

Improving the Performance of Polyvinyl Alcohol Membrane by Deposition of Graphene Oxide Nanostructures Using Non-Thermal Plasma

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Article Info

Article type:
Research Article

Keywords:

Membrane nanofiltration,
plasma modification,
graphene oxide nanostructures,
non-thermal plasma jet,
desalination performance.

ABSTRACT

In this research, membranes based on polyvinyl alcohol polymer were surface modified using graphene oxide nanostructures, and the effect of this layering on the performance and properties of the membrane was investigated and evaluated. Graphene oxide nanostructures were produced using a non-thermal plasma jet. The surface morphology and hydrophilicity of prepared membranes were studied using the AFM, SEM, FTIR, and CA analyses. The analyses showed a rougher surface with more hydrophilicity for the modified membranes than the pure PVA membrane. Also, the separation performance of membranes using a cross-flow filtration system was investigated for different salt solutions. The modified nanofiltration membrane with graphene oxide, showed good separation performance for Na₂SO₄ and NaCl salts, 94% and 38%, respectively. On the other hand, the high-water flux for the two salts mentioned above, 60 L/m².h and 73 L/m².h respectively, was obtained for the modified nanofiltration membrane with graphene oxide. In sum, this study shows that the separation performance of nanofiltration membranes can be effectively improved by adjusting the different factors in the groups that changed the graphene oxide surface. Also, membranes modified using graphene oxide nanostructures have more suitable antibacterial properties compared to other unmodified membranes.

INTRODUCTION

Desalination is when salts and minerals are separated from salty sources such as seawater, sewage, and industrial effluents. Two major technologies are used for water desalination: thermal desalination (distillation) and membrane desalination. Saltwater is heated to produce pure water vapor in the thermal desalination process. However, since a lot of energy is needed to heat water, in recent decades, the use of membrane desalination technology has been widely considered due to its high efficiency and lower energy consumption. In membrane desalination, ions dissolved in water can be easily separated from water by a membrane with tiny openings (about nanometers) [1].

Nevertheless, choosing a membrane that can separate a high percentage of salts in water and at the same time has a high permeability for the passage of water molecules has always been a challenge for researchers in this field [2]. To solve this problem, regulating the physicochemical properties by introducing porous materials as fillers and searching for new membranes with advanced properties are the main priorities of research in this field. Until now,

different types of nanomaterials such as zeolites, metal oxides, materials based on carbon compounds, graphene, aquaporin, clay, and polyelectrolyte nanoparticles have been studied and investigated in the water desalination process [3]. Among the filler nanomaterials, graphene oxide (GO) has been used as a filler in the construction of nanofiltration membranes due to its special properties such as having a thickness of one atom and the presence of hydrogen functional groups [4].

Graphene oxide is an atomically thin layer of carbon plates with a sp² bond with oxygenated functional groups. These functional groups include carboxylic and carbonyl at the edge of the plates and epoxy and hydroxyl at the surface of the carbon plates. Despite these factors, GO layers are well dispersed in polar solvents such as water. Many articles have reported that the percentage of water permeability into the membrane and the separation efficiency of solutes and salts in water increases by adding graphene oxide to the selectable layer of polyamide (PA) [5, 13]. But the graphene oxide nanosheets cannot spread uniformly inside the membrane and this factor limits the performance of the membrane. For the uniform dispersion of nanosheets GO, Kang et. al synthesized sulfated

How to Cite this paper: Kiamehr Z. Improving the Performance of Polyvinyl Alcohol Membrane by Deposition of Graphene Oxide Nanostructures Using Non-Thermal Plasma. *Challenges in Nano and Micro Scale Science and Technology*. 2023; 11(2): 1-6. DOI: 10.22111/cnmst.2025.49565.1253



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DOI: 10.22111/cnmst.2025.49565.1253

graphene oxide (SGO) nanosheets and used them as fillers in the construction of nanofiltration membranes. They observed that the membrane properties of thin-layer nanofiltration such as hydrophilicity, surface roughness, charge density, and water permeability, and the anti-fouling ability of the membrane depends on the amount of SGO nanosheets added to the membrane. The many features and applications of GO nanostructures caused various methods to be proposed for their production [6]. These methods are usually associated with wasting materials and spending a lot of time and energy. In recent decades, plasma methods for the production and deposition of nanomaterials have received more attention [7, 8]. Due to the presence of active species such as ions, electrons, radicals, and high-energy photons, the plasma environment provides the necessary conditions for the advancement of chemical and physical reactions for the deposition of nanomaterials at low temperatures [9]. Plasma electrons play an effective role in reducing metal precursors to produce desired nanomaterials. In recent research, it was found for precursors in the liquid phase that plasma electrons in contact with the fluid surface can penetrate it and directly regenerate the dissolved metal ions so that the desired nanomaterials are finally produced [10-12].

In this research, the nanofiltration membrane was prepared based on commercial polyvinyl alcohol polymer and then coated with graphene oxide nanostructures. In the following, the effect of adding GO nanostructures on membrane properties such as blockage and its desalination rate from salt water is evaluated. It seems that the plasma modification of GO causes uniform dispersion of nanoparticles inside the PVA membrane and significantly increases the amount of desalination and purification of salt water.

Materials and Devices

Laboratory materials from Merck, Germany were used in this work. the commercial polyvinyl alcohol (PVA) membrane with a thin layer of polyamide nano filter, sulfuric acid (H_2SO_4), phosphoric acid (H_3PO_4), nitric acid (HNO_3), hydrochloric acid (HCl), potassium permanganate ($KMnO_4$), hydrogen peroxide (H_2O_2), and sodium sulfate (Na_2SO_4). Field emission scanning electron microscope (SEM) made by TE-SCAN company was performed. The Fourier Transfer Infrared Spectroscopy (FTIR) model AVATAR 9 from Thermo, USA was used to study the molecular structure and determine the functional groups of nanopowders.

Preparatory Preparation

First, graphene oxide is obtained by the modified Hummer method. For the synthesis of graphene oxide, first, 67.5 ml of H_2SO_4 , 10 ml of H_3PO_4 , and 22.5 ml of HNO_3 were poured into a beaker and placed on a magnetic stirrer for 20 minutes to mix well. Then, 1 gram of graphite powder was added to this acidic solution and stirred for 30 minutes until the graphite powder was well eaten with acid. In the next step, the obtained solution was placed in an ice bath and 6 grams of $KMnO_4$ was slowly added to it over 1.5 hours. Then, the solution was placed in a bath with a

temperature of $45^\circ C$ for 2 hours. Then, 100 ml of deionized water was slowly added to the solution by burette, the temperature of the bath was brought to $85^\circ C$ and the solution was kept at this temperature for 1 hour. The solution obtained in this part is cocoa milk in color. After this period, 15 ml of H_2O_2 and 120 ml of deionized water were added to the solution at the same time, and the solution was centrifuged for 3 minutes at a speed of 750 rpm so that the unreacted graphite was separated from the solution, and deposited at the bottom of the container. These black deposits are thrown out and the remaining yellow solution is passed through filter paper. Then, 4 ml of HCl was diluted with 40 ml of deionized water and the obtained graphene oxide deposits were poured into this prepared solution and placed in an ultrasonic bath. Finally, the solution was centrifuged for 10 minutes at a speed of 8000 rpm to separate the graphene oxide from the acid, and then this washing was repeated several times until its pH was between 5 and 7. The final precipitate was dried at room temperature. To prepare the precursor material required for the layering of graphene oxide nanostructures, 0.06 g of graphene oxide was poured into 40 ml of deionized water and placed in an ultrasonic device with a probe for 30 minutes to obtain a completely uniform solution. This solution is converted into droplets with a size of 2 to 10 micrometers by the nebulizer. These tiny droplets are suspended in the plasma for a very short time. Active plasma species, especially electrons, hit the surface of the droplets suspended in the plasma and penetrate the surface of the substrate [14-16].

METHOD

Figure 1 shows the non-thermal plasma jet for modification. This jet works with a dielectric barrier discharge configuration that has two copper electrodes wrapped on the surface of a quartz tube at a distance of 1 cm from each other. This quartz tube with an inner diameter of 2 mm and an outer diameter of 4 mm is used as a dielectric in this plasma jet. Plasma gas flows inside this quartz tube along with aerosol carrier gas. To produce plasma, these electrodes are connected to a high-voltage power supply, between 5 to 10 kV, and a working frequency of 20 kHz, and the voltage applied to the plasma is measured by a high-voltage probe and an oscilloscope. In this system, argon gas is used as plasma gas and aerosol carrier gas. The plasma gas flow rate is 2 SLPM (The standard liter per minute) and the carrier gas is 0.5 SLPM. After passing through the pneumatic nebulizer, the carrier gas creates an aerosol of graphene oxide solution droplets is added to the plasma gas, and flows along with it into the quartz tube of the plasma jet. After passing through the gap between two electrodes, this gas becomes a plasma and is radiated to the surface of the membrane substrate. The distance between the substrate and the plasma nozzle is 3 mm. The surface of the membrane substrates is completely cleaned and free of any contamination with acetone, ethanol, and deionized water [7-11].

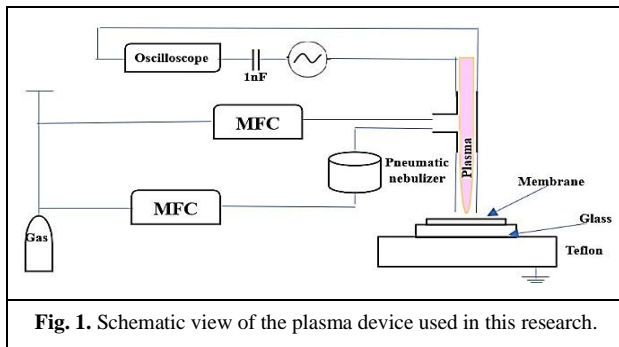


Fig. 1. Schematic view of the plasma device used in this research.

Evaluation of the Performance of the Modified Membranes

Contact angle measuring system model OCA 15 plus manufactured by Dataphysics company measured the hydrophilicity of membranes with different percentages of filler and for each membrane, the average of three times of the test related to the contact angle of water drop on the membrane surface was reported [17]. A cross-flow filtration system was used to investigate membranes with different nanofillers. Each membrane with an effective surface area of 36 square centimeters was placed in contact with deionized water under a pressure of 1.5 MPa to be integrated and compressed and a stable flux was removed from it. Then, feed solutions containing Na_2SO_4 , and NaCl were added to the stock, respectively. The operating pressure was reduced to 1 MPa, and the amount of salt removal and membrane repulsion (R) and water flux through the membrane (J) were obtained from the following relations:

$$R = \left(1 - \frac{c_p}{c_f}\right) * 100 \quad (1)$$

In this regard, c_p is the salt concentration in the solution passing through the membrane and c_f is the salt concentration in the feed solution.

$$J = \left(\frac{V}{A \cdot \Delta t}\right) \quad (2)$$

V is the volume of flux passing through the membrane in liters (l), A is the effective surface area of the membrane in square meters (m^2), and Δt is the duration of the separation process in hours (h) [18].

RESULTS AND DISCUSSION

In Figure 2, the chemical structure of pure PVA and the modified membranes was examined using FTIR in the range of $600\text{--}4000\text{ cm}^{-1}$ wave number [19]. The spectrum of pure PVA membrane, the peaks of 842 and 1118 cm^{-1} are related to the symmetric stretching of the C–O–C bond in the ether functional group. Peaks 1732 , and 2898 cm^{-1} are related to the stretching vibration of carbonyl C=O, and $-\text{CH}_3$ group, respectively. Also, the peaks of 1445 , and $3300\text{--}3500\text{ cm}^{-1}$ were attributed to the movement of H–N–C=O, and N–H groups, respectively. The peak at 3267 cm^{-1} indicates the hydroxyl-OH group and the peak at 2912 cm^{-1} is related to the symmetric vibrations of C-H. The observed vibrational peak in the range of 1419 cm^{-1} was attributed to CH_2 bending. By combining GO with PVA, the FTIR spectrum shows the formation of common features such as O-H, C=O, C=C, $-\text{CH}_2$, and C-O

stretching at 842 , 1118 , 1689 , 1419 and 3841 cm^{-1} , respectively. It can be seen that the peak in C=O stretching, and C=C stretching became more obvious compared to single element GO. The same applies to the $-\text{CH}_2$ stretch, C-O stretch, and O-H stretch compared to pure PVA. The presence of polar oxygenated functional groups such as carboxyl and hydroxyl on the GO surface is very useful for hydrophilicity, dispersion in aqueous solution, and generation of negative surface charge on quantum dots.

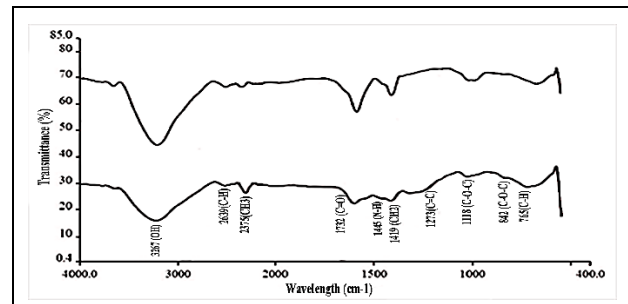


Fig. 2. The FTIR analysis results of the PVA control (Downer), and plasma-modified membrane (Upper).

Microscopic properties of PVA and GO-PVA thin films are well investigated using AFM analysis and determining the root mean square of surface roughness [20]. As it is clear from Figure 3, the control membrane has a rough, compact, and nodular surface with $R_{\text{rms}}=27.07\text{ nm}$. It shows nm. For the modified membranes, the formation of knots is increased and a rougher surface is observed for these membranes. Therefore, $R_{\text{rms}}=29.41\text{ nm}$ was obtained for the membrane modified with GO.

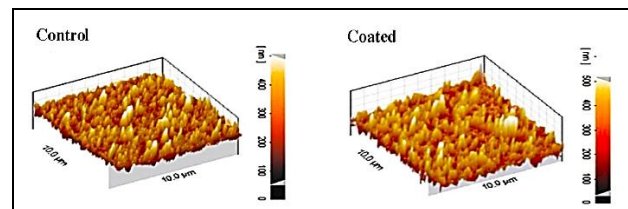


Fig. 3. The AFM analysis results of the PVA control and plasma-modified membrane.

The results of SEM analysis (Fig. 4) are in good agreement with AFM analysis and as expected, the modification by non-thermal plasma jet after hitting the surface of the membrane has more roughness than the control sample. The changes in this roughening of the PVA membrane surface can be seen after modification.

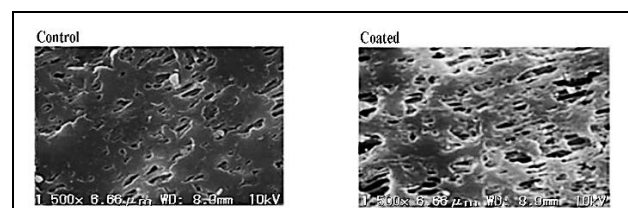


Fig. 4. The SEM analysis results of the PVA control membrane and plasma-modified membrane

In general, the contact angle depends on different factors, including surface hydrophilicity and roughness [21]. Compared to the pure PVA membrane, the contact angle for modified membranes gradually decreases with increasing plasma treatment (Fig. 5). The results of AFM analysis show the roughening of the surface and the results of FTIR analysis show the presence of polar functional groups on the surface of the modified membranes. According to the results of the contact angle of the contact drop, it can be concluded that the effect of chemical compounds occurring on the surface of the modified membranes prevails over the morphological effects and finally the effect of plasma modification leads to making the membranes more hydrophilic [22]. If, contrary to the results of this research, the effects of surface roughness were dominated by the chemical composition of the surface, the contact angle of the modified samples showed hydrophobicity. Therefore, it seems that the presence of multiple oxygenated functional groups confers high hydrophilic properties on the membranes.

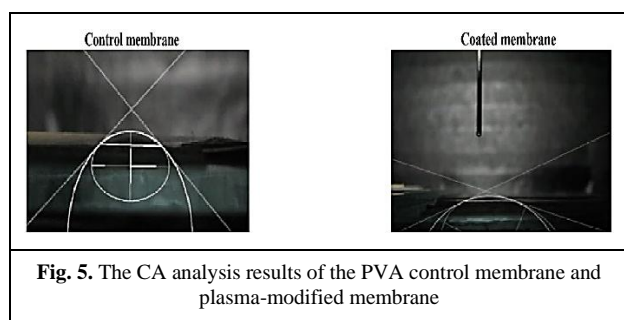


Fig. 5. The CA analysis results of the PVA control membrane and plasma-modified membrane

The Effect of Plasma Modification on the Desalination Performance of Membranes

The obtained values of saltwater flux and repulsion of Na_2SO_4 and NaCl salts for pure and plasma-modified membranes are presented. It can be seen that the water flux increases with the GO plasma treatment. For pure PVA, the average water flux for NaCl and Na_2SO_4 reaches $60.1 \text{ L/m}^2 \text{ h}$ and $73.9 \text{ L/m}^2 \text{ h}$, respectively. The modified membrane shows the high Na_2SO_4 and NaCl passing flux. For the membrane modified by the plasma method, the appropriate hydrophilicity of the membrane surface along with the formation of more water channels in the border between the nanoparticles and the outer surface layer has increased the flux passing through the membrane. For the membranes with more plasma treatment, although the hydrophilicity of the membranes increased, it seems that the accumulation of GOs and their clumping on the surface prevented the passage of water flux inside the membrane.

After checking the filtration process, it was observed that the removal process of salt Na_2SO_4 is more than that of salt NaCl . Although the mechanism of salt separation in nanofiltration membranes is complex, nevertheless, the repulsion in these membranes can be related to the molecular sieve (which depends on the average size of the nanofiltration membrane pores) and Donnan's limitation effect [23]. The Na_2SO_4 salt repulsion for the modified membrane is maintained at a high level and relatively stable. Also, negatively charged nanofiltration membranes

show more repulsion for divalent anions such as SO_4 than monovalent anions such as Cl , because these membranes have a stronger electrostatic repulsion. Considering the effect of Donnan's limitation, the concentration of ions on the surface of the membrane is not equal to their concentration in the solution. In other words, the concentration of ions opposite to the membrane charge on the membrane surface is higher than their concentration in the solution, and then the concentration of ions with the same name as the membrane charge on the membrane surface is lower than their concentration in the solution. When an external pressure is applied to the membrane, water can pass through the membrane while the ions of the same name are repelled due to the Donnan repulsion effect. At the same time, due to the electrostatic balance, ions opposite to the charge of the membrane are also repelled. Therefore, due to the stronger electrostatic attraction of divalent cation compared to monovalent cation, it leads to less salt rejection. Also, for monovalent anions, the radius of hydration is greater; Therefore, the withdrawal increases [24]. In the plasma-modified membrane, Na_2SO_4 salt repulsion shows a significant increase after treatment. With the increase of more functional groups on the surface of the modified membranes, the surface charge has increased, and correspondingly, the repulsion between the solutes and the membrane surface has increased due to the Donnan effect, and therefore, salt repulsion has improved.

Graphene oxide nanostructures have been widely used as an inorganic filler in the structure of polymer membranes due to their properties such as electrical conductivity, high mechanical/thermal resistance, and suitable surface absorption. Since Graphene oxide nanostructures have excellent electrical conductivity, they also have catalytic/disinfection applications. To investigate the antibacterial properties of these membranes, the author addresses challenges related to dairy processing, where the production of quality lactose-free milk products is a critical issue.

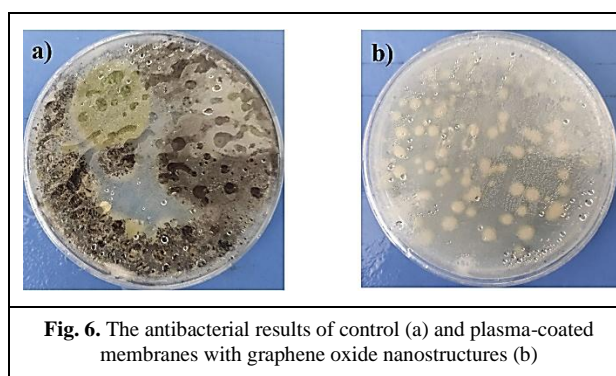


Fig. 6. The antibacterial results of control (a) and plasma-coated membranes with graphene oxide nanostructures (b)

- Modified membranes have antibacterial properties
- In the standard conditions of $\text{pH} = 6$, the filtration test of one liter of unfrozen milk with natural contaminants was performed.
- In the control membrane (Fig. 6a), fungi (black and green parts) and bacteria (yellow dots) grew
- In the modified membrane (Fig. 6b), no fungi grew and only bacteria grew.

CONCLUSION

Saline waters make up 97% of the earth's water and more than 3% of the rest is in the form of ice in the poles, natural glaciers, and underground waters. Therefore, a very small percentage of the total water on earth is usable and available. Considering the importance of the topic, in this research, modification of PVA-based thin layer nanofiltration membrane was studied to desalinate salt water. Plasma modification of thin layer nanofiltration membrane was used to improve hydrophilicity and increase separation performance membranes. Graphene oxide nanostructures were directly deposited by a non-thermal plasma jet at atmospheric pressure on the surface of the membrane substrate and the effect of functional groups on membrane properties such as chemical structure, and separation performance was systematically investigated. AFM and SEM images showed that due to the launch of graphene oxide nanostructures with different sizes and densities, the surface roughness increases after plasma treatment. FTIR analysis confirmed the increase of polar groups on the surface after modification. The simultaneous effect of the two led to a more hydrophilic surface of the PVA nanofiltration membrane after GO plasma modification compared to the control membrane. Using the results obtained from the analysis, it was found that the membrane modified with graphene oxide nanostructures shows a higher water flux while maintaining high salt repulsion. Graphene oxide-modified membranes showed better surface properties including higher hydrophilicity compared to pure PVA membranes. The best separation efficiency of the modified membrane was obtained for Na₂SO₄. It was also observed that membranes modified using graphene oxide nanostructures had more suitable antibacterial properties than other unmodified membranes.

The presence of defects, the size of the pores, and the chemistry of the membrane surface are among the factors that can be studied further. Also, the long-term stability of GO-based membranes has always been a major challenge for researchers. The stability test time of this type of membrane is only from a few hours to a few months, which is very short for practical applications. Therefore, the long-term stability of nanofiltration membranes based on GOs should be further investigated. Also, modification of the surfaces of the resin membranes to change the nature of hydrophilic/hydrophobicity using different methods such as plasma irradiation is another issue that needs a detailed discussion. We hope that this review will provide a promising motivation and a clear vision for researchers to overcome the existing limitations to fabricate high-performance membranes in the future.

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