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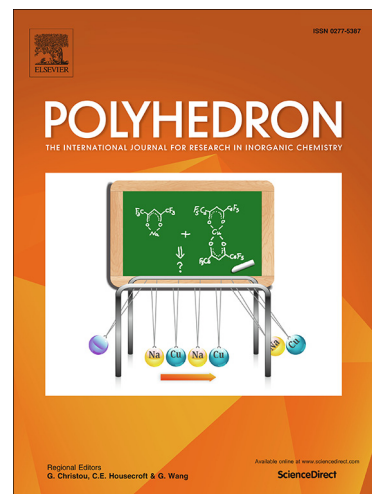
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Polyamide-zinc oxide-based thin film nanocomposite membranes: Towards improved performance for forward osmosis

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Abstract

In this study, polyamide (PA)-zinc oxide (ZnO) nanocomposite membrane was fabricated by embedding various amounts of ZnO nanoparticles (ranging from zero to 1.0 wt%) into the membrane matrix. Interfacial polymerization method was used to prepare thin film nanocomposite (TFN) membranes for forward osmosis (FO) application. The effect of ZnO nanoparticles on the chemical structures and compositions of synthesized membranes was studied using FT-IR spectroscopy, atomic force microscope (AFM) and scanning electron microscopy (SEM). Moreover, the performance of the fabricated membranes (water flux) and hydrophilicity were investigated using NaCl powder solution and contact angle measurements, respectively. Experimental results showed that the water flux of prepared membranes remarkably enhanced after the addition of ZnO nanoparticles. All as-prepared TFN membranes exhibited an enhanced desalination performance in FO systems as compared to the original thin-film composite (TFC) polyamide membrane.

Keywords: Zinc oxide nanoparticles; Interfacial polymerization; Desalination; Forward osmosis

1. Introduction

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In the past decades, the increasing world population and rapid industrialization led to rising enormous global demand for fresh and clean water. The water purification process using a separation membrane has gained remarkable growing attention. This method could be considered as an ideal alternative to thermal separation techniques [1, 2]. Because of some serious limitations in energy consumptions from non-renewable sources, it is not reasonable to utilize the reverse osmosis (RO) technique for water softening. Moreover, the RO process has two main shortcomings namely applying high hydraulic pressure and low water recovery ability that limited extremely its practical applications [3, 4]. Compared to the RO process, forward osmosis (FO) as a novel membrane process has demonstrated a lot of advantages including, low cost [5], easy cleaning [6], low fouling tendency [7] and so forth. Therefore, massive efforts have been devoted by membrane scientists in order to develop the forward osmosis (FO) process as a potential desalination technique to compete with RO technique [8, 9]. Despite great advances in FO process, there remains a strong challenge for improving the properties of thin film composite (TFC) FO membrane, especially at the top active skin layer in order to decrease important restrictions of FO process in terms of the internal concentration polarization (ICP) and fouling. Generally, ICP is a critical and inescapable issue which limits the performance of FO membrane [10]. It worth to mention that in the most cases the interfacial polymerization (IP) method has been used in order to form an ultra-thin polyamide (PA) active layer on top of the porous substrate of TFC-FO membranes [11]. By tuning the IP parameters such as reactive monomers, monomer concentration, nanofillers as additives, and reaction temperature and time, it is possible to adjust the membrane performance in which meet the desired standard [12]. Among them, through incorporating nanoparticles (NPs) during the IP process, both substrate and active thin layer could be modified by increasing porosity, improving hydrophilicity and decreasing ICP effect. Therefore, Recently, great attempts have been devoted to the nanotechnology as a versatile platform for developing of new nanostructured materials such as zeolite [13], carbon nanotubes [14], graphene oxide [15], silver NPs [16], TiO₂ NPs [17], ZrO₂, SiO₂ and Al₂O₃ NPs [18-20] which could be used in the fabrication of novel FO membranes. Apart from the aforementioned nanoparticles, the incorporation of zinc oxide NPs (ZnO) in the membrane matrix might be considered as a promising approach due to their outstanding physical and chemical properties [21]. In addition, higher hydrophilicity as well as high surface area of the ZnO NPs not only has made them as one of the most suitable materials for construction of

composite membranes but also could solve the fouling problem of membranes [22-24]. In this work, the incorporation of ZnO NPs into a thin film polyamide membrane via interfacial polymerization in order to prepare novel TFN-FO membranes have been reported. The membrane performance was investigated in terms of water flux and desalination. Furthermore, the chemical alteration and membrane composition were analyzed using FT-IR, SEM, EDX, AFM and contact angle measurements.

2. Experimental

2.1. Materials

In the synthesis of sublayer, polyethersulfone (PES with a molecular weight of 58 kDa from BASF, Germany), dimethylformamide (DMF, Merck) and polyethylene glycol (PEG-300, Merck) were used as polymer, solvent and additive, respectively. Chemicals used for the fabrication of active thin layer via interfacial polymerization (IP) technique included *m*-phenylenediamine (MPD, >95%, Merck), trimesoyl chloride (TMC, >98%, Merck) and *n*-hexane (>99%, Merck). Sodium chloride (NaCl, Merck) was used for the salt permeability test. ZnO NPs were synthesized according to our previous report [25].

2.2. Preparation of PES support membrane

The PES support membrane was prepared using the phase inversion method [26]. Briefly, PES (2 g) and PEG-300 (5.6 g) were dissolved in DMF and stirred until the solution became homogeneous and, the solution was kept in the open atmosphere for 24 h for the elimination of the trapped air bubbles. Then, the prepared solution was cast on a flat glass plate in a uniform thin film using a casting knife at a thickness of 120 μm . After that, the glass plate was immersed in a bath containing water at room temperature to initiate the phase inversion process. Finally, the PES support membrane was dried at room temperature for 24 h.

2.3. Fabrication of TFC and TFN membranes

To fabricate the TFC membrane, the obtained PES support membrane was immersed in MPD solution (2.0 wt%) for 60 min followed by soaking in a 0.15 wt/v% TMC in *n*-hexane for 50 s. Finally, the obtained TFC membrane was kept in DI water prior to use. The PA-ZnO TFN membranes were prepared similar to TFC membrane with a difference in aqueous solution.

Typically, ZnO NPs in various concentrations (0.1, 0.2, 0.5 and 1.0 wt%) were added in the MPD solution and dispersed homogeneously via sonication for 20 min. Finally, these solutions were utilized as aqueous solutions for interfacial polymerization. The resultants TFN membranes were assigned as TFN-ZnO-0.1, TFN-ZnO-0.2, TFN-ZnO-0.5 and TFN-ZnO-1, depending on the ZnO loading in the MPD solution.

2.4. Membrane characterization

The FTIR spectrum of the TFC and TFN membranes were obtained with a Bruker Tensor-27 FTIR spectrometer on a KBr disc over the range 400-4000 cm^{-1} . Top surface and cross-section morphologies of the TFC and TFN membranes were imaged with a scanning electron microscope (SEM, Tescan MIRA3). Roughness and surface morphologies of the synthesized membranes were investigated by atomic force microscopy (AFM, Park Systems XE-100, USA). The water contact angles were measured using the sessile drop method.

2.5. Evaluation of membrane performance

A cross-flow filtration apparatus was used in order to evaluate the separation performance (water flux and reverse salt flux) of the prepared TFC and TFN membranes. Separation experiments were carried out using DI water as a feed solution (FS) and 1M NaCl aqueous solution as a draw solution (DS) in both active layers facing feed water (FO) and active layer facing draw solution (PRO) orientations. When the system reached a steady state, separation performance data were collected. Finally, two parameters, J_v and J_s as water flux and reverse salt flux were calculated using the following equations:

$$J_v = \frac{\Delta V}{A_m \Delta t} \quad (1)$$

$$J_s = \frac{\Delta(CtVt)}{A_m \Delta t} \quad (2)$$

Where, ΔV (L) is the volume of permeated water over a time interval Δt (h), A_m is the effective area of the membrane for permeation, C_t and V_t are the concentration of NaCl and the volume of permeated water at the final time t , respectively. All membrane performance results were obtained from 3 replicate measurements of 3 independent membrane samples.

3. Results and discussion

3.1. Characterization of chemical structure and composition of TFC and TFN membranes

The chemical structures of TFC and TFN membranes with various amount of ZnO NPs i.e. TFN-ZnO-0.1, TFN-ZnO-0.2, TFN-ZnO-0.5 and TFN-ZnO-1 were investigated using FT-IR spectra. Figure 1 shows the FT-IR spectra of TFC and TFN-ZnO-1 membranes. In all spectra, the existence of bands at 1135, 1336, 1410 and 1500 cm^{-1} is belonging to the symmetric and asymmetric O=S=O stretching, C-O-C stretching, C=C aromatic ring stretching and CH₃-C-CH₃ stretching, respectively in which confirmed the chemical structure of PES substrate [27, 28]. Furthermore, the peak at 1651 cm^{-1} is assigned to the vibrational bands of the polyamide layer and could be as a clear witness for the formation of this layer on the top of PES substrate [29]. As it could be seen, there is no distinguished difference between the bands of TFC and TFN which incorporated with various amount of ZnO NPs. Introduction of ZnO NPs in the polyamide structure leads an initial blue shift in the stretching vibration of carbonyl groups from 1651 to 1660 cm^{-1} for TFN-ZnO-0.1 as compared to the TFC. However, by increasing ZnO NPs amounts, a red shift occurs from 1660 to 1652 cm^{-1} (Figure S1, See supporting information). The initial blue shift could be ascribed as a lesser extent of H-bondings for TFN-ZnO-0.1. In contrary, the red shifts may happen because of much more ZnO NPs dispersed in the open gaps of the polyamide matrix resulting more H-bonding between the hydroxyl groups of ZnO NPs and the carbonyl groups of the polyamide [30].

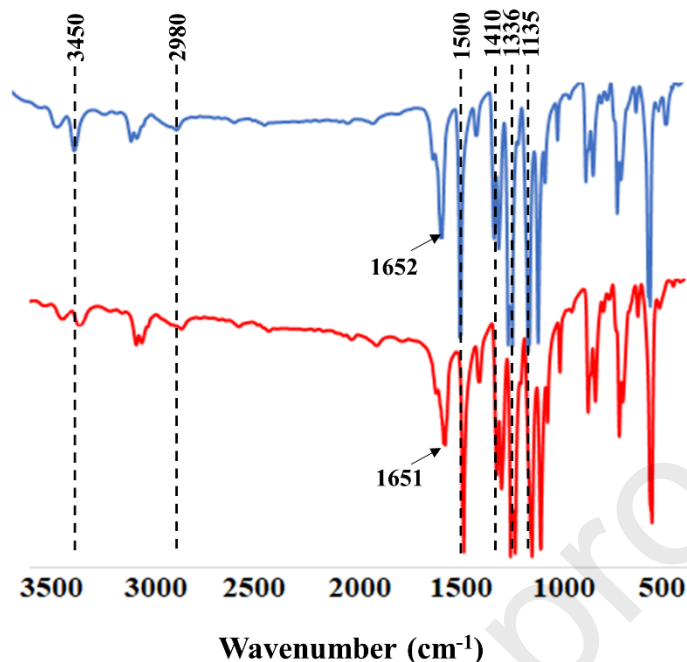


Figure 1. FTIR spectra of TFC and TFN-ZnO-1 membranes

The surface morphologies of TFC and TFN membranes with different ZnO NPs loading visualized by SEM are depicted in Figure 2. All images showed that the surfaces were ridge-valley, which are very common for TFC and TFN membranes prepared by the interfacial polymerization method [31]. For TFN-ZnO-0.1 and TFN-ZnO-0.2 membranes, ZnO NPs are fairly well distributed over the whole membrane surface although aggregates of some particles could be still seen visible in the case of TFN-ZnO-1 membrane which was distinguished with red circles in Figure 2E. These aggregations could be related to the hydrophilic-hydrophobic interactions between ZnO NPs with high concentrations in hydrophobic media of PES casting solution.

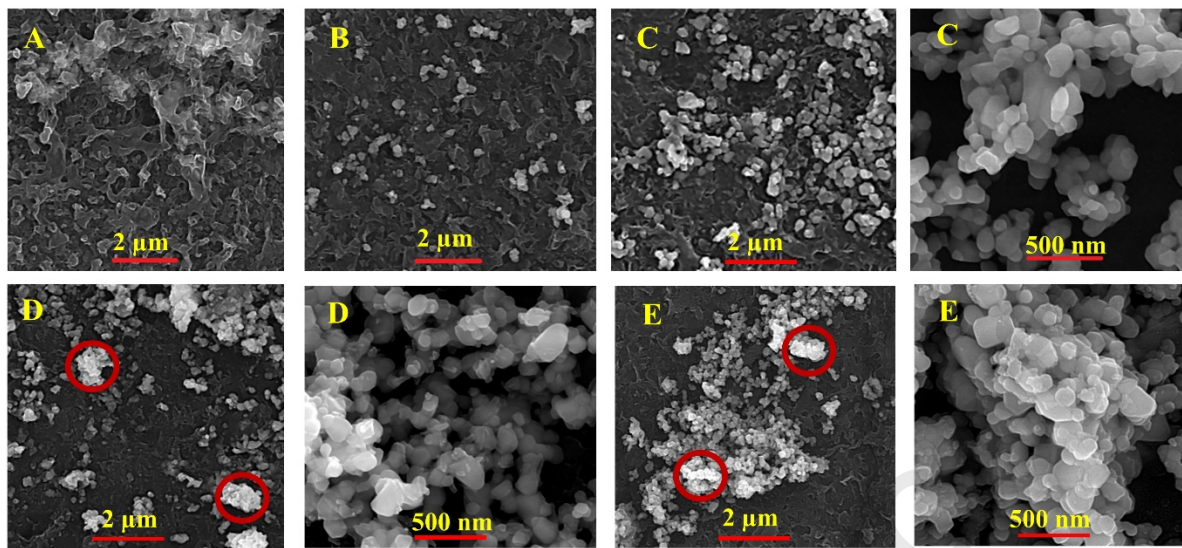


Figure 2. Surface SEM images of A) TFC, B) TFN-ZnO-0.1, C) TFN-ZnO-0.2, with two different magnifications D) TFN-ZnO-0.5 with two different magnifications and E) TFN-ZnO-1 with two different magnifications

Moreover, the elemental composition of synthesized membrane was determined using EDX analysis. The EDX results were depicted in Figure S2 (See supporting information), which confirmed the existence of Zn element in the structure of TFC, TFN-ZnO-0.1, TFN-ZnO-0.2, TFN-ZnO-0.5 and TFN-ZnO-1 with the concentration of 0, 4.15, 6.51, 8.66 and 12.34, respectively. The cross-section SEM micrographs of the synthesized membranes TFC, TFN-ZnO-0.1, TFN-ZnO-0.2, TFN-ZnO-0.5 and TFN-ZnO-1 are depicted in Figures 3 A, B, C, D and E, respectively. From these SEM images, it could be visually seen that finger-like structures were formed for TFC and all TFN sublayer from the top of sublayer to the bottom. It is generally well recognized that the nanoparticle addition not only increased the casting solution viscosity but also affected significantly the finger-like pore morphology of membranes [32]. From Figure 3, in comparison to TFC, more interconnected or finger-like pore structure could be observed for all of the TFN membranes. In principle, this type of morphology could efficiently facilitate water passing, leading to enhance pure water permeability [33].

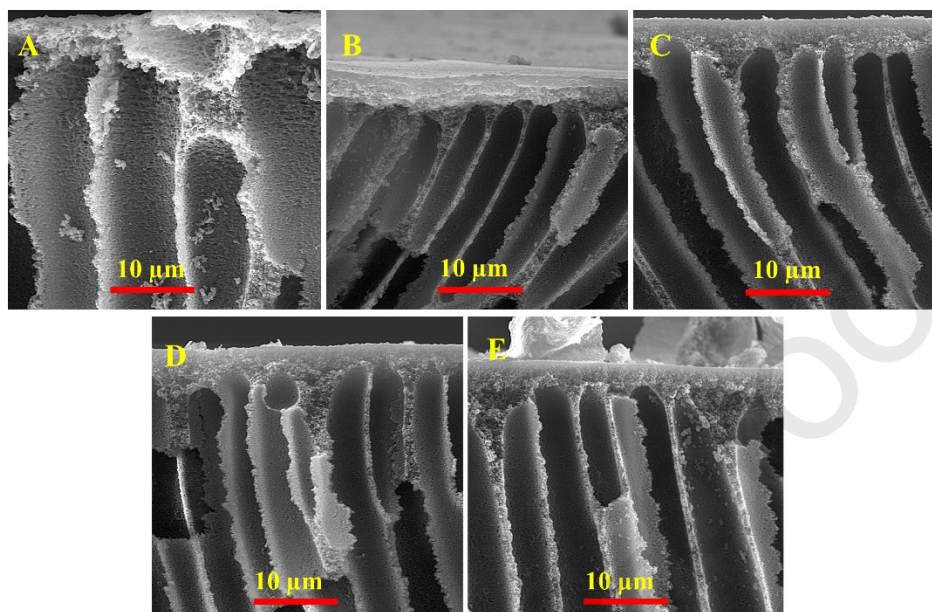


Figure 3. Cross-section SEM morphologies of synthesized membranes, A) TFC, B) TFN-ZnO-0.1, C) TFN-ZnO-0.2, D) TFN-ZnO-0.5 and E) TFN-ZnO-1

Figure 4 A, B, C, D and E illustrate the 3D surface AFM images as well as the related height histograms of TFC and TFN-ZnO-0.1, TFN-ZnO-0.2, TFN-ZnO-0.5 and TFN-ZnO-1 membranes, respectively. As it could be seen, TFC membrane shows different surface topology than TFN membranes. On the other hand, the surface topographies of the membranes alter remarkably from a relatively smooth surface for TFC to rougher surfaces for TFN membranes which made with the embedding of ZnO nanoparticles. According to the corresponding height histograms, TFC shows a uniform and very broad distribution of 200-300 nm. However, in the case of TFN-ZnO-0.1, the height histogram is relatively greater than TFC, with most occurring at 150-350 nm, corresponding to a much wider distribution pattern. With a higher concentration of ZnO NPs in TFN-ZnO-0.5 and TFN-ZnO-1, the peak heights of the synthesized membrane lost their uniformity and distributed in a much wider region from 500-800 nm.

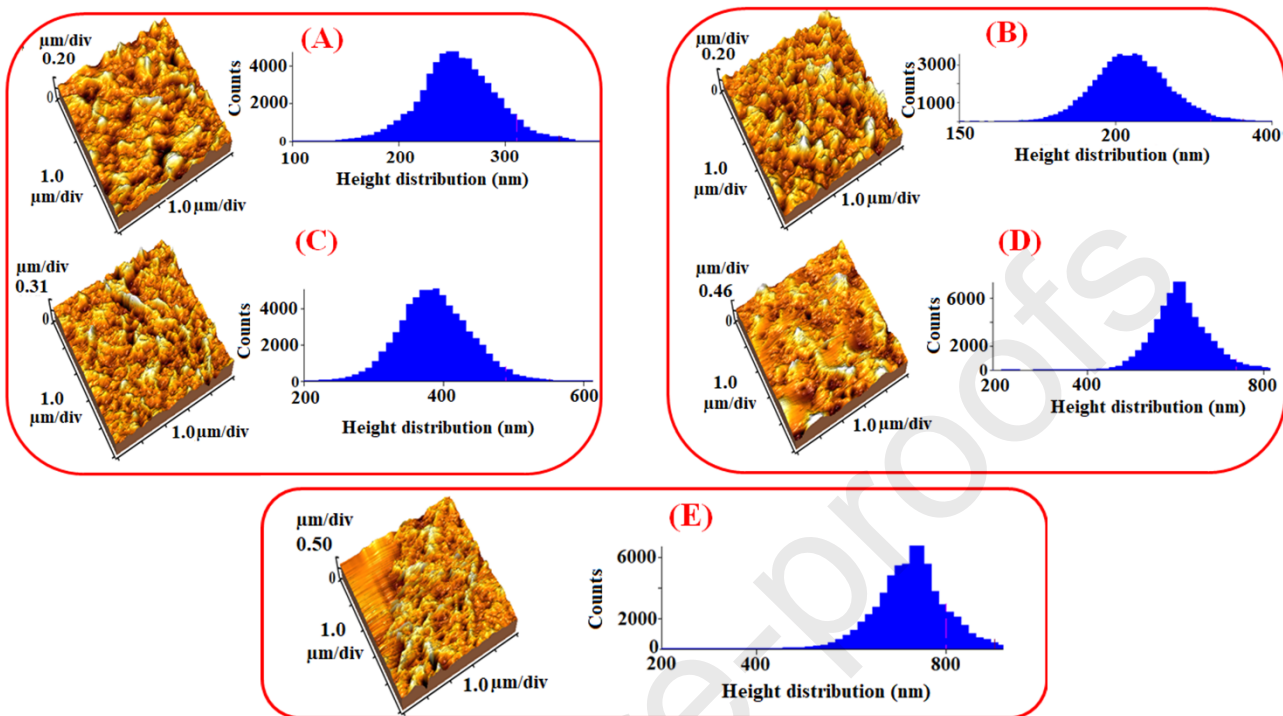


Figure 4. 3D AFM images and height histograms of A) TFC, B) TFN-ZnO-0.1, C) TFN-ZnO-0.2 D) TFN-ZnO-0.5 and E) TFN-ZnO-1

The calculated amplitude parameters of the synthesized membranes were summarized in Table 1. From Table 1, the average roughness (R_a) of the prepared membranes increases from 28.27 nm for TFC to 37.43, 41.04, 48.84 and 58.73 nm for TFN-ZnO-0.1, TFN-ZnO-0.2, TFN-ZnO-0.5 and TFN-ZnO-1, respectively. In addition, the root mean square roughness (R_q) of the membranes increases from 36.41 to 47.33 nm and finally reached to 80.77 nm. These are in good agreement with the topographical features discussed in their 3D AFM images. Finally, the mean difference between the highest peaks and lowest valleys (R_{10z}) which shows the contrast between the heights and depth of peaks and valleys on the surface of membranes increases from 360 to 403.80 nm. However, incorporation of a higher amount of ZnO NPs results to much higher R_{10z} for TFN-ZnO-0.2 (613.50 nm), TFN-ZnO-0.5 (925.10 nm) and TFN-ZnO-1 (1603 nm) which implied the existence of greater contrast in heights and depths because of the formation of isolated high peak and low valley on the membrane surface [30].

Table 1. Calculated amplitude parameters for synthesized membranes by AFM

Membrane	Amplitude parameters		
	R_a (nm)	R_q (nm)	R_{10z} (nm)
TFC	28.27	36.41	390
TFN-ZnO-0.1	37.43	47.33	403.80
TFN-ZnO-0.2	41.04	52.98	613.50
TFN-ZnO-0.5	48.84	64.97	925.10
TFN-ZnO-1	58.73	80.77	1603

Surface hydrophilicity is one of the most substantial factors which could significantly affect the flux and antifouling ability of membranes. In this regard, water contact angle measurement technique could be considered as a versatile method for determining the surface hydrophilicity [34]. Images of contact angle measurements are exhibited in Figure 5. Compared to the TFC, which had a contact angle of 102.7 °, all TFN membranes showed enhanced surface hydrophilicity with reduced water contact angles. It is worth to mention that the water contact angle of the membranes incorporated with ZnO NPs remarkably declined when the amount of ZnO NPs increased in the membrane structure. On the other hand, by addition of 0.1, 0.2, 0.5 and 1 wt.% ZnO NPs, the contact angles fell to 87.7 °, 79.3 °, 70.7 ° and 62.9 ° for TFN-ZnO-0.1, TFN-ZnO-0.2, TFN-ZnO-0.5 and TFN-ZnO-1 membranes, respectively. This fact could be explained that the surface hydroxyl groups and the hydrophilic nature of ZnO NPs increased the hydrophilicity of the aforementioned membranes. Moreover, it is well-known that the existence of hydrophilic nanoparticles in the casting solution transfer towards the top layer of synthesized membranes leading to the decoration of nanoparticles on the top surface of the membrane and increases the membrane hydrophilicity [35].

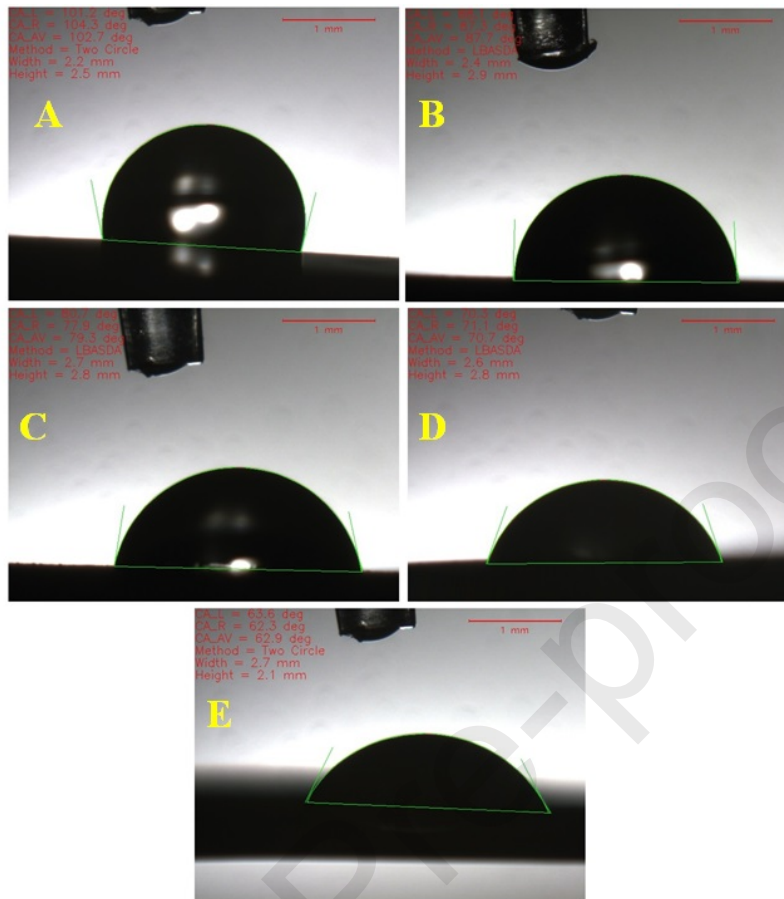


Figure 5. Contact angles of A) TFC, B) TFN-ZnO-0.1, C) TFN-ZnO-0.2, D) TFN-ZnO-0.5 and E) TFN-ZnO-1

3.2. As prepared membranes performances in FO and PRO modes

Cross-flow processes at FO and PRO modes were utilized to investigate the water flux and reverse salt flux of TFC and TFN membranes, and results were illustrated in Figures 6 (A and B) and 7 (A and B). In both cases, two different concentrations of NaCl (1M and 0.5M) salt were used as FS and DS solutions. As it could be observed in Figure 6 A and B, compared to the TFC membrane, TFN membrane with various amount of ZnO NPs demonstrated higher water flux in both FO and PRO modes. On the other hand, with 0.5 wt% ZnO NPs added, the water flux of the TFN membrane in FO mode was enhanced from 12 L/m² h (for 0.5M NaCl) and 22.22 L/m² h (for 1M NaCl) to 47.25 L/m² h and 84.74 L/m² h, respectively. The same trend (increasing water flux ability) could be also seen in PRO mode. This increase in water flux ability could be ascribed by several parameters in which were related to the hydrophilic nature of ZnO NPs. In

another word, the incorporation of ZnO NPs in the membrane structure not only increases the porosity of membranes but also increases the hydrophilicity of the synthesized membranes, which could absorb water into the membrane structure and transports water molecules through the membrane easily. It worth to mention that due to the aggregation of ZnO NPs in the membrane matrices, the water flux of TFN-ZnO-1 with highest ZnO NPs loading decreased slightly in both FO and PRO modes. These outcomes are in compromise with the membrane characterization results described earlier.

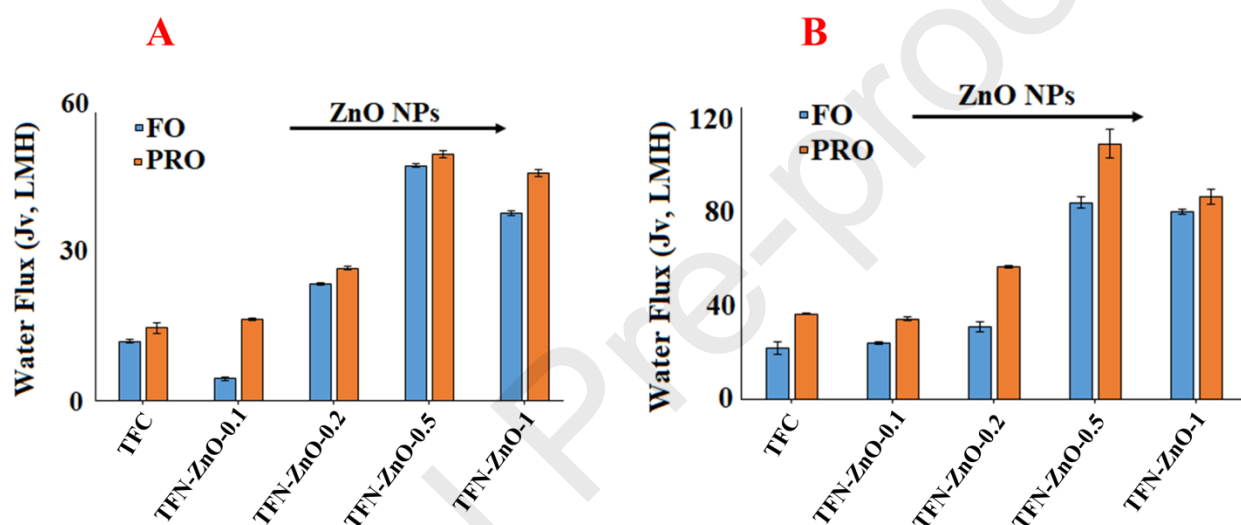


Figure 6. water flux performance of TFC and TFN membranes A) 0.5M NaCl and B) 1M NaCl salt. Error bars were based on the standard deviations of 3 replicate measurements of 3 independent membranes

Moreover, the reverse salt flux of TFC and TFN membranes in FO and PRO modes with two different concentrations of NaCl solution (0.5 and 1M) were depicted in Figure 7 A and B. it is clear that by increasing the amount of ZnO NPs in the membrane matrix, the reverse salt flux of TFN membranes increased, which could be related to the solute permeability of the membrane [28].

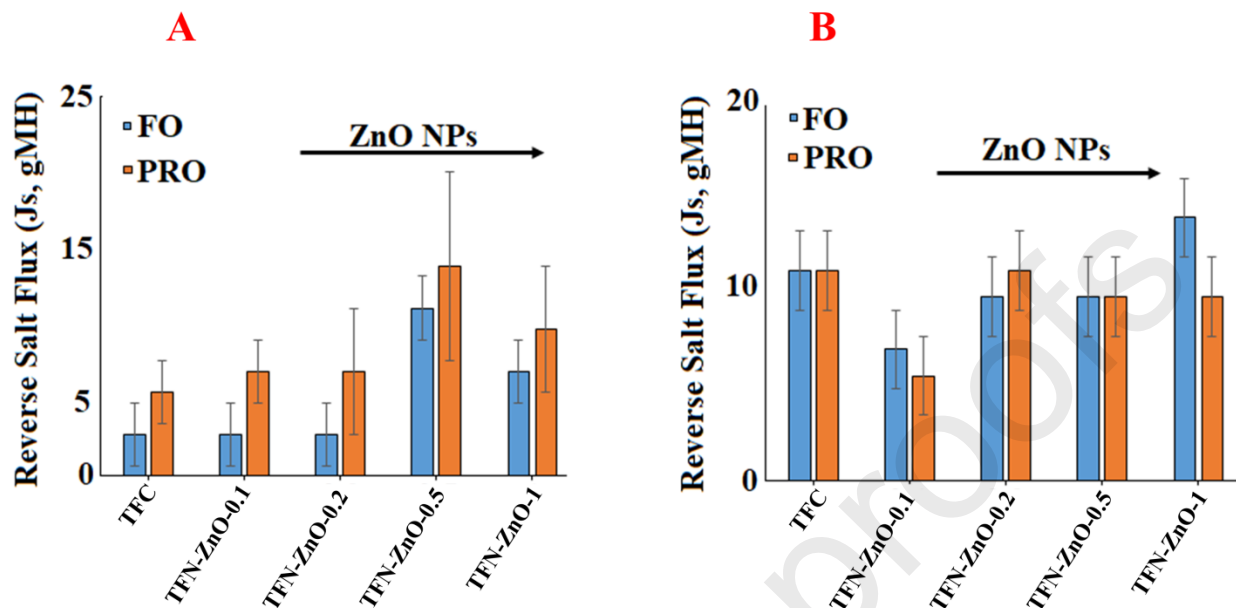


Figure 7. Reverse salt flux performance of TFC and TFN membranes A) 0.5M NaCl and B) 1M NaCl salt. Error bars were based on the standard deviations of 3 replicate measurements of 3 independent membranes

Draw solution (DS) concentration has an important effect on the osmotic water flux performance of the membranes. In this regard, the TFN membrane with 0.2 wt% of ZnO NPs (TFN-ZnO-0.2) was selected and the effect of DS concentration on its water osmotic performance was studied. Experimental results were shown in Figure 8. As it could be seen in the both FO and PRO modes, the water flux increased by increasing the salt concentration from 0.5 to 1M due to the existence of high osmotic force across the membrane in the high concentration of NaCl solution.

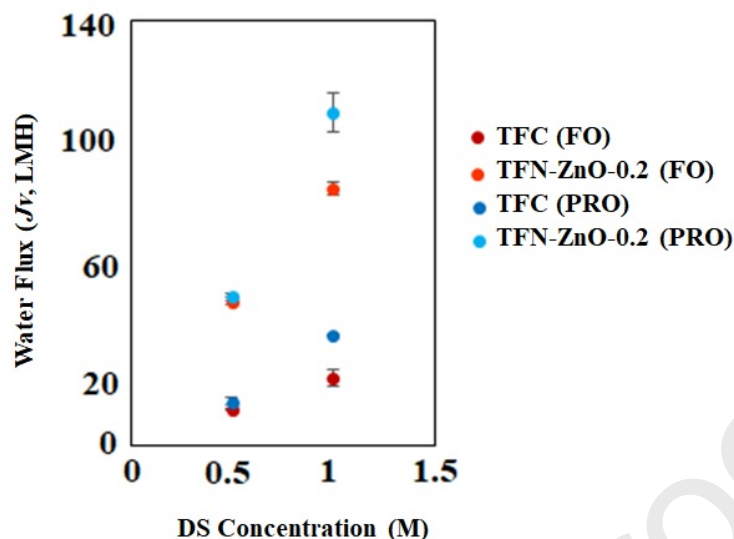


Figure 8. Effect of DS concentration on the FO water flux performance of TFN-ZnO-0.2

To date, there are few studies focusing on the utilization of various type of nanoparticles as an additive in the membranes. Table 2 provides a comparison of the obtained results for TFN-ZnO-0.5 membrane with those reported in the literatures.

Table 2. Comparison of TFN-ZnO-0.5 membrane with previously reported membranes

Nanoparticles	Polymeric substarte	Water Flux (L/m ² h)	Refs
Silver	PES	9.6	[36]
MoO ₃	PES	68.7	[37]
Al-doped ZnO	PSf ^a	32	[38]
MWCNTs ^b	PSf	40.0	[26]
Silica (1.6 wt%)	PEI ^c	42.0	[39]
TFN-ZnO-0.5	PES ^d	84.74	This work

^aPolysulfone beads, ^bMulti-wall carbon nanotubes, ^cPolyetherimide, ^dPolyethersulfone

The data illustrated in Table 2 clearly demonstrated that the incorporation of ZnO NPs increased the water flux performance of the synthesized membranes.

4. Conclusion

In summary, TFN membranes with ZnO NPs- embedded polyamide matrix were fabricated via *in situ* interfacial polymerization of reactive monomers. Synthesized membranes were characterized using FT-IR, SEM, AFM. By the addition of ZnO NPs, the hydrophilicity and porosity of the polyamide-ZnO nanocomposite substrate was enhanced. On the other hand, the higher the ZnO NPs loading the lower contact angle value was obtained. Regarding membrane performance, water flux and reverse salt flux of all samples were investigated using crossflow filtration technique. Experimental results showed that the membrane performances increased by the incorporation of ZnO NPs. Among the TFN membranes synthesized in this study, TFN-ZnO-0.5 membrane (0.5 wt% of ZnO NPs) was demonstrated to be the best performing membrane for the water desalination process.

Acknowledgments

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Author Contributions Section

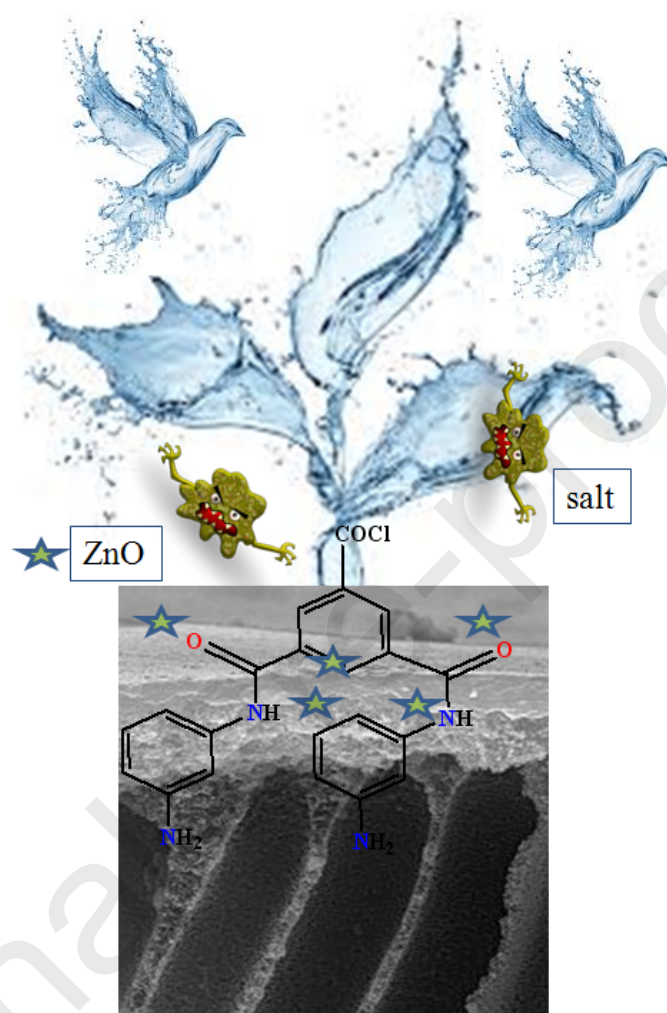
Mojtaba Amini: Supervision, **Maryam Seifi:** Conceptualization & Methodology, **Ali Akbari:** Writing - Original Draft, **Mojtaba Hosseinifard:** Software

Declaration of interests

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests:

Graphical abstract



Graphical abstract-S

A polyamide (PA)-zinc oxide (ZnO) nanocomposite membrane was fabricated by embedding various amounts of ZnO nanoparticles into the membrane matrix for using in forward osmosis (FO) process.