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Removal of boron from water through soluble polymer based on N-methyl-D-glucamine and regenerated-cellulose membrane

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ABSTRACT

This paper presents a systematic study of boron removal from simulated and real aqueous systems in northern Chile by means of polymer-enhanced ultrafiltration. Poly(glycidyl methacrylate-N-methyl-D-glucamine), P(GMA-NMG), was used to form complexes with boron, and a cellulose-regenerated membrane was used as a complex separator. The first tests were performed using two simulated water samples from northern Chile with the same pH and concentrations of boron, arsenic, and chloride. P(GMA-NMG) showed a maximum of 60% boron retention for these artificial waters. Studies of boron enrichment using P(GMA-NMG) with simulated water showed that the soluble polymer reached maximum retention capacity values between 2.0 and 4.0 mg of B retained per gram of polymer. SEM images and FTIR spectroscopy confirm the deposition of the polymer on the surface of the membrane after polymer-enhanced ultrafiltration, which explains the permeate flux decay observed in retention experiments. Finally, sorption–desorption tests of boron from real water samples show that it is possible to use the polymer P(GMA-NMG) to remove boron from waters of northern Chile. It is also possible to release the retentate and regenerate the water-soluble polymer.

Keywords: Boron; Cellulose membrane; Polymer; Ultrafiltration; Water

1. Introduction

Boron is an element widely distributed in aqueous environments and can be toxic depending on its concentration [1–3]. Excessive consumption of boron can cause certain diseases, and therefore, boron concentrations must be regulated [4]. The World Health Organization establishes a maximum permissible limit of 2.4 mg B/L in drinking water [5]. Boron is derived from natural and anthropogenic sources. In the region of Antofagasta, northern Chile, it is possible to find water contaminated with high concentrations of boron, arsenic, and other elements, which affects the quality of water used for irrigation and consumption [6–9].

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Various technologies have been developed for the removal of boron from natural water or wastewater. The implementation of membrane technology is a promising strategy for boron separation. The literature describes the use of supported liquid membranes, reverse osmosis, and electrodialysis [10–12]. However, such processes are pH-dependent, and in the case of reverse osmosis, high pressure is required [13].

We have recently investigated the separation of low molecular weight species by complexing them with water-soluble polymers and the separation of complexes by ultrafiltration [14]. This method is known as *liquid-phase polymer-based retention* (LPR) [15,16], and it allows for the removal or separation of such inorganic ions as arsenate, chromate, and borate [17–19]. LPR is a type of homogeneous method and can be selective toward a species of interest. The literature on the removal of boron by LPR is limited [14,19–26]. Dilek et al. used poly(vinyl alcohol) as a chelating agent for boron [21]. Doğanay et al. used hydroxyethyl aminoglycerol-functionalized poly(glycidylmethacrylate) or poly(4-vinyl-1,3-dioxalan-2-one-co-vinyl acetate) as polychelatogens for boron retention [23].

The ligand N-methyl-D-glucamine (NMG) is known as an efficient boron-chelating group. This ligand has been incorporated into different matrices to study borate removal from aqueous environments [27–29].

Recently, we synthesized and characterized the soluble polymer poly(glycidylmethacrylate-N-methyl-D-glucamine), P(GMA-NMG), and studied its capacity to remove borate and arsenate [14,30]. Boron removal was analyzed by LPR as a function of pH, the polymer:boron molar ratio, and the presence of interfering ions (chlorides and sulfates).

The present study focused on the separation of boron from water of northern Chile that contains boron, arsenic, and chloride. First, we optimized the separation parameters with simulated water and then studied the direct removal of boron from water samples of northern Chile via sorption–desorption cycles using P(GMA-NMG) coupled to a regenerated-cellulose membrane.

2. Experimental

2.1. Materials

N-methyl-D-glucamine (NMG), glycidyl methacrylate (GMA), and ammonium persulfate (PA) were purchased from Sigma-Aldrich. Boric acid and azomethine were obtained from Merck. Ultrafiltrationmembranes composed of regenerated cellulose (50- and 10-kDa MMCO) were obtained from Millipore.

2.2. Synthesis of P(GMA-NMG)

The synthesis of P(GMA-NMG) was conducted as follows: 60 mL of NMG solution (0.83 M) was placed in a three-neck round-bottom flask. Then, 6.8 mL (50 mmol) of GMA was added slowly to the solution. The reaction mixture was heated to 70˚C with vigorous stirring for 5 h. The product was washed with diethyl ether to remove excess GMA. The monomer solution was submitted to radical polymerization using 1 mol% of ammonium persulfate as an initiator at 70˚C for 24 h in an inert atmosphere. After polymerization, the polymer solution was fractionated using an ultrafiltration membrane with a molecular weight cut-off of 50 kDa to eliminate any unreacted monomer. The procedure has been described in detail elsewhere [30]. The polymer structure is shown in Fig. 1.

2.3. Analysis of FTIR and SEM

FTIR spectra were obtained using a Nicollet Nexus spectrometer. For analysis, 1 mg of sample was mixed with 100 mg of KBr. Spectra were obtained before and after the removal of boron.

Scanning electron microscopy (SEM) images were recorded using a Cameca model SU-30 SEM Probe to analyze the morphology of the regenerated-cellulose membrane before and after the ultrafiltration process.

2.4. LPR technique

The LPR system consisted of a filtration cell (Amicon 8050 stirred cell volume of 50 mL) equipped with a regenerated-cellulose ultrafiltration membrane with a molecular weight cut-off of 10 kDa and a diameter of 44.5 mm, a liquid reservoir with a volume of 800 mL, and a pressure source. A detailed description of the system is provided elsewhere [16]. The boron removal capacity of P(GMA-NMG) was studied by LPR via the washing and enrichment methods.

In the washing method, the P(GMA-NMG) was dissolved in 20 mL of artificial water containing suitable amounts of boron, arsenic, and chloride to mimic natural water. After dissolving the polymer, the pH

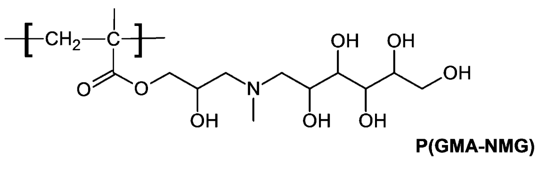

Fig. 1. Chemical structure of water-soluble polymer based on N-methyl-D-glucamine.

value was adjusted to 9.5 to ensure that boron occurred in its anionic form. This solution was washed with water at the same pH from the reservoir. The pH was adjusted using 0.1 mol L^{-1} of NaOH or HNO_3. Ultrafiltration studies were performed at 1.5 bar of pressure, room temperature, a polymer:boron molar ratio of 40:1, and the molecular weight fraction of P (GMA-NMG) above 50 kDa. The total volume (20 mL) in the cell was kept constant during ultrafiltration, and fractions of 10 mL were obtained in the filtrate. The boron concentration in the filtrate was measured by the azomethine-H method [14]. The arsenic concentration was measured by atomic absorption spectrometry using a Perkin–Elmer 3100 spectrometer.

Results are systematically presented as percent retention (% R) vs. filtration factor (Z). Retention R is defined as follows:

$$R = [B_{\text{cell}}]/[B_{\text{initial}}] \tag{1}$$

where $[B_{\text{cell}}]$ is the amount of boron retained in the cell and $[B_{\text{initial}}]$ is the initial amount of boron in the feed. The filtration factor (Z) is the ratio between the total volume of the permeates (V_p) and the retained volume (V_r) in the ultrafiltration cell.

$$Z = V_p/V_r \tag{2}$$

The second method used for boron removal is the enrichment method. With this method, it is possible to determine the maximum retention capacity (MRC) of a soluble polymer. MRC is the maximum amount of boron (mg) retained per gram of polymer. To determine the MRC of the polymer used in this study, we defined the measure as follows:

$$\text{MRC} = MV/P_m \tag{3}$$

where P_m is the amount of polymer (g), M is the initial concentration of boron (mg L^{-1}), and V is the volume of the permeate (mL). The amounts of polymer used were 75 mg for 3 mg L^{-1} of B and 125 mg for 5 mg L^{-1} of B. The total volume of the permeate varied from 100 to 200 mL.

In this evaluation, artificial water containing boron, arsenic, and chlorides was passed from the reservoir into an ultrafiltration cell containing the polymer solution. The artificial water was simulated according to the concentrations observed in water samples of northern Chile (see Table 1). The sorption–desorption method was then applied directly to the real water samples using the same procedure.

In both methods, washing and enrichment, a blank experiment (in the absence of soluble polymer) was performed.

2.5. Water samples from Antofagasta Region, Chile

Water samples from the region of Antofagasta were received and analyzed according to their concentrations of boron, arsenic, and chloride and pH (see Table 1).

This study was conducted on three water samples: The first was from the Loa River in Conchi reservoir (#1), the second was tap water from Calama (#2), and the third was an aqueous solution containing boron (#3).

3. Results and discussion

In previous studies, we determined the ability of P (GMA-NMG) to retain up to 60% boron from a solution containing 2 mg L^{-1} boron. The optimum molar ratio of polymer to boron was specified as 40:1. To evaluate the selectivity of P(GMA-NMG), we used the optimized conditions determined previously to compare the boron removal from simulated water to that from aqueous solution without interfering species.

3.1. Boron removal from water of northern Chile

3.1.1. Washing method

The evaluation of LPR efficiency was performed by the washing method. First, a blank run was performed without soluble polymer. The results demonstrated the effect of the membrane material on boron removal. The collected results show that the membrane used retained a certain amount of boron at the beginning of the experiment. However, the retention decreased rapidly, reaching 5% by the end of the experiment. This retention was improved by the presence of P(GMA-NMG). The water-soluble polymer showed approximately 60% boron retention for different artificial waters (see Fig. 2). P(GMA-NMG) contains N-methyl-D-glucamine ligands able to complex borate ions at pH 9.5, and the interfering ions did not affect the complexation.

The results obtained for simulated water samples 1, 2, and 3 can be observed to be similar. The retention of boron in sample 1 showed that the polymer is efficient in separating boron even in the presence of high concentrations of chloride. This experiment demonstrated the high selectivity of P(GMA-NMG) toward interacting and retaining boron.

Table 1
Concentrations of boron, arsenic, and chloride in water samples of Antofagasta Region, Chile

Sample	Station name	pH (at 25°C)	Boron (mg L^{-1})	Arsenic (mg L^{-1})	Chloride (mg L^{-1})
1	Loa River water	8.3	3.00	0.056	1,200
2	Tap water, Calama	6.8	5.00	0.005	300
3	Aqueous solution	9.5	2.00	–	–

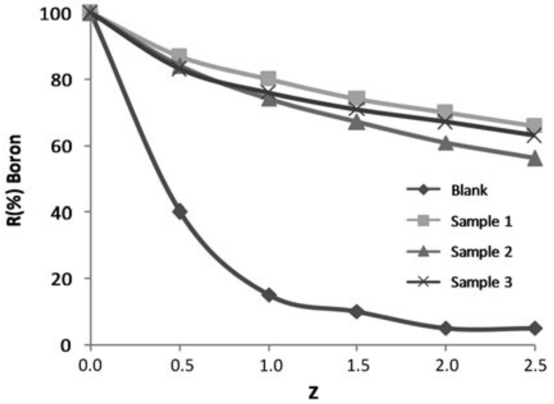

Fig. 2. Retention profile of boron from simulated samples 1, 2, and 3 at pH 9.5. Blank represents the experiment performed in the absence of P(GMA-NMG).

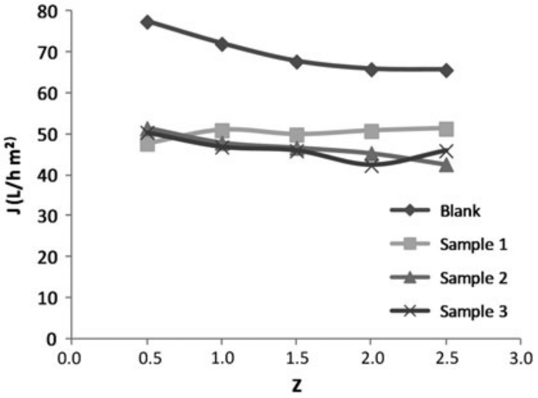

Fig. 3. Permeates flux of simulated samples containing P(GMA-NMG) at 1.5 bar of pressure. Blank represents the experiment performed without P(GMA-NMG).

Comparing our results with other membrane techniques such as reverse osmosis, the results obtained are not more efficient in removal capacity.

Results obtained using reverse osmosis showed maximum retention between 55 and 90% depending on the pH and NaCl concentration [31]. However, in the reverse osmosis, higher working pressures were used compared to LPR.

3.1.2. Permeate flux

Simultaneously, an analysis of flux was conducted on different samples of water at pH 9.5 in both the absence and presence of soluble polymer. As expected, the permeate flux in the blank in all water samples was higher than the flux permeate in the presence of P(GMA-NMG) (see Fig. 3). This finding could be attributed mainly to interactions between the polymer and the membrane. Fouling is a complex phenomenon involving sorption on the membrane surface, deposition on the adsorbed layer, compression of the layer, and possible gelation of the deposit. However, under our experimental conditions, the flux remained constant throughout the diafiltration process. Moreover, the permeate flux obtained in the experiment performed with the polymer in simulated waters was similar to that obtained in the experiment performed with the polymer in aqueous solution, and these values are in accord with those reported in the literature [23].

In addition, we studied the evolution of the flux permeates as a function of pressure at different polymer:boron molar ratios. In this experiment, the boron concentration was 2 mg L^{-1} at pH 9.5 in the absence of interfering ions. The results show that the permeate flux increased with increasing pressure and with decreasing polymer concentration. Similar permeate fluxes for the three polymer:boron molar ratios studied (40:1, 60:1, and 80:1) were obtained at pressures between 1 and 2 bar (see Fig. 4). Therefore, in this pressure range, the permeate flux is independent of polymer concentration. This concentration independence of the flux may be explained by the existence of a sufficiently high shear force due to a sufficiently high cross-velocity, which thus overcomes a possible concentration polarization [24,25]. However, at pressures above to 2 bar, the permeate flux increased at a polymer:boron ratio of 40:1 compared to the fluxes observed at ratios of 60:1 and 80:1.

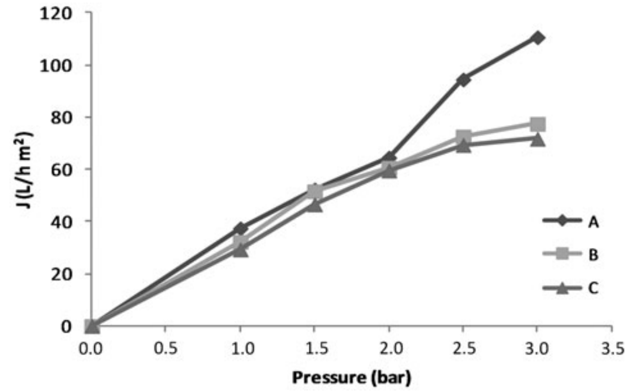

Fig. 4. Permeate flux as a function of pressure at different polymer:boron molar ratios. Curve A (40:1), curve B (60:1), and curve C (80:1).

3.1.3. Enrichment method

The MRC of boron was evaluated by the enrichment method. Fig. 5 shows the changes in the boron concentration in the permeate in relation to the permeate volume. To calculate the MRC, a blank experiment without the water-soluble polymer was performed. Using the difference in the slopes of curves obtained, the MRC can be calculated as the amount of boron (mg) complexed by the polymer (g). According to the data obtained from the blank test, it can be observed that the boron concentration reached the feed concentration within the first 20 mL of permeate added. Therefore, the retention by the membrane was considered negligible under our experimental conditions.

A study of the enrichment of boron with P(GMA-NMG) showed that sample 1 reached the saturation level of the polymer at 100 mL of permeate, whereas sample 2 reached saturation at 50 mL of permeate. On the other hand, sample 3 reached saturation at 180 mL of permeate. The values of the MRC were 4 mg B/g of polymer, 2 mg B/g of polymer, and 12 mg B/g of polymer for samples 1, 2, and 3, respectively. Despite containing a high salt content, sample 1 exhibited more efficient boron removal than did sample 2. However, the MRC measured in simulated water was lower than that measured in aqueous solution.

The literature shows results of boron removal using different insoluble polymeric sorbents containing ligands of N-methyl-D-glucamine. The sorption capacity for these materials is between 7 and 32 mg of boron retained per gram of sorbent [32]. There is a demand for more investigation, as there are not other studies on boron removal using natural water from the north of Chile.

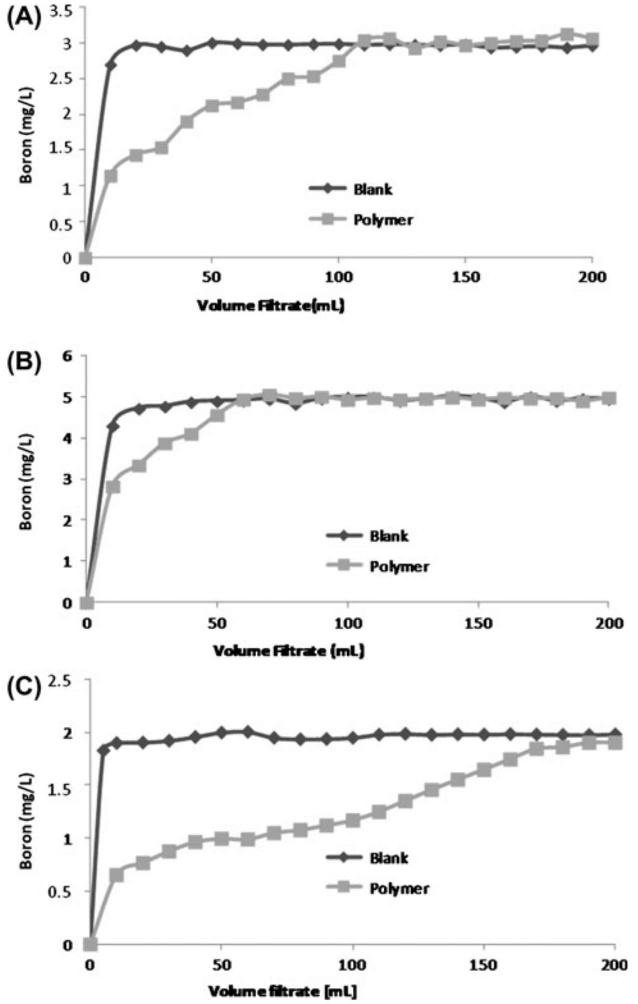

Fig. 5. Boron concentration in filtrate vs. volume of filtrate using P(GMA-NMG) as extracting agent at pH 9.5 in (A) sample 1, (B) sample 2, and (C) sample 3. Blank represents the experiment performed without polymer.

3.2. Fourier transformed infrared spectroscopy

Fig. 6 shows the spectra of (A) P(GMA-NMG), (B) P(GMA-NMG) loaded with boron, (C) the regenerated-cellulose membrane, and (D) the regenerated-cellulose membrane after enrichment. Fig. 6(A) shows absorption bands corresponding to the polymer containing methacrylate and N-methyl-D-glucamine groups. The signal at 3,398 cm^{-1} is a characteristic band of the –OH groups. For GMA, the vibrational bands include one at 1,718 cm^{-1} due to the stretching of C=O and another at 1,562 cm^{-1} corresponding to the stretching of CO–O. In the case of NMG, the characteristic bands include overlapping bands at 1,079 cm^{-1} due to the stretching of C–O, at 1,035 cm^{-1} due to the stretching of C–N, and at 1,181 cm^{-1}

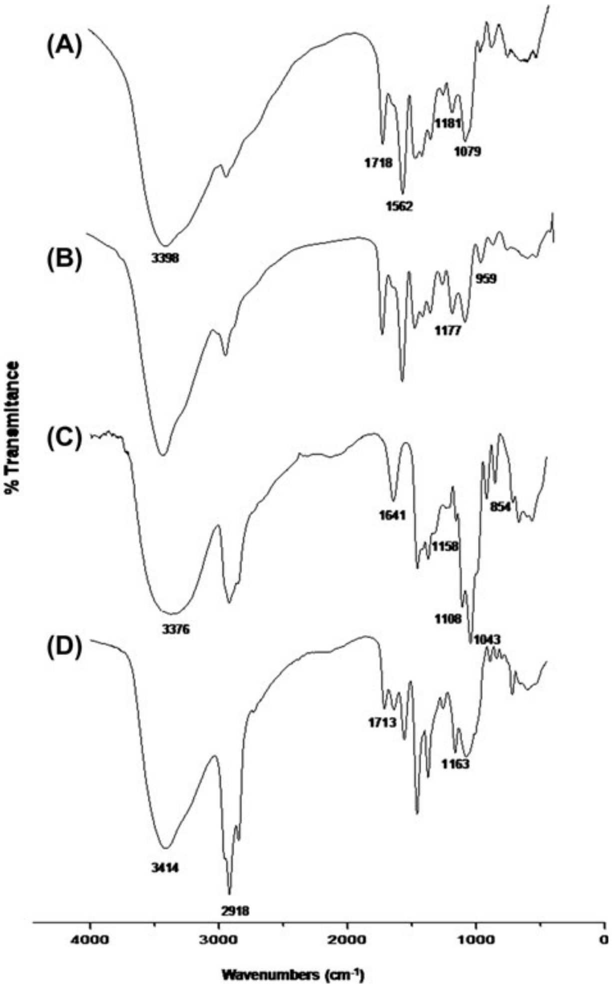

Fig. 6. FTIR spectra of (A) P(GMA-NMG), (B) P(GMA-NMG) loaded with boron, (C) cellulose-regenerated membrane, and (D) cellulose-regenerated membrane after enrichment.

corresponding to the stretching of CH–OH. The absorption bands of the epoxy ring are not present in the spectrum, confirming that N-methyl-D-glucamine was incorporated by ring opening [30].

After loading borate, some changes were observed in the absorption bands of the spectrum (see Fig. 6(B)). Boron was removed at pH 9.5; therefore, almost all of the boron occurred in its tetrahedral form, which yielded characteristic bands at 1,177 cm^{-1} assigned to the bending of tetrahedral B–OH. The band at 959 cm^{-1} is assigned to the asymmetric stretching of tetrahedral boron [33].

Fig. 6(C) shows the spectrum of the regenerated-cellulose membrane. The broad peak located at 3,376 cm^{-1} corresponds to the –OH stretching vibration. Signals around 1,641 cm^{-1} are attributed to C=O stretching vibrations. The signal at 1,158 cm^{-1} is due to the asymmetric stretching of C–O–C corresponding to the saccharine structure. Skeletal vibration is associated with the signals at 1,108 and 1,043 cm^{-1} due to C–O stretching. The band at 854 cm^{-1} corresponds to the amorphous regions of the regenerated-cellulose membrane.

Fig. 6(D) shows the spectrum of the regenerated-cellulose membrane obtained after having performed the enrichment method. The intensity of the hydroxyl peaks at 3,414 cm^{-1} corresponds to the cellulose membrane and the glucamine-based polymer. The decreased signal shown in the Fig. 6(C) is likely due to the coverage of the surface of the membrane by the soluble polymer after diafiltration. The band at 2,918 cm^{-1} is assigned to the asymmetric and symmetric stretching vibrations of $–CH_2$ and $–CH_3$ groups. Two new absorption bands are observed at 1,713 and 1,163 cm^{-1}, attributed to C=O and N–H stretching vibrations of P(GMA-NMG), respectively.

3.3. Characterization of membrane by SEM

The regenerated-cellulose membrane is a membrane formed by two layers, an active layer of cellulose deposited on a layer of polypropylene. Fig. 7 shows SEM images of the surface and a cross section of the regenerated-cellulose membrane and its modification after enrichment.

Fig. 7(A) shows the surface morphology of the virgin membrane with well-defined pores. The cross-sectional view (see Fig. 7(B)) shows the active layer of the virgin cellulose membrane, which is quite compact and homogeneous.

After performing the enrichment method, changes in the morphology of the membrane could be observed due to the presence of the soluble polymer. Fig. 7(C) shows the active surface of the membrane after diafiltration using 50 mg of P(GMA-NMG). In this image, it can be observed that the pores disappeared with the deposition of the polymer or polymer-boron on the membrane surface. Fig. 7(D) shows a cross-sectional view, which clearly shows the presence of a homogeneous layer and the accumulation of P(GMA-NMG) on the surface of the active layer of cellulose. Fig. 8(E) and (F) shows the change in the morphology of the membrane after using 300 mg of P (GMA-NMG) for diafiltration via the enrichment method. This image shows the deposition of large amounts of polymer, forming a layer containing agglomerates. The SEM images confirm the deposition of the polymer on the surface of the membrane after diafiltration, which explains the decay in the permeate flux during retention experiments and the differences in the FTIR spectra.

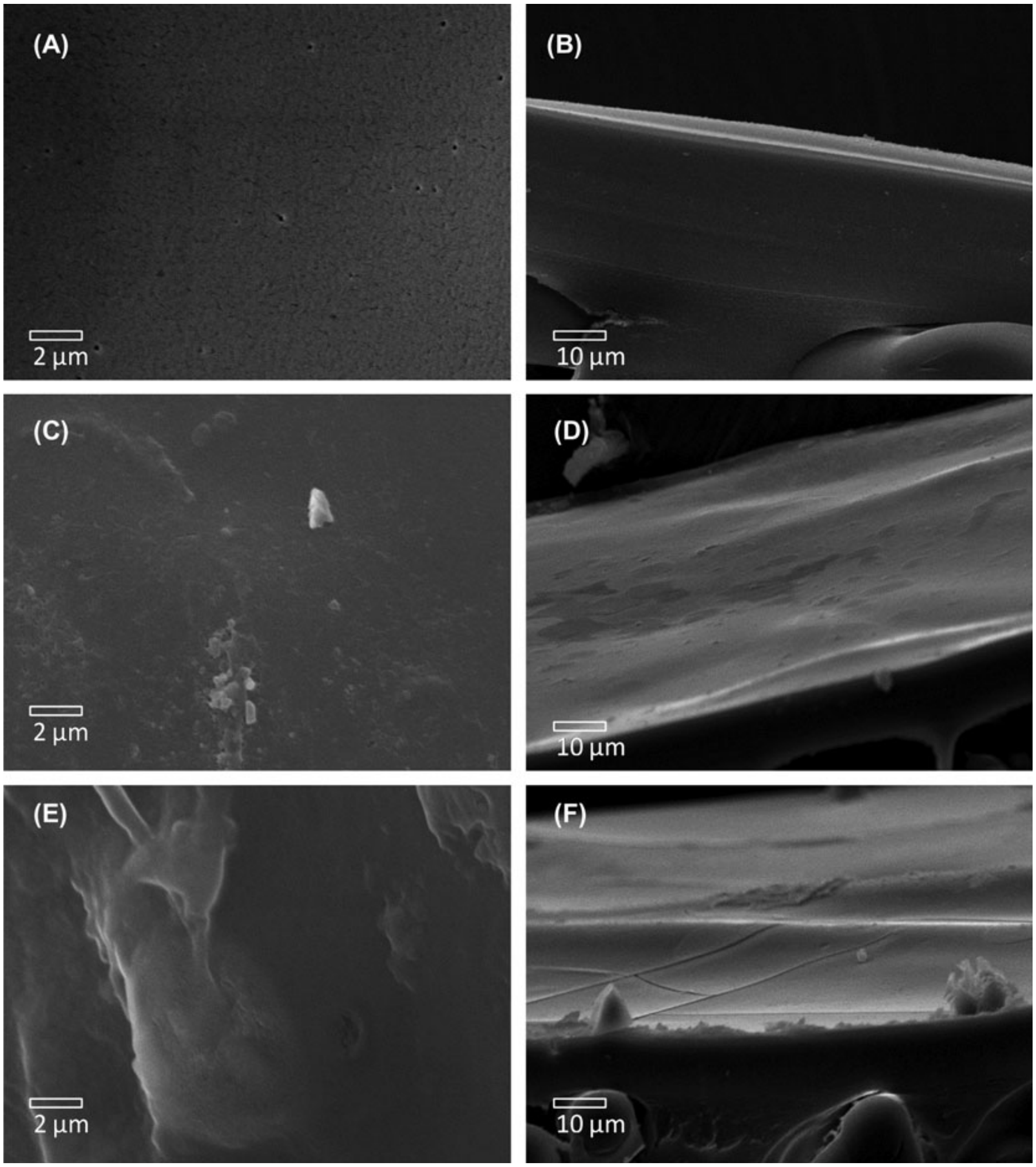

Fig. 7. SEM images of surface and cross section of (A) and (B) regenerated-cellulose membrane, (C) and (D) regenerated-cellulose membrane after enrichment using 50 mg of P(GMA-NMG), and (E) and (F) regenerated-cellulose membrane after enrichment using 300 mg of P(GMA-NMG).

3.4. Sorption–desorption of boron from real water

Finally, in this study, sorption–desorption cycling of boron was conducted using drinking water from the region of Antofagasta (sample 2). First, the sorption experiment was carried out with real water samples obtained in northern Chile adjusted to pH 9.5. Then, boron was desorbed at pH 3 under the same conditions, with the boron released considered to be that retained by the polymer. Finally, a second sorption process was performed to evaluate the extent to which the polymer could be regenerated. The experiment with natural water from the Loa River (sample 1) presented a flux that was excessively low for our experimental conditions, probably due to the presence of high salinity and organic matter; therefore, we did not submit this sample to enrichment.

Fig. 8(A) shows the saturation of the polymer for sample 2. At the beginning of the enrichment process, a high boron concentration in the filtrate was

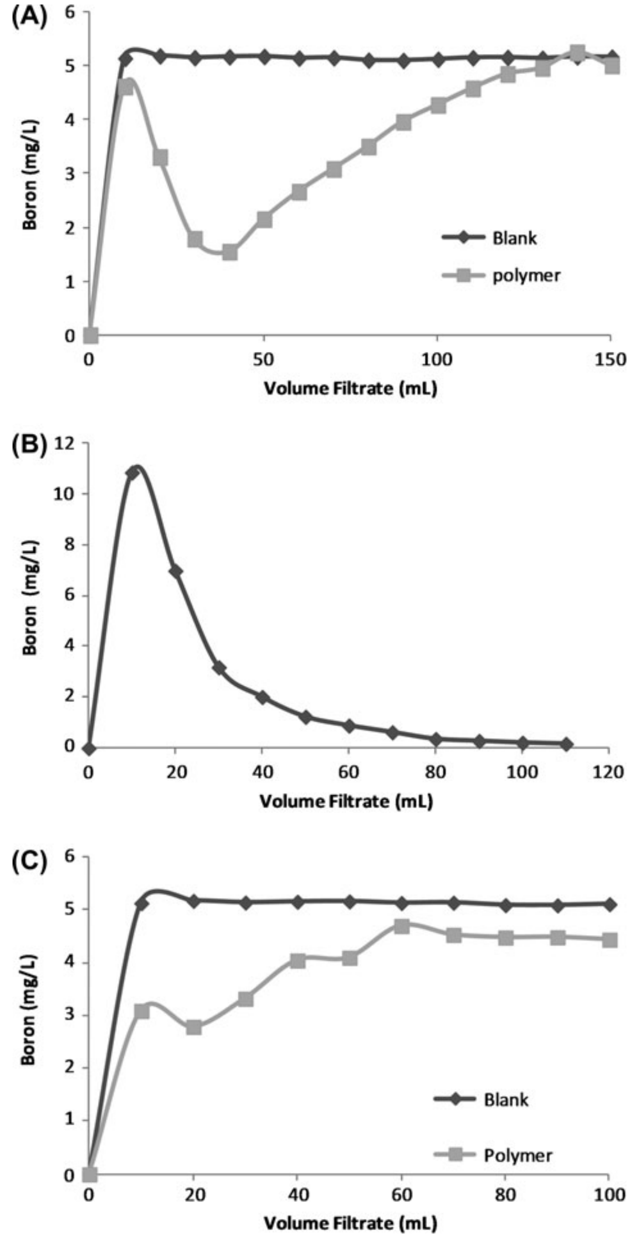

Fig. 8. Sorption–desorption of boron in real water (sample 2) using P(GMA-NMG) as extracting agent. (A) Enrichment of boron at pH 9.5 and (B) desorption profile of boron at pH 3. (C) Second enrichment of boron at pH 9.5. Blank represents the experiment performed without polymer.

observed. The concentration of boron decreased with the first 40 mL of permeate. The enrichment curve then reached saturation at 140 mL of filtrate. Thus, the enrichment showed different behaviors compared to that observed for the simulated water sample probably due to the presence of other ions in tap water. This study suggests that some adjustments can be made to the system before being used. The results of the blank experiment show that the sorption of boron by the membrane is also negligible.

Fig. 8(B) shows the delivery of boron retained by the polymer. It can be that the polymer released the highest concentration of boron within the first 10 mL of filtrate, which is sufficient to allow for the regeneration of P(GMA-NMG). Fig. 8(C) demonstrates the ability of the regenerated polymer to be used for a second sorption run. The results indicate that boron can be removed from water by P(GMA-NMG) and that the polymer can be recycled.

4. Conclusions

This research focused on the use of P(GMA-NMG) for quantitative boron removal from simulated and real aqueous environments in northern Chile.

Boron removal studies were carried out with simulated water samples. P(GMA-NMG) showed a maximum of 60% boron retention for these artificial waters.

In studies of the enrichment of boron, P(GMA-NMG) reached MRC values between 2.0 and 4.0 mg of B retained per gram of polymer.

SEM images and FTIR spectra confirm the deposition of the polymer on the surface of the membrane after diafiltration, which explains the permeate flux decay observed during retention experiments.

Finally, sorption–desorption tests demonstrated that it is possible to use P(GMA-NMG) to remove boron from real waters of northern Chile. It is also possible to release the boron retained and recover the removal capacity of the synthesized polymer.

Acknowledgments

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