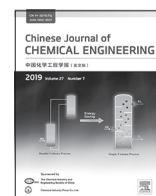




Contents lists available at ScienceDirect

Chinese Journal of Chemical Engineering

journal homepage: www.elsevier.com/locate/CJChE

Perspective

Emerging R&D on membranes and systems for water reuse and desalination



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ARTICLE INFO

Article history:

Received 22 February 2019

Received in revised form 7 April 2019

Accepted 10 April 2019

Available online 17 April 2019

Keywords:

Membrane technology

Water reuse

Desalination

Hybrid system

Nanomaterials

ABSTRACT

Sustainable production of clean water is a global challenge. While we firmly believe that membrane technologies are one of the most promising solutions to tackle the global water challenges, one must reduce their energy consumption and fouling propensity for broad sustainable applications. In addition, different membranes face various challenges in their specific applications during long-term operations. In this short review, we will summarize the recent progresses in emerging membrane technologies and system integration to advance and sustain water reuse and desalination with discussion on their challenges and perspectives.

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1. Introduction

The growth of population and industries has driven the global water demand rapidly. Seawater desalination and water reuse are essential solutions to meet the high water demand and sustain our prosperity. However, the current dominant membranes for water reuse and seawater desalination have drawbacks such as fouling, high energy consumption and weak resistance to chlorine. Breakthroughs on membrane materials and system design are urgently needed to increase their performance. This review aims to (1) briefly introduce the recent progresses on membrane science and materials with great sustainability and (2) highlight the emerging R&D directions for next-generation membranes in the fields of water reuse and seawater desalination. We will start from thin-film composite (TFC) and thin-film nanocomposite (TFN) membranes because they are not only the dominant membranes for reverse osmosis (RO) seawater desalination but also for forward osmosis (FO) and nanofiltration (NF) in the last two decades [1–7]. Since the fabrication processes for TFC membranes have been well established, any breakthrough on membrane materials can be potentially scaled up easily for commercialization. Then, we will discuss other emerging membrane technologies such as membrane distillation (MD) and hybrid systems for sustainable water reuse and seawater desalination.

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2. Optimization of TFC and TFN Membranes

Since Cadotte and Petersen made the first TFC membrane based on the reaction between phenylene diamine and trimesoyl chloride, extensive efforts have been made to develop better TFC and TFN membranes for RO and other osmosis processes [2–25], including surface modifications [8–11], manipulation of interfacial polymerization process [12–15], solvent treatment [16,17], incorporation of aquaporin (AQP) [18,19] and porous nanoparticles [20–25]. Among them, the modification of the selective polyamide layer has attracted greater attention since it is the dominant factor in determining the water permeability, salt rejection and energy consumption for osmosis membranes. The incorporation of AQP and porous nanoparticles into the polyamide layer with enhanced water transport properties can be considered as biomimetic or nature mimic membranes. The major difference between AQP and porous nanoparticles is that the former is a water-channel protein produced from living species, while the latter are synthesized from either inorganic or organic and inorganic hybrid materials. Since proteins would denature under harsh environments [26–29], the AQP-based membranes may have a narrow operation window and low sustainability. They also need special storage conditions to rejuvenate and keep their separation performance. Hydrophilic porous nanoparticles may function as good as AQP but with superior sustainability.

Therefore, researchers have incorporated a variety of porous nanoparticles, such as zeolites [20–24], carbon nanotubes [25] or

metal–organic frameworks (MOFs) [30–33], into the polyamide layer as TFN membranes for seawater desalination and water reuse. Some of them showed encouraging results. For example, Dong *et al.* incorporated 0.15 wt% NaY zeolite nanoparticles into the polyamide layer during interfacial polymerization. The resultant RO membranes showed a pure water permeability of $48.5 \text{ L}\cdot\text{m}^{-2}\cdot\text{h}^{-1}\cdot\text{MPa}^{-1}$ ($\text{LMH}\cdot\text{MPa}^{-1}$), a salt permeability of $0.913 \text{ L}\cdot\text{m}^{-2}\cdot\text{h}^{-1}$ (LMH) and a NaCl rejection of 98.8% [23]. Duan *et al.* embedded hydrothermally stable MOF-zeolitic imidazolate framework-8 (ZIF-8) into the selective polyamide layer of RO membranes for water desalination. They obtained pure water permeability, salt permeability and NaCl rejection values of $33.5 \text{ LMH}\cdot\text{MPa}^{-1}$, 0.79 LMH and 98.5%, respectively [30].

In addition to porous nanoparticles, non-porous nanoparticles such as silica [34], titanium dioxide (TiO_2) [35], nanosilver [36] and graphene oxide (GO) [37,38] also exhibit synergistic effects when being embedded into the polyamide layer of RO membranes for seawater desalination. These positive results may arise from the fact that these non-porous nanoparticles possess many functional groups with high affinity towards water molecules. They may also interact with the monomers and form the polyamide layer together. In addition, some of these 3-dimensional (3D) nanoparticles could behave like “meatballs” strewn among the polymer chains and force the polymer chains apart with a higher free volume and an increased permeability [39].

Therefore, future researches on TFC and TFN membranes should focus on the incorporation of porous or non-porous nanoparticles in the polyamide layer. Porous and water stable nanoparticles with tunable particle size, pore size and chemical functionality are preferred. For example, UiO-66 is a very promising porous nanoparticle because it has excellent chemical and thermal resistance. Its sub-nanometer pores of $\sim 0.6 \text{ nm}$ may behave like water channels to facilitate water transport while blocking hydrated cations such as Na^+ , K^+ , Ca^{2+} , Mg^{2+} , etc. Its hydrophilicity and favorable stability in water also make it suitable to be incorporated into the polyamide layer of RO membranes for seawater desalination [30–33,40]. Carbon quantum dots (CQDs) are another example of non-porous nanoparticles [41–46]. By incorporating hydrophilic nanocarbon dots with a quantum size of 6.8 nm into the polyamide layer, Li *et al.* showed that the resultant RO membranes had pure water per-

meability, salt permeability and NaCl rejection values of $56 \text{ LMH}\cdot\text{MPa}^{-1}$, 1.05 LMH and 98.8%, respectively [41]. These interesting results may arise from (1) high affinity between CQDs and water molecules, (2) strong interactions between them and monomers for interfacial polymerization and (3) the reduced chain packing among polymer chains [39]. In addition, CQD-modified TFC membranes exhibited higher chlorine resistance because the hydrogen bonds between CQDs and the polyamide layer tightened the polymer chains and inhibited the replacement of amidic hydrogens. Furthermore, the charge exclusion between COO^- from electron-rich CQDs and active chlorine (OCl^-) not only impeded the contact of OCl^- with the membrane surface but also reduced the diffusion rate of OCl^- in the polyamide matrix [45]. In terms of fabrication method for interfacial polymerization, Ma *et al.* recently found that the addition of bicarbonate with the aid of ultrasound during the thin-film polymerization can lead to remarkable improvements in both water permeability and salt rejection [47]. This breakthrough may result in next-generation RO membranes with a better permeability–selectivity relationship.

3. Hybrid Systems for Energy-efficient Desalination Processes

Although there have been many progresses in the development of RO membranes, high pressure pumps and energy recovery devices, the current seawater RO (SWRO) plants consume 2 to $3.5 \text{ kW}\cdot\text{h}$ to produce 1 m^3 of freshwater [2,7]. A major part of the operating expenditure (OpEx) comes from the high energy consumption because a high hydraulic pressure must be employed to overcome the osmotic pressure of seawater. In addition to continuously improve the energy recovery devices, there are mainly two methods, as shown in Fig. 1, to further reduce the energy consumption of SWRO: (1) dilute the seawater feed in order to increase the recovery or reduce the RO operating pressure and (2) recycle and/or reuse the energy from the brine [48,49].

Forward osmosis (FO) can be integrated with SWRO by using seawater and wastewater as the draw and feed solutions, respectively [50–52]. FO takes the advantage of the osmotic gradient and induces water transport across the semi-permeable membranes, and therefore requires no high pressure hydraulic pumping and has more reversible fouling [53–55]. Since the seawater feed is osmotically

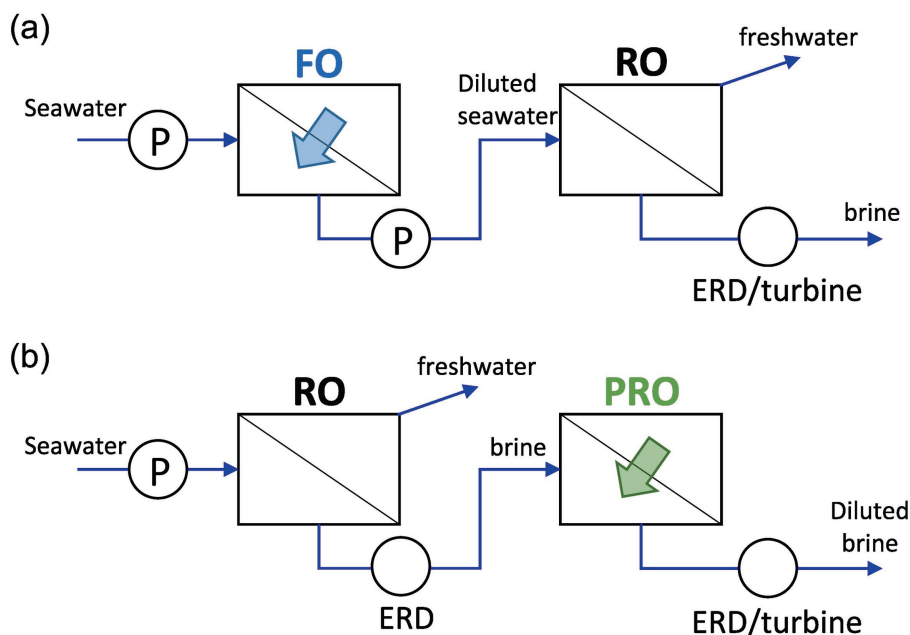


Fig. 1. (a) FO–RO and (b) RO–PRO integrated processes for more energy-efficient desalination.

diluted by the wastewater with the aid of FO, the osmotic pressure of seawater becomes lower and hence SWRO can be operated at (1) a much lower pressure to achieve the same seawater recovery or (2) a much higher process recovery if a constant pressure is employed. More favorably, FO not only dilutes the salts but also reduces the concentration of foulants in the seawater feed. Therefore, less RO membrane fouling and scaling are expected in the integrated process. The FO membranes can also act as a pre-treatment of the wastewater that mixes with the feed seawater and reduces the pre-treatment cost of SWRO. However, the wastewater source needs to be carefully chosen to reduce the membrane fouling in FO [56,57]. It was reported that fouling caused 25% and 50% declines in water flux when biologically treated wastewater and primary effluent were used as FO feeds, respectively, while the fouling from the secondary effluent was negligible [56]. Moreover, recent techno-economic analyses revealed that the energy saving from the FO–RO integration would significantly outrun the additional capital expenditure (CapEx) required for the integration, resulting in a 16% reduction in the total cost of seawater desalination [58,59]. These findings assure both the technical and economic feasibilities of the FO–RO integration.

The RO brine after SWRO not only possesses a high pressure, but also contains a high concentration of salts and a warm temperature of about 30 °C. In addition, it is relatively clean because of the SWRO pre-treatment. The conventional energy recovery devices (ERDs), such as pressure exchangers, only recover the energy from its high pressure. The high osmotic energy of the warm RO brine is left unutilized. Loeb *et al.* pioneered in harvesting the osmotic energy from the concentrated brine via pressure-retarded osmosis (PRO) [60,61]. Statkraft (Norway) set up the first pilot-scale osmotic power plant [62]. However, both failed due to the lack of effective membranes [63]. Severe membrane fouling was also observed in the Statkraft pilot [48,64]. Significant advances in PRO membranes with higher transport properties, higher mechanical properties and lower structural parameters have been made recently [46,63–66]. For example, Zhang and Chung synthesized TFC membranes upon polyethersulfone (PES) hollow fiber supports and found that a lower salt permeability (*i.e.*, towards a lower internal concentration polarization (ICP)) was essential to enhance the maximum power density to as high as 24.3 W·m⁻² when 1 mol·L⁻¹ NaCl and deionized (DI) water were used as feeds [65]. Later, the PES-based PRO membrane comprising QDs in the polyamide layer displayed a power density of 34 W·m⁻² at 2.2 MPa using the same feed pair [46]. The PES-based PRO membrane was further improved by incorporating CaCl₂ into the support layer, it showed a power density of 38 W·m⁻² at 3 MPa using DI water and 1.2 mol·L⁻¹ NaCl as the feed solutions [66].

It was estimated that by effectively recovering the osmotic energy from the RO brine, the specific energy consumption of SWRO could be reduced by 40%–50% to approximately 1.1 kW·h·m⁻³ [50,67,68]. However, membrane fouling in PRO is much more severe and complicated than those in FO and RO because the porous support of TFC membranes faces the wastewater feed. The accumulation of foulants would take place deeply inside the porous and tortuous substrate beneath the polyamide layer [69–77]. It not only reduces the power density, but also creates difficulties for effective cleaning. For example, when a municipal wastewater retentate was utilized as the feed solution and a 0.8-mol·L⁻¹ NaCl solution at 2 MPa was used as the draw solution, the power density of the TFC-PES membrane dropped from 21.6 W·m⁻² to 5.7 W·m⁻² due to the severe calcium phosphate fouling inside the porous membrane substrate [69,70]. By means of acidifying the municipal wastewater retentate and conducting coagulation pre-treatment to mitigate the scaling, the power density was recovered to 9–13 W·m⁻² [69,70]. Therefore, to sustain the high power density of PRO membranes, (1) study of fouling

mechanisms [69–73], (2) development of effective pre-treatments [69,70,72], (3) development of new membrane cleaning strategies [73,74] and (4) design of anti-fouling membranes [44,75–77] are the four directions for future PRO researches. Despite of the challenges, both RO–PRO pilots at the National University of Singapore and Japan Megaton water project generated average power densities higher than the breakeven power density of 5 W·m⁻², which was estimated set by Statkraft for PRO to be commercially feasible [78–81]. Therefore, the RO–PRO integration has the great potential to make SWRO energetically and economically more competitive.

Moreover, MD-PRO and SWRO-MD-PRO have also been proposed because membrane distillation (MD) produces a more saline and warmer brine than that by SWRO [50,82–84]. Thus, the hybrid MD-PRO system could almost double the maximal power density when the draw solution to PRO increased from 0.6 mol·L⁻¹ to 2 mol·L⁻¹ [82]. Currently, SWRO-MD-PRO is a less popular hybrid system than SWRO–PRO. However, it can potentially further reduce the cost of seawater desalination if the heating source of MD can be cheaply acquired [50,83,84]. The Korean GMVP project adopted this SWRO-MD-PRO hybrid system with aims to recover valuable resources from SWRO brine and mitigate its adverse environmental effects during the brine discharge [83,84].

Since FO has a lower fouling propensity, a FO–PRO hybrid system has been proposed [85]. The inter-loop solution serves as both the draw solution in FO and the feed solution to PRO. By choosing the NaCl concentration of the inter-loop solution as 0.1 mol·L⁻¹, a power density greater than 5 W·m⁻² could be achieved. Meanwhile, the concentrated brine used in PRO as the draw solution could be diluted back to the seawater level for easy disposal. Under this condition, FO possessed a comparable water flux with conventional pretreatment methods using pressure-driven membrane processes at a possibly lower operation cost.

4. Revitalized Research on Membrane Distillation

Membrane distillation (MD) used to be considered as an energy intensive process for seawater desalination. However, with the advances in solar panels, nanotechnology and membrane technology, it has been reborn as an emerging technology that draws significant attention from both academia and industries. In addition to the traditional MD-crystallizer (MD-C) systems [86–90], and aforementioned MD-PRO and SWRO-MD-PRO [82–84], totally new integrated processes such as FO-MD, freeze distillation (FD)-MD, FD-vacuum membrane distillation (VDM), FD-MD-C, submerged vacuum membrane distillation crystallization (VMDC) and MD-solid hollow fiber cooling crystallization (MD-SHFCC) systems have been recently proposed for a wide range of applications that totally open up new perspectives for future MD research [90–96]. Using FO as an example, it is not a low energy process unless there is no need to regenerate the draw solution [3,48,97]. In addition, a draw solution with a higher osmotic pressure may encounter more difficulties to be regenerated using the traditional pressure-driven processes. To economically regenerate the draw solution, the FO-MD integration provides a potential solution [98–101]. With the aid of solar energy and waste heat, FO-MD may be more cost-effective than those using RO or NF to recycle the draw solution [101].

The revitalization also comes because MD combines some uniqueness of both membrane and distillation processes. It is modular and space-saving, operates at a mild temperature (50–90 °C) and pressure (atmospheric pressure or vacuum), and has theoretically 100% rejection to salts. Different from other pressure-driven membrane processes, its separation performance is less affected by the feed concentration and its energy source can be solar energy or waste heat instead of electricity. Therefore, MD is one of the key

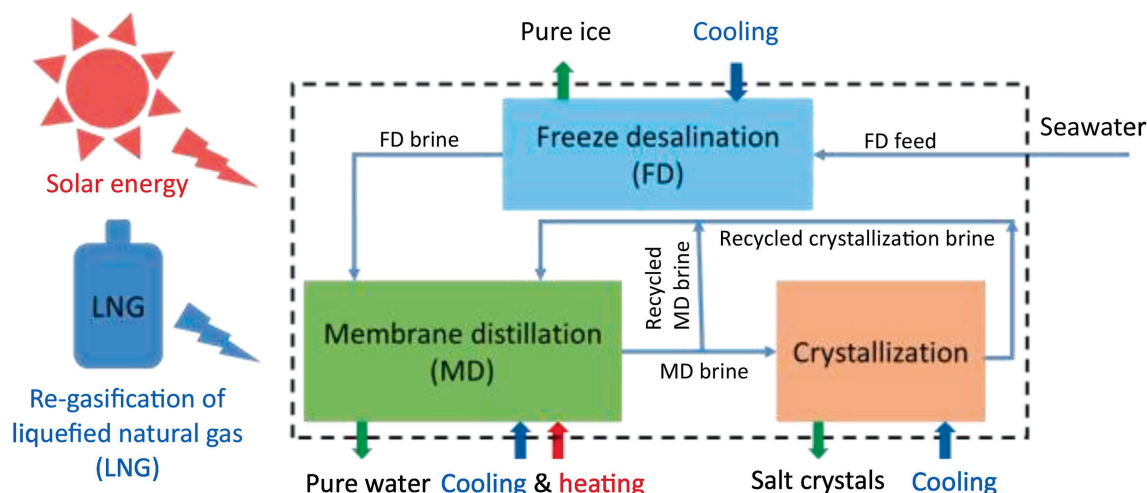


Fig. 2. An economical hybrid ZLDD system consisting of FD, MD and crystallization powered by solar energy and LNG, copyright (2019) Elsevier.

process elements to achieve the goal of zero liquid discharge desalination (ZLDD) that has characteristics of high water recovery, zero waste generation, wide energy source and valuable salt production [96,102,103]. Fig. 2 illustrates a hybrid ZLDD system consisting of FD, MD and crystallization powered by solar energy and liquefied natural gas (LNG) [104]. Briefly, FD is the first step to harvest clean ice by freezing seawater using the cold energy during the re-gasification of LNG, then, the concentrated FD brine is treated by MD with the aid of solar energy to increase the total water recovery [91,96,105]. Finally, salt crystals are recovered from the leftover brine by means of a crystallizer and the remaining brine is recycled and concentrated again in the MD unit [86–89,92,94].

To develop a sustainable solution and meet the zero liquid discharge, one must convert seawater into pure water and valuable salts without generating a waste stream. The concept of ZLDD becomes more realizable today if MD is included in its process. The MD unit is not only able to produce pure water but also brings the brine to a much high concentration for easy crystallization in the subsequent crystallizer. On the other hand, in ZLDD systems, MD concentrates the brines to a near-supersaturated status. Though high salinity has less impact on the driving force for MD than other pressure-driven membrane processes such as RO, scaling is a serious issue at such a high salt concentration [88,94,106,107]. Moreover, temperature and concentration polarization also accelerate the crystal growth on the membrane surface. Inorganic scales can block the vapor transport path, reduce the surface energy and eventually lead to the membrane wetting. Therefore, ground-breaking designs of MD membranes and modules to mitigating the scaling issue and novel restoration techniques to regenerate MD membranes must be advanced in order to have sustainable ZLDD systems.

Traditionally, microporous PP, PVDF and PTFE membranes and their modified ones are employed as MD membranes [86–90,92,108–110]. They have good short-term performance, but their long-term performance is a concern because of scaling, wetting and complexity of the feed streams. Since nowadays both seawater and wastewater may comprise trace amounts of low surface tension materials due to oil-tank leakages and industrial pollutions, future MD membranes must have characteristics of omniphobicity so that they are robust and able to repel both high and low surface tension compounds [111–114]. Two factors are important to achieve surface omniphobicity; namely, the re-entrant structure and the low surface energy [114]. The former is often created by nano/microparticle deposition while the latter is realized by coating, mixing, or plasma-treating using fluorinated materials. Omni-

phobic membranes have been developed in various configurations including flat sheets, hollow fibers and nanofibers on different support materials such as glass fibers and polyvinylidene difluoride (PVDF). They have been tested in direct contact membrane distillation (DCMD), air gap membrane distillation (AGMD) and vacuum membrane distillation (VMD), and shown extremely stable performance using feeds containing high salinity, low surface tension surfactants and organic foulants [112–120]. However, the chemical inertia of typical hydrophobic materials causes great difficulties for membrane scientists to effectively manipulate the surface morphology and energy of omniphobic membranes. Current fabricating methods consist of several steps and take long hours or even days to prepare the omniphobic layer. Future research must simplify the fabrication process. The use of 3D printing technology to design the omniphobic re-entrant structure would be one of the approaches. To minimize environmental impact, green solvents during the fabrication should be also explored [121].

In terms of system configuration, future research should explore the potential of AGMD and VMD more than DCMD [108,121–126]. Even though AGMD has a lower flux than DCMD, the former has greater sustainability than the latter because of less fouling propensity, lower conductive heat loss and better energy recovery [125,126]. In contrast, VMD may have a higher flux and energy efficiency than DCMD because vacuum not only facilitates the water vapor transport but also lowers the thermal conduction across the membrane [123,127,128]. To withstand the vacuum pressure, MD membranes with a sandwich cross-sectional structure consisting of (1) two sponge-like inner and outer porous layers and (2) a thin middle layer full of small-size macrovoids have been designed for VMD and they exhibit better wetting resistance [129]. In addition, to lower the energy consumption, MD membranes made of solar heating or photo-thermal elements may be the future direction because this may minimize the purchase of expensive solar panels [130–134].

5. Graphene Oxide (GO) and CQD-based Nanofiltration (NF) Membranes

It is well known that NF is a promising technique to remove contaminants especially heavy metal ions and dye solutes from wastewater because it can reject them not only by size exclusion but also through charge repulsion [6,135–139]. NF membranes consisting of GO may have great potential in separation fields because they can further improve separation performance, mechanical stability and chemical stability. For GO laminates

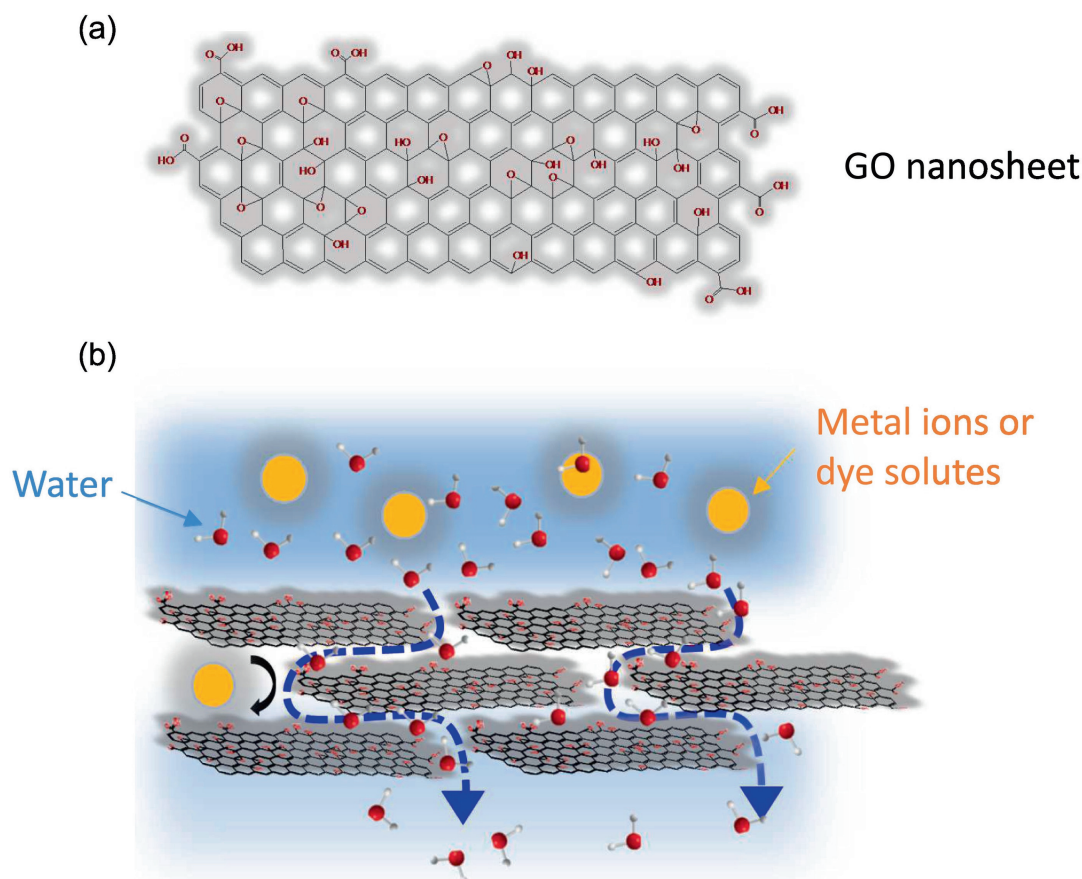


Fig. 3. (a) A possible chemical structure of a GO nanosheet and (b) water transport paths and size exclusion mechanism in a GO membrane.

soaked in water, the hydration effect will increase the d-spacing of the pristine GO membranes to $\sim(1.3 \pm 0.1)$ nm by inserting water layers into the laminates. Since the effective thickness of graphene is 0.34 nm, the size of the nanochannels between graphene sheets is ~ 0.9 to 1 nm, which may allow faster permeation of molecules with sizes smaller than 0.9 nm but block those ions or molecules larger than that [140–151]. Since GO is normally negatively charged over a range of pH due to the presence of carboxyl and hydroxyl groups, the pristine GO laminates also have better rejections to negatively charged molecules and divalent anions [141]. In addition, it has good antimicrobial properties, which may effectively decrease the fouling propensity of the formed membranes [140–144]. Thus, GO has been utilized for the formation of high effective NF membranes for water treatments. Fig. 3 presents a possible chemical structure of a GO nanosheet, water transport paths and size exclusion mechanism in GO membranes.

Till now, quite a few impressive studies have used GO to remove metal ions and dye solutes from wastewater. For example, Han and his co-workers deposited a thin layer of base-refluxing reduced GO (brGO) on top of commercialized MF membranes. By reducing the thickness of the brGO layer to 22 nm, the ultrathin graphene nanofiltration membrane had a rejection of 99.2% to methyl blue with a molecular weight (MW) of 800 and a pure water permeability (PWP) of $218.7 \text{ LMH} \cdot \text{MPa}^{-1}$ [145]. Zhang *et al.* fabricated a GO selective layer on top of Torlon® hollow fiber substrates via layer-by-layer deposition and showed high rejections of about 95% to heavy metal cations, such as Pb^{2+} , Ni^{2+} , and Zn^{2+} , with a PWP of $47 \text{ LMH} \cdot \text{MPa}^{-1}$ [146]. The GO layers formed by vacuum-assisted deposition and cross-linked with boron compounds had a 98.74% rejection to methylene blue (MW: 320) and an impressive PWP exceeding $70 \text{ LMH} \cdot \text{MPa}^{-1}$ [147]. Therefore, GO is a promising

material in water treatments for selective molecular seizing and transport. However, how to effectively manipulate the interlayer spacing among GO graphitic layers remains challenging [139,148–150] because pure GO laminates may swell in water that enlarges their d-spacing and nanochannels. One must identify effective methods to suppress the swelling so that they can separate small ions [151,152]. Thus, future research work could focus on manipulating and optimizing the interstitial space among GO layers for rejecting small ions.

On the other hand, CQDs may also be a promising material that can be used to fabricate NF membranes for water treatments. Similar to GO, it is hydrophilic and has good anti-fouling and anti-biofouling properties. So far, no wholly integral asymmetric membranes consisting of CQDs have been demonstrated for NF applications; they were only explored in the configuration of TFN membranes [153,154]. Since the zero-dimensional CQDs have small dimensions and rich functionalities, the dispersion of CQDs in a TFN layer is normally homogeneous. The CQDs may decrease pore size and enhance hydrophilicity of the formed membranes, resulting in an increased water permeability without compromising the rejection. They also show good anti-fouling properties compared to the membrane without CQDs [153–155]. Therefore, CQD-based NF membranes may show great potential in NF membranes.

6. Conclusions

In this paper, we have briefly examined the recent progresses of emerging membrane technologies and system integration for sustainable water reuse and desalination. In summary, TFN membranes consisting of hydrophilic and stable MOFs and CQDs may likely to be the dominant research directions for seawater

desalination and water reuse in the next decade. Additionally, more accurate control of materials and membrane morphology are needed to meet the requirements of a specific membrane process [156,157]. Hybrid systems comprising FO, MD, MD-C, PRO and SWRO will be the trend in modern membrane technologies in order to lower the overall energy consumption, operation costs and meet the target of ZLDD. Advances in omniphobic membranes are essential for sustainable MD operations. However, their fabrication processes must be significantly simplified. Both low-cost GO and CQDs may become popular in the development of next-generation NF membranes because of their unique properties.

Acknowledgments

This research is supported by PUB, Singapore's National Water Agency under the project "Development of 8 inch Novel High Efficiency Pressure-Retarded Osmosis (PRO) Membrane Modules towards Potential Pilot Testing and Field Validation" with NUS grant No. R-279-000-555-592. The authors would also like to thank Singapore National Research Foundation for supporting the project entitled, "Using Cold Energy from Regasification of Liquefied Natural Gas (LNG) for Novel Hybrid Seawater Desalination Technologies" (Grant number: R-279-000-456-279). The authors also thank BASF SE, Germany for partially funding this project with a grant number of R-279-000-363-597. Special thanks to Dr. Yu Zhang, Miss Wenxiao Gai, Mr. Dangchen Ma, Mr. Jian Chang, Dr. Yiming Xu, Dr. Zhenlei Cheng, Dr. Lin Luo and Prof. Sui Zhang of NUS as well as Dr. Natalia Widjojo of BASF for their very kind support.

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