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Experimental Evaluation of Graphene Oxide-Modified Membranes for Enhanced Water Purification and Heavy Metal Separation

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Abstract--- Contamination of surface and groundwater with heavy metals still stands out as one of the major environmental problems caused by effluent discharge from industries, mining operations and inadequate wastewater treatment. The use of polymeric membranes can help in the purification of water but their efficiency has been hindered by poor hydrophilicity, fouling and poor selectivity towards metal ions dissolved in the water. In this research work, the effect of polyethersulfone (PES) membranes coated with varying amounts of graphene oxide (GO) (0, 0.1, 0.25, 0.5 and 1.0 wt%) on water purification and separation of heavy metals has been experimentally assessed. The graphene oxide has been prepared using a modified Hummers process and then embedded in the polymeric matrix by non-solvent induced phase inversion technique. The most promising results were obtained using the 0.5 wt% GO modified membrane; the water contact angle was decreased from 78° to 58° and the pure water flux was increased from 42 to 95 L m^{-2} h^{-1} bar^{-1}. The removal of Pb^{2+} ions reached 94.5%, whereas the control PES membrane showed only 48.0% rejection of this ion. The membrane exhibited the enhanced fouling resistance and maintained the rejection level of 83.7% after five successive filtration cycles. These results suggest that GO modification could be used for the enhancement of embrane permeability and efficiency at the same time.

Keywords--- Graphene Oxide, Polyethersulfone Membrane, Heavy Metal Removal, Water Purification, Membrane Fouling, Phase Inversion, Metalion Rejection.

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I. Introduction

Access to safe water is constrained by industrial discharge, mining, agriculture and urban runoff, all of which release toxic metal ions into aquatic systems. Lead, cadmium, copper and nickel are of particular concern because they are non-biodegradable, bioaccumulate and cause renal, neurological and carcinogenic effects at low exposure (Vasseghian et al., 2021; Zunita et al., 2020; Ayub & Othman, 2023). Conventional treatments such as chemical precipitation, coagulation and ion exchange are widely used, but they generate sludge, consume reagents and struggle to meet increasingly strict discharge limits (Chai et al., 2021). Membrane processes offer continuous operation and tunable selectivity (Moradi et al., 2020).

Polymeric membranes, and PES in particular, are attractive because of their mechanical strength, chemical stability and ease of fabrication. Their intrinsic hydrophobicity, however, lowers water flux and promotes fouling, and their pore structure gives limited selectivity toward small hydrated metal ions Ahmad et al., (2020). Embedding nanomaterials in the polymer matrix is a well-established way to correct these weaknesses. Graphene oxide is especially suitable because it is a two-dimensional sheet carrying epoxy, hydroxyl and carboxyl groups that raise hydrophilicity, provide binding sites for cations and create narrow transport channels between stacked layers (Ahmad et al., 2020; Liu et al., 2022).

This has been recently supported by scientific findings where modified synthesis procedures under low temperatures enhance the adsorption capabilities of the GO sheets (Goyat et al., 2022). PES-GO blend membranes exhibit superior permeability and antifouling performance than PES only, while the latter decorated by GO can remove more than 90% of iron and manganese ions in one pass of filtration (Giwa & Hasan,

2020; Marjani et al., 2020; Goyat et al., 2024). However, most researches are conducted to address the effect on one response variable at a time.

Consequently, this study assesses GO–PES composite membranes in a systematic loading scheme and attempts to connect structure and function using a theoretical model. The specific goals of the study are (i) to determine the impact of GO loading on hydrophilicity and porosity, (ii) to determine the flux and rejection efficiency of four metal ions, (iii) to determine the effect of feed pH, and (iv) antifouling and reuse potential.

Moreover, the success of membrane technology is also dependent on ensuring that there is a good balance between the permeability and selectivity of the membrane. While increasing the porosity of the membrane can increase the permeation rate of water, this should not affect the ability of the membrane to retain the contaminants in solution. Thus, the modification of nanomaterials should be conducted with care so that improvement in filtration does not affect the membrane itself.

II. Conceptual Framework

The work is built on the conceptual framework in figure 1, based on the flow of materials from their input into the processes of separation. GO loading in combination with the PES matrix results in the construction of membrane architecture, which is determined by hydrophilicity, porosity, interlayer spacing, and surface charge. The structural characteristics define the processes of transport and separation – water flux and separation of metal ions. The separation process occurs because of three coupled effects: size exclusion in nanochannels, electrostatic interactions between charged surface groups, and cation coordination with carboxyl and hydroxyl sites (Ahmad et al., 2020; Khan et al., 2021). The operation parameters, including feed pH, pressure, concentration and number of filtration cycles, influence the mechanisms and result in the desired performance.

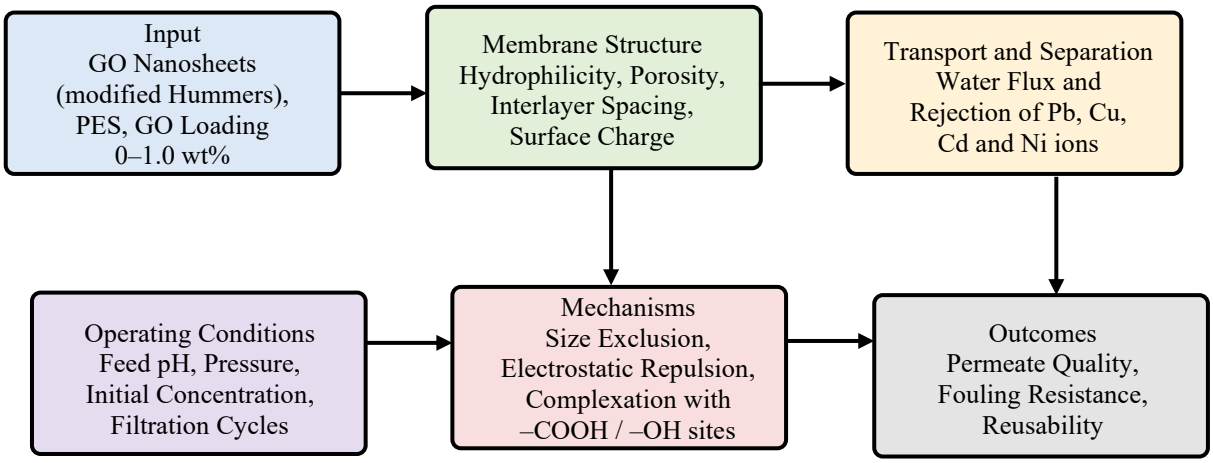


Figure 1: Conceptual Framework Linking GO Loading, Membrane Structure, Separation Mechanisms and Performance Outcomes

Hypothesis one is that moderate loading will increase both flux and metal rejections. Hypothesis two states that heavy loading results in agglomeration, which lowers both parameters.

Relationship between Membrane Properties and Performance

Structural properties of the membrane affect filtration behavior. Water flow through the membrane is achieved by hydrophilicity while porosity affects the pathways for the process of permeation. Functional groups on the surface affect the interaction with metal ions that are dissolved. In general, all of these structural properties affect the filtration properties and thus help determine the effect of GO concentration.

III. Materials and Methods

Materials

The analytical grade graphite powder, H2SO4, H3PO4, KMnO4, H2O2, DMF and nitrates of Pb, Cu, Cd, and Ni were used as purchased. Pellets of polyethersulfone (PES) were the polymer matrix, while bovine serum albumin (BSA) was used as the model foulant.

Synthesis of GO and Membrane Fabrication

GO was synthesized from graphite using an improved Hummers method where graphite was oxidized in a H2SO4/H3PO4 mixture containing KMnO4 followed by quenching in an ice-cold water/H2O2 mixture and washing and drying (Goyat et al., 2022; Goyat et al., 2024). The GO was dispersed in DMF through probe sonication for 30 min and mixed with the PES solution giving the GO content at 0%, 0.1%, 0.25%, 0.5% and 1.0% (wt%) with respect to the polymer weight. The membranes were cast on glass plates using a doctor blade technique and submerged in a deionized water coagulation bath resulting in the formation of the porous structure.

Characterization

The surface hydrophilicity was evaluated using contact angle measurement of sessile drop for five points on each sample. The porosity was determined gravimetrically based on wet and dry weights of the membrane. All experiments were done in triplicate, and data is presented as means with SD less than 4%.

Filtration and Separation Experiments

The experiments were carried out using a dead-end stirred cell under a pressure of 2 bar. Pure water flux was measured after 30 minutes of compaction. Metal removal efficiency was evaluated with 50 mg L-1 solutions of ions, and the concentration of permeates was estimated by atomic absorption spectroscopy. Removal efficiency was calculated by the formula: R (%) = (1-Cp/Cf) ×100, where Cp is the permeate concentration and Cf is the feed concentration (Goyat et al., 2024). The pH was varied in the range of 2-7 by dilution of HNO3 and NaOH solutions. The anti-fouling properties were evaluated by the flux recovery ratio (FRR) during filtration of 1 g L-1 BSA solution and rinsing with deionized water.

Experimental Consistency

For consistency during the evaluation of membranes, the same operating parameters were applied for each of the different membrane types. The measurement of contact angles was made from several different locations on the surface, while the filtration tests were made at constant pressures. This method is used to reduce variability and increase accuracy of the results obtained. This enables comparison of the performance of modified membranes with the unmodified PES membrane.

IV. Results and Discussion

Experiments on GO modified PES membranes have been conducted to understand the effect of graphene oxide concentration on various membrane characteristics such as water flux, heavy metals rejection, pH dependence, anti-fouling property, and reusability. The findings show that adding GO enhances the membrane properties until an optimum level of 0.5 wt% is reached.

Effect of GO Loading on Structure and Permeability

Properties of the membranes were compiled in table 1. The contact angle was reduced from 78° for the pristine PES membrane to 58° for 0.5 wt% loading of GO. Likewise, the porosity of the membranes was increased from 62.0% to 75.1%. This shows that the surfaces became more hydrophilic and the membrane structures were modified due to incorporation of GO. Pure water permeate flux was raised from 42 to 95 L m^{-2} h^{-1} bar^{-1} at 0.5 wt% loading of GO. This increase can be attributed to the presence of hydrophilic functional groups of GO and enhanced transport channels for water molecules. However, for 1.0 wt% loading of GO, the flux dropped to 81 L m^{-2} h^{-1} bar^{-1}. The flux and contact-angle trends are presented in figure 2.

Table 1: Structural and Transport Properties of the PES and GO–PES Membranes

GO loading (wt%)	Contact angle (°)	Porosity (%)	Flux (L m^{-2} h^{-1} bar^{-1})	BSA flux recovery (%)
0 (neat PES)	78	62.0	42	71.2
0.1	71	66.4	58	79.8
0.25	64	71.3	76	86.5
0.5	58	75.1	95	92.4
1.0	61	73.2	81	88.1

At 1.0 wt%, both performance indices became poor, as evidenced by an increase in the contact angle to 61° and a decrease in flux to 81 L m-2h-1bar-1. Such behavior can be explained by the stacking and agglomeration of GO sheets, leading to the hiding of hydrophilic groups and some blocking of pores, and this is consistent with optimal loading behavior in GO mixed matrix membranes (Giwa & Hasan, 2020; Marjani et al., 2020).

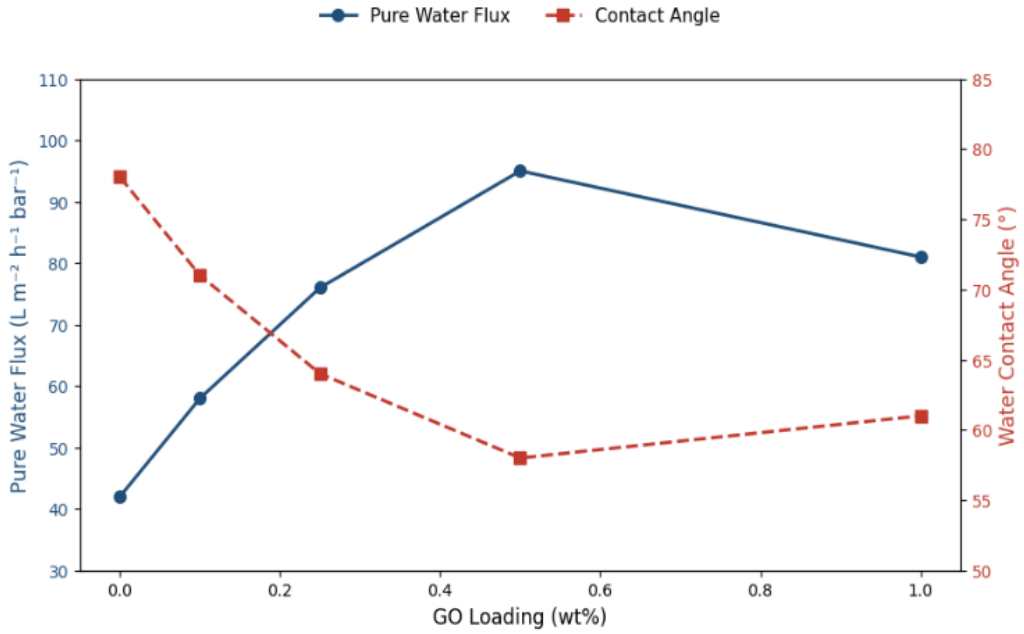


Figure 2: Pure Water Flux and Water Contact Angle of the Membranes as a Function of GO Loading

Heavy Metal Rejection Performance

Table 2 describes the rejection properties of the Pb^{2+}, Cu^{2+}, Cd^{2+}, and Ni^{2+} by the GO-modified membranes were found to be higher than those of the PES membrane. For 0.5 wt% of GO content in the membranes, the rejections were 94.5%, 91.2%, 88.6%, and 84.3%, respectively. This is because the presence of oxygen-containing functional groups in the GO allows the interactions between GO and the metal ions. Size exclusion and interaction effects were the factors responsible for the rejection. The rejection sequence was Pb^{2+} > Cu^{2+} > Cd^{2+} > Ni^{2+}. At 1.0 wt% GO concentration, the rejections were lower. The comparative rejection trends are shown in figure 3.

Table 2: Rejection (%) of Metal ions at pH 6 and 50 mg L-1 Feed Concentration

GO loading (wt%)	Pb^{2+}	Cu^{2+}	Cd^{2+}	Ni^{2+}
0 (neat PES)	48.0	44.5	39.8	35.2
0.25	86.1	81.7	77.4	72.9
0.5	94.5	91.2	88.6	84.3
1.0	90.2	87.4	83.5	79.1

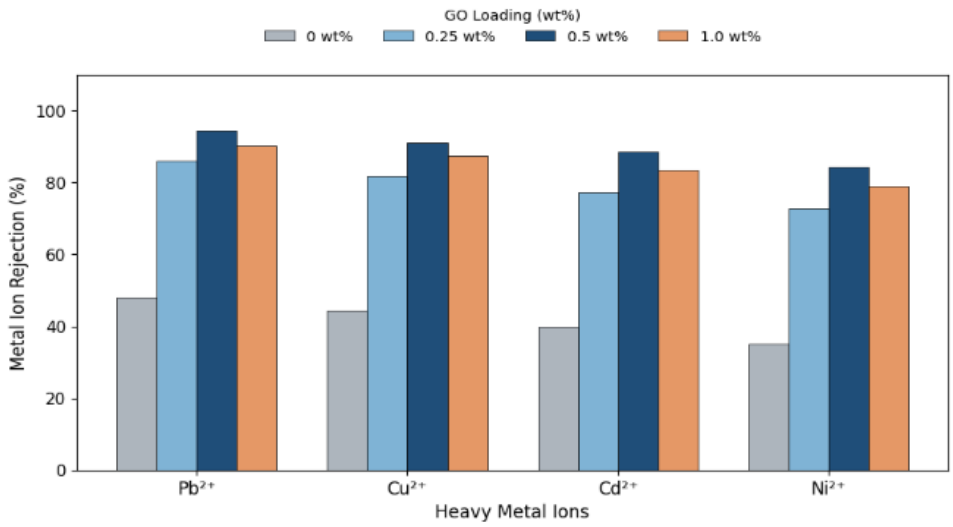


Figure 3: Rejection of Pb, Cu, Cd and Ni ions by Membranes with Different GO Loadings

Influence of Feed pH

In figure 4(a), the rejection of Pb2+ with 0.5 wt% membrane was found to increase from 31.4% at pH 2 to 94.5% at pH 6 and remain almost constant at pH 7. The effect of feed pH was studied using the membrane with 0.5 wt% GO. The rejection of Pb^{2+} was observed to increase from 31.4% at pH 2 to 94.5% at pH 6 and remain almost constant at pH 7. With low pH, there is protonation of carboxyl groups, which results in competition with the hydrogen ions, thus leading to the unavailability of the binding site for metals. An increase in pH leads to the deprotonation, thereby facilitating the strong electrostatic interaction and complexation between metal ions and the membrane surface.

Antifouling Behaviour and Reusability

The antifouling properties were tested by using BSA as a model foulant. The non-functionalized PES membrane had a flux recovery ratio of 71.2%, while the modified one with 0.5 wt% GO showed a flux recovery ratio of 92.4%. It can be stated that higher surface hydrophilicity provides enhanced water permeation recovery after membrane cleaning. In the course of five successive filtration cycles, the Pb^{2+} rejection rate decreased from 94.5% to 83.7%, and the flux recovery rate dropped from 97.0% to 88.6%. It might have been caused by slow filling of the active sites on the surface and partial pore clogging. The filtration-cycle performance is shown in figure 4(b).

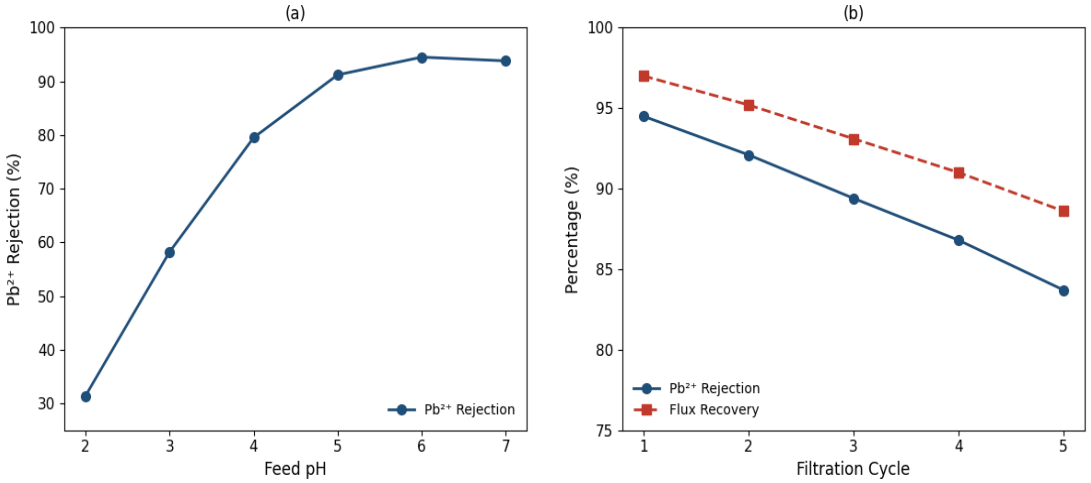


Figure 4: (a) Effect of feed pH on Pb2+ Rejection and (b) Pb2+ Rejection and Flux Recovery Over Five Filtration Cycles for the 0.5 wt% GO–PES Membrane

Comparison with Previously Reported Membranes

Comparison between the optimum membrane and the latest heavy metal membranes is provided in table 3. The optimum membrane attained heavy metals rejection ranging between 84.3% and 94.5% at 0.5 wt% GO-PES membrane composition. These results are equivalent to those obtained by other membrane systems shown in table 3, including GO-PES, GO-MnO_2 modified PES and polymer-based nanocomposites membranes. The above comparison reveals the possibility of using GO to enhance the separation capacity of polymeric membranes. However, factors such as feed concentration, pressure and configuration of experiment need consideration.

Table 3: Comparison of Metal Rejection with Recent Membranes

Membrane	Ions	Rejection (%)	Ref.
GO–PES	Cu, Zn, Cd	65–80	Marjani et al., (2020)
GO–MnO_2 on sulfonated PES	Cu, Zn, Ni	64.0–81.1	Ibrahim et al., (2022)
Polyamide–TiO_2 thin-film	Cu, Hg, Pb	77.0–86.9	Karki & Ingole, (2022)
Polysulfone–mesoporous silica	Cd, Zn	91–94	Alotaibi et al., (2021)
GO–PES, 0.5 wt% (this work)	Pb, Cu, Cd, Ni	84.3–94.5	This work

Practical Significance of the Proposed Membrane

Out of all five membrane compositions that were studied, 0.5 wt% GO gave the best results overall. This composition had the smallest contact angle, the highest porosity, maximum water flux, and the highest metal ion rejection. Lowered efficiency at 1.0 wt% illustrates the significance of the right concentration control of the nanomaterial when producing membranes.

All results prove that the efficiency of the membrane depends on the connection between its hydrophilic properties, porosity, transport paths, and metal ions interaction. The antifouling properties and cyclic stability confirm the prospective usage of GO modified PES membranes for wastewater treatment.

V. Conclusion

Five loadings of GO–PES membranes were fabricated and their performance for purification of water and metals separation was analyzed. At 0.5 wt% loading, the optimal parameters of membranes were obtained; the contact angle decreased to 58°, porosity was increased up to 75.1%, and pure water permeation was increased to 95 L m-2 h-1 bar-1 and rejection reached 94.5% of Pb2+. The sequence of metals rejection by the membranes was Pb2+ > Cu2+ > Cd2+ > Ni2+ and was the highest for pH 5–7 according to the conceptual framework due to the influence of size exclusion, electrostatic interactions, and complexation. At 0.5 wt% loading, the flux recovery from BSA fouling reached 92.4%, while rejection from five cycles was 83.7%.

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