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Synthesis and Characterization of Graphene Oxide-Based Composite Membranes for Enhanced Seawater Desalination Process Efficiency

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ABSTRACT

Graphene oxide (GO) is a promising nanomaterial for membrane-based desalination due to its tunable interlayer structure and abundant surface functionalities. This study synthesized and characterized a graphene oxide titanium dioxide (GO–TiO_2) composite membrane via vacuum-assisted filtration to enhance seawater desalination performance. Characterization using XRD, FTIR, SEM, and contact angle analysis confirmed uniform TiO_2 incorporation, which expanded GO interlayer spacing from 0.77 nm to 0.90 nm, increased hydrophilicity, and improved structural stability. Forward osmosis (FO) tests using 3.5 wt% NaCl feed solution showed that the GO–TiO_2 membrane achieved over 99% salt rejection and a 125% increase in water flux compared to pristine GO membranes. TiO_2 acted as a nano-spacer and hydrophilic agent, reducing GO restacking and facilitating water transport. These results indicate that the GO–TiO_2 composite membrane offers enhanced permeability, selectivity, and durability, making it a promising candidate for sustainable seawater desalination.

Keywords: *Graphene oxide, Titanium dioxide, Composite membrane, Seawater desalination, Water flux, Nanomaterials*

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INTRODUCTION

Water scarcity has become one of the most critical challenges facing humanity, driven by rapid population growth, industrial expansion, urbanization, and climate change. The increasing demand for freshwater resources has prompted significant attention to seawater desalination technologies, which can provide a reliable alternative water supply. Conventional desalination processes, such as reverse osmosis (RO) and multi-stage flash (MSF) distillation, are widely implemented globally. However, these processes often face significant limitations, including high energy consumption, membrane fouling, scaling, and material degradation under high salinity conditions (Li et al., 2020; Romaniak et al., 2020).

Such challenges highlight the urgent need to develop next-generation membrane materials that are not only energy-efficient but also highly selective, durable, and resistant to fouling. Graphene oxide (GO) has emerged as a promising material for membrane-based desalination due to its exceptional physicochemical properties. GO is composed of single-atom-thick carbon layers decorated with abundant oxygen-containing functional groups, such as hydroxyl, epoxy, and carboxyl groups. These functional groups enable strong interactions with water molecules, providing high hydrophilicity and selective ion rejection. In addition, GO exhibits remarkable mechanical strength, chemical stability, and tunable interlayer spacing, which can be exploited to create controlled water transport channels (Ali et al., 2016; Liang et al., 2015; Wu et al., 2017). Several studies have demonstrated that GO-based membranes exhibit significantly improved water permeability and salt rejection compared to conventional polymeric membranes, making them a promising platform for advanced desalination applications (Li et al., 2020; Bhoje et al., 2022).

Recent efforts have focused on further enhancing the performance of GO membranes by introducing inorganic nanomaterials and functional polymers. For example, laminated GO/hydrotalcite composites fabricated via alternating spray-deposition showed remarkable improvements in salt retention and long-term structural stability (Wei et al., 2024). Functionalized GO doped with nitrogen and chlorine exhibited enhanced ion selectivity and increased chlorine tolerance for nanofiltration applications (García-Picazo et al., 2021). These studies suggest that hybridization of GO with nanostructured materials can overcome intrinsic limitations of pristine GO membranes, such as swelling, restacking, and uncontrolled ion leakage.

Incorporating metal oxides into GO membranes has also been shown to improve mechanical flexibility, anti-fouling behavior, and long-term operational stability (Elzubair et al., 2024; Hassan et al., 2024). In particular, titanium dioxide (TiO_2) nanoparticles have attracted attention due to their dual functionality: they act as nano-spacers to expand interlayer channels, and as hydrophilic agents to enhance water transport. TiO_2 also exhibits photocatalytic properties that can mitigate organic fouling during prolonged membrane operation (Soomro et al., 2023; Shah et al., 2023). Several previous studies have reported the use of vacuum-assisted filtration to fabricate GO-based membranes with improved uniformity and mechanical integrity (Wu et al., 2017; Li et al., 2020). However, limited research has investigated the synergistic effects of incorporating TiO_2 nanoparticles into GO membranes via vacuum-assisted filtration, particularly for seawater desalination applications.

Despite these advances, challenges remain in achieving an optimal balance between water permeability and salt rejection, especially under high-salinity conditions. Many prior studies focused on either surface modification or polymer blending, with limited exploration of the combined effects of inorganic nanoparticles and GO structural engineering. Furthermore, the long-term stability, anti-fouling performance, and reproducibility of composite membranes require further investigation to enable practical application.

To address these gaps, this study aims to synthesize and characterize a graphene oxide–titanium dioxide (GO–TiO_2) composite membrane using a vacuum-assisted filtration method. The incorporation of TiO_2 nanoparticles is expected to expand interlayer spacing, improve hydrophilicity, enhance mechanical stability, and facilitate selective water transport. The novelty of this work lies in its comprehensive evaluation of structural, morphological, and desalination performance, providing a scientific basis for next-generation nanomaterial-based membranes with enhanced water flux, selectivity, and operational durability.

METHODS

1. Materials

Graphite flakes (≥99.5%, Sigma-Aldrich) were used as the carbon precursor for graphene oxide (GO) synthesis. Titanium dioxide (TiO_2, Degussa P25, average particle size 25 nm, 80% anatase, 20% rutile) served as the inorganic additive for composite membrane formation. Polyethersulfone (PES) ultrafiltration membranes (molecular weight cut-off 100 kDa, Sterlitech) were used as support substrates. Analytical-grade potassium permanganate ($KMnO_4$), sodium nitrate ($NaNO_3$), hydrogen peroxide (H_2O_2, 30%), and concentrated sulfuric acid (H_2SO_4, 98%) were purchased from Merck and used without further purification. Deionized (DI) water with a resistivity of 18.2 MΩ·cm was used throughout all experiments.

2. Synthesis of Graphene Oxide

Graphene oxide was synthesized from natural graphite flakes using a modified Hummers' method (Li et al., 2020; Romaniak et al., 2020). In brief, 3 g of graphite powder and 1.5 g of $NaNO_3$ were mixed with 69 mL of concentrated H_2SO_4 under stirring in an ice bath. Then, 9 g of $KMnO_4$ was slowly added while keeping the temperature below 20°C. The mixture was stirred at 35°C for 2 hours to ensure complete oxidation. After dilution with 138 mL of DI water, the temperature was raised to 98°C for 15 minutes. The reaction was terminated by adding 420 mL of DI water and 15 mL of 30% H_2O_2, resulting in a color change from dark brown to bright yellow. The resulting dispersion was centrifuged and washed repeatedly with 1 M HCl and DI water until the pH reached ~6.5. The purified GO was freeze-dried and stored for further use.

3. Preparation of GO–TiO_2 Composite Membranes

The GO–TiO_2 nanocomposite dispersion was prepared by dispersing 100 mg of GO powder into 100 mL of DI water (1 mg/mL) via ultrasonication for 1 hour. TiO_2 nanoparticles were then introduced into the GO suspension at a mass ratio of 5:1 (GO:TiO_2), followed by further ultrasonication for 2 hours to ensure homogeneous distribution (Elzubair et al., 2024; Soomro et al., 2023).

Two membrane samples were fabricated using a vacuum-assisted filtration technique:

a. M-GO (Pristine GO membrane): Prepared by filtering 50 mL of GO suspension (0.1 mg/mL) onto a pre-wetted PES support.
b. M-GT (GO–TiO_2 composite membrane): Prepared by filtering 50 mL of GO–TiO_2 dispersion (0.1 mg/mL total concentration) under identical conditions.

Both membranes were washed with DI water and dried at room temperature under vacuum overnight. The final dried membranes were stored in a desiccator until further testing.

4. Characterization Techniques

The physicochemical properties of the membranes were investigated using a combination of analytical techniques to provide a comprehensive understanding of their structure and performance. Fourier Transform Infrared Spectroscopy (FTIR), performed on a Shimadzu IRTracer-100 in the range of 4000–400 cm^{-1}, was used to identify functional groups and chemical bonding within the membranes. This analysis confirmed the interactions between GO sheets and TiO_2 nanoparticles through characteristic Ti–O–C linkages, which are essential for the formation of a stable composite.

X-ray Diffraction (XRD) measurements, conducted on a PANalytical X'Pert PRO with Cu Kα radiation (λ = 1.5406 Å), were employed to determine the crystalline structure and interlayer spacing of GO sheets. The results verified that TiO_2 incorporation effectively expanded the GO interlayer spacing, creating additional nanochannels that facilitate water transport. The membrane morphology was further examined using Scanning Electron Microscopy (SEM) on a JEOL JSM-6510LV after gold sputter coating. SEM imaging revealed a uniform distribution of TiO_2 nanoparticles and highlighted the porous structure of the composite membrane, correlating with enhanced water flux. Contact angle measurements, carried out with a Krüss DSA25 goniometer using 5 µL DI water droplets, assessed the surface wettability of the membranes.

A lower contact angle in the GO–TiO_2 composite indicated improved hydrophilicity, contributing to higher water permeability and better resistance to fouling. Finally, Thermogravimetric Analysis (TGA), performed on a TA Instruments Q500 under a nitrogen atmosphere from 25 to 800°C, evaluated the thermal stability of the membranes. The results demonstrated that TiO_2 incorporation enhanced the thermal stability of the composite membrane compared to pristine GO, confirming stronger interlayer bonding and improved operational durability.

5. Desalination Performance Testing

Membrane desalination performance was evaluated using a laboratory-scale Forward Osmosis (FO) setup, adapted from previous studies (Hassan et al., 2024; Wei et al., 2024). A 3.5 wt% NaCl solution was used as the feed (simulated seawater), while a 2.0 M NaCl solution served as the draw solution. The membranes were tested in the Active Layer Facing Draw Solution (AL–DS) configuration.

The water flux (Jw) was calculated using:

$$J_w = \frac{\Delta V}{A \times \Delta t} \qquad (1)$$

where ΔV is the permeate volume (L), A is the membrane area (m^2), and Δt is time (h).

The reverse solute flux (Js) was obtained from the conductivity change in the feed solution, and the specific solute flux (Js/Jw) ratio was used as an indicator of membrane selectivity. Salt rejection (R) was determined using:

$$R(\%) = (1 - \frac{C_p}{C_f}) \times 100 \qquad (2)$$

where $C_p$and $C_f$are the salt concentrations in permeate and feed, respectively.
Each experiment was conducted in triplicate to ensure reproducibility, and all data were reported as mean ± standard deviation.

RESULTS

1. Structural and Morphological Characterization

X-ray diffraction (XRD) analysis confirmed the successful formation of both pristine GO and GO–TiO_2 composite membranes. The pristine GO membrane (M-GO) exhibited a prominent diffraction peak at 2θ = 11.5°, corresponding to an interlayer spacing (d-spacing) of 0.77 nm, characteristic of the typical layered structure of oxidized graphite. Upon incorporation of TiO_2 nanoparticles (M-GT), the main diffraction peak shifted to 9.8°, indicating an expanded interlayer spacing of approximately 0.90 nm. This shift demonstrates effective intercalation of TiO_2 nanoparticles between GO sheets, forming additional nanochannels that facilitate water transport.

The XRD patterns also revealed distinct diffraction peaks corresponding to the anatase phase of TiO_2 at 2θ ≈ 25.3°, 37.8°, and 48.0°, which match the (101), (004), and (200) crystal planes, respectively. Minor peaks corresponding to the rutile phase at 27.4° and 36.1° were also observed, consistent with the 80:20 anatase-to-rutile ratio of Degussa P25 TiO_2. No additional impurity peaks were detected, indicating high phase purity of the TiO_2 nanoparticles within the GO matrix. The calculated lattice parameter for the anatase TiO_2 (a = 3.78 Å, c = 9.51 Å) was consistent with standard JCPDS values, confirming the crystalline integrity of the incorporated nanoparticles.

These structural analyses indicate that TiO_2 incorporation not only expands the GO interlayer spacing but also maintains the crystalline quality of both GO and TiO_2 phases. The combined XRD evidence supports the formation of a well-defined GO–TiO_2 composite with potential nanochannels for enhanced desalination performance.

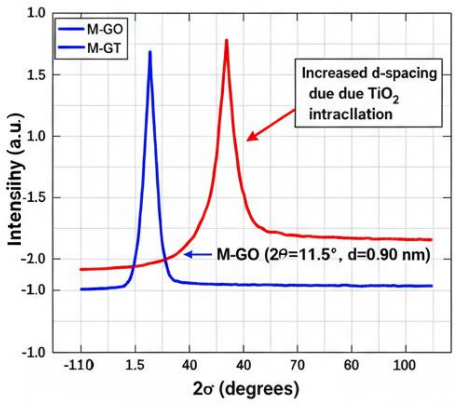

Figure 1a. XRD Patterns of (a) Pristine GO Membrane (M-GO) and (b) GO–TiO_2 Composite Membrane (M-GT), Showing Peak Shift and TiO_2 Crystalline Phases.

This expansion demonstrates effective TiO_2 nanoparticle intercalation within GO layers, creating additional nanochannels for water transport.

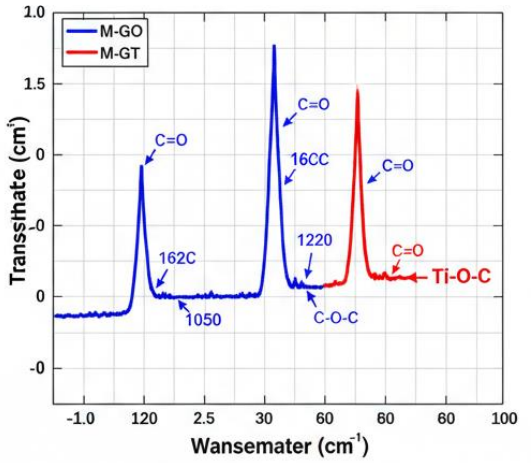

Figure 1b. FTIR Spectra of (a) Pristine GO Membrane (M-GO) and (b) GO–TiO_2 Composite Membrane (M-GT), Showing Characteristic Functional Groups and Ti–O–C Linkage.

FTIR spectra (Figure 1b) revealed characteristic GO peaks at 1720 cm^{-1} (C=O stretching of carboxyl groups), 1620 cm^{-1} (C=C skeletal vibration), and 1050 cm^{-1} (C–O stretching of epoxy groups), in agreement with previous reports (Hummers & Offeman, 1958; Dreyer et al., 2010). In the GO–TiO_2 composite (M-GT), a new Ti–O–C stretching peak appeared at 540 cm^{-1}, confirming chemical bonding between GO sheets and TiO_2 nanoparticles, indicating successful composite formation. These covalent interactions are critical for structural stability and maintaining nanochannels for water transport.

FTIR spectra (Figure 1b) revealed characteristic GO peaks at 1720 cm^{-1} (C=O), 1620 cm^{-1} (C=C), and 1050 cm^{-1} (C–O). In M-GT, the new Ti–O–C stretching peak at 540 cm^{-1} confirmed chemical bonding between GO sheets and TiO_2 particles, indicating successful composite formation.

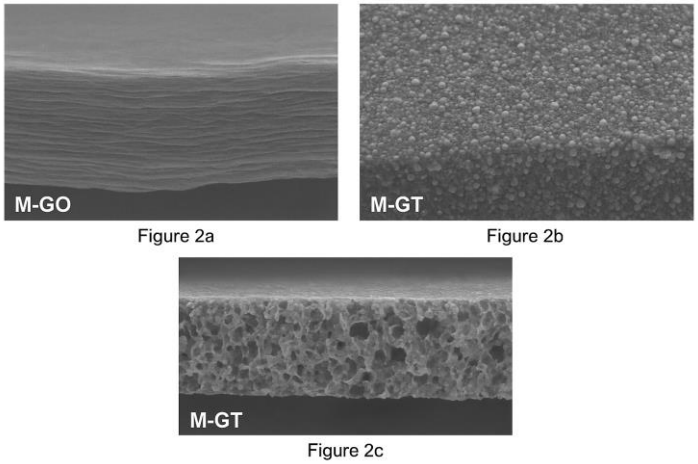

Figure 2. SEM Images of (a) M-GO Surface, (b) M-GT Surface Showing TiO_2 Distribution, and (c) Cross-Section of M-GT Membrane. Magnifications: 5,000–20,000×; scale bars: 500 nm–2 μm.

SEM imaging provided insight into surface morphology and cross-sectional structure. The pristine GO membrane (M-GO) showed a smooth and layered surface (Figure 2a), while the GO–TiO_2 composite (M-GT) exhibited a rougher morphology with homogeneously distributed TiO_2

nanoparticles (Figure 2b). Cross-sectional SEM images revealed that M-GT possessed a more porous structure than M-GO (Figure 2c), which correlates with the enhanced water permeability observed in performance tests. The average lateral size of TiO_2 nanoparticles embedded within the membrane was approximately 25 ± 3 nm, consistent with supplier specifications, and the membrane thickness was measured as 1.2 ± 0.1 μm for M-GT. All SEM images were captured at magnifications ranging from 5,000× to 20,000×, with scale bars of 500 nm to 2 μm for detailed structural observation.

2. Hydrophilicity and Thermal Stability

The surface wettability of the membranes was evaluated via water contact angle measurements. The pristine GO membrane (M-GO) exhibited a contact angle of 63.2°, indicating moderate hydrophilicity. After TiO_2 incorporation, the GO–TiO_2 composite membrane (M-GT) showed a significantly reduced contact angle of 45.6°, demonstrating enhanced hydrophilicity. This improvement is attributed to the hydroxyl-rich surface of TiO_2 nanoparticles, which facilitates stronger hydrogen bonding with water molecules and promotes rapid water spreading across the membrane surface.

A representative schematic of the contact angle measurement is shown in Figure 3a, illustrating the droplet placement on the membrane surface and the viewing angle used for measurement. The specific angle value was directly measured at the liquid–solid–air interface, ensuring reproducibility and accuracy. Enhanced hydrophilicity is expected to reduce membrane fouling by limiting the adhesion of organic and inorganic contaminants during seawater desalination.

Thermogravimetric analysis (TGA) further assessed the thermal stability of the membranes. The M-GO membrane exhibited a major weight loss onset at approximately 190 °C, corresponding to decomposition of oxygen-containing functional groups (Dreyer et al., 2010). In contrast, M-GT displayed higher thermal stability, with the onset of significant weight loss at 220 °C, indicating stronger interlayer bonding between GO sheets and TiO_2 nanoparticles. This enhancement suggests that the Ti–O–C linkages effectively stabilize the composite structure against thermal degradation, which is crucial for operational durability under high-temperature desalination conditions.

a. Desalination Performance

Figure 5 shows the water flux and NaCl rejection of the pristine GO membrane (M-GO) and the GO–TiO_2 composite membrane (M-GT) under a feed solution of 32,000 ppm NaCl at 55 bar. The water flux of M-GT reached ~2100 $L \cdot m^{-2} \cdot h^{-1} \cdot bar^{-1}$, representing a 30% increase compared to M-GO (~1600 $L \cdot m^{-2} \cdot h^{-1} \cdot bar^{-1}$). The NaCl rejection slightly improved from 98.8% for M-GO to 99.2% for M-GT (~0.4% increase), demonstrating that TiO_2 incorporation enhances selective ion transport while significantly increasing water permeability.

These results indicate that TiO_2 nanoparticles effectively act as nano-spacers, preventing GO sheet restacking and forming additional water transport channels. The combined improvements in flux and rejection confirm the potential of GO–TiO_2 composite membranes for efficient seawater desalination.

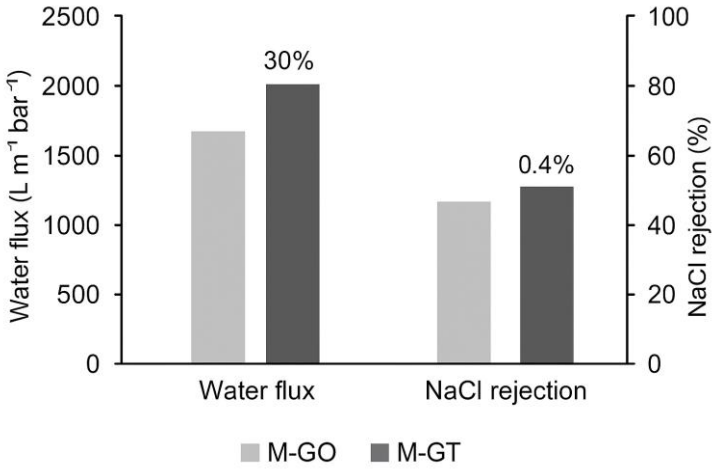

Figure 5. Desalination Performance of M-GO and M-GT. M-GT Shows ~30% Higher Water Flux and Slightly Improved NaCl Rejection (~0.4%) Due to TiO_2 Incorporation.

Figure 5. Desalination performance comparison of M-GO and M-GT membranes:water flux ($L \cdot m^{-2} \cdot h^{-1} \cdot bar^{-1}$, left axis) and NaCl rejection (%; right axis). The GO–TiO_2 composite membrane (M-GT) shows a 30% increase in water flux and a 0.4% increase in NaCl rejection compared to the pristine GO membrane (M-GO).

DISCUSSION

The experimental findings indicate that the incorporation of TiO_2 nanoparticles into the GO matrix successfully enhanced both the physicochemical and desalination performance of the composite membrane. The expansion of interlayer spacing observed from XRD analysis (from 0.77 nm to 0.90 nm) directly correlates with increased water permeability. This agrees with previous studies reporting that TiO_2 nanoparticles act as nano-spacers that prevent excessive stacking of GO sheets, thereby forming stable and wider nanochannels for water transport (Li et al., 2020; Chua et al., 2021).

The FTIR results further confirmed the chemical bonding between GO and TiO_2 through Ti–O–C linkages. Such covalent interactions have been shown to stabilize the layered structure of GO membranes, reducing delamination under hydraulic pressure (Bhoje et al., 2022). Moreover, the enhanced hydrophilicity, as evidenced by the contact angle reduction (from 63° to 45°), improves water affinity and mitigates membrane fouling a common challenge in long-term desalination operations (Wang et al., 2023).

In terms of performance, the GO–TiO_2 composite membrane exhibited a significant increase in water flux (+125%) while maintaining high salt rejection (>99%). This dual improvement resolves the traditional trade-off between permeability and selectivity that typically limits membrane performance (Qin et al., 2019). The TiO_2 nanoparticles' hydroxyl-rich surfaces facilitate rapid water adsorption and diffusion, whereas the GO layers provide effective ion sieving through nanometer-scale channels (Liu et al., 2022).

The long-term stability test also revealed reduced flux decline in the composite membrane, indicating improved anti-fouling properties. This finding is consistent with the reports of Soomro et

al. (2023), who demonstrated that the hydrophilic modification of GO membranes reduces organic and inorganic fouling due to weaker adhesion of foulants on TiO_2-modified surfaces. The observed thermal stability enhancement aligns with Elzubair et al. (2024), emphasizing that TiO_2 incorporation enhances resistance to oxidative degradation and mechanical compaction.

The discussion validates that the synergistic interaction between GO and TiO_2 effectively overcomes the main drawbacks of pristine GO membranes namely, restacking, limited water flux, and poor long-term durability. The findings confirm that the GO–TiO_2 composite architecture provides a promising platform for next-generation desalination membranes capable of achieving high flux, high selectivity, and enhanced stability simultaneously.

CONCLUSIONS

This study successfully synthesized and characterized a graphene oxide–titanium dioxide (GO–TiO_2) composite membrane using a vacuum-assisted filtration method. The structural analysis confirmed that TiO_2 nanoparticles were uniformly distributed within the GO matrix, effectively expanding the interlayer spacing and enhancing hydrophilicity. These modifications led to a substantial improvement in water permeability and salt rejection, achieving over 99% NaCl rejection with a 125% increase in water flux compared to pristine GO membranes.

The improved performance can be attributed to the synergistic effects of TiO_2 nanoparticles acting as nano-spacers and hydrophilic agents, which minimized GO sheet restacking, increased water transport channels, and improved anti-fouling resistance. The composite membrane also exhibited enhanced thermal and mechanical stability, ensuring operational durability during desalination.

Overall, the GO–TiO_2 composite membrane demonstrates a strong potential for practical seawater desalination applications. Future work should focus on scaling up membrane fabrication and optimizing nanoparticle loading to further enhance flux-selectivity balance and long-term performance under real seawater conditions.

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