Research Paper

A Novel Electrodialysis Membrane, Modified by Polydopamine and Carbon Nanofibers, Removes Toxic Heavy Metal Ions From Wastewaters

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ABSTRACT

Background: In light of severe and growing shortages of clean water and the rising environmental pollution in many countries, seawater desalination has been an effective method to produce freshwater. Cationic membranes have enabled environmental scientists to effectively remove toxic heavy metals from wastewater and to regenerate freshwater.

Methods: We used a novel method, involving electro- and physico-chemical membranes to successfully remove toxic heavy metal ions (copper & chromium) from wastewater samples. Specifically, Fourier-transform infrared spectroscopy, field emission scanning electron microscopy, surface wettability, flux, selectivity and electrical resistance measurements were applied to conduct this study.

Results: The obtained results illustrated relatively uniform foaming of the modified membranes. Also, electron microscopic images indicated almost even distribution of the particles. The dada indicated that applying polydopamine layer and incorporating nanofibers in monomer solution caused surface hydrophilic enhancement. Also, increased carbon nanofibers loading ratio to 0.07% raised the ionic flux. The data also showed a higher capacity in the modified membranes for the removal of copper and chromium ions from the wastewater samples. Although the surface modified membranes displayed a higher flux and lower permselectivity to some extents, utilizing nanoparticles led to a steady trend of ion elimination. Generally, carbon nanofibers incorporation to the membrane surface modified samples up to 0.1% weight, resulting in nearly a constant areal electrical resistance.

Conclusion: The novel method developed by this study is an excellent candidate with high potential for the removal of toxic heavy metal ions from wastewater samples.

Keywords: Membranes, Cations, Water pollutant, Heavy metals

Intrpduction

Over the past decade, the environmental pollution and the severe shortage of water resources have grown considerably. It is very clear that seawater desalination is considered as a worthy method of regenerating freshwater suplies for public consumption [1, 2]. In recent years, advanced functional ion exchange membranes (IEM) prepared with nanoparticles have provided an excellent opportunity among various fields in industries and human life, for the separation of various types of liquid and gas contaminations [3, 4]. Membrane-based seperations have been developed rapidly to generate clean water from various waste resources due to their greatly promising advantages [5, 6].

Reverse osmosis (RO) technology has received much attention globally, among all other water desalination methods [7]. However, recently RO developement has become limited due to the environmental problems arising from the resultant concentrates [8]. Alternatively, electrodialysis (ED) has been preferred for the treatment of salty wastewaters, in which greater dilute brackish water is produced from the efficiency perspective, as an appropriate choice. The main reasons are its nonpolluting and environmental friendliness compared to RO and thermal methods [9-12]. The ED and membrane deionization methods utilize ion exchange membranes to produce fresh water, involving materials with charged groups. Developing an effective IEM requires improved seperation properties, such as permselectivity, ionic conductivity and chemical stability [13].

Nanocomposite ion exchange membranes are prepared by incorporating particle additives into polymeric membranes. These products have demonstrated considerable advantages for their mechanical strength and seperation charactristics, compared to the homogeneous counterparts [14]. Recently, typical additives include carbon nanotubes [15], silica [16], graphene-based nanomaterials [17], and copper ferrit [18]. However, more recent studies, using IEM for wastewater desalination, have been concerned with the rational design of nanopore memranes. As indicated by a number of recent studies [19-22], the most ion diameters in salty wastewaters vary from 0.4 to 0.9 nm [17], while the size of small organic molecules ranges 1−2 nm. In this context, the optimal size of membrane nanopores should be 0.9-1 nm to reach a high permselectivity for salt ions and organic molecules.

Further, utilizing nanoparticles at higher percentages can result in undesireable challenges, which may diminish the electrochemical characteristics of the resultant IEM [19, 20]. In recent years, application of polydopamine (PDA) has recieved significant attention in the process of membrane modification [21, 22]. Dopamine monomer is freely oxidized and polymerizes to create a crosslinked PDA [23]. An earlier study [24] has found that surface modification of membranes based on polyethylene (PE), polyvinylidene fluoride (PVDF) and polytetrafluoroethylene (PTFE) with polydopamine (PDA) may increase their hydrophilicity [21]. Also studies have demonstrated that the interspaces between the accumulated PDA nanoaggregates can make nanopores sized 0.5 to 2 nm, in which transport mechanism can be controlled by the formation of PDA membranes [24].

Aim of the study: This study aimed at developing a novel electrodialysis method to remove toxic heavy metals, such as copper and chromium, from wastewaters. We used polyvinylchloride (PVC) due to its desireable properties, mainly the chemical and biological resistance, and its easy processing.

Novelties of the study: Since PVC membranes show low permeability and high selectivity [25], PDA may control and modify the seperation properties, improve the permeability and selectivity of PVC-PDA-CEMs. It can be obtained by the incorporation of such materials, as carbon nanofiber (CNF) to achieve a novel doublelayer cationic exchange Membrane (DLCEM). Other advantages of filtration with CNF membranes are attributed to their porous and interconnected structure, large surface area to volume ratios, and the submicron pore sizes. These features allow cationic exchange membranes to be permeable with superior filtration efficiency [26].

Materials and Methods

Materials: All solvents and reagents were commercially available and used per the suppliers' instructions. Polyvinylchloride (PVC; S-7054) was purchased from BIPC (Tehran, Iran), and used as a polymerbinder. Tetrahydrofuran (THF; 72.11g/g mol) was utilized as the base solvent. Dopamine hydrochloride and carbon nanofiber (CNF), used as the nanofiller, were purchased from Sigma-Aldrich. (Darmstadt, Germany).

Preparation of doublelayer cationic exchange membrane: The cationic exchange membranes (CEM), used as the substrate, were prepared by casting solution procedure. The PVC polymer, used as the main body,was dissolved in THF (1:20; w/v). Next, powdered resin particles (Amberlyst 15, >1.7 meq/g; Merck Inc., Germany) was added to the polymer solution at equal ratio (w/w). To break up the particle aggregates evenly, the mixture was sonicated for 90 min on an ultrasound apparatus (Parsonic, 28 KHz; Tehran, Iran). The solution was then casted on glass plates and were left at room temperature to complete dryness.

To make the ultimate modified membranes, dopamine hydrochloride powder and varying ratios of CNF, as the filler, were dissolved in 30 mL deionized water and sonicated for 2 hr. See details in Table 1. The clumps of PVC-CEMs with the area of 12.56 cm^2 were prewetted in deionized water for 1 hr, and immersed into the prepared solution at pH 10 for dopamine polymerization on the membrane surface. Also, a few drops of galic acid (GA) was added to the solution as the crosslinking agent.

The reaction continued for 3 hours at 90˚C. Finally, the surface modified DLCEMs were rinsed with deionized water to clean the surface from the alkaline substances.

Membrane characterization: The surface morphology, structure and uniformity of the CEMs were examined under electron microscopy (FESEM, model: MIRA3TESCAN-XMU; Razi Foundation, Tehran, Iran). Also, the samples were chemically characterized by a single beam Fourier transform infrared spectrophotometer (FTIR, Galaxy series 5000). To evaluate the surface hydrophilicity, hydrophobicity, photocatalystic effects and surface self-cleaning of the membranes' contact angel, these features were measured in deionized water at room temperaure.

Water content: The samples were dissolved in deionized water for 24 hr to measure the water uptake (10^{-4}g), using an OHAUS apparatus (New Jersey, USA). The membranes' wet weight (W_{wet}) was determined after removing the extra water droplets on a Wattman paper filter. Wet membranes were dried in an oven at 70˚C for 4 hr, after which the water content was determined based on Equation 1 as follows [27, 28]:

$$1.\quad Water\ Content\ (\%) = \left(\frac{W_{wet}-W_{dry}}{W_{dry}}\right) \times 100$$

Heavy metal ions removal The performance of the membranes for heavy metal ions removal was examined by an ED test cell in three parts. The concentrations of and cations were assayed in the aqueous solutions of $CuSO_4$ and $CrSO_4$, by atomic emission spectroscopy. The residual concentrations of the cations were used to estimate the percentage of the ions' separation. The pH of the feed was set at 7. A simple schematic illustration of the ED setup is shown in Figure 1.

Measurement of ionic flux: To measure the ionic flux, a sodium chloride solution was used in test cells with platinum electrodes, with the terminals connected to direct current. The ionic flux in the membranes was measured at different pH settings of the compartments [29, 30] .

Transport number and permselectivity: The current fraction carried by a special ion is considered as the transport number for that ion, which was determined based on Equation 2 as follows:

$$2.\quad t_i = \frac{z_i J_i}{\sum_i z_i J_i}$$

The above equation was also applied to determine the mean dynamic transport number (MDT) of the ions under study. This parameter was compared with static ones derived by Donnan potential. The permselectivity as assessed based on the counter ions' migrations in membrane, using Equation 3 as follows:

$$3.\quad P_s = \frac{t_i^m - t_0}{1 - t_0}$$

Where is the transport number of each counter ion [30, 31].

Electrical resistance: The membranes were placed in a cell containg electrolyte solution to determine the initial electrical resistance (R_1). Then, the second resistance (R_2) was measured, utilizing the apparatus without a sample. Finally, the membrane resisitance was determined, using the following Equations 4 and 5:

4. $R_m = R_1 - R_2$

5. $r = R_m A$

Where "r" is the real resistance and "A" is the surface area.

Rsults and Discussion

Characterization of the membranes

Morphological study: Figure 2 illustrates the FTIR spectra of PVC-PDA-DLCEM membranes. The spectrum for each sample was prepared to better describe the analysis of bonding fomation. The results indicated that the bonds at 3200-3500/cm were attributeable to the OH and NH groups in the modified membranes, and the presence of polydopamine on the surface. The peaks identified near the 1500 cm^{-1} and 1600 cm^{-1} represent the dopamine in the membranes, and denote the aromatic rings in its molecule. Also the peak at 1636 cm^{-1} is attributed to the OH symmetrical stretching vibration [32, 33], and the peak at 1197.69 cm^{-1} is related to the carbon-chloride bonds in the PVC. The carbone-nitrogen bonds in the dopamine provide for the partial inter-chain interactions [34]. It sould be noted that the CH_2 and CF_2 groups in PVC are characterized in 1330–1429 cm^{-1} and 1117–1250 cm^{-1} areas of the plots, respectively [35, 36].

The FESEM images illustrated in Figures 3 and 4 define the structural homogeneity, unifomity and integrity of the double-layer membranes with surface modifications, and incorporated additives. The resin particles and CNF additives were uniformly distributed on the mem-

Table 1. Composition of the CEM samples

Membrane Sample (M_x)	Membrane Sample	Dopamine (W/W)	CNF (W/W)
M_1	PVC-CEM	0.00/100	0.00/100
M_2	PVC--DLCEM	7.00/100	0.00/100
M_3	PVC-PDA-CNF-DLCEM$_1$	7.00/100	0.07/100
M_4	PVC-PDA-CNF-DLCEM$_2$	7.00/100	0.10/100
M_5	PVC-PDA-CNF-DLCEM$_3$	7.00/100	0.30/100

Abbreviations: M: Membrane; CEM: Cation exchange membrane; PVC: Polyvinyl chloride; PDA: Polydopamine; DLCEM: Double layers cation exchange membrane; CNF: Carbon nanofiber.

branes' surfaces. It is worth noting that the incorporation of nanofibers into the membranes casting solution (Figure 3 C, M_3 sample) caused the formation of cavities and voids. These occurred due to the sudden appearance of CNF additives and the increased nanofibers loading ratio up to 0.3% of the weight. This led to the development of sample, with a high concentration of CNF additives in the casting solution, which improved the integrity of the nanofibers' agglomeration (Figure 3 D).

Figure 4 represents the increase in surface roughness of the membranes modified with dopamine, which had negative effect on the antifouling property [37]. In this study, it was noted that the most optimal loading ratio of dopamine was achieved by a correlation between surface hydrophilicity and roughness.

Water contents: Table 2 presents the water contents of the membranes we developed, and the effects of incorporating PDA and CNF in them. The increasing trend of water uptake may be related to the hydrophilic nature of PDA and nanofibers versus the hydrophobicity of the membrane surface [38]. Results in Figure 3 indicate that a rise in the nanoparticle concentration in the membranes icreases their water content. Thus, it may be concluded that the increase in the structural heterogeneity of the membranes may be linked to the presence of nanofibers (Figure 3) [39]. By further increasing the amount of the CNF nanofibers from 0.07% to 0.1%, the membranes' water content showed a declining trend. It is likely that adding similar materials to the membranes' matrix may lead to further decline in the water uptake capacity [40].

Removal of heavy toxic metals: As shown by the data presented in Figure 5, both M_2 and M_3 membranes had a greater capacity for the removal of Cu^{2+} and Cr^{2+} compared to M_1. The higher efficiency of the membranes in the removal of the toxic ions, compared to M_1, may be due to the greater hydrophilicity of PDA surface modification, and presence of –NH and –OH groups. Also, the increased porosity of the memberanes due to incorporating nanofibers, improves their ability to eliminate the toxic heavy metal ions from the wastewater samples. The electrostatic exclusion of the toxic ions by the negatively charged membranes' surface is another reason for the observed improvement in their efficiency [41, 42].

As shown in Figure 5, with further increase in the nanofiber loading ratio of the membranes, the capacity to uptake and began to decline. This change in behavior may be due to the declining trend in the water content, seconday to a higher CNF loading ratio (Table 2). Also, the nanofibers agglomeration led to lower porosity at the site due to a lower adsorption behavior [34, 43]. Further, a higher ionic valence was likely to cause the greater ions repulsion from the membrane surface.

The elimination of the toxic ions increases by a rise in the valence of co-ions. Since the removal of the heavy metal ions improves with changes in the hydrated radius and charges of the anions, they lead to lower ions diffusion into the nanofibers and membrane surface. Indeed, the ionic removal capacity improves with an increase in the valence of co-ions and hydrated radius, because the ions repulsion occurs better at higher ionic valences. Also a high hydrated radius combined with lower ionic radius lead to ions lower diffusion into the nanoparticles and membranes surface. Therefore, Cr^{2+} with a higher hydrated radius and lower ionic radius than those of the Cu^{2+} is believed to have a higher removal capacity for ions than does Cu^{2+}. This is likely to be caused by the steric hindrance effects and the ions difficulty in passing across nanoparticles [41, 44].

The permselectivity: As reflected by the data in Table 3, lower permselectivity for compared to the unmodified membrane , can be attributed to the enhanced water content, which may lead to a decline in the co-ions percola-

Table 2. Water content of the membranes developed in this study.

Membrane Sample (M_x)	Membrane Sample	Water Content (%)
M_1	PVC-CEM	33.24
M_2	PVC--MMCEM	36.77
M_3	PVC-PDA-MMCEM$_1$	45.54
M_4	PVC-PDA-MMCEM$_2$	36.64
M_5	PVC-PDA-MMCEM$_3$	60.90

Abbreviations: M: Membrane; CEM: Cation exchange membrane; PVC: Polyvinyl chloride; PDA: Polydopamine; DLCEM: Double layers cation exchange membrane; CNF: Carbon nanofiber.

Table 3. Permselectivity for the prepared membranes.

Membrane Sample (M_x)	Permselectivity
M_1	>96
M_2	>87
M_3	>86
M_4	>83
M_5	>81

M: Membrane

tion. Under such conditions, greater amounts of water uptake can facilitate the ionic passage through the pathways [18]. With an increase in the CNF concentration in the casting solution, the permselectivity remain almost constant, although has shown a greater reduction compared to . This behavior may also be attributed to the water content decline, as reflected in Table 2. The filling of ion pathways by the CNF can narrow down the channels. Thus, the ionic agent groups transcend ion percolation, which may finally result in an increase in permselectivity or at least keep them constant.

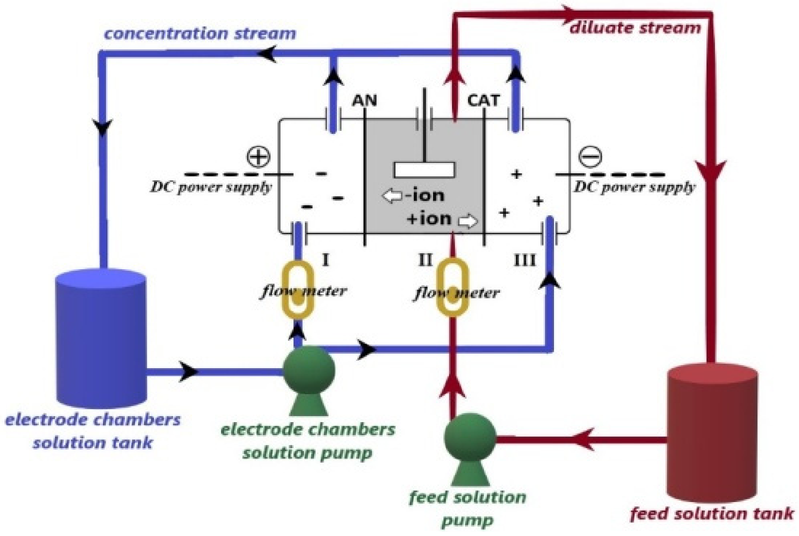

Figure 1. A simple schema of the electrodialysis setup

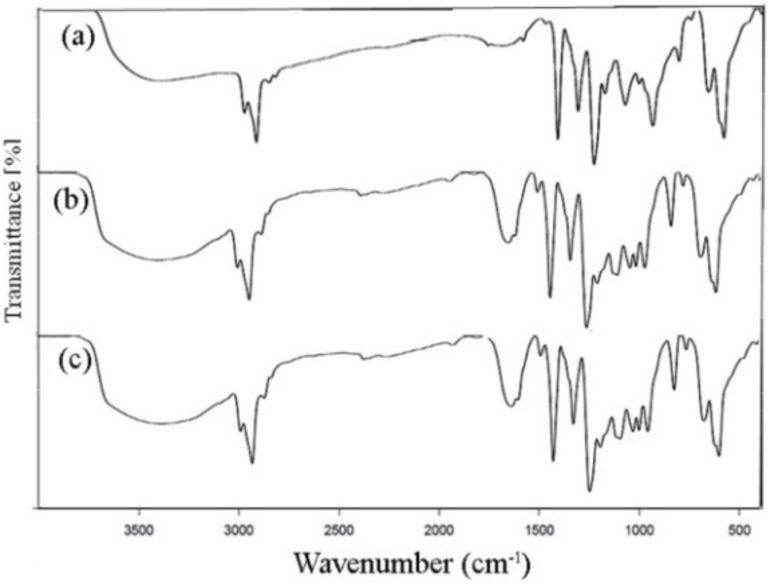

Figure 2. The Fourier-transform infrared spectroscopic spectra of a) Pure PVC, b) PVC/PDA, c) PVC/PDA/CNF.

Abbreviations: PVC: Polyvinyl chloride; PDA: Polydopamine; CNF: Carbon nanofiber.

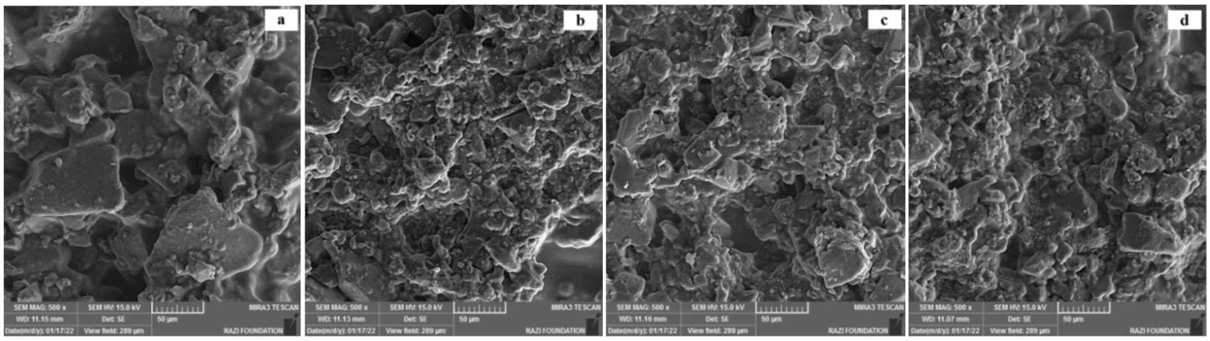

Figure 3. The FESEM images of the membrane samples at 500x magnification

Micrograph panels: a) M_1, b) M_2, c) M_3, d) M_5

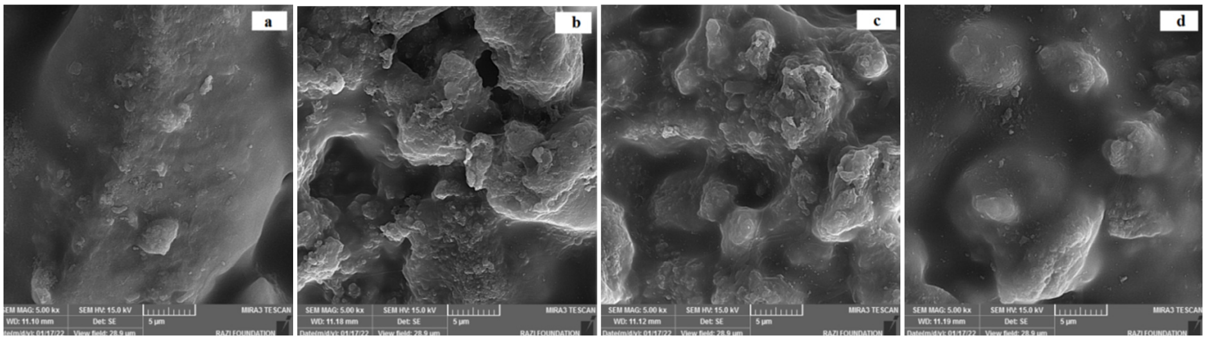

Figure 4. The FESEM image of the membrane samples at 5000x magnification

Micrograph panels: a) M_1, b) M_2, c) M_3, d) M_5

Ionic permeability and flux: As illustrated in Figure 6, membrane surface modification with PDA increased

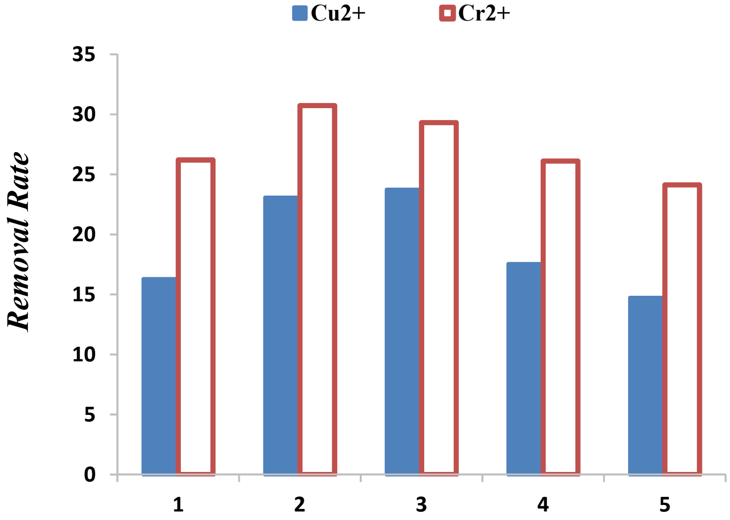

Figure 5. The removal rates of Cu^{+2} and Cr^{+2} ions from the wastewater samples

both the ionic permeability and flux. This may be due to the hydrophilic properties of PDA and controlled interspaces between the accumulated PDA and nanoaggregates [21, 24]. Furthure, the membrane ionic permeability and flux increased with a rise in the CNF nanofibers' loading ratio up to 0.07% in weight. This may be associated with the appropraite water content, and ionic pathways formation in the membranes. With greater increases in the nanofiber concentration from 0.07% to 0.3%, the ionic permeability and flux began to decline. This may be due to the lower percentage of water content. This event may also result from narrow channel formation restricting ion transport through the membranes. Moreover, this may also be due to nanofibers agglomeration with higher concentration of nanofibers in the casting solution, decreasing the membranes charge density.

Electrical resistance: Figure 7 displays the samples'areal electrical resistance (ER). As seen in the Figure, the membranes surface modification with PDA plus the CNF incorporation to the surface up to 0.1% resulted in nearly a constant areal electrical resistance. This may be due to appropraite antifouling potential and the greater hydrophilicity of PDA [38]. With a greater increase in CNF nanofibers concentration in the modified layers up to 0.3%, the membranes' electrical resistance increases clearly, likely due to restriction against the oinic transport. The rise in the samples heterogeneity in the presence of added nanofibers increased the ionic transport but decreased the resistance (up to M_3). With further increases in the CNF loading ratio and decline in the water content (Table 2), the porous spots can be surrounded by nanofibers, hence a decline in the ionic

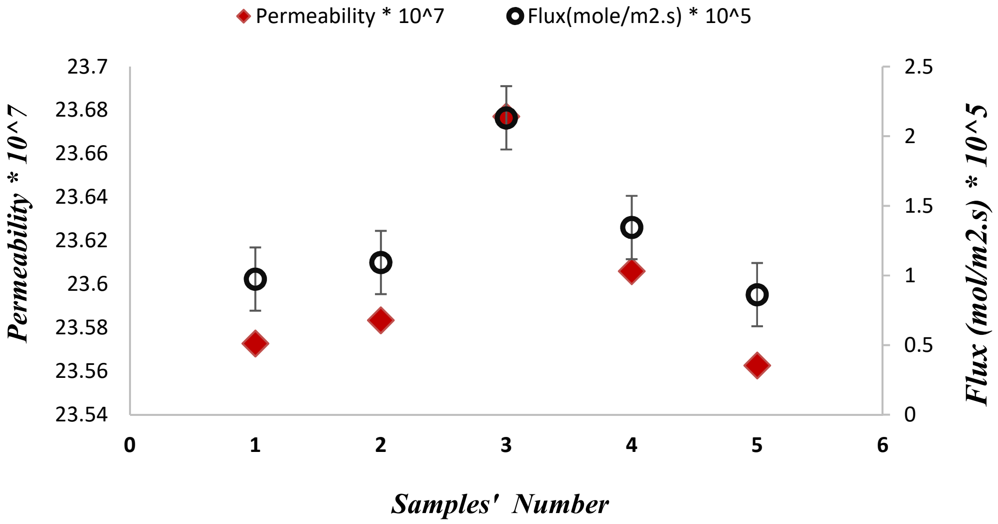

Figure 6. The prmeability and flux of the membranes

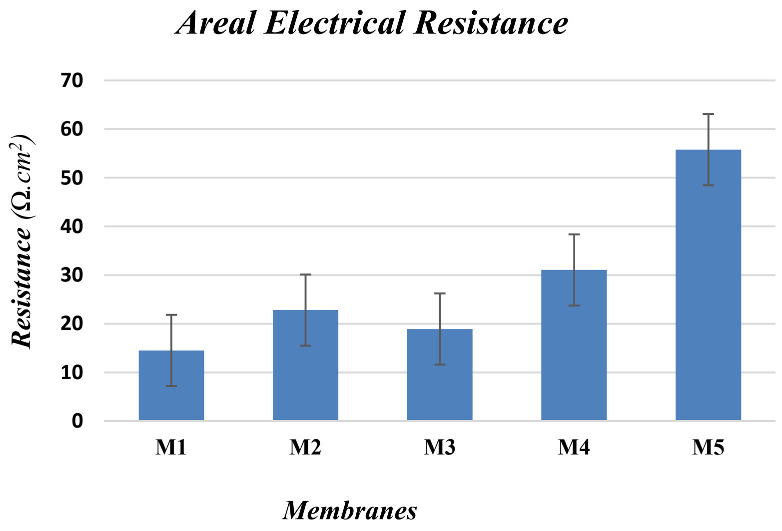

Figure 7. The areal, electrical resistance of the membranes

transport. Therefore, under such conditions, the electrical resistance rises with further increases in the nanofibers concentration in the modified layers.

Conclusions

The surface hydrophilicity of the samples increased in the presence of PDA together with PVC-PDA-CNF. The modified membrane developed in this study offers a greater sodium flux compared to that achieved by unmodified membranes. Also the modified membranes had appropriate water content, higher permeability and ion exchange capacity. Application of membranes with the surfaces modified with PDA can improve the elimination efficiency of toxic heavy metal ions from wastewaters. In general, PVC-PDA-DLMs in lower loading ratio of CNF are desireable for water desalination and removal of heavy metal ions from wastewaters.

Name of the institution where the work was done: Department of Chemical Engineering, Faculty of Engineering, Arak University, Arak, Iran.

Ethical Considerations

Compliance with ethical guidelines

There were no animals or humans used in this study; therefore, ethical considerations were not applicable.

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Authors' contributions

Conceptualization and supervision: Ezzatollah Joudaki and Sayedmohsen Hosseini; Investigation, writing - original draft: Amin Seidypoor and Samaneh Bandehali; Methodology, data analysis, writing - review & editing: All authors; Data collection: Amin Seidypoor; Funding acquisition and Resources: Amin Seidypoor. Ezzatollah Joudaki.

Conflict of interest

The authors declared no conflict of interests.

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