Review:

Fabrication of Nanocomposite Membrane with Nanomaterial Filler for Desalination

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Abstract: *This review aims to provide a complete overview on the modification of polymer and biopolymer membranes into nanocomposite membrane materials. Fabrication of nanocomposite membranes is carried out by incorporating inorganic filler materials in nanoparticle sizes. Nanomaterials refer to the class of materials that consist of particulate substances with any dimension of less than 100 nm at least. The properties of nanomaterials include large specific surface area, crystalline structure, shape (that regulates most of its properties as well as their unique attributes), surface morphology, and assembling phenomena. This review primarily concentrates on the recent nanotechnology-based practices to enrich the outcomes of desalination on the footings of nanocomposites, developed practicing distinct nanomaterials. A classification for various forms of nanomaterials used for building nanocomposites has also been illustrated. Special emphasis has been given to the usage of nanocomposites constructed from several nanomaterials such as nanoparticles, nanotubes, nanoshells, nanofibers, nanocapsules, nanosheets, and quantum dots, and how these nanocomposites have been utilized for desalination purposes.*

Keywords: *desalination; filler; membrane; nanocomposite; nanomaterials*

■ INTRODUCTION

Water is one of the most essential substances on the earth. Consumption of large amounts of water is needed in various human activities such as agriculture, power generation, sanitation, drinking water needs, and others. Therefore, the lack of clean water is a major problem in various parts of the world, especially in coastal areas [1-4]. The shortage of clean water is caused by many factors, such as the rapid increase in human population over the last few decades which has resulted in a drastic increase in demand for clean water [4], unequal distribution of clean water [5], as well as climate change and rainfall accompanied by a decrease in the amount of surface and groundwater reserves [6]. Therefore, the latest innovations and solutions are needed to overcome this case. Desalination of seawater and brackish water is the alternative approach to produce potable fresh water, which can be utilized for agricultural and industrial applications [5]. The area of the earth's waters is estimated at 361 million km^2, which indicates that the outer seas control about 71% of the earth’s total area [6]. This is the basis that the potential of desalinating sea water into fresh water is the best solution. Several desalination industries have been established worldwide in recent years, and this trend is expected to continue [7-10].

Desalination is a technique for removing salt content from water, first discovered by Thomas Jefferson in 1791. Generally, seawater and brackish water are treated by desalination. Several seawater desalination methods have been researched and developed to obtain fresh water from salt. The desalination process can remove salts including Na_2SO_4, NaCl, $MgCl_2$, LiCl, and $MgSO_4$. Numerous techniques for seawater desalination have been carried out for a long time: thermal, electric, and pressure desalination [11-13]. Up to now, the three desalination techniques are still used, but each has its weaknesses, especially in terms of energy requirements in the operation process. Therefore, a desalination technique that has high performance and efficiency, but low cost is needed to convert seawater into fresh water.

Desalination using membrane materials is a desalination technique that is currently being developed. One method of desalination is reverse osmosis (RO), which can absorb water molecules but reject salts and organic compounds [10]. However, the RO operating system requires a relatively higher pressure when compared to desalination techniques using nanomembranes or so-called nanofiltration (NF) membranes. In addition, using low-pressure, NF membranes also has several advantages such as high flux, high rejection of divalent ions, and low energy consumption [11]. However, commercial NF membranes widely used in desalination processes based on synthetic polymer materials are relatively expensive. Therefore, using biopolymer materials as raw materials for membrane formation is a very appropriate solution. Several biopolymers such as chitosan [5] and cellulose [14-17] have been used as a matrix of membranes.

Many cellulose-based membranes or other biopolymer-based membranes have been modified to improve the membrane performance [18-21]. Some of these studies showed good performance in terms of rejection of salt which reached above 90% [22-24]. On the other hand, when the salt rejection percentage reaches a high value, the water flux through the membrane decreases. This phenomenon has been seen in several studies [23,25-26]. Of course, this is one of the main problems of this study. Ideally, in the application of desalination of seawater to fresh water, the membrane should be able to pass water with high fluxation and at the same time have a high rejection of salt compounds contained in seawater used as a feed solution. This phenomenon is caused by the accumulation of salt on the surface of the membrane which is faster when the water feed passes through the membrane, which in turn causes the rate of water flux to be suspended [27]. In addition, the decrease in water flux is caused by fouling on the surface of the membrane by materials other than salts such as bovine serum albumin (BSA), bacteria and other microorganisms. The fouling occurs because the salt filtering mechanism and water passage are in the same system. Fouling causes the permeating flux to decrease, so frequent membrane replacement and washing are required [28].

To improve the performance of biopolymer-based membranes in seawater desalination, especially in passing water molecules, retaining salts and antifouling; several modifications have been made by researchers [27-28]. One approach that has been widely developed is adding nanomaterial filler to the surface of a pristine membrane to produce a nanocomposite membrane [22-24]. In this review, the authors review in detail the role of nanomaterials in the development of polymer or biopolymer-based nanocomposite membranes. The authors describe the methods, results, and performance of membranes that have been combined with nanomaterials used in the desalination of seawater into freshwater. Thus, from this review, recommendations can be obtained regarding the types of nanomaterials that are most suitable for use in developing membranes that have the best performance for the desalination process.

■ METHODS USED FOR LITERATURE COLLECTION

This article review is compiled from the latest articles in the last 5 years. Articles were obtained from journals published by ScienceDirect, membranes journal, MDPI, Google Scholar, Springer Link, and others. Reviews are arranged based on the order of references and the order of topics. This paper comprehensively reviews the latest developments of these modifications in pristine membrane for desalination, especially about using nanomaterials in membrane modification alongside cutting-edge research advancements. The search approach is centered on principal keywords that are utilized in different combinations, such as surface coating, membrane modification, nanomaterials, polymers, and biopolymers.

■ MEMBRANE CHARACTERISTICS FOR DESALINATION

The membrane properties required for desalination applications are critical for achieving high efficiency in removing salt content from saltwater or brine. Desalination membranes must have a number of unique features in order to work properly in the process. High water flux and salt rejection are two key features of membranes that contribute significantly to good desalination performance [29]. Certainly, these two features are influenced by the membrane's intrinsic properties such as porosity, hydrophilicity, contact angle, solubility, thickness, thermal stability, and mechanical properties [30]. Therefore, research on membranes for desalination processes is highly focused on increasing water permeation while also considerably retaining salt.

Water flux refers to the flow rate of water that can pass through a membrane per unit surface area of the membrane. Several things that need to be considered regarding the properties of membrane water flux include water flux rate, water flux stability, and water flux control. The high rate of water flux means the membrane is capable of producing a lot of fresh water in a short time [31]. This is important to increase the productivity of the desalination process. The membrane water flux rate is greatly influenced by porosity, membrane thickness, operating pressure, and feed water quality. Regarding porosity, membranes with a high porosity allow water to pass through more easily because of their big pores [29]. Water flow is increased as a result of the faster water passage across the membrane. However, it is important to achieve the right balance in the degree of membrane porosity. High porosity can cause susceptibility to membrane fouling (blockage) because particles and contaminants can more easily pass through the membrane [30].

Apart from the intrinsic properties of the membrane, the presence of membrane fouling also affects the membrane's performance in terms of water flow and salt removal. Membrane fouling can be caused by pore blocking, cake formation, organic adsorption, inorganic precipitation, and biological fouling, leading to temporary or permanent flux decline [32]. Membrane fouling can be reversible and irreversible. Reversible fouling can be easily removed by rinsing the membrane, but irreversible fouling cannot be treated simply by rinsing due to the interaction between the foulant and the membrane surface [32-34]. In many cases, foulants not only interact physically with the membrane surface but also chemically degrade the membrane material [33]. Therefore, apart from focusing on increasing water flux properties and salt rejection, membrane development for desalination applications also needs to pay attention to membrane fouling aspects by developing membranes that have high antifouling properties. In numerous research, various methods, such as surface coating, surface grafting, surface bioadhesion, physical blending, and surface segregation, have been explored to prepare different kinds of antifouling membranes [34]. Membrane modification using nanomaterials as membrane fillers has attracted the attention of many researchers. In the next section, various nanomaterials that have been used for the improvement of membranes used for desalination purposes will be reviewed further.

■ ROLE OF NANOMATERIAL IN MEMBRANE MODIFICATION

Nanomaterials have emerged as a significant area of research in membrane technology for desalination applications. Their incorporation into membrane structures offers several advantages, such as increased selectivity, improved permeability, and enhanced fouling resistance. Nanomaterials, such as nanoparticles or nanotubes, can be integrated into the membrane matrix to increase the selectivity of the membrane. These nanomaterials can selectively block certain ions or molecules, allowing the passage of fresh water while rejecting salts and impurities. Nanomaterials can enhance the permeability of the membrane. By incorporating nanoscale materials, researchers have been able to create membranes with larger pore sizes, which allows for a higher flux of water [35-36]. This is particularly important for increasing the efficiency of the desalination process. Nanomaterials can also provide antifouling properties to membranes. They can create a more hydrophilic surface, making it difficult for foulants, such as organic matter or particles, to adhere to the membrane. This reduces fouling and extends the lifespan of the membrane [36].

Various nanomaterials have been used to modify polymer and biopolymer-based membranes used for desalination purposes, especially in improving water flux, salt rejection, and antifouling properties. The addition of nanomaterials to polymer membranes produces nanocomposite membranes that have better performance. Nanocomposites are materials formed from more than one solid phase that has at least one dimension on the nanometer scale. Usually, all solid phases are on the nanometer scale (1–20 nm) [37]. In addition to size, nanocomposites can also be seen from the aspect of composition which usually consists of organic, inorganic, or even a mixture of both compounds. The nanocomposite solid phase can be in the form of semi-crystalline, crystalline, amorphous or also in a combination of both [38]. Nanocomposites are formed in nanoparticle sizes (i.e., sizes between 1–100 nm), which makes nanomaterials very attractive because they work between bulk materials and atomic scale materials [39-40]. Nanocomposite membrane fabrication as a modified form of polymer-based membranes for desalination applications has been developed for a long time. From several studies that have been developed [41], nanomaterials added to polymer membranes are classified into four categories, namely nanoparticles, nanofibers, nanosheets, and nanotubes, as shown in Fig.1 and will be further discussed in the next section.

■ MODIFICATION OF MEMBRANE WITH NANOPARTICLES

Metal Oxide Nanoparticles

Modification of biopolymer-based membranes with the addition of metal oxide nanoparticles has been developed in recent years. Previous work [41-43] reported the synthesis of nanocomposite membranes based on cellulose and chitosan biopolymer materials incorporating TiO_2 and ZIF-8 nanoparticle fillers. The method used is the phase inverse method combined with the sol-gel method [42-44]. The results of the study showed that the addition of TiO_2 metal oxide had a significant effect on increasing water flux and salt rejection [42]. Kadhon et al. [43] reported the synthesis of nanocomposite thin-layer membranes modified by the addition of bentonite nanoparticles using the solvothermal method. Pure

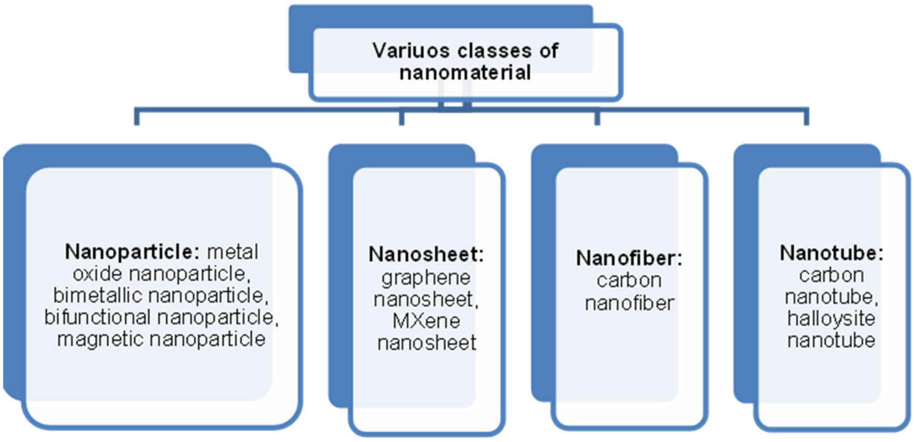

Fig 1. Various types of nanomaterials for nanocomposite fabrication

membranes were prepared by an interfacial polymerization process between *m*-phenylenediamine and trimesoyl chloride. The results showed that the addition of bentonite nanoparticles influenced the performance of the entire membrane under test conditions of 2,000 ppm NaCl, temperature of 25 °C, and pressure of 300 psi. The addition of 0.1% bentonite nanoparticles resulted in a water flux increase from 45.6 to 58.8 L/m^{2}h. Table 1 shows the several types of membranes with modified metal oxide nanoparticle materials.

Table 1 provides a valuable overview of various types of membranes that have been modified with metal oxide nanoparticle materials. These modifications play a crucial role in influencing the performance of the membranes, as demonstrated by the reported water flux and salt rejection data. Membrane PES/CA/PVP with TiO_2 nanoparticles exhibits an impressive water flux of 89.6 L/m^{2}h, indicating its ability to process a substantial volume of water [41]. The salt rejection rate of 76.8% suggests its effectiveness in desalination applications. PES-CNT composite membrane with ZIF–8 nanoparticles also exhibits an impressive water flux [21]. While the water flux is slightly lower at 53.51 L/m^{2}h compared to the previous membrane (PES/CA/PVP/TiO_2 membrane), it still demonstrates efficient water processing capabilities. A high salt rejection rate of 95% is achieved, making it suitable for desalination processes.

From some of the data in Table 1, it can be seen that TiO_2 nanoparticles are one of the nanomaterials most frequently used in modifying biopolymer-based membranes and synthetic polymers, and show promising results. Apart from that, the data in Table 1 also shows that the combination of TiO_2 nanoparticles with cellulose acetate biopolymer produces a higher water flux than the combination of polyamide (which is a type of synthetic polymer) with TiO_2. Overall, the overview in this section exhibits the versatility and potential of modifying membranes with metal oxide nanoparticles to enhance their performance in water treatment and desalination processes.

Bimetallic Nanoparticles (BNPs)

Bimetal nanoparticles (BNPs) are nanoparticles produced by combining two different metals [46-48]. Nanoparticles are made in various sizes, shapes, and structures. The number of BNPs depends on the ratio in which the two metals activate their respective physiognomies throughout the combination. BNPs play a significant part in nanotechnology due to their unique characteristics [49]. Conversely, desalination-related applications also employ BNPs. Recently, utilizing bimetallic organic framework (BMOF) as a precursor, porous carbon nanostructures were created for high-performance capacitive membrane deionization (CMDi) [46]. In this work, BMOFs of various molar ratios of Zn and Co were synthesized. The different particle sizes and degrees of graphitization of porous carbon derived from BMOF are obtained by looking at the molar ratios of Zn and Co. To evaluate the desalination performance, a series of batch-mode experiments using symmetrical electrodes passed through a water feed at different initial concentrations over a wide range of electrical potentials (0.8 to 1.4 V) were carried out. The results of the membrane test showed that the nanocarbon-Zn-Co-3 membrane showed the highest salt rejection value among all samples 32.29 mg/g [38].

Table 1. Several types of membranes with modified metal oxide nanoparticle materials

Type of membrane	NPs	Water flux (L/m^{2}h)	Salt rejection	Ref.
Polyethersulfone, cellulose acetate, and polyvinylpyrrolidone (PES/CA/PVP)	TiO_2	89.6	76.8%	[41]
Polyethersulfone and CNT composite	ZIF–8	53.51	95.0%	[21]
Cellulose acetate	TiO_2	47.42	-	[20]
Polyamide nanocomposite	TiO_2	9.10	95.0%	[42]
Polyamide	Silica nanoparticle	44.00	41.0%	[44]
Cellulose acetate	Silver nanoparticle	-	56.0%	[45]

In another study, Arumugham et al. [50] reported the dechlorination of trichloroethene (TCE) in water was accomplished using a new composite membrane with membrane holes loading acrylic acid microgels covered with Fe-Pd bimetallic nanoparticles. This membrane was organized by initially immobilizing acrylic acid microgels in poly(vinylidene fluoride) (PVDF) membrane pores. Then, fabrication of Fe-Pd bimetallic nanoparticles through *in situ* synthesis. Overall, the membrane immobilized by bimetallic Fe-Pd nanoparticles showed better dechlorination results than the membrane immobilized Fe. Assuming the effect based on the change of Fe or Fe-Pd metal nanoparticle on the TCE dechlorination process can be ignored. In this study, however the water flux and salt rejection were not identified, but the result of dechlorination indicated the good performance of the membranes.

Chlorination is one of the problems in the desalination process of seawater because the presence of chlorine in water bodies can damage the membrane. To prevent chlorination, membranes with reactive nanoparticles (Fe and Fe/Pd) fixed in a polymer film are fabricated [51]. This membrane was fabricated via a new method which is illustrated in Fig. 2. Firstly, through *in-situ* polymerization of acrylic acid, PVDF membranes were given poly(acrylic acid) functionality. The polymerization process took place in the aqueous phase, and the polymerization solution contained 40 mL of deoxygenated DIUF, 0.6 g, or around 1 wt.%, of potassium persulfate, 20 mL of acrylic acid, and 1 mL of ethylene glycol, which was used to cross-link the molecules. Secondly, to transform the-COOH into COONa form prior to Fe^{2+} ion exchange, PAA-functionalized PVDF membranes were immersed in a NaCl solution (5–10 wt.%) at pH 10 (adjusted with 0.1 M NaOH) for at least 3 h. Then, the membrane was washed with DIUF in a subsequent stage until the pH of the washing solution reached neutral. In the next step, the membrane was immersed for 4 h in a $FeCl_2$ solution with a pH of 5.5. The volume and concentration of the feed solution were typically 200 mL and 180 mg/L Fe^{2+}, respectively. Then, nitrogen gas was bubbled to reduce Fe^{2+} oxidation. Finally, the Fe^{2+} modified membrane was immersed in green tea extract (50 mL, 20 g/L) at pH 5, to ensure the creation of Fe nanoparticles. Table 2 shows on modification of a biopolymer or polymer-based membrane with the BNPs.

Magnetic Nanoparticles (MNPs)

Magnetic nanoparticles (MNPs) are nanomaterials that can be controlled through magnetic fields. In general, MNPs consist of two components. The first component plays a role in membrane function, while the other component plays a role in the scope of the magnetic field [55]. Studies that focus on desalination mostly use magnetite (Fe_3O_4) nanoparticles such as Ni and Co nanoparticles which have also been used for desalination of brackish water and seawater. The advantage of using MNPs in the desalination process is the presence of a magnetic field which helps the free separation of the post-desalination voltage current. In a recent study, Fe_3O_4 nanoparticles were prepared which were coated with gelatine and applied as a solute in a forward osmosis (FO) system [55]. In this study, the

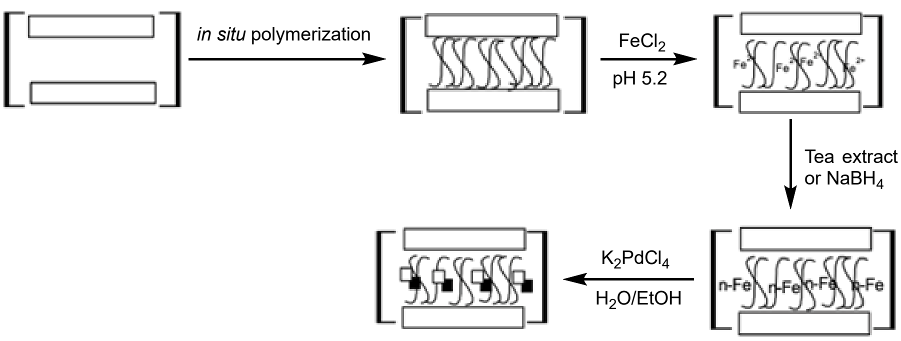

Fig 2. Schematic of PAA coating and Pd/Fe nanoparticle synthesis in PVDF membranes [51]

Table 2. Type of bimetallic nanoparticle-based nanocomposite membrane

Type of membrane	Synthesis method	Nanoparticle	Result	Ref.
PVDF membrane	*In situ* polymerization, coating	Fe and bimetallic Fe-Pd NPs	Water flux value of 2.2 L/m^{2}h and a rejection factor of 96%	[51]
Polyethersulfone (PES) ultrafiltration membranes	The phase-inversion procedure	Fe-Mn bimetallic oxides	The highest pure water flux (398 L/m^{2}h), exhibited the highest porosity (74%)	[52]
PVDF ultrafiltration membranes.	Bimetallic hydroxide prepared via reverse microemulsion, blended method used for membrane fabrication	Co-Fe hydroxide nanoparticles	The optimal water flux of the PVDF hybrid membranes (MD-10) reached 328 LMH, high BSA rejection rate (91.04%)	[53]
PVDF	Coating method	Fe-Pd bimetallic nanoparticles	The membrane immobilized by bimetallic Fe-Pd nanoparticles showed better dechlorination	[50]
Honeycomb-like porous chitosan bead	A facile one-step coprecipitating method	Mn-Fe bimetallic oxide nanoparticles	Transport of MnO_2-Fe_3O_4/CH consequently enhanced the catalytic performance	[54]

crosslinking interaction between Fe_3O_4 nanoparticles and gelatine was strengthened by the presence of *Persicaria bistorta* root extract which was used as a natural crosslinker, whose efficiency was then compared to that of a glutaraldehyde crosslinker.

The optimal results achieved through the utilization of *P. bistorta* root extract include a peak osmotic pressure of 1.01 bar, with concentrations of the crosslinking solution and gelatin being 14.25 mg/L and 7.70% w/v, respectively. Simultaneously, under comparable experimental conditions, the use of glutaraldehyde yielded an osmotic pressure of 1.06 bar, closely resembling the 1.01 bar achieved through the application of *P. bistorta* root extract. This observation provides empirical support for the efficacy of *P. bistorta* root extract as a favorable cross-linking agent [56]. Moreover, gelatin-coated Fe_3O_4 nanoparticles were utilized as the draw solution in the FO system as shown in Fig. 3. Meanwhile, 0.01 M NaCl solution and deionized water were employed as the feed solutions, maintaining an osmotic pressure differential of 1.0 and 1.5 bar, respectively. When deionized water was employed as the feed solution, the water flux attained a rate of 1.54 L/m^{2}h, whereas the utilization of NaCl as the feed solution resulted in an initial water flux of 0.62 L/m^{2}h. Subsequently, the gelatin-coated Fe_3O_4 nanoparticles experienced a twofold enhancement in their separation from water when subjected to a magnetic field. Nevertheless, the desalination process remained unaffected by the presence of the magnetic field [55].

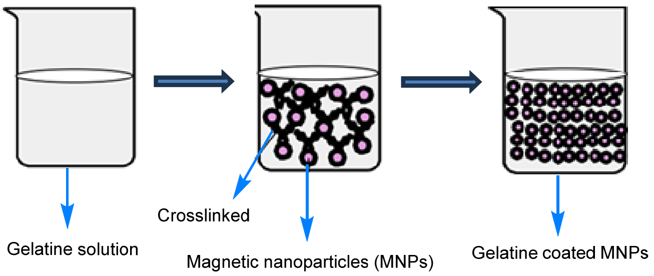

Fig 3. Crosslinking mechanism of Fe_3O_4 magnetic nanoparticles with gelatine [55]

In another study, modified PVC polymer-based nanocomposite membranes were synthesized by adding MNPs at different composition ratios. In addition to improving the standard performance of PVC membranes in desalination applications, the addition of Fe_2O_3 MNPs was carried out to improve the physiochemical characteristics of PVC membranes. The results of his research showed that the addition of magnetic Fe_2O_3 nanoparticles produced an exfoliated structure on the membrane and significantly increased the thermal stability of the membrane. Furthermore, the use of membranes in RO and FO devices to demonstrate increased antifouling properties, elimination of solutes and water flux of up to 10% with membranes modified with MNP material. Composite membranes with the addition of 6% MNPs gave optimal results with an increase in water flux of 55%, an increase in salt rejection of 40% for NaCl, LiCl, and $MgSO_4$, and by 37% for Na_2SO_4 when compared to pure PVC membranes. Also, a flux recovery of 89% (the highest increase) was received which exceeds the flux recovery obtained through pure PVC membranes [56].

Increasing the water flux in the desalination process with membranes is the center of attention for researchers in this field. In addition, the recovery of nanoparticles which are used as a towing component in assisting the fluxation of water through the membrane is also being studied continuously, especially in the application of membranes in the FO system. One of the advantages of using MNPs is their ability to perform very easy recovery of nanoparticles due to a magnetic field. Research by Ge et al. [57] shows how the comparison of the role of Fe_2O_3 MNPs coated with pectin and Fe_2O_3 MNPs without being coated with pectin in helping water fluxation, rejection of salt, and its ability to process nanoparticle recovery. The results showed that the synthesized nanoparticles have a cylindrical Fe_3O_4 crystalline phase and are superparamagnetic, which allows the recovery of MNPs using simple magnets. On the membrane with Fe_3O_4 MNPs without pectin coating, a water flux of 35.7 L/m^{2}h was obtained using deionized water as the feed solution. The use of feed NaCl solutions with different salinity levels was also studied in this research. The results obtained showed a decrease in water flux of 14, 58, and 76% observed for each 0.25 0.50 and 1.00% NaCl solution. Furthermore, the effect of pectin coating (0.5 and 1.0 g%) on the use of MNPs as a withdrawing component proved to have a significant effect on pure water flux. A water flux of 2.6 LMH was obtained against 5.5 g%, 55,000 ppm, NaCl solution as a feed solution indicating the potential use of MNPs as a withdrawal solution for brine management. Rejection of salt on all membrane samples showed a significant value that exceeded 95%.

The amount of water fluxation and membrane rejection of ions in the feed solution is strongly influenced by the surface condition (morphology) of the membrane [58-59]. The incorporation of MNPs material on the surface of the membrane through a layer-by-layer system can have a significant effect in increasing the performance of the membrane in the desalination process, especially with regard to the state of the membrane surface. Tayefeh et al. [42] reported the results of the synthesis of polyamide-based membranes modified with Fe_3O_4 and TiO_2 MNPs. This polyamide-based membrane is a type of commercial membrane that has been used extensively in conventional RO systems. In this study, polyamide membranes were coated with Fe_3O_4 and TiO_2 nanoparticles in organic solutions of trimesoyl chloride (TMC) and metaphenylene diamine (MPD). TiO_2 nanoparticles are used to modify the properties of the substrate. Fe_3O_4 nanoparticles were synthesized using the coprecipitation method which was then used in making TMC and MDP dope solutions that would coat the base of the polyamide membrane.

Polyamide membrane coating was carried out in 2 types, namely coating with TMC solution containing Fe_3O_4 nanoparticles at various concentrations of weight percent (wt.%) and coating with MDP solution containing TiO_2 nanoparticles, Fe_3O_4, and an alloy of TiO_2/Fe_3O_4, and without nanoparticles. The results of his research indicated that polyamide membranes could be modified into nanocomposite membranes modified by magnetic nanoparticles Fe_3O_4 and TiO_2 either through colloidal TMC solutions or MPD aqueous solutions through interfacial polymerization processes. The amount of nanoparticles added has an effect on the degree of interfacial polymerization and the type of nanoparticles can be a controlling factor in modifying the surface characteristics of the membrane. Increasing the number of Fe_3O_4 nanoparticles shows a decrease in the level of hardness (roughness) of the membrane and increases the hydrophilicity of the membrane [60].

The recently formulated MNPs are enveloped in three distinct hydroacids (citric acid, oxalic acid, and EDTA), each possessing abundant carboxylic groups. These carboxylic groups contribute to the enhanced dispersibility and ionization of the hydroacid-coated MNPs in water. These properties empower hydroacid-coated MNPs in aqueous solutions to generate osmotic pressures and FO water fluxes similar to conventional draw solutions, while concurrently demonstrating minimal reverse flux. The experimental findings suggest that enhanced FO performance was observed when employing hydroacid-coated MNPs with smaller dimensions in the FO process. When utilizing a model seawater solution containing 3.5 wt.% NaCl as the feed solution, the MNPs coated with citric acid yielded a water flux of 8.5 L/m^{2}h, surpassing reported values for MNPs used as draw solutes in FO for seawater desalination [60].

The development of membranes to obtain the best performance in overcoming fouling is carried out continuously. In a study by Zarandona et al. [61], the effectiveness of humic acid removal by a magnetic-sensitive Fe_3O_4-PES nanocomposite membrane is examined. The amount of end-capped Fe_3O_4 was varied by adjusting the concentration of suspended magnetite nanoparticles from 100 to 2,500 ppm. According to the study, Fe_3O_4 nanoparticles have a twisting action that lowers concentration polarization near the membrane's top surface, which lowers the risk of membrane fouling. The results of this study indicate that the membrane can maintain a normal water flux of 0.96 ($J = 32.8$ Lm^{-2} h^{-1}) with a salt rejection of 99.47%.

The ability of Fe_3O_4 MNPs to enhance the antifouling properties of membranes was also shown in a previous study by Arshad et al. [62] who reported the synthesis of a novel mixed matrix membrane. This mixed matrix membrane was synthesized using three types of self-synthesized magnetic nanofiller materials. The three nanofillers are neat Fe_3O_4, polyaniline coated Fe_3O_4 (PANI), and Fe_3O_4 coated multi-wall carbon nanotubes (MWCNT) mixed with PES solution in *N*,*N*-dimethylacetamide (DMAc). Smooth membrane morphology with a high degree of hydrophilicity of the mixed matrix membrane provides better antifouling properties. Magnetically fabricated PANI/Fe_3O_4/PES mixed matrix membrane with repeatability and outstanding resistance to whey protein fouling.

Apart from overcoming contamination by whey protein, MNPs also show the ability to prevent fouling due to the presence of oil in water which was reported earlier [63]. In this study, four coupling techniques for treating emulsions were formed by applying MNPs to the membrane separation process in various ways. The four coupling procedures outperformed direct ultrafiltration in terms of chemical oxygen demand (COD) rejection and flux for sample waste-cutting emulsions. In particular, the flux of the ideal process was 41.5 times higher than the control process, at 83 L/m^{2}h. The introduction of MNPs actively affected the membranes; the MNPs avoided direct oil-membrane contact via the coating of oil drops and a cake layer that formed on the membrane surface, which then enhanced ultrafiltration and improved flux significantly [64].

In a recent study, a chitosan-based membrane that was modified by incorporation of Fe_3O_4 MNPs was prepared via the casting solution method for antimicrobial activity. According to the result of research, a membrane with adding 10% Fe_3O_4 reached 43% inhibition in the case of *S. epidermidis*, while 98% was reached for *E. coli* bacteria. The positive charge of the amino group in chitosan interfered with the negative charge of cell membranes to produce an antibacterial effect by causing the loss of protein and other intracellular components. On the other hand, the ability of iron nanoparticles to interact with cell membranes and enter cells, resulting in cell damage and bacterial inactivation, was associated with the antibacterial action [65].

■ MODIFICATION OF MEMBRANE WITH NANOSHEET

Nanosheet materials are two-dimensional nanostructured materials with a thickness that oscillates between 1 and 100 nm. The incorporation of nanosheet material provides an opportunity to produce the expected membrane with the potential to modify the structural properties of the membrane, hydrophilicity, charge and charge density, thermal and mechanical stability, porosity, and roughness, which of course aims to improve the balance between membrane permeability and selectivity, besides it can minimize the tendency for fouling to occur which can be a major problem in membrane separation [66-68]. There are three categories of nanosheet membranes: blended, stacked, and monolayered. Single-layered nanosheets with intrinsically porous features or newly formed pores are used to separate ions. The incorporation of the nanosheet material into the polymer substrate produces a mixed matrix membrane (MMM), in which the distribution of nanostructures occurs in the dope solution during the fabrication process. The dissolution of nanostructures in the polyamide top layer of thin film composite (TFC) membranes is an alternative method. However, it would be regarded as a thin film nanocomposite (TFN) membrane in this instance [69-70].

The nanosheet material used in modifying the membrane has been developed by several researchers. Recent research by Nakagawa et al. [67] reported the results of the synthesis of a porous PVDF membrane modified by the addition of MoS_2 nanosheet material which was previously synthesized via the hydrothermal method using L-cysteine as a sulfur precursor. The synthesis results show that the process of forming nanosheet materials goes through several sequences that produce different structural dimensions of MoS_2 materials, namely nanospheres, nanoplatelets, and nanosheets which can be obtained under different chemical and physical conditions during blending. Besides the MoS_2 nanosheet material, graphene is an early example of a nanomaterial in the nanosheets category which is a two-dimensional material with a dimension size of 0.34 nm [67].

Graphene-based membrane fabrication is the focus of extensive ongoing research and has been commonly used for water purification applications [71-72]. Graphene-derived materials such as fullerene, graphene oxide (GO), and reduced GO (rGO) are conductors similar to metals, with zero band gaps in their 2D nanosheet structures. A widely researched substance with beneficial properties for various applications and a cheaper substitute for pure graphene is GO [72]. The structure of GO resembles a honeycomb lattice with functional groups around the holes or at the edges. Fig. 4 shows randomly distributed oxidized sp^3 and pure graphitic sp^2 patches in the basal plane of the nanosheet. Defects in GO nanosheets result from either their synthesis or later alterations. Compared to carbonyls, which are found at the edges and in holes, hydroxyl and epoxy groups have the most functionality [73].

The utilization of graphene oxide nanosheet materials has been developed in several areas, including applications in seawater desalination processes, altering the flexibility of tissue culture, altering DNA surfaces, and enhancing the quality of drug structure binding. Several researchers have employed graphene nanosheet material as an addition to enhance membrane performance [66,74-77]. In a particular experiment, a nanosheet was formed through the hybridization of exfoliated with GO (EHT/GO) via flocculation deposition. Furthermore, this nanosheet exhibited uniform dispersion in a PEI solution, serving as the aqueous phase in the interfacial polymerization process. An analysis of the deflocculation and flocculation technique employed for EHT/GO was conducted to understand its mechanism. This technique resulted in the formation of discriminating layers of thin films composed of the EHT/GO fusion membranes beyond the surface of the PES substrate [78]. Stylianakis et al. [66] identified the effect of graphene nanosheet barriers on acid resistance in TFC membranes. In this study, the impact of GO nanosheets implanted in a TFC polyamide (PA) membrane was compared to the impact of oxidized

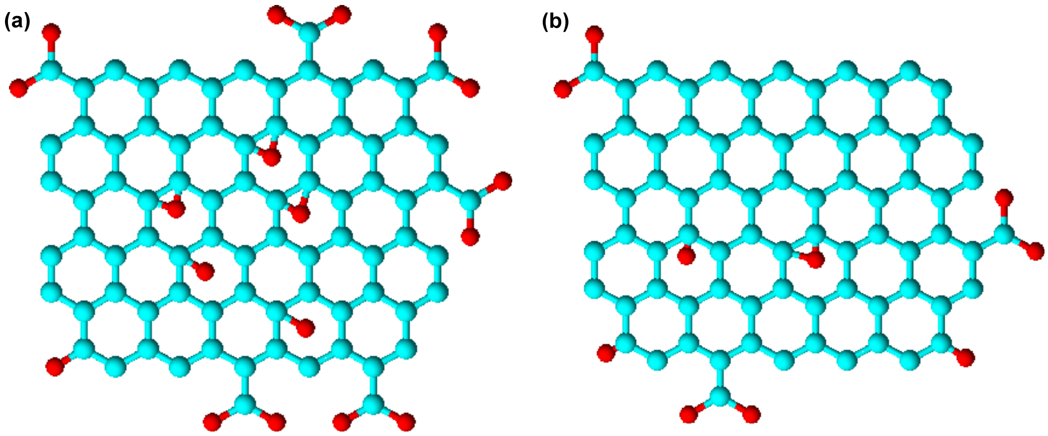

Fig 4. Molecular structures of different 2D nanomaterials GO and rGO nanosheets

single-walled carbon nanotubes on the membrane's acid resistance (o-SWNTs). The nanosheet shape of the GONs appears to have a major barrier effect that contributes to the acid resistance of the GONs-embedded membranes, along with a sacrifice role played by the PA layer that protrudes due to the addition of GONs and a reduction in acid reaction sites due to the hydrogen bonding between GONs and PA. Without taking into account GON aggregation, a high concentration (300 ppm) of the GONs was used to produce the TFC PA membrane, which resulted in 4.7 times longer membrane selectivity than the control TFC membrane.

The highly flexible material GO develops hydrophobic interlayer nanochannels and nanowrinkles with hydrophilic holes. This arrangement increases the water flux while causing electrostatic repulsion of materials like salt ions [79]. Longer hydrophobic nanochannels created by large GO sheets allow water to go through them more quickly, increasing the flow. When pressure is applied, the flexible GO nanosheets compact linearly. Due to this effect, the separation performance can be tuned so that the interlayer spacing changes in response to pressure [80]. Other nanomaterials, besides GO, that have been applied to membrane alterations for desalination applications as nanosheet materials are shown in Table 3.

■ MODIFICATION OF MEMBRANE WITH NANOFIBERS

Nanofibers are fibers with a diameter in the region of a nanometer. The most striking characteristics of nanofibers are their compellingly high surface area to volume ratio and significant porosity, which set them apart from the other nanomaterials [88-90]. Nanofibers can be synthesized using various methods, such as centrifugal spinning, phase separation, template synthesis, bicomponent extrusion, and self-assembly processes. Recently, some researchers developed nanofiber material through electrospinning technique. A large variety of spinnable materials, low spinning costs, precise and regulated processes, and straightforward manufacturing equipment are all benefits of ES technology [23-24]. Due to these benefits, ES technology has emerged as one of the most widely used techniques for the efficient manufacture of nanofiber materials. The electrospun nanofibers possess several exceptional qualities, including flexibility, a tiny diameter, a big surface area, a high aspect ratio, and special physiochemical properties [91].

Nanofibers provide several advantages in their application, so they are widely used in desalination and water purification processes. Recently, a novel cation exchange membrane for electrodialysis using a mixed matrix (MM) structure has been developed by incorporating carbon nanofibers (CNFs) attached to a polyvinyl chloride (PVC) membrane matrix. The incorporation of CNFs into the membrane structure resulted in the enhancement of various properties such as transport number, ionic conductivity, hydrophilicity, and potential, accompanied by a reduction in water requirements. Comparative analysis between the PVC-CNF membrane and a standard pristine membranes revealed that the PVC-CNF membrane exhibited a smoother surface than the pristine counterpart. This revealed a homogenous arrangement of resins and CNFs that were in charge of creating superior regions and

Table 3. Various types of nanosheet modifier of desalination membranes

Type of nanosheet	Matrix of membrane	Foulant	Highlights	Ref.
GO, rGO or MoS_2 Stacked	PES(S)	BSA, SA	MoS_2 has highest flux and FRR	[81]
Ti_3C_2Tx	PA(TF), PSf(S)	BSA	11.1% flux decrease, resistance against chlorination	[82]
Ti_3C_2Tx	$AgNO_3$, PVDF(S)	BSA, MB	FRR = 97%	[83]
WS2	Cellulose acetate	BSA	FRR = 99%	[84]
gCN(H)	PDA(C), PA(TF), PES(S)	BSA	FRR > 95%	[85]
PMSA-GO MMM	PVDF(S)	BSA	zwitterions incorporated, better dispersion of GO, FRR = 95.3%	[86]
gCN(H), rGO	TiO_2-NP, PVDF(S)	BSA	FRR = 86.1%	[87]

ensuring that counter ions could flow across the channels comfortably. The PVC-CNF membrane's connection to electrodialysis also resulted in the revelation of a ratio of 2.14 for K^+/Na^+ flow. Additionally, the mechanism for the transverse shift of Na^+ and K^+ across the membrane was explored by considering several variables, including hydration potential, hydration-free energy, hydration entropy, etc. The maximum transport number (> 93) and selectivity were achieved with membranes containing 0.5 wt.% CNFs [79].

A limited number of foundational studies were conducted to revise the *in-situ* modification of the surfaces of electrospun nanofibers. One specific investigation aimed to produce bead-free, superhydrophobic electrospun nanofibers through the incorporation of a hydrophilic polymer host with small quantities of oligomeric fluorinated polyurethane additive (FPA) and polysulfone (PSF) (3% FPA in the PSF mixture). The examination of FPA segregation to the exterior of nanofibers during the electrospinning process resulted in the development of nanofibers with enhanced hydrophobic properties. This discovery facilitated the identification of fluorine-rich surfaces and the optimization of mechanical attributes. Electrospun nanofiber mats' morphological and physical properties are affected by the addition of fluorinated additives. Increased FPA volume significantly affects the PSF mixture's surface rigidity by increasing the diameter, interfacial space, contact angle, and electrical conductivity. The luminosity of the light-emitting polymer (LEP) was observed to be higher in FPA incorporated PSF electrospun nanofiber membranes (ENMs) compared to PSF ENMs without FPA (PSF = 0), but it was later discovered that LEP level degraded as FPA concentration was increased [92]. Direct contact membrane distillation (DCMD), a desalination method, proved to be effective with a 4.5% FPA combined with PSF ENMs (PSF = 1.5, 3.0, and 4.5). When subjecting a feed solution of NaCl (30 g/L) to DCMD, the ENMs produced with 4.5 wt.% FPA in the PSF mixture (4.5 wt.%) achieved a permeate flux of 53.8 kg/m^{2}h at a feed temperature of 80 °C and a permeate temperature of 20 °C. The permeate's electrical conductivities were seen to be steady (5.1 S/cm) and to have extremely high dismissal efficiencies (> 99.99%) [93-94].

Another study newly synthesized Janus fibrous membranes by mixing NH_3-H_2O, 17-FAS, and PVDF powder to PVA/PAA solutions to manufacture PVA/PAA ENMs. After heat treatment, a high-water flux of over 27 L/m^{2}h and a high desalination efficiency of 100% were reached when the simulated hypersaline wastewater constituted of 3.5 wt.% of NaCl, 0.1 g of SDBS, and 1.0 g of lubricating oil was desalinated. Table 4 demonstrates various nanofiber membrane types used in the treatment of water.

■ MODIFICATION OF MEMBRANE WITH NANOTUBES

Recently, modification of polymer-based membranes with nanotube materials has also been developed. Nanotube materials used in modifying membranes can be classified into several types. However, in this review, the authors focus on types of carbon nanotube materials as a filler. Carbon nanotubes are carbon wall structures that have a diameter of under 100 nm. SWCNTs are a type of carbon nanotube that only contains a single layer of carbon atoms in the nanotube.

Table 4. Type of various nanofibers membrane

Type of membrane	Synthesized method	Water flux	Rejection	Ref.
Electrospun nylon 6,6/ZIF-8 Nanofiber membrane	Electrospinning	1967 L/m^{2}hbar	highest oil rejection (89%)	[95]
Electrospun silk Nanofibrous/PMMA	Electrospinning, interfacial polymerization, heat curing	3.62 LMH	99 %	[96]
FPA with PSF (3 wt.% of FPA in the blend of PSF)	Spinning	53.8 kg/m^{2}h (4.5 wt.% FPA + 4.5 wt% PSF) at 80 °C	> 99.9% (NaCl)	[97]
Superhydrophobic/organophilic electrospun/PDMS/PMMA	Electrospinning	-	alt rejection of 99.95%	[98]

On the contrary, MWCNTs are the name given to nanotubes that are created by the sequential arrangement of many single-walled carbon layers. Currently, two types of carbon nanotubes have been commercialized, namely vertically aligned carbon nanotubes (VA-CNT) membranes and mixed-matrix carbon nanotubes (MM-CNT) membranes. CNTs exhibit a wide range of outstanding qualities, including exceptional electrical conductivity, high thermal conductivity, and incredible tensile strength [99]. A general comparison between the VA-CNT membranes and the MM-CNT membranes is shown in Table 5. In this review, the authors will examine the research on these two types of carbon nanotube membranes.

A study conducted by Yousef et al. [100] reported the ability of VA-CNT membranes to significantly improve the salt rejection rate. In this study, carbon nanotubes were grown on Si wafers and coated with poly(dimethylsiloxane) (PDMS) 50% (w/b), then sliced and fabricated into VA-CNT membranes. The main causes of salt rejection are the inner diameter of CNTs and the surface charge of the membrane. Because the PDMS is negatively charged, the Na^+ ions are attracted to the surface and are trapped, which lowers the salt concentration. This technique displayed an astounding result of 96% salt rejection by all the examined membranes. The results of this study indicate that the density of carbon nanotubes affects the flux velocity of the feed solution through the membrane and enhanced anti-biofouling properties without damaging the salt rejection. The membrane with the highest CNTs density (1×10^{11} cm^{-2}) resulted in a maximum flux of 1203 L/m^2h.

Another study investigated the water flux and salt rejection capabilities of membranes incorporated with carbon nanotubes through computational studies using molecular dynamics simulations [101]. The study discovered that salt rejection is significantly reliant on the hydrostatic pressure applied, indicating that more rejection can be accomplished with larger tubes than previously thought, whereas membrane permeability depends on salt content. The optimal CNT size for desalination applications was discovered to be around 1.1 nm in diameter, producing good permeability and high salt rejection. The effect on the CNT membranes toward salt rejection was investigated with used pressure ranging from 5 to 400 MPa.

Table 5. Comparison between VA-CNT and MM-CNT [99]

CNT (VA-CNT)	MM-CNT membrane
Vertical CNT arrangement	Mixed CNT arrangement
Compact CNT network	CNT network loosely fit
High water flux rate	Moderate water flux rate
Complicated fabrication	Simple fabrication

In other studies, the synthesis of membranes with the type of MM-CNT membrane is also being developed for desalination applications. The use of matrix-polymer functionalized carbon nanotubes in the construction of water permeation routes in the membranes has been shown in the research by Tseng and Liu [102] to be a successful method for increasing the water permeation fluxes of the pervaporation desalination membranes. When using PVA as the membrane fabrication matrix polymer, a small amount of PVA-functionalized CNTs (0.06 wt.%) successfully increases the water permeation fluxes of the PVA membranes from 1,630 to 6,140 g/m^2h (feeding solution: 3.5 wt.% NaCl at 25 °C) without sacrificing salt rejection, equating to a 3.77. The membrane can also be used with a 15 wt.% NaCl solution of concentrated salt. Some types of carbon nanotube membranes and their applications in the desalination process are presented in Table 6.

In another research, a novel RO nanocomposite membrane based on polypyrrole (PPy) coated MWCNTs was synthesized for brackish water desalination. The combination of MWCNT and PPy was mixed in *m*-phenylenediamine solution at various concentrations, so that a polyamide RO membrane was obtained using the interfacial polymerization method. Based on the results, the MWCNTs-PPy membrane showed an increase in water flux from 21.5 to 30.4 and 34.3 L/m^2h, respectively. Moreover, the blending of the two materials can improve the antifouling properties, especially in the 0.002 wt.% oxidized MWCNTs-PPy membrane [110].

A recent study by Roslan et al. [111] reported a modification of a novel nanofiltration membrane with

Table 6. Different types of carbon nanotube membranes and their performance

Type of membrane	Target salts	Removal efficiency of salt	Reduction of water flux	Ref.
Hybrid aerogel NSA (N-Gr-MWCNT) (Gr-MWCNT)-GO membrane	NaCl	Water flux 89.6 L/m^{2}h	20.1-22.5 mg/g	[103]
CNTF-Porous ESF membrane	NaCl	> 99.9%	stable	[104]
PPy functionalized MWCNTs-PA membrane	NaCl	-	34.3 L/m^{2}h	[105]
PVA-assisted (HNT) TFN membrane (halloysite nanotube type)	Na$_2$SO$_4$	97.8% at 4 bar pressure	34.5 Lm^{-2}h^{-1}bar^{-1} (0.05% TFN membrane)	[106]
Nitrogen doped quantum dots-HNT membrane	NaCl	44 L/m^{2}h	20.05 mg/g (EC at 1.2 V in 500 mg/L NaCl solution)	[107]
N-GOQD	NaCl	97.0%	3.36 LMH/bar	[108]
Na-CQD	NaCl	98.6%	2.56 LMH/bar (1 wt.% Na-CQDs)	[109]

the incorporation of a double-walled carbon nanotube (DWCNT) interlayer supported by a PES substrate. The development of the polyamide layer was significantly influenced by the DWCNT interlayer when the DWCNT dosage was increased. DWCNT was modified with dopamine hydrochloride to help it firmly attach to the substrate surface. Generally, increasing the dose of DWCNT enhanced the water flux but lowered the salt rejection. The phenomenon of increasing water flux with high doses of DWCNT is influenced by three factors. Firstly, according to the findings of XPS, contact angle, and zeta potential, DWCNT was added to the membrane to increase the amount of carboxyl groups on the NF membrane, which enhanced the hydrophilicity of the membrane surface. Secondly, the addition of DWCNT increased the specific surface area, which provided more penetration sites for water molecules passing through the polyamide layer and improved the transmission efficiency.

The last reviewed study related to the synthesis of VACNT wall membrane with its ultrafast water permeability shows a great promise as a novel support layer for membranes because of its ultrahigh porosity and hydrophobicity. This RO membrane delivers a flux of 128.6 L/m^{2}h and 98.3% salt rejection at 15.5 bar, opening an avenue for a leap over the water permeability at the rejection level that has been pegged at 5 L/bar m^{2}h over the past ten years. Formation of a thin PA layer by a modified preparation method and ultrahigh porosity of VACNT support membrane are the main factors for the high-performance RO membrane [112].

■ CHALLENGES AND FUTURE OF NANOMEMBRANE

Based on the results of several studies reviewed previously, most membranes with modified nanoparticles, nanosheets, nanofibers and nanotubes still show weaknesses in terms of membrane selectivity related to the balance between water flux performance and salt rejection. Several studies show that there is a contradiction in values between water flux and salt rejection, where when the water flux value is high, the salt rejection increases as the filtering time increases, and when the salt rejection is high, the water flux decreases. One of the main factors causing this is the fouling of the membrane by various types of foulants found in seawater. From the intrinsic aspect of the membrane, fouling occurs because the membrane separation system is one-way so when the membrane surface is covered by foulant, water permeation will decrease. Therefore, to overcome fouling, the desalination membrane filtering system should be multidirectional. The water permeation and salt retention systems should not be in the same area. Of course, this is a challenge for researchers in this field who must find a solution.

In the future, in developing membranes for seawater desalination applications, researchers must consider separating the function of the membrane part between the water flux system, salt filtering, and other components in the seawater feed solution. Membranes, with the addition of modified nanomaterials, need to be combined with a multilayer membrane system that can separate membrane functions. Several studies related to the development of membranes with multilayer systems have been developed and have shown positive results in terms of membrane selectivity.

CONCLUSION

Membrane modifications have been carried out using nanomaterials as filler materials. Some of the nanomaterials that have been used such as metal oxides, magnetic, bimetallic, and bifunctional nanoparticles are proven to improve membrane performance. The costs and time spent on the desalination process have been greatly reduced by the nanomaterials mentioned. Modification of biopolymer-based membranes can be carried out through several methods such as phase inversion, sol-gel, interfacial polymerization, dip coating, spin coating, electrospinning, etc. Zeolite, carbon nanotube, and graphene-based nanomaterials are currently being used to make further breakthroughs. Nanotechnology could be used to improve these traditional desalination methods, which would also help with applications like waste purification and waste-water treatment by improving effectiveness and lowering costs.

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CONFLICT OF INTEREST

In compiling this review, the author does not have a conflict of interest that could intentionally provide benefits to other parties. The preparation of this review is purely because the author wants to review more deeply regarding membrane modifications for desalination applications.

AUTHOR CONTRIBUTIONS

Muhammad Nur Alam, Indah Raya, Ahyar Ahmad, Paulina Taba, and Suriati Eka Putri designed research ideas, conducted review, and wrote manuscripts. Harningsih Karim did the review and editing.

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