Journal of Environmental Economics & Chemical Processes (JEECP)

https://doi.org/10.22034/jeecp.2026.2088035.1017

Journal Homepage: *WWW.JEECPJournal.com*

Journal of Environmental Economics & Chemical Processes (JEECP)

ORIGINAL RESEARCH PAPER

Thin-Film Composite Forward Osmosis Membranes with Graphene Oxide Interlayer for Efficient Seawater Desalination

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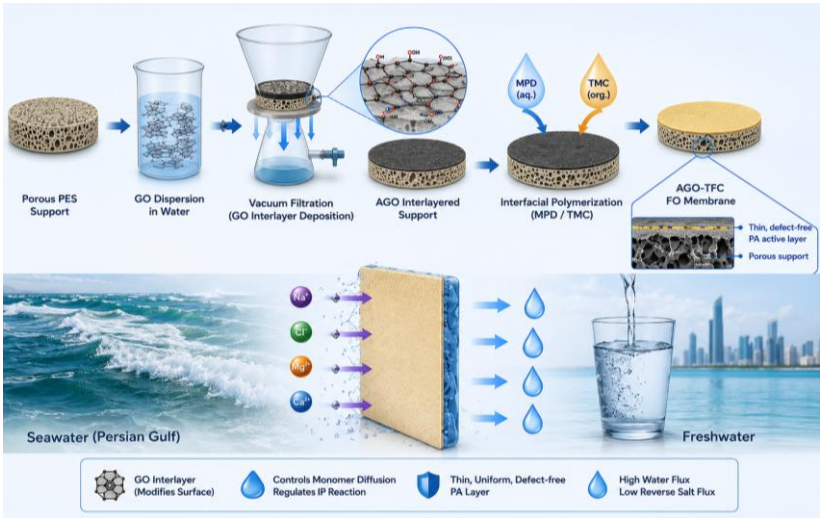
P A P E R I N F O

Paper history:
Received: 05/30/2026
Revised: 06/15/2026
Accepted in revised form: 06/25/2026
Published: 06/30/2026

Keywords:
Forward Osmosis;
Graphene Oxide;
Interlayer;
Desalination.

A B S T R A C T

Forward osmosis (FO) has attracted considerable attention as a membrane-based desalination technology because of its low energy requirement and reduced membrane fouling compared with conventional pressure-driven processes. Nevertheless, achieving highly selective and permeable thin-film composite (TFC) membranes remains challenging due to uncontrolled interfacial polymerization and the formation of defective polyamide (PA) layers. In this study, a graphene oxide (GO) interlayer was deposited onto a porous PVDF substrate via vacuum-assisted filtration to regulate the interfacial polymerization process. The GO interlayer enhanced substrate hydrophilicity, controlled monomer diffusion, and promoted the formation of a thin, uniform, and defect-free PA active layer. Consequently, the GO-modified TFC membrane exhibited significantly improved FO performance, with water flux increasing from 7.3 to 13.3 LMH and reverse salt flux decreasing from 6.9 to 3 gMH compared with the pristine membrane. These results demonstrate that incorporating a GO interlayer effectively enhances the permeability–selectivity balance and provides a promising strategy for improving FO membrane performance in seawater desalination applications.



1. Introduction

Freshwater scarcity has become one of the most critical global challenges due to rapid population growth, industrialization, climate change, and increasing water pollution [1]. Desalination technologies have therefore attracted considerable attention as sustainable solutions for producing freshwater from seawater and other saline resources. Among various desalination processes, reverse osmosis (RO) has dominated the commercial market owing to its high salt rejection and technological maturity [2,3]. Nevertheless, RO processes require substantial hydraulic pressure and energy, which increase operational costs and the likelihood of membrane fouling [4]. Forward osmosis (FO) has recently emerged as a promising alternative membrane technology for desalination and water treatment applications because it utilizes osmotic pressure difference rather than hydraulic pressure as the driving force for water transport. Compared to pressure-driven membrane processes, FO offers several advantages, including lower energy consumption, reduced membrane fouling, higher contaminant rejection, and milder operating conditions [5,6]. These advantages make FO highly attractive for seawater desalination, wastewater reclamation, food processing, and osmotic power generation. The performance of FO systems strongly depends on the properties of the membrane. Thin-film

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ISSN: 3116-0050
URL: https://www.jeecpjournal.com/article_735389.html

Please cite this article as: H. Salehi, & A. Shakeri, *Journal of Enviromental Economics & Chemical Processes (JEECP)*, 2026, *3*(2), 71-74. https://doi.org/10.22034/jeecp.2026.2088035.1017

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composite (TFC) membranes are widely considered the most suitable membrane configuration for FO applications due to their superior permeability-selectivity balance [7]. A typical TFC-FO membrane consists of a dense polyamide (PA) selective layer formed on a porous support substrate through interfacial polymerization (IP). The porous substrate provides mechanical support and minimizes internal concentration polarization (ICP), while the PA layer governs water permeability and salt-rejection characteristics [8,9]. Despite significant advances in TFC membrane fabrication, forming an ideal PA-selective layer on highly porous microfiltration substrates remains challenging [10]. In traditional interfacial polymerization, aqueous amine monomers readily penetrate large substrate pores, leading to uncontrolled reactions with acyl chloride monomers and producing rough, thick, and defective PA layers with poor selectivity and increased transport resistance [11,12]. Moreover, the intrusion of polyamide into substrate pores reduces membrane permeability and exacerbates internal concentration polarization [13]. To overcome these limitations, introducing an intermediate layer between the porous substrate and the PA selective layer has proven an effective strategy for regulating the interfacial polymerization process. Interlayers can modify substrate surface properties, control monomer diffusion, improve substrate wettability, and suppress PA formation within substrate pores. Consequently, interlayer-based TFC membranes generally exhibit thinner and smoother PA layers with enhanced water permeability and selectivity. Among interlayer materials, graphene oxide (GO) has attracted considerable attention due to its unique physicochemical properties. GO nanosheets possess abundant oxygen-containing functional groups such as hydroxyl, epoxy, and carboxyl groups, which provide excellent hydrophilicity and tunable surface chemistry [14,15]. In addition, the two-dimensional structure and high surface area of GO facilitate rapid water transport while simultaneously regulating the diffusion behavior of monomers during interfacial polymerization . Recent studies have demonstrated that graphene oxide-based interlayers can significantly improve membrane morphology and FO performance by producing defect-free, ultrathin PA-selective layers [16,17]. The enhanced membrane performance is generally attributed to controlled monomer transport, inhibition of PA intrusion into substrate pores, and formation of additional water transport pathways. However, achieving an optimal interlayer structure that simultaneously enhances permeability, selectivity, and membrane stability remains a major challenge.

In this work, a GO-based interlayer was introduced onto a porous poly (vinylidene fluoride) (PVDF) substrate to fabricate a high-performance TFC-FO membrane for seawater desalination. The GO interlayer was designed to regulate the interfacial polymerization reaction, control monomer diffusion, and inhibit PA growth within the substrate pores. The effects of the GO interlayer on substrate properties, PA layer morphology, and FO desalination performance were systematically investigated. The fabricated GO-interlayered membrane demonstrated substantially enhanced water flux, reduced reverse salt flux, and selectivity compared to the pristine TFC membrane.

2. Methods

2.1. Methodology

Porous PVDF microfiltration (PVDF-MF) membranes with an average pore size of 0.22 μm were employed as the support substrate for the fabrication of TFC-FO membranes. The PVDF substrates were purchased from Millipore (Germany). GO nanosheets (supplied by Graphenea) with an average lateral size of 13–15 μm (D50), monolayer structure, and an oxygen content of 41–50% (C/O ratio ≈ 1.0–1.4) were utilized as the interlayer nanomaterial. m-Phenylenediamine (MPD, 99%) and trimesoyl chloride (TMC, 98%) were purchased from Sigma-Aldrich and used as monomers in the aqueous and organic phases, respectively, for interfacial polymerization. n-Hexane (Merck) was used as the organic solvent to dissolve TMC. Sodium chloride (NaCl, analytical grade) was used to prepare the draw solution during FO performance evaluation.

2.2. Fabrication of GO Interlayered Support Layer

The GO interlayer was deposited onto the PVDF substrate surface using a vacuum-assisted filtration method. Initially, the PVDF support membrane was fixed inside a vacuum filtration unit. Then, a predetermined volume (5 mL) of an aqueous GO suspension (0.01 wt.%) was poured onto the membrane surface and filtered under mild vacuum (500 mbar). During this process, GO nanosheets were uniformly assembled on the substrate surface, forming an ultrathin interlayer structure. After vacuum filtration, the modified substrates were rinsed gently with DI water to remove loosely attached nanosheets and dried at room temperature overnight before further use.

2.3. Fabrication of TFC-FO Membranes

TFC membranes were fabricated through interfacial polymerization on both pristine PVDF substrates and GO-modified PVDF substrates. In a typical procedure, the support membrane was first immersed in an aqueous MPD solution (2.0 wt.%) for 2 min to allow complete impregnation of amine monomers into the substrate surface. Excess aqueous solution was subsequently removed using a rubber roller, and the membrane surface was air-dried for approximately 60 s. Afterward, a TMC solution (0.10 wt.%) dissolved in n-hexane was carefully poured onto the MPD-saturated substrate surface to initiate the interfacial polymerization reaction. The reaction was allowed to proceed for 90 s, resulting in the formation of a polyamide-selective layer at the water/organic interface. The excess organic solution was then removed, and the freshly prepared membrane was thermally cured in an oven at 70 °C for 3 min to complete polymer crosslinking. Finally, all fabricated membranes were thoroughly rinsed with DI water and stored in fresh DI water at 4 °C prior to characterization and FO testing.

2.4. Membrane Characterization

The surface morphologies of the membranes were examined using field-emission scanning electron microscopy (FESEM, MIRA3 TESCAN). The hydrophilicity of membrane surfaces was investigated by measuring water contact angles (WCAs) with a contact angle analyzer (Dataphysics OCA 20). In each measurement, 2 μL water droplets were placed on different regions of the membrane surface, and the average value was reported from at least five independent measurements.

2.5. FO Performance Evaluation

The FO performance of the prepared membranes was investigated using a laboratory-scale crossflow FO system operated in active layer facing feed solution (AL-FS) orientation. During the experiments, deionized water was employed as the feed solution (FS), while a 1.0 M NaCl solution was used as the draw solution (DS). Both solutions, each with a volume of 1.0 L, were continuously circulated on opposite sides of the membrane cell using peristaltic pumps at a flow rate of 100 mL/min. The effective membrane area was 4.9 cm^2, and all experiments were carried out at 25 ± 1 °C. The volume of permeated water was monitored by recording the weight change of the FS using a digital balance over a 1.0 h test period. The water flux was calculated according to:

$$J_w(LMH) = \frac{\Delta V}{A_m \times \Delta t} \quad (1)$$

where J_w represents the water flux (LMH), ΔV is the permeated water volume (L), A_m is the effective membrane area (m^2), and Δt is the operating time (h).

The reverse salt flux was determined from the conductivity increase in the FS using a conductivity meter (Adwa 330) and calculated using:

$$J_s(gMH) = \frac{\Delta V.\Delta C}{A_m\,\Delta t} \quad (2)$$

where J_s denotes the reverse salt flux (gMH) and ΔC corresponds to the variation in salt concentration within the feed solution.

3. Results and Discussion

3.1. Role of the GO Interlayer in TFC-FO Membrane Fabrication

The fabrication of defect-free PA selective layers on highly porous microfiltration substrates remains one of the major challenges in TFC-FO membranes. In conventional interfacial polymerization, the large surface pores and low wettability of porous substrates promote uncontrolled diffusion of aqueous amine monomers into the organic phase. As a consequence, rapid polymerization occurs not only at the interface but also inside substrate pores, resulting in rough, thick, and defective PA structures with poor separation properties [10]. The introduction of an ultrathin interlayer between the porous support and the PA selective layer can effectively regulate the interfacial polymerization process. In this study, the GO-based interlayer served multiple vital functions in enhancing the membrane's structure and performance. First, the GO nanosheets significantly enhanced the hydrophilicity of the PVDF substrate due to the abundance of oxygen-containing functional groups such as hydroxyl and carboxyl groups. Improved surface wettability facilitated uniform spreading of the MPD aqueous solution across the substrate surface and reduced localized monomer accumulation. Second, the two-dimensional structure of GO partially covered the substrate's large surface pores and created a more controlled interfacial region for polymerization. Furthermore, the GO interlayer served as a physical barrier, confining the interfacial polymerization reaction near the membrane surface. Such regulation resulted in a thinner, more homogeneous PA-selective layer with fewer structural defects. In addition, the hydrophilic nanochannels created by stacked GO nanosheets could facilitate water transport across the membrane, thereby improving membrane permeability. Therefore, the incorporation of the GO interlayer is expected to simultaneously improve membrane selectivity and permeability.

3.2. Surface Morphology of Support Layers

The surface morphology of the pristine and GO-modified PVDF substrates was investigated using SEM images. As shown in the SEM images in Fig. 1a, the pristine PVDF substrate exhibited a highly porous surface with relatively large, irregular pores. Such morphology is generally unfavorable for controlled interfacial polymerization because large pores can absorb excessive amounts of aqueous monomers, thereby promoting uncontrolled PA growth. After deposition of the GO interlayer via vacuum-assisted filtration, as shown in Fig. 1b the surface morphology changed considerably. The GO nanosheets were uniformly distributed across the substrate surface and partially covered the pores, resulting in a smoother, denser morphology. No obvious aggregation of nanosheets was observed, indicating successful formation of a homogeneous ultrathin interlayer. The reduction in apparent pore size after GO deposition is highly beneficial for controlling monomer transport during interfacial

polymerization. The interlayer can effectively reduce monomer intrusion into substrate pores and localize the reaction near the membrane surface. Moreover, the wrinkled nanosheet structure of GO may provide additional transport pathways for water molecules while maintaining a highly hydrophilic surface environment.

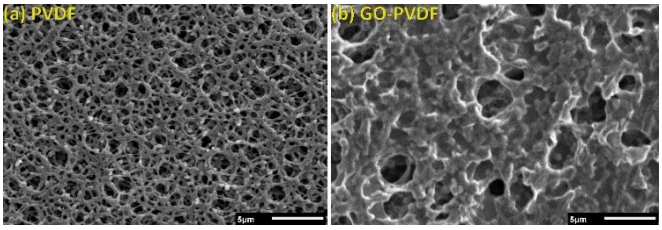


Figure 1. SEM images of support layers, (a) pristine PVDF and (b) GO-interlayered PVDF.

Source: Research findings

3.3. Effect of GO Interlayer on PA Layer Morphology

The morphology of the PA selective layer was strongly influenced by the presence of the GO interlayer. SEM surface images revealed substantial differences between the pristine TFC membrane and the GO-interlayered membrane. The PA layer formed on the bare PVDF substrate (Fig. 2a) exhibited a typical ridge-and-valley morphology with relatively large nodular structures and high surface roughness. Such morphology is commonly associated with rapid, uncontrolled interfacial polymerization driven by excessive MPD diffusion from substrate pores into the organic phase [18]. In contrast, as shown in Fig. 2b, the GO-interlayered membrane exhibited a significantly smoother, more uniform surface structure. The ridge-and-valley features became smaller and more compact, indicating that the interfacial polymerization process was effectively regulated by the GO interlayer. The improved morphology can be attributed to several synergistic effects. First, the enhanced hydrophilicity of the GO-coated substrate enabled a more uniform distribution of the aqueous monomer solution. Second, the interlayer slowed down the diffusion of MPD monomers into the organic phase, which helped prevent aggressive polymerization reactions. Additionally, the smoother, more uniform PA layer is expected to exhibit fewer defects, resulting in lower reverse salt flux and enhanced membrane selectivity. Overall, the SEM observations clearly demonstrate that the GO interlayer effectively tailors the interfacial polymerization process and enables the fabrication of a thin, defect-free, and highly selective PA active layer suitable for FO desalination applications.

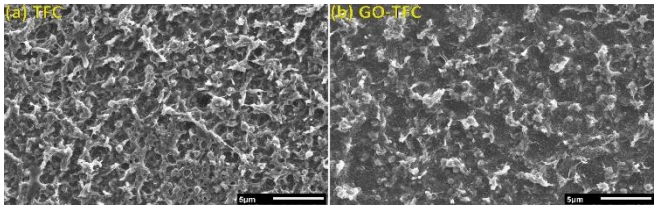


Figure 2. SEM images of TFC-FO membranes, (a) control TFC membrane without interlayer, and (b) GO-interlayered TFC membrane.

Source: Research findings

3.4. Effect of GO Interlayer on FO Performance

The FO performance of the fabricated membranes was systematically evaluated in terms of water flux and reverse salt flux using DI water as the FS and 1.0 M NaCl as the DS. The results in Fig. 3 clearly demonstrated that incorporating the GO interlayer significantly improved the overall FO desalination performance of the TFC membrane. Compared with the pristine TFC membrane, the GO-interlayered membrane exhibited a substantially higher water flux. The enhanced water permeability is primarily attributable to the formation of a thinner, more uniform PA-selective layer. In conventional TFC membranes, uncontrolled interfacial polymerization and PA intrusion into substrate pores generate a thick active layer with high transport resistance [19,20]. However, the GO interlayer effectively regulated the diffusion of MPD monomers and confined the interfacial polymerization reaction near the membrane surface, leading to the formation of an ultrathin PA layer with lower resistance against water transport. Furthermore, the highly hydrophilic nature of GO nanosheets significantly enhanced water permeation. The abundant oxygen-containing functional groups on GO improved surface wettability and facilitated rapid adsorption and transport of water molecules across the membrane structure. In addition, the stacked two-dimensional GO nanosheets may create interconnected nanochannels that provide preferential pathways for water transport [21]. Another important factor contributing to the improved water flux is the suppression of internal concentration polarization (ICP). The porous and hydrophilic interlayer can promote more efficient mass transfer near the substrate surface and reduce solute accumulation inside the support structure [22]. Consequently, the effective osmotic driving force across the membrane is better preserved during FO operation. In addition to improving water permeability, the GO interlayer significantly reduced reverse salt flux. The pristine TFC membrane prepared on the bare PVDF substrate exhibited relatively higher reverse salt flux due to the presence of structural defects and non-uniform PA morphology. In contrast, the GO-modified membrane formed a denser and more homogeneous selective layer with fewer non-selective transport pathways. The controlled diffusion of MPD monomers through the GO interlayer was crucial to producing a highly crosslinked PA structure with improved integrity. Moreover, the interfacial interactions between GO functional groups and polyamide chains likely enhanced the compactness of the selective layer and minimized defect formation. As a result, salt diffusion from the draw side toward the feed side was effectively suppressed. The simultaneous enhancement of water flux and reduction of reverse salt flux indicate a substantial improvement in membrane perm-selectivity after incorporation of the GO interlayer. Such behavior is highly desirable for practical FO desalination applications because high water productivity and low solute leakage are both essential for efficient process performance. Notably, the GO-interlayered membrane demonstrated stable FO operation and maintained high permeation performance during desalination experiments, confirming the structural robustness of the fabricated membrane. The results suggest that the proposed GO interlayer strategy is highly effective in developing advanced FO membranes for seawater desalination.

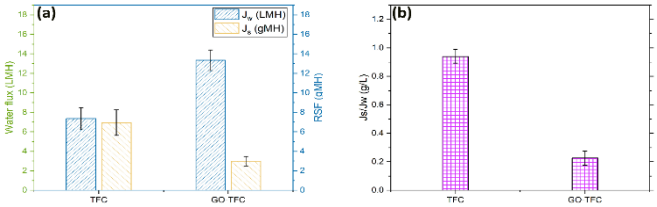


Figure 3. FO performance of TFC membranes in terms of (a) water flux and salt flux, and (b) selectivity. The test was conducted with 3 membrane samples, and the average data was reported.

Source: Research findings

3.5. Application of GO-TFC Membranes for Seawater Desalination

The practical applicability of the fabricated membranes for seawater desalination was further evaluated by assessing their permeability-selectivity performance during FO. This test used a feed solution of 35 $g \cdot L^{-1}$ model seawater and a draw solution of 1.0 M NaCl. The results clearly indicate that the GO-interlayered membrane exhibits superior desalination performance compared to the pristine TFC membrane. In desalination applications, especially for highly saline feed streams such as seawater, an ideal FO membrane should simultaneously exhibit high water flux and low reverse salt flux. High water flux ensures efficient freshwater production, while low salt leakage is essential to maintain osmotic driving force and minimize draw solute loss during operation. The pristine TFC membrane prepared on the bare PVDF substrate exhibited relatively limited desalination performance, with a water flux of 3.2 LMH due to the formation of a rough, partially defective PA layer. The uncontrolled interfacial polymerization on the porous substrate generated non-selective transport pathways and increased mass transfer resistance, leading to lower water permeability and higher reverse salt diffusion. By contrast, the GO-interlayered membrane demonstrated significantly improved desalination behavior, with a water flux of 5.1 LMH. The ultrathin GO interlayer enabled the formation of a highly uniform, defect-free PA-selective layer with reduced thickness. Consequently, the membrane exhibited substantially higher water flux together with reduced reverse salt flux. Given the high salinity and harsh operating conditions of Persian Gulf seawater, the fabricated GO-TFC membrane shows strong potential for practical desalination applications in this region. Persian Gulf seawater typically has higher salinity than that of many other natural bodies of seawater, posing substantial challenges for membrane processes [23]. Therefore, membranes with high water permeability and low reverse salt flux are critically required. The present results suggest that the GO-interlayered membrane can effectively operate under such demanding conditions due to its improved permeability-selectivity balance and robust PA structure. Furthermore, the low hydraulic pressure requirement and reduced fouling tendency of the FO process make this membrane highly attractive for sustainable seawater desalination in arid regions such as the Middle East. Overall, the developed GO-TFC membrane provides a promising platform for next-generation FO desalination technologies and could potentially contribute to freshwater production from Persian Gulf seawater with improved efficiency and lower energy consumption.

4. Conclusions

In this work, a high-performance TFC-FO membrane was successfully fabricated by incorporating a GO interlayer onto a porous PVDF substrate. The ultrathin GO interlayer played a crucial role in regulating the interfacial polymerization process and in tailoring the morphology of the PA-selective layer. The incorporation of the GO interlayer significantly improved substrate hydrophilicity and controlled the diffusion behavior of MPD monomers during PA formation. As a result, a thinner, smoother, and more uniform PA active layer with fewer structural defects was successfully obtained. SEM analyses confirmed the successful formation of the GO interlayer and its strong influence on membrane structure and morphology. The GO-interlayered membrane demonstrated superior FO performance compared with the pristine

TFC membrane. The membrane showed a 90% increase in water flux, rising from 7.3 LMH in the control TFC membrane to 13.3 LMH in the GO-TFC membrane. The reverse salt flux decreases to 3.0 gMH in the GO-TFC membrane, demonstrating a significant enhancement in membrane selectivity. The improved desalination performance was mainly attributed to reduced transport resistance, suppression of PA intrusion into the substrate pores, and the formation of a highly crosslinked, defect-free selective layer. Moreover, the developed membrane showed promising capability for seawater desalination applications under FO conditions. The GO-TFC membrane exhibited a water flux of 5.1 LMH, which is 59% higher than the control TFC membrane's 3.9 LMH. Given the high salinity of Persian Gulf seawater, the fabricated GO-TFC membrane is a promising candidate for practical desalination processes due to its high permeability, low salt leakage, and stable membrane structure. Overall, this study demonstrates that GO-based interlayers offer an effective, scalable strategy for fabricating next-generation FO membranes with improved desalination performance and reduced energy demand.

Author Contributions

All authors contributed equally to the conception, preparation, and writing of this manuscript.

Conflict of Interest

The authors, while observing publication ethics in the referencing, declare the absence of interest of conflict.

Acknowledgement

The Authors gratefully acknowledges the support provided by theUniversity of Tehran in conducting this research.

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