better chemical stability, and reasonable antibacterial properties. Similar to ZnO, by dispersing TiO_2 to the polymer solution, both aggregation and agglomeration can occur at high concentrations, which contribute to pore structure defects and reduction of permeability and hydrophilicity. Thus, reduced GO has been introduced to facilitate better dispersion and distribution of TiO_2 in the PVDF membrane matrix. PVDF nanocomposite membranes containing 0.05 wt.% reduced GO/ TiO_2 with reduced GO to TiO_2 ratio of 70/30 have shown the highest hydrophilicity, pure water flux, and antifouling ability. In a related study, membrane hydrophilicity, and permeability were improved due to the various oxygenated hydrophilic groups on the reduced GO/ TiO_2 surface. The results revealed that by increasing the hydrophilicity of the membranes, the water permeability will increase by attracting water molecules inside the membrane matrix and facilitate their permeation through the membrane [46].

From the mechanical performance perspective, high tensile strength and elongation at break values mean better toughness and sustained integrity of membranes, which will assure a comprehensive performance for a long-term operation. Quan et al. [47] reported that the enhancement in the mechanical strength of GO/polymer nanocomposite membranes can be much higher than that of other carbon-based nanocomposite membranes. Such properties could be achieved at very low GO content in the polymer matrix. It was reported that superior enhancement in tensile strength and elongation at break of 43.94% and 39.24%, respectively, were achieved for GO/PVDF nanocomposite membranes [44]. In another study, the tensile strength measurement of the 3-aminopropyltriethoxysilane functionalized GO/PVDF membrane was improved by 69%, while the elongation at break was 48% more in comparison with GO/PVDF membrane [48].

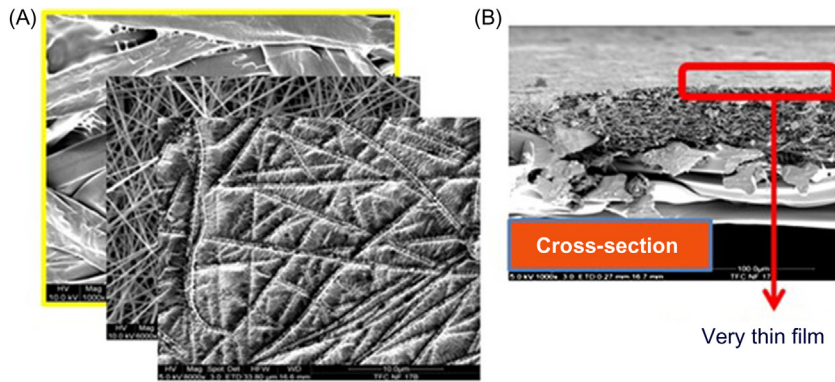


Fig. 6

A three tier architecture of (A) thin-film nanofibrous composite (TFNC) membrane and (B) its cross-sectional view [49].

Recently, TFC FO membranes, which are composed of polyamide layer (top layer) and dual-layered PSF (substrate layer), have been developed by incorporation of GO into the membrane substrate. The conceptual illustration of such membrane preparation is shown in Fig. 6. FO is one of the attractive osmotic-driven membrane processes for brackish and seawater desalination. The main challenge in FO is low water fluxes caused by internal concentration polarization (ICP). ICP occurs inside the substrate layer, which provides resistance to the diffusion of draw solutes within the substrate layer. To minimize the ICP effects, a high porous finger-like structure is required at the bottom of the substrate layer. It is revealed that an incorporation of 0.25 wt.% GO in the substrate layer can exhibit higher water permeability, and ion selectivity can be enhanced by the presence of well-dispersed hydrophilic GO in the membrane substrate. Lim et al. [50] obtained the highest water flux of $30.3 \text{ Lm}^{-2} \text{ h}^{-1}$ for this membrane. The presence of GO nanosheets has been found to improve the chemical resistance, wettability, porosity, and antifouling properties of the fabricated TFC FO membranes [51,52]. In another study, Yin et al. [53], embedded GO into the PA thin layer of TFN membrane by in situ IP. The permeate water flux under 20 bar was found increasing from 39 to $59 \text{ Lm}^{-2} \text{ h}^{-1}$, while rejections of salt slightly decreased from 95% to 93% when the loading amount of GO was increased from 0 to 0.02 wt.% [53].

4.3 Carbon Nanofibers Nanocomposite Membranes for Desalination Application

Global demand for water continues to increase with increasing population and weather variability whilst freshwater sources are becoming scarcer due to seasonal variations in rainfall. The rise in demand is anticipated among of the residents when the water supply disruptions had occurred. To increase the water catchment capacity, seawater desalination has been considered as an alternative water source treatment that is not climate dependent

[54]. The water desalination process technology is a membrane separation process that is a green and effective separation technology to remove solutes from water [55]. Nanofiltration (NF), RO, and FO membranes are the most dominant technology used for desalination and water softening applications. This technology widely uses the TFC PA membrane.

TFC membranes consisting of three fundamental layers have been developed to reduce the resistance and enhance the flux of the NF, RO, and FO membranes. The three fundamental layers consist of a top ultrathin selective barrier layer, middle porous support layer, and bottom nonwoven fabric [49]. The top and middle layer can be carefully altered to achieve the optimal separation performance [56]. Although the bottom nonwoven fabric does not influence the separation characteristics, it has been used to provide mechanical strength during handling. In recent years, efforts have been made to employ nanofibers in TFNC membranes as can be seen in Fig. 6.

The use of electrospun nanofibrous membranes (ENMs) as the middle layer in TFC has gained much interest in recent years due to several attractive attributes, such as their highly porous and interconnected pore structure, micron-scaled interstitial space, controllable thickness, and a large surface area to volume ratio [57,58]. The higher permeate flux compared with the conventional membranes has been their added advantage [49]. In an effort to improve the mechanical integrity, chemical and thermal stabilities of the ENMs as the intermediate substrate layer, converting the ENMs into carbon nanofibers (CNFs) nanocomposites membrane also have been taken into consideration by several researchers recently. The mechanical, chemical, and thermal stabilities, as well as antibacterial properties of carbon-based nanomaterials, are better than polymer materials. These favorable features make carbon nanofibrous nanocomposite membranes the right emerging candidate for water treatment and desalination [59].

4.3.1 Fabrication of electrospun nanofibrous membranes via electrospinning method

Several techniques such as chemical vapor deposition and high-temperature annealing treatment of electrospun polymer fibers have been employed to achieve CNFs. Among these technologies, electrospinning is a highly versatile method that produces continuous polymer fibers with diameters ranging from a few nanometers to several micrometers under the influence of external electric field applied to a polymer solution or melt [60].

Electrospinning is a process using electrostatic forces, which makes it a unique method to produce superfine fibers by forcing a solution through a spinneret with an electric field. There is also evidence of sizable static charges in electrospun fibers that could effectively handle producing three-dimensional structures. There are three main components for electrospinning setup as shown in the generic design below (Fig. 7) which are the high voltage power supply, nozzle/spinneret, and grounded collector [61].

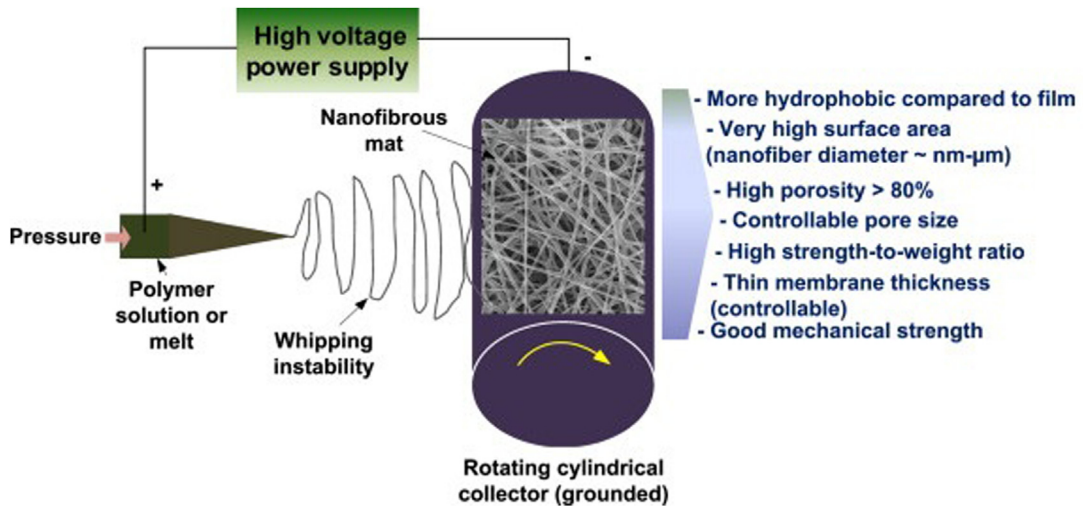


Fig. 7

Schematic of typical setup for electrospinning [61].

In most electrospinning processes, generally a metallic needle and syringe were used as spinneret and the polymer solution is loaded into the syringe that connected to the metallic needle while the aluminum foil was used as a grounded collector. Typically, the polymer solutions are prepared by electrospinning by dissolving the polymer in some solvents [62]. This versatile technique enables to produce CNFs from various polymer precursors, such as polyacrylonitrile (PAN) [63], pitch [64], polyimide [64], cellulose [65], phenolic resin [66], and PVDF [67] by suitable thermal treatment. PAN has been frequently used as the precursor of carbonaceous material for various applications by electrospinning because of its good spinnability and high carbon yield [68].

4.3.2 Conversion of electrospun nanofibers to carbon nanofibers through pyrolysis process

Electrospun NFs are converted into CNFs through carbonization in inert condition with a high temperature in the range from 800°C to 3000°C to eliminate the noncarbon elements as volatile gases. Carbonization process was done under an inert condition to avoid the access of atmospheric air and oxidation at a high temperature as well as to dilute the toxic waste gases in the gas extract system [69]. For this purpose, inert gases such as nitrogen and argon have been used. During carbonization, it was believed that around 50 wt.% of the nanofibers were volatilized into noncarbon elements including methane, nitrogen, water, ammonia, and various other gases [70]. Moreover, the performance of the CNFs highly depends on the heating rate of carbonization where higher carbonization rate will lead to the defects on the structure of the CNFs while lower carbonization rate causes the loss of too much nitrogen at the early stages of carbonization [71]. The atomic structure of a CNF

is similar to that of graphite, consisting of carbon atom layers (graphene sheets) arranged in a regular hexagonal pattern.

4.3.3 *Thin film carbon nanofibrous nanocomposite membrane*

4.3.3.1 Thin film carbon nanofibrous nanocomposite membranes by coating

The intrinsic high porosity with an interconnected pore structures makes nanofiber mats promising candidates for TFC membrane supports. However, hydrophilic nanofibers undergo a dramatic decrease in strength when exposed to water [72]. The drastically reduced strength in the hydrophilic nanofibers is due to the high degree of swelling. Coating a nonswelling hydrophobic fiber to make it hydrophilic and fully wettable without negatively impacting the mechanical properties of the material is a smart approach for desalination application. Huang et al. [72] introduced the IP of nylon 6,6 directly onto electrospun hydrophobic PVDF fibers. The modified nanofibers exhibited significantly lower swelling propensity due to the hydrophobic core fiber, and are thus able to retain strength in an aqueous environment and exhibited excellent flux performance. Zhao et al. [73] also applied the coating of polymer solutions by incorporation of chitosan and glutaraldehyde, terephthaloyl chloride as a barrier layer on the nanofibrous membrane to enhance the performance of UF or NF medium with better flux rate and higher rejection.

4.3.3.2 Thin film carbon nanofibrous nanocomposite membranes by interfacial polymerization technique

Recently, there have been many reports documented on the modifications of the barrier layer on top of the porous polymer membrane. Yoon et al. [57] reported the IP at three different ratios of piperazine and bipiperidine on PAN nanofiber membranes. Meanwhile a study by Yung et al. [74] has revealed that the TFNC prepared by IP of piperazine (PIP) using ionic liquids (IL) exhibited twice higher permeation flux compared with that of commercial NF membrane with comparable salt rejection ratio. Meanwhile, the work demonstrated that modifications of IP using β -cyclodextrin has increased the value of water flux, salt rejection, surface charge, and antifouling properties compared with normal polyester membranes [75]. This enhanced flux behavior in TFNC membranes was due to the large open pore structure and low hydraulic resistance of the nanofibrous membranes support.

A facile method for preparing ultrathin polymeric nanofiltration membrane using surface modification of ultrafine cellulose nanofiber (UCN) membrane via IP has been recently reported [76]. The resultant membranes comprising an ultrathin selective layer intertwined with cellulose nanofiber matrix are smooth and allow fast permeation of water. Typically, the 77.4 nm-thick membrane with a mean pore size of about 0.45 nm and molecular weight cut-off of 824 g mol⁻¹ has a high pure water flux of 32.7 L m⁻² h⁻¹ bar⁻¹ that is an order of magnitude higher than those of previously reported similar nanofiltration membranes.

4.3.3.3 Self-supporting membranes made from carbon nanofibers

The self-supporting CNF membranes have been fabricated using the electrospinning of PAN nanofiber followed by their peroxidation, carbonization, and posttreatment with ZnCl_2 [75]. The prepared membrane has demonstrated relatively high capacitance and hence relatively high salt sorption while testing with 500 mg/L NaCl solution in capacitive desalination [75]. The freestanding and highly microporous phenolic resin CNF membranes were successfully prepared by Chen et al. [76] for capacitive deionization (CDI). The resultant membranes exhibited higher CDI capacity (50.1 mg g^{-1}) toward 2000 mg L^{-1} brackish water at 1.2 V and excellent reproducibility. Another work on the carbon-based composites was reported by Dong et al. [77] utilizing the rGO and activated CNFs (ACNFs) for capacitive deionization. It was found that the nanofibers help to immobilize graphene and avoid the agglomeration of graphene, which resulted in an increase in conductivity and improved pore width distribution of the electrospun composite. The composite RGO/ACNF has shown a high desalination capacity of 7.2 mg g^{-1} and an excellent reversibility for CDI.

4.3.3.4 Functionalization of carbon nanofibers through carboxylation

Materials such as carbon in the shape of fibers are of great practical and scientific importance. The combination of high specific area, flexibility, superior chemical properties, and high mechanical strength with their unique nanostructures allows nanofibers to be used as reinforcing fillers in polymeric composites [78,79]. In addition, CNFs have superior separation capability as well as excellent physical properties including high tensile moduli and strength, which can be used as potential fillers in the fabrication of polymeric composites such as asymmetric membranes [80,81]. However, to harness the exceptionally smooth channels of CNFs for fast water transport, the CNFs are likely to be chemically modified to increase hydrophilicity. Unlike related carbon materials such as multiwalled carbon nanotubes, the sidewalls of CNFs are more chemically reactive and there exist more options for chemical functionalization, which improves compatibility with the polymer matrix. Fibers with exposed edge planes along the entire interior and exterior surfaces of the nanofibers have a hollow core that is surrounded by a cylindrical fiber, which makes a unique carbon nanostructure. These edge sites of CNFs are reactive and facilitate chemical modification of the fiber surface for compatibility improvement with polymer matrix [82]. However, the low dispersion and chemical inertness of carbon nanostructures are the main processing limitations for the preparation of mixed matrix membranes. Furthermore, the uniform dispersion of CNFs in a polymer matrix has been identified as a major issue that must be addressed in the preparation of high-performance membranes [83]. Carboxylation is the most common and effective functionalization method through which COOH functional groups attached on CNFs surfaces and CNFs were found to have excellent dispersibility in water and many common polar solvents [84]. The functionalization procedure is schematically shown in Fig. 8 using sulfuric and nitric acids [86].

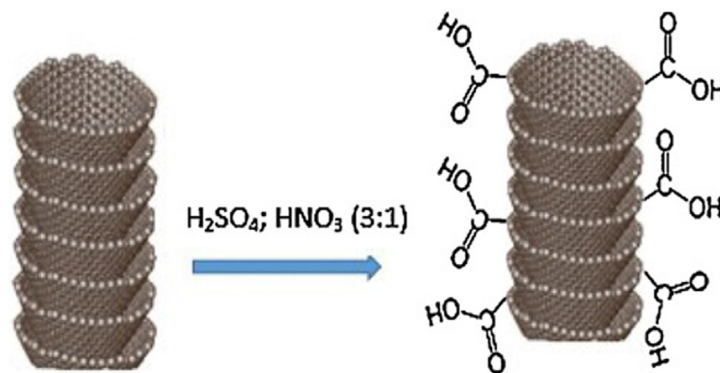


Fig. 8

Procedure for the preparation of carboxylated CNFs [85].

Dabaghian et al. [79] found that the modification of FO membranes with amine functionalized CNFs possessed good performance in seawater desalination, i.e., high FO flux and low reverse solute flux. It is also found that the amine functionalized CNFs FO membranes possessed higher mechanical strength. The ICP also significantly reduced by addition of low content of hydrophilic modified carboxylated CNFs in the membrane structure [86].

4.3.4 The performance of carbon nanofibrous and electrospun nanofibrous membranes in thin-film nanocomposite membrane for desalination application

Carbon-based nanomaterials have grown significant interest in the fabrication of TFC membrane due to their promising characteristics such as high mechanical strength, thermal stability, and antibacterial properties. The utilization of nanofibers in TFN membrane with improved performance is shown in Table 1. It was found that by utilizing ENMs and CNFs produced by electrospinning in the TFC configuration in various desalination configurations, higher permeate fluxes and higher salt rejection than conventional membranes were achieved. This has been ascribed to the high porosity, low transmembrane pressure and interconnected pore structure of the nanofibrous structure. In addition, the nanofibers layer prepared by electrospinning has high porosity and through-hole structure, so when the water molecules pass the skin layer, they can quickly evaporate [87]. This unique structure overcomes the defects in the traditional support layer prepared by phase inversion and can increase the flux of the composite membrane [87].

5 Conclusions and Future Trends

Application of carbon-based membranes is of tremendous advantage in the field of desalination. Recent advancements make it realistic to support that carbon-based

Table 1 The incorporation of CNFs and ENMs in TFN membrane for desalination application

Substrate	Additive	Optimum Performance	Application	References
PAN nanofiber	No	<ul style="list-style-type: none"> The optimum membrane exhibited 2.4 times permeate flux and rejection for almost 98% 	NF	[57]
PES nanofiber	Ionic liquid	<ul style="list-style-type: none"> Pure water flux increased by 50 LMH¹ MgSO₄ rejection is increased by 97.8% 	NF	[74]
Cellulose nanofiber	No	<ul style="list-style-type: none"> Exhibited higher water flux by 44.7 LMH MgSO₄ rejection is 99% compared with NF270 (97.4%) 	NF	[88]
PAN nanofibrous scaffold	No	<ul style="list-style-type: none"> The flux of low concentration NaCl solution (5000 ppm) can reach 8.53 LMH The salt rejection was higher than 99.5% 	Pervaporation	[87]
Cellulose triacetate	Carbon nanofibers–COOH	<ul style="list-style-type: none"> Pure water flux is increase up to 15.5 LMH Hydrophilicity is increase NaCl rejection is increase to 87% 	FO	[85]
Cellulose triacetate	Carbon nanofibers–NH ₂	<ul style="list-style-type: none"> Osmotic water flux is improved to 18 LMH Improved in membrane hydrophilicity Increase reverse solute flux by 0.5 g/m² h 	FO	[81]
PAN nanofiber	Silica	<ul style="list-style-type: none"> Osmotic water flux is improved approximately to 60 LMH Increased the reverse salt flux approximately to 8 g/m² h 	FO	[89]

BSA, bovine serum albumin; FO, forward osmosis; LMH, L/m².h; NF, nanofiltration; PAN, polyacrylonitrile; PES, polyethersulfone; UF, ultrafiltration.

membranes (CNTs, graphene, CNFs) might be one of the most reliable solutions to lower energy consumption for more sustainable development of desalination technology. However, several critical issues still remain open. One of the most critical challenges that may hinder their further development is associated with their complicated synthesis and processing [59]. Although the experimental studies from previous researchers have provided substantial evidence that carbon-based membranes can be used to fabricate high flux and high selectivity membrane, it is generally difficult to synthesize CNTs, CNFs, and graphene with well-controlled diameter, length, and tailored pore size.

Furthermore, to harness the exceptionally smooth channels of carbon membranes for fast water transport, the carbon-based membranes are likely to be chemically modified to increase its hydrophilicity [85,86]. However, chemical functionalization might deteriorate the carbon-based membranes' favorable transport characteristics. Therefore, further

understanding of the influences of various forms of functionalization on the potential application of carbon-based membranes in desalination is of great importance for the future development of this technology.

Another major hindrance to the readily usage of CNTs, CNFs, and graphene in desalination technology is their costs and operational issues [16]. As the manufacturing cost of carbon-based membranes might impose a significant cost to the overall desalination process, the combined savings benefited from the enhanced flux and salt rejection must be significantly evidenced or the application of carbon-based membranes in desalination will remain economically unfavorable. Moreover, the integration of this material into large area membranes and measurement of the transport characteristics remains the main hurdle. Challenges arise when scaling up the technology from lab scale to industrial level processes. Therefore, the platform for experimentally verifying these predictions is urgently needed. However, it is strongly believed that the scale-up issue will be resolved over time if the performance enhancement proves practically achievable.

List of Acronyms

CNT	Carbon nanotube
RO	Reverse osmosis
GO	Graphene oxide
SWCNT	Single-walled carbon nanotubes
MWCNT	Multiwalled carbon nanotubes
rGO	Reduced graphene oxide
GOF	Graphene oxide framework
NPG	Nanoporous graphene
ZCNTs	Zwitterion functionalized CNTs
PSB	Poly(sulfobetaine)
UCNTs	Ultralong CNTs
MCE	Mixed cellulose ester
VA	Vertically aligned
MD	Molecular dynamic
FO	Forward osmosis
PRO	Pressure retarded osmosis
TFN	Thin-film nanocomposite
MMMs	Mixed matrix membranes
PSF	Polysulfone
PVDF	Polyvinylidene fluoride
PCL	Polycaprolactone
PES	Polyethersulfone
PMMA	Poly(methylmethacrylate)
TFC	Thin-film composite
PVA	Polyvinylalcohol
UF	Ultrafiltration
PA	Polyamide
PAI	Polyamide-imide

PEI	Polyetherimide
ICP	Internal concentration polarization
NF	Nanofiltration
TFNC	Thin-film nanofibrous composite
ENMs	Electrospun nanofibrous membranes
CNFs	Carbon nanofibers
PAN	Polyacrylonitrile
IL	Ionic liquids
CD	Cyclodextrin
UCN	Ultrafine cellulose nanofiber
CDI	Capacitive deionization
ACNFs	Activated carbon nanofibers
BSA	Bovine serum albumin

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