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FABRICATION OF PES@TiO_2-GO MEMBRANE FOR DESALINATION PROCESS

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ABSTRACT

The membrane filtration process has been a rapidly emerging technology for removing different sizes of pollutants from untreated industrial wastewater owing to their desired merits relative to conventional treatment processes such as lower operating expenses, higher separation efficiency, smaller carbon footprint, and stable product quality. Presently, thin-film composite, blended ultrafiltration, and nanocomposite membranes provide excellent separation performance for pressure-driven water treatment techniques. In this perception, polyethersulfone (PES) based blended polymer nanocomposite (BPN) ultrafiltration membranes were fabricated for desalination applications. PES layers as support were fabricated with a blended mass of titanium dioxide (TiO_2) particles and then graphene oxide (GO) particles in the nano range were combined to enhance the rejection rate and water flux of the membrane. Phase inversion and Modified Hummer's methods were adapted to prepare the layers of PES as support and graphene oxide. Blended polymer nanoparticle membrane-PES@TiO_2(2%)-GO (0.5%) exhibited enhanced water flux (86.5 l m^{-2} h^{-1} bar^{-1}), mean radius of the pore (15.9 nm), porosity (90.625%) and 86% salt rejection which proves that the membrane stands good for desalination.

Keywords: Membrane, Desalination, Blended Polymer Nanocomposite, Water Flux, Rejection Rate.

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INTRODUCTION

The exigency for pure water has highly escalated with rapid development in industrialization, global climate change, an increase in the world's population, and increasing scarcity of ground and surface water resources.[1] The shortage of pure water has thus raised efforts towards enhancing modern treatment methods and advancement of novel practices to sustainably make the water potable, which we obtain from industrial, municipal wastewater, and seawater desalination.[2,3,4] Among various Earth's water resources only 2.5% pure water can be seen. On the other hand, 0.003% of pure water sources are only available as a source of utilization. Installation of desalination plans will reduce shortages of clean water in dry areas. Sea water also contains various rich sources like Magnesium, Bromine, Sodium Chloride, and other minerals.[5,6] Amidst 70% oceans and seas, 96.5% water is a complex mixture of sea water which contains 2.5% salts, with organic and inorganic materials and a lesser number of other substances and atmospheric gases. A rich source of profitable significant chemicals was found in seawater. 99% of sea salts comprise of Sodium, Sulphate, Magnesium, Chloride, Potassium, and Calcium. Other major substances are Bromide, inorganic Carbon, Fluoride, Strontium, and Boron.[7,8,17] In general, the salinity range is between 33 to 37 gm/kg of seawater.[11] This can be lowered by the nearby freshwater resources by dilution. In addition, the removal of water by evaporation has no change in the composition. However, in the regions with a high rate of evaporation mild alteration in composition was observed.[18,21] The use of sea or ocean water by proper treatment will definitely enhance the source of fresh water.[30] In the present study, a blended polymer nanocomposite (BPN) membrane was fabricated using TiO_2 and graphene oxide nanoparticles for desalination to enhance freshwater sources.

Membrane-based Desalination

The capacity to distinguish particulates in gas and liquid streams of feed describes the efficiency of the membrane. Membranes are widely used in various processes. The pressure-driven methods of membrane separation are simple. The basic membrane filtration process is given in Fig.-1. Here, the membrane acts

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as a semipermeable impediment and these processes are related with greater flux when compared with concentration-based and thermal separation processes.[9,30] Fouling of membrane can be divided into four types: They are (a) microbial fouling, (b) scaling fouling, (c) colloidal fouling or particulate, and (d) organic fouling. Microbial or biofouling is a serious problem in the membrane water and wastewater separation process as it highly compromises the filtration efficiency of treatment processes.[6,7] Over the membrane surface, microbes are attached and it produces extracellular polymers (biofilm), which eventually declines the filtration performance of the membrane. Reverse osmosis and nanofiltration membranes are exposed for biofouling.[8,9,10] Colloidal or particulate fouling consists of bacteria, algae, and natural organic matter (NOM) that fall into the size of colloids and particles. Colloidal fouling is the most important in micro and ultrafiltration.[11,12] This section explains various membranes used for desalination, their fabrication, and their characteristics.

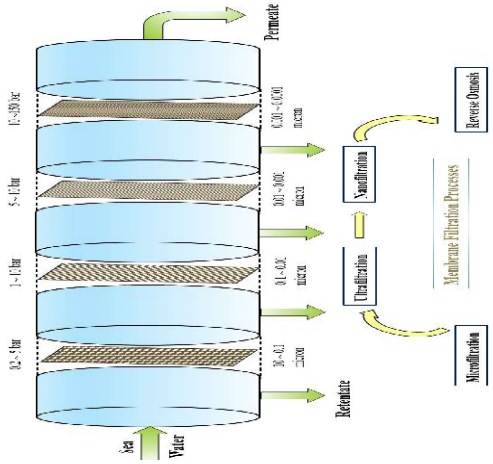


Fig.-1: Classification of Membrane Filtration Processes

Polymeric Membranes

Flexibility, chemical stability, selectivity, mechanical strength, and good film-forming property were observed in polymeric membranes. The polymeric membranes have enormous functions in the areas of water treatment, gas separation, drug delivery, and other commonly seen filtration process.[13,14] Polymers like polyether sulfone, polyethylene, polyimide, polypropylene, polyvinyl alcohol, polyamide, polyvinylidene fluoride, and polyvinyl chloride were commonly used in the fabrication of membranes. Polyether sulfone has been used for the preparation of membranes due to its hydrolytic, thermal, and mechanical stability.[15,16,19,20]

Graphene Oxide Based Polymeric Nano-Composite Membranes

The derivatives of graphene are graphene oxide and its outstanding characteristics are the reason for its application in the polymeric matrices. Blended membranes of graphene oxides of various compositions were made using methyl-2-pyrrolidone with water and ethanol. Characteristic studies of prepared membranes were made. The result infers that graphene oxide with 1% weight showed 82.2% porosity, permeability of 52 L/m^2 h bar, 163.71 L/m^2 h water flux, and enhanced hydrophilicity.[26] The mechanical possessions of the membrane were enhanced when graphene oxide was added and this polyether sulphone mixed matrix membrane displayed extraordinary anti-bacterial and anti-fouling performance. It exhibited 90% removal of *Escherichia coli* and a fouling recovery of 94.2%.[27] Graphene oxide nanoplates and glycidyl polyhedral oligo silsesquioxane were used to prepare polyetherimide nano-membrane. The membrane exhibited substantial separation performance and anti-fouling properties.[28]

EXPERIMENTAL

Material and Methods

Graphite powder (extra pure 98%, Otto), 98% Sodium nitrate (Spectrum chemicals), 99% Potassium permanganate (Spectrum chemicals), 30% Hydrogen peroxide (Spectrum chemicals), 98% Sulphuric acid and 35% Hydrochloric acid. PES (Polyether sulfone) as the polymeric substrate was purchased from (BASF Co, Ultra SonE). N,N-Dimethylformamide (Fisher Scientific) and (PVP K30) Polyvinyl pyrrolidone

(LOBA Chemie) exercised as a solvent and agent for the formation of pores. Entire chemicals were purchased with analytical grade and were utilized devoid of purification. Titanium dioxide (99%, Otto) is used as a modifier. The nanocomposite blended membranes were prepared using distilled water in our laboratory.

Method of Synthesis

Expansion and delamination of Graphene Oxide will result in case of rapid heating. The properties and structure of GO are based on the synthesis approach. Modified Hummer's method is utilized for the GO synthesis.

Modified Hummer's Approach

Modified Hummer's approach includes exfoliation and oxidation because of the suspension's thermal treatment.[29] The preparation method is mentioned in a stepwise manner as follows (Fig.-2):

1. Graphite flakes and Sodium Nitrate each 2 g were dispersed in 98% Sulphuric acid of 90 ml in a 1000 ml flask, maintained in an ice bath with constant stirring.
2. The temperature was maintained and stirring was given for 4 hrs. Potassium Permanganate of 12 g was supplemented and the reaction was controlled by maintaining the temperature at 15^0C.
3. 184 ml of distilled water was added to dilute the suspension and continuous stirring was given for 2 hrs. And the suspension was stirred for another 2 hrs at 35^0C (a).
4. This suspension is reflexed for 10 to 15 min at 98^0C and cooled to 30^0C to obtain a brown-colored suspension.
5. This suspension was again reduced to 25^0C and this temperature was maintained for 2 hrs.
6. The solution color got changed to yellow after treatment with Hydrogen Peroxide of 40 ml.
7. Two individual beakers were taken with 200 ml of water and the prepared solution was added equally with continuous stirring for 1 hr.
8. The obtained mixture was kept idle for 3 to 4 hrs until the particle settles down and filtered (b).
9. The filtered mixture was washed and centrifuged with DI water and HCl (10 %) until the suspension formed gel-like consistency (c).
10. After centrifugation the obtained substance was dried in a vacuum at 60^0C for 6 hrs to get Graphene Oxide powder (d).

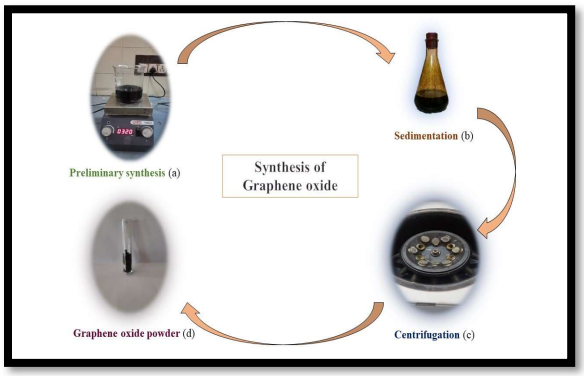


Fig.-2: Preparation Process of Graphene Oxide Powder

General Procedure

Pure Polyether Sulfonate (PES) and blended Polyether Sulfonate support membranes (M1 to M5) were prepared using the immersion precipitation technique by the method of phase inversion.[23,24,25] The solution compositions (Homogenous) for the fabrication of blended membranes were tabulated in Table-1. Along with PES, Polyvinyl Pyrrolidone (PVP K30) was added. Graphene Oxide and Titanium Oxide were sonicated (e) and dispersed in N,N-Dimethylformamide. The prepared suspension was stirred for 8 hrs at 40^0C at 750 rpm to get a polymer solution (f). This polymer suspension was kept idle for 4 hrs to eliminate air bubbles at room temperature. The blended suspension was poured on a flat plate (glass). The thickness was adjusted (100 µm) using a thin film applicator (g) and steel knife. After the development of slender film (40 s), the film was engrossed in water at 20^0C immediately. This initiates the phase separation process.

The prepared pure PES, PES@TiO_2, and PES@GO membranes were sustained in DI water prior to usage (g). The prepared membrane (h) can be seen in Fig.-3.

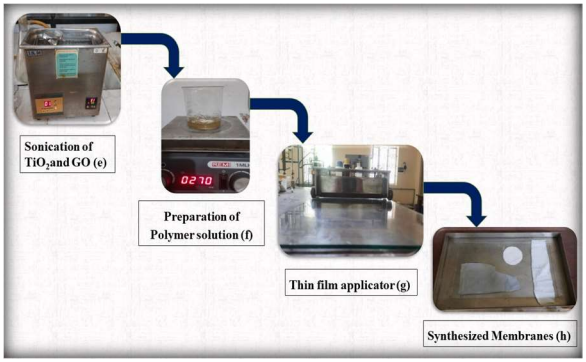


Fig.-3: Process of Membrane Synthesis

Table-1: Concentration of the Dope solution

Sample Code	Membrane code	PES support layer (wt %)				GO loading (wt%)
		PES	DMF	PVP K30	TiO_2	
M1	Pure Polyether Sulfonate	18	82	0	0	-
M2	PES/PVP-3%	17	80	3	0	-
M3	PES/PVP@TiO_2(2%)	17	78	3	2	-
M4	PES/PVP-GO (0.5%)	17	80	2.5	0	0.5
M5	PES@TiO_2(2%)-GO (0.5%)	17	78	2.5	2	0.5

Properties of Membrane

In this section detection of membrane properties like water permeation rate, porosity, salt rejection, and mean pore radius were discussed.

Water Permeation Rate

The rejection performance and water permeation rate were measured through the apparatus as shown in Fig.-4. The complete set-up is constructed of stainless steel. It consists of a collection tank, a feed solution holding setup, a high-pressure compressor, and a membrane housing unit. Tap water was utilized as feed and was driven through the unit of membrane by using a compressor. Retentate and permeate solutions were drawn in individual stainless-steel vessels. The pressure was adjusted using a needle valve which is placed at the inlet of the housing.13000 ppm of NaCl was taken as a feed sample. Throughout the experiment, a pressure of 4 bar was maintained constantly throughout the experiment and 6.4 x 10^{-3} m^2 of the cross-sectional area of the membrane is effectively used. The permeation flux can be estimated using,

$$J_w = QA\Delta t \quad (1)$$

Where J_w is the permeation flux of water in L/m^2hr, permeate is 'Q' which is measured in liters, the membrane cross-sectional area is depicted as 'A' which is determined in m^2, and permeate is collected at time 't' in hour.

Determination of Porosity

ε- porosity was estimated by obtaining the capacity of water-absorbance of the test membrane (sample of membrane) by immersing test (24 hours in water) and then oven drying at 65°C. The mass of the membrane at wet condition (M1) and dried (m2) was utilized for the P- porosity calculations using the below equation (2).

$$\text{Porosity } (\epsilon) = \frac{w_w - w_d}{\rho \text{Al}} \times 100\% \quad (2)$$

Fig.-4: Dead-End Filtration Setup

Mean Pore Radius

The Guerout- Elford- Ferry expression was sourced in calculating the mean radius of the pore (R_P) on the basis of porosity and flux data of pure water of membranes.

$$R_P = \sqrt{\frac{(2.9 - 1.75\varepsilon)8Ql\eta}{\varepsilon\, A\, \Delta P}} \quad (3)$$

Where Q is the permeate flow rate (m^3/s), the pressure drops ΔP and the water viscosity η in Pascal and l is the support layer thickness of PES (m).

Salt Rejection

Dead-end filtration apparatus was utilized to study the salt rejection performance. The apparatus is basically a batch cell with a sample holding capacity of 1 lit. The synthesized membranes (M1 to M5) were taken one by one and tested for their rejection capability using water containing 13000 ppm of NaCl as a feed sample. The pressure was maintained constant at 4 bar. The permeation flux and the conductivities of feed, permeate and reject were measured for each experimental run, and the outcomes are illustrated in Fig.-7. Salt rejection and rejection % of sample feed were assessed by,

$$\%\ \text{Rejection} = 1 - \frac{C_p}{C_f} \times 100\% \quad (4)$$

Where the concentration of permeate is C_p and feed samples is C_f respectively.

RESULTS AND DISCUSSION

Pure Water Flux

Experiments were performed for the synthesized membranes using tap water as a feed sample. The permeation flux of these synthesized membranes (M1 to M5) was presented below in Fig.-5. It is evident that GO-based blended UF membrane (M4) showed a much higher flux (an increase of 35%) when compared with TiO2-based blended UF membrane. Also, the membrane made with a combination of PES/PVP/ TiO_2/GO (M5) showed a much higher permeation flux which was 2.5 times higher than the permeation flux recorded for pure PES membranes.

Effect of TiO2 and GO on Pure Water Flux as Modifiers

The enhancement of membrane performance is distinct when a pure PES sample is compared with the other fabricated membranes. The supplement of inorganic substances like TiO_2 and GO nanoparticles in the PES membranes exhibited great water flux as an impression of a synergic effect. The maximum water permeation of 86.5 ($lm^{-2}h^{-1}bar^{-1}$) and pressure of transmembrane as 4.5 bar, was observed in the M5 BPN membrane (0.5wt% GO and 2 wt% of TiO_2) which was raised thrice higher than M1 pure PES membrane.

Porosity and Mean Pore Radius

Figure-6 represents the mean radius of pore and porosity of the prepared pure and BPN membrane samples. They were calculated by the previous equations (2) and (3). It is acknowledged that the rate of water permeation will be greater for membranes that have substantial pores.[81] From Fig.-6, the membrane-M1(pure PES) possess the least porosity since, polymer concentration is high and M5 BPN membrane has the highest porosity of 90.625%.

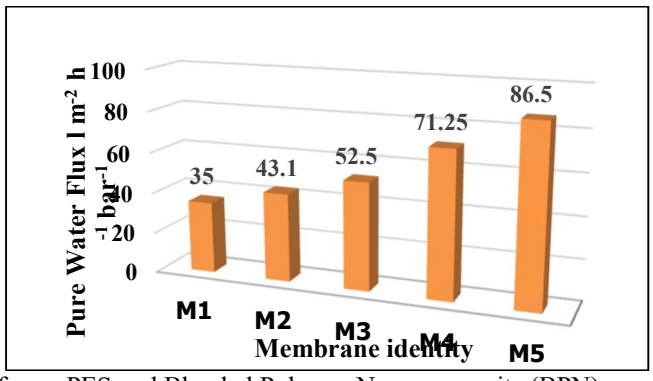
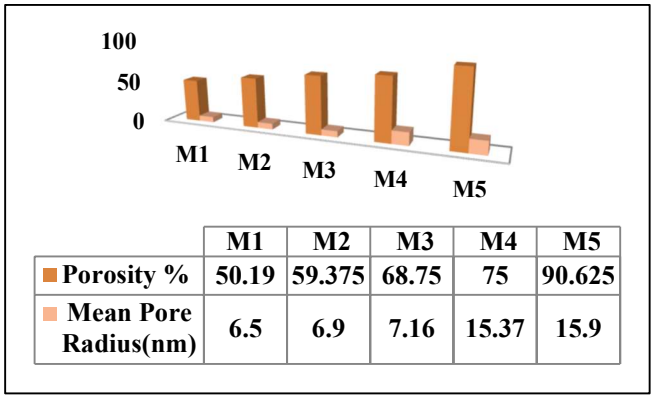


Fig.-5: Discrepancy of pure PES and Blended Polymer Nanocomposite (BPN) membranes by Water Flux



	M1	M2	M3	M4	M5
Porosity %	50.19	59.375	68.75	75	90.625
Mean Pore Radius(nm)	6.5	6.9	7.16	15.37	15.9

Fig.-6: Mean Radius of Pore and Porosity of Pure and BPN Membranes

Effects of TiO_2 and GO

These two significant parameters mean radius of pore and porosity are not directly postulating the hydrophilicity of the membrane. The addition of TiO_2 and GO in the M5 membrane displays high surface porosity of 90.625% and a comparatively large average pore size of 15.9 nm. Further, the M5 BPN membrane porosity and mean pore size were increased by two times respectively, compared to that of the M1 pure PES membrane, due to the increase in the concentration of both TiO_2 (2wt %) and GO (0.5wt %).

Salt Rejection

A feed sample containing NaCl of 130000 ppm was used for the assessment of salt rejection using dead-end filtration apparatus. Salt rejection efficacy of pure Polyether Sulfonate and BPN membranes are calculated using equation (4.4). The rejection functioning of BPN membranes was enhanced by TiO_2 addition and GO nanocomposites related with pure PES membrane as shown in Fig.-7.

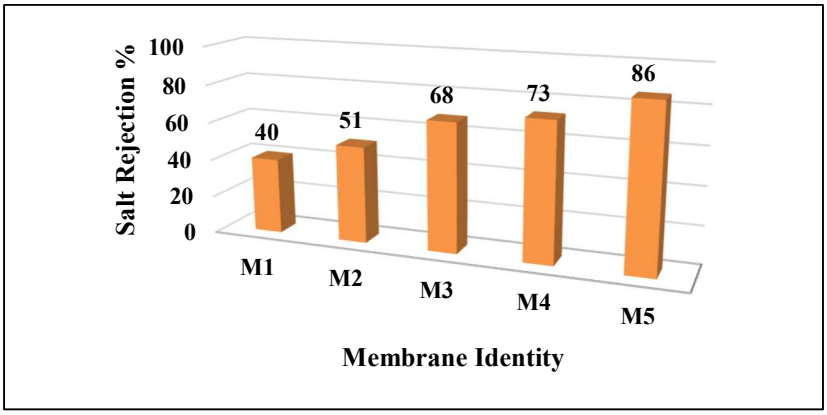


Fig.-7: Salt Rejection of Pure and BPN Membranes

Effect of TiO_2 and GO on Salt Rejection

The rejection efficacy of feed samples by the fabricated membranes is examined to vary between 40% to 86%. Salt rejection efficiency of the M5 BPN membrane (86%) is increased by 2 times assessed with M1 pure Polyether Sulfonate membrane (40 %) and it is observed because of the increase in TiO_2 and GO concentration (2 and 0.5 wt. %). The results evidently depict those membranes (M2 -M5) have greater salt rejection when assessed with pure polyether sulfonate (M1) membrane. The resulting increase of salt rejection is because of the porosity and hydrophilicity of the membranes by the addition of 2wt% (TiO_2) and 0.5 wt. % (GO).

CONCLUSION

Graphene oxide from graphite was synthesized using a modified Hummer's method. Pure Polyethersulfone (PES) membrane (M1), PES/PVP blended membrane (M2), Modified PES/PVP blended Ultrafiltration membranes using TiO_2 as modifiers (M3), Modified PES/PVP blended Ultrafiltration membranes using GO as modifiers (M4) and Novel UF membranes with PES/PVP as support layers with TiO_2 and GO as modifiers (M5) were produced by the method of phase inversion. Salt rejection rate and water flux of pure Polyethersulfone (M1) and blended nanocomposite (M2-M5) membranes were examined. The synthesized M5- PES@TiO_2(2%)- GO (0.5%) membrane produced satisfactory results for the treated feed sample. The TiO_2 and GO nanoparticles (2% and 0.5%) exhibited a synergistic effect which enhanced the water flux (86.5 l $m^{-2}h^{-1}bar^{-1}$), mean radius of the pore (15.9 nm), porosity (90.625%), and 86% salt rejection M5-PES@TiO_2(2%)- GO (0.5%) membrane. The results of the experiment indicated that the blended polymer nanocomposite membrane separation performances increased due to the synergic effect of 2% TiO_2 and 0.5% GO nanoparticles. Hence, the M5 BPN membrane is a novel type of blended polymer nanoparticle membrane, and because the inorganic materials blended in the membrane [PES@TiO_2(2%)-GO(0.5%)] which exhibits a synergistic effect proves that this membrane stands good for desalination.

Future Perspectives

The nanocomposite membranes synthesized using the novel composite polymeric material have been proven to have excellent hydrophilicity and solute rejection. However, all these experimental studies using the synthesized membranes were carried out in batch filtration mode. The laboratory-scale experimental studies prove that the nanocomposite membranes synthesized in our laboratory are technically feasible for the treatment of seawater. However, the data obtained from the laboratory batch studies only proves the technical feasibility and does not provide enough interpretable data for scaling up the process, for continuous treatment of seawater. Therefore, it is necessary to develop a continuous pilot-scale flow membrane module that can handle large volumes of effluent on a day-to-day basis. Hence, this can be taken as a future scope of work that will lead to new process development. It is proposed to carry out RSM and ANOVA analysis for optimum pH, pressure, and feed concentration for maximum removal. This can be used in the future to plan pilot-scale studies. The membranes developed from this novel nanocomposite polymer will have immeasurable potential in both fundamental research and practical use. Water treatment systems with these new and improved membranes using novel nanomaterials like reduced graphene oxide will have an increased scope of environment-related applications in the future.

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CONFLICT OF INTERESTS

The authors declare that there is no conflict of interest regarding the publication of this article.

AUTHOR CONTRIBUTIONS

All the authors contributed significantly to this manuscript, participated in reviewing/editing, and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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