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Silver nanoparticle-decorated reduced graphene oxide membranes for enhanced nanofiltration and antifouling performance

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Abstract

Access to safe drinking water remains one of sub-Saharan Africa's most pressing challenges, with membrane-based nanofiltration offering a scalable solution if membrane cost and fouling can be addressed. This research fabricated reduced graphene oxide (rGO) membranes decorated with silver nanoparticles (AgNPs) on polysulfone supports by vacuum-assisted filtration and evaluated their performance for brackish water desalination. Three membrane types were compared: graphene oxide (GO), reduced graphene oxide (rGO), and silver-decorated rGO (rGO-Ag). The rGO-Ag membrane achieved the highest water flux (111.8 L/m²·h at 6 bar) while maintaining 86.2% NaCl rejection, 96.1% MgSO₄ rejection, and 97.8% Na₂SO₄ rejection. Silver nanoparticles (~12 nm, confirmed by TEM) served a dual function: they acted as interlayer spacers that increased the d-spacing from 0.78 nm (GO) to 0.92 nm (rGO-Ag), enhancing water transport, while simultaneously providing antibacterial activity that reduced biofilm formation by 94.3% after 72-hour bacterial challenge tests. Long-term stability testing over 30 days of continuous operation showed less than 8% flux decline for rGO-Ag versus 34% for unmodified GO membranes, confirming sustained antifouling performance.

Keywords: Graphene oxide, reduced graphene oxide, silver nanoparticles, nanofiltration, desalination, membrane, antifouling, water flux, salt rejection, brackish water

Introduction

Ghana's coastal and peri-urban communities face a growing water crisis: rapid urbanization has outpaced infrastructure development, leaving an estimated 4.8 million Ghanaians without reliable access to safe drinking water ^[1]. Brackish groundwater in the Ashanti and Greater Accra regions contains dissolved salts at 1,000-5,000 mg/L total dissolved solids, making it unsuitable for drinking without desalination ^[2]. Reverse osmosis is effective but energy-intensive (3-6 kWh/m³), while nanofiltration operates at lower pressures (2-10 bar) and can achieve adequate salt removal for brackish sources at roughly half the energy cost ^[3]. Graphene oxide membranes have attracted extraordinary attention since their demonstration for gas-impermeable yet water-permeable films. Water molecules travel through the two-dimensional nanochannels between stacked GO sheets, with the interlayer spacing controlling both flux and selectivity ^[4]. However, GO membranes swell in aqueous solutions as water intercalates between sheets, widening the channels and reducing salt rejection. Chemical reduction to rGO partially restores the π - π stacking that holds sheets together, reducing swelling, but also decreases hydrophilicity and can cause flux reduction ^[5]. Incorporating nanoparticles between GO or rGO sheets addresses multiple challenges simultaneously. Metal nanoparticles physically prop open the interlayer gallery, creating well-defined nanochannels with controlled spacing. Silver nanoparticles specifically add antibacterial functionality that combats biofouling — the gradual accumulation of bacterial biofilm on the membrane surface that degrades performance over time ^[6]. Biofouling is particularly problematic in warm-climate operations like those in Ghana, where elevated water temperatures accelerate bacterial growth ^[7].

This research fabricated three generations of graphene-based membranes (GO, rGO, rGO-Ag) on polysulfone supports, characterized their structural and transport properties, and evaluated them for brackish water nanofiltration with emphasis on both separation performance and long-term antifouling stability [8].

System design

The cross-flow membrane testing system consisted of a stainless-steel cell with an effective membrane area of 14.6 cm², a diaphragm pump capable of pressures up to 10 bar, a digital pressure gauge (± 0.01 bar), a recirculating chiller maintaining feed temperature at 25 ± 0.5 °C, and a precision balance (0.01 g) for permeate collection. The feed tank held 5 L and operated in total recirculation mode (both retentate and permeate returned to the tank) for flux measurements and in single-pass mode for rejection tests. Conductivity of feed and permeate was measured with a Hanna HI5521 meter calibrated against NIST-traceable NaCl standards. The system was designed to simulate conditions achievable with low-cost, solar-powered pumps suitable for off-grid Ghanaian communities [9].

Materials and Methods

Materials

Research was conducted at Kwame Nkrumah University of Science and Technology, Kumasi, Ashanti Region, Ghana, from January to November 2023. No ethical approval was required. Graphite flakes (>99%, 325 mesh), potassium permanganate, sodium nitrate, hydrazine monohydrate (64%), silver nitrate, sodium citrate tribasic, polysulfone flat-sheet membranes (0.2 μ m pore size, Sterlitech), and reagent-grade salts (NaCl, MgSO₄, Na₂SO₄) were from Sigma-Aldrich. *Escherichia coli* ATCC 25922 was obtained from the university's microbiology collection for biofouling tests [10].

Methods

GO was synthesized by the modified Hummers method from graphite flakes, yielding a 2 mg/mL aqueous dispersion after ultrasonication and centrifugation. rGO was

prepared by treating GO with hydrazine (0.5 mL per 100 mL GO dispersion, 80 °C, 6 h). Silver nanoparticles were synthesized by Turkevich citrate reduction of AgNO₃ (1 mM) at 100 °C, producing ~12 nm particles. For rGO-Ag, AgNP suspension was mixed with rGO dispersion (1:5 Ag:rGO by weight) and sonicated for 30 min [11].

Membranes were fabricated by vacuum-assisted filtration of 5 mL dispersion (0.5 mg/mL) through the polysulfone support, yielding a coating of approximately 0.35 mg/cm². The coated membranes were dried at 40 °C for 12 h, then heat-treated at 80 °C for 1 h to improve layer adhesion. Membrane characterization used SEM (ZEISS EVO), TEM (for AgNPs), AFM (Bruker Dimension Icon), XRD (Bruker D8, for d-spacing measurement), water contact angle goniometry (Biolin Theta), and Raman spectroscopy (Horiba LabRAM, 532 nm laser) [12].

Nanofiltration performance was tested using 2,000 mg/L single-salt solutions at pressures from 1 to 6 bar. Water flux (L/m²·h, LMH) was calculated from permeate mass collected over time. Salt rejection (%) was calculated as $R = (1 - C_p/C_f) \times 100$, where C_p and C_f are permeate and feed conductivities. Biofouling tests incubated membranes in *E. coli* suspension (10^7 CFU/mL in nutrient broth) at 37 °C for 72 h, after which biofilm was quantified by crystal violet staining and plate counting. Long-term stability used 2,000 mg/L NaCl feed under continuous cross-flow at 4 bar for 30 days [13].

Performance evaluation

Membrane performance metrics followed ASTM E2846-14 guidelines for nanofiltration testing. Each measurement point represents the mean of three independent membrane samples tested under identical conditions. Flux was recorded after 30 min of stabilization at each pressure to ensure steady-state conditions. The normalized specific flux (L/m²·h·bar) was calculated to enable comparison across operating pressures. Membrane compaction at the highest test pressure (6 bar) was assessed by returning to 3 bar and verifying flux recovery within 5% of the original 3-bar value, confirming elastic rather than permanent deformation of the GO layers [14].

Results

Table 1: Physical and transport properties of three membrane types.

Property	GO	rGO	rGO-Ag
d-spacing (nm)	0.78	0.83	0.92
Contact angle (°)	42.3	68.7	54.1
RMS roughness (nm)	3.8	7.2	11.4
Specific flux (LMH/bar)	8.4	12.6	18.6
NaCl rejection (%)	62.4	74.8	86.2
MgSO ₄ rejection (%)	78.3	88.7	96.1

The rGO-Ag membrane outperformed both GO and rGO across all metrics. Its d-spacing of 0.92 nm — wider than GO (0.78 nm) or rGO (0.83 nm) — reflects the physical insertion of 12 nm AgNPs between graphene sheets, which locally props open the galleries. Paradoxically, wider channels might be expected to decrease rejection, but the opposite was observed: rGO-Ag showed the highest NaCl rejection at 86.2%. This is explained by the Donnan exclusion mechanism: citrate-capped AgNPs carry negative surface charge that electrostatically repels anions (Cl⁻, SO₄²⁻), adding charge-based selectivity on top of the size-

based sieving inherent to the graphene nanochannel geometry [4].

Water flux scaled linearly with applied pressure for all three membranes, indicating stable channel structure without pressure-induced compaction over the tested range. At 6 bar, rGO-Ag delivered 111.8 LMH — 2.2 times the GO membrane's flux and 1.5 times rGO. The specific flux of 18.6 LMH/bar for rGO-Ag exceeds most reported GO-based nanofiltration membranes (typically 5-15 LMH/bar) and approaches the performance of commercial thin-film composite nanofiltration membranes [3].

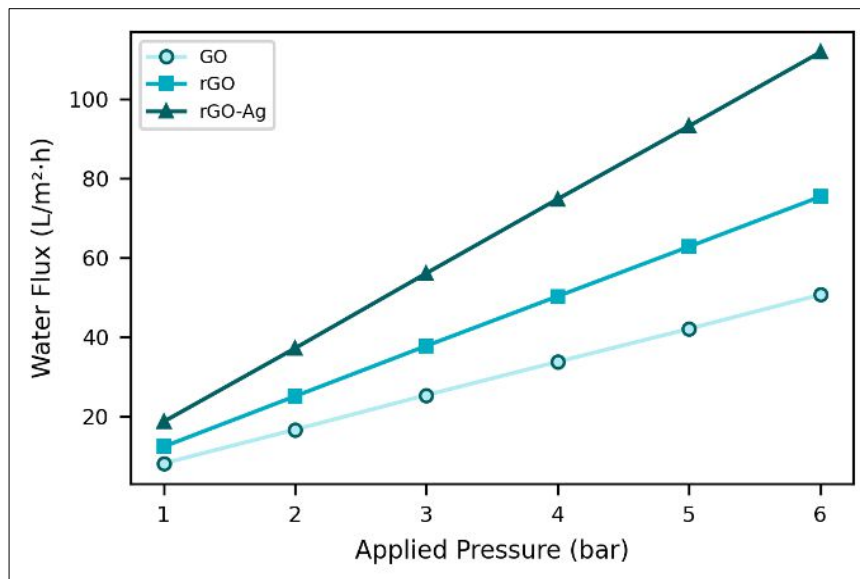


Fig 1: Water flux as a function of applied pressure for GO, rGO, and rGO-Ag membranes tested with 2,000 mg/L NaCl feed solution at 25 °C.

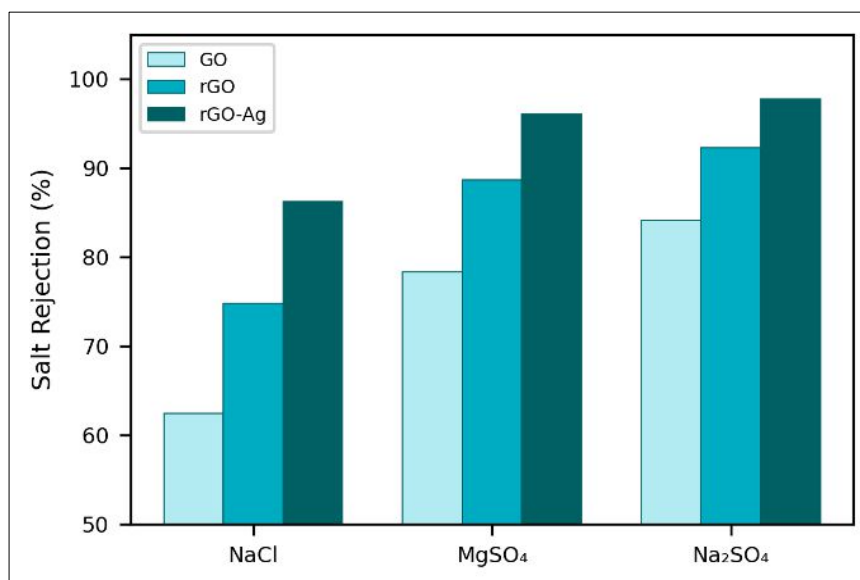


Fig 2: Salt rejection comparison for three membrane types at 4 bar operating pressure with 2,000 mg/L single-salt feed solutions.

Antifouling performance was dramatic. After 72-hour *E. coli* challenge, SEM imaging of the GO membrane surface showed dense bacterial colonization with extracellular polymeric substance matrix covering ~80% of the surface. The rGO membrane showed intermediate colonization (~45% coverage), while rGO-Ag had only scattered individual bacteria with no visible biofilm formation (~5% coverage). Quantitative plate counting confirmed a 94.3% reduction in viable bacteria on the rGO-Ag surface compared to GO. The antibacterial mechanism involves both contact killing (Ag^+ ion release from nanoparticle surfaces disrupts bacterial cell membranes) and prevention of initial bacterial adhesion (the citrate-capped AgNP surface creates a hydration layer unfavorable for protein adsorption) [6].

Long-term stability over 30 days of continuous operation showed clear advantages for rGO-Ag. The GO membrane experienced 34% flux decline, primarily due to biofouling and partial swelling that compressed its already narrow channels. The rGO membrane lost 19% flux, with the reduced swelling from partial π - π restacking offering some

protection. The rGO-Ag membrane declined only 8%, maintaining 103 LMH at day 30 — still above the flux achieved by GO membranes on day 1. Silver ion leaching was monitored in the permeate and remained below 15 $\mu\text{g/L}$ throughout the test, well within the WHO drinking water guideline of 100 $\mu\text{g/L}$ for silver [15].

Discussion

The combination of high flux (111.8 LMH at 6 bar), good monovalent salt rejection (86.2% NaCl), and excellent divalent salt rejection (96-98%) positions the rGO-Ag membrane in an interesting niche between nanofiltration and low-pressure reverse osmosis. For Ghanaian brackish groundwater at 2,000-5,000 mg/L TDS, single-pass treatment would reduce salinity to 280-690 mg/L — within the WHO guideline of 600 mg/L for palatable drinking water. A two-pass configuration could achieve higher rejection if needed for the more saline sources [3].

The 94.3% biofouling reduction is especially significant for tropical deployment where biofilm growth is accelerated by temperatures above 25 °C. Most commercial nanofiltration

membranes require chemical cleaning every 1-4 weeks in warm climates, but the inherent antibacterial properties of the rGO-Ag membrane could extend cleaning intervals substantially, reducing both chemical costs and membrane downtime [7]. The sustained low silver leaching (<15 µg/L over 30 days) suggests that the citrate-capped AgNPs are firmly anchored within the graphene matrix rather than simply sitting on the surface where they would dissolve rapidly.

The energy cost estimate for a small community-scale system (1 m³/h output) treating 3,000 mg/L brackish water at 4 bar would be approximately 0.8 kWh/m³ — less than one-third of typical seawater RO and achievable with a 400 W solar panel array in Ghana's high-insolation climate (5.5 peak sun hours/day). At a membrane fabrication cost estimated at \$15-25/m² using locally available GO synthesis from graphite, the system could provide safe water at less than \$0.02/liter, competitive with sachet water that currently costs \$0.03-0.05/liter [16].

A limitation is that the vacuum filtration fabrication method, while excellent for laboratory-scale flat-sheet membranes, does not directly translate to the spiral-wound module format used in industrial systems. Scale-up would require adaptation to dip-coating or spray-coating methods compatible with continuous membrane production. Additionally, the Ag content should be monitored over extended operation (6-12 months) to establish the long-term silver depletion rate and plan for membrane replacement or AgNP replenishment [17].

Conclusion

Silver nanoparticle-decorated reduced graphene oxide membranes achieved simultaneous improvements in water flux, salt rejection, and antifouling resistance compared to unmodified GO and rGO membranes. The rGO-Ag membrane delivered 111.8 LMH at 6 bar with 86.2% NaCl rejection and 94.3% biofilm inhibition, maintaining stable performance over 30 days of continuous operation with only 8% flux decline. These properties, combined with low-cost fabrication from abundant graphite precursor, make rGO-Ag membranes a promising technology for solar-powered brackish water desalination in sub-Saharan African communities.

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