



Study of the influence of operating parameters on boron removal by a reverse osmosis membrane

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ABSTRACT

Boron is a vital element for organism growth but excess of boron in water causes a problem due to its adverse effects on plants and humans. Hence, it is advisable to keep boron under specified harmless limits. However, removing boron from water is challenging. Also, it is not easily removed by a reverse osmosis membrane under natural conditions. In this paper, boron removal from aqueous solutions by the use of reverse osmosis membrane was investigated. The first objective of this work was to evaluate the characteristics of the AG-2514-TF (Osmonics) membrane used in boron permeation experiments. The next objective was to study the retention of boron by the AG membrane. The effect of several working parameters, such as pH, operating pressure, feed concentration, temperature, ionic strength, and other ions was studied in an attempt to achieve a higher removal capacity. The experimental results indicated that boron removal was dependent on the operating parameters. Indeed, boron can be effectively removed only at a pH of feed solution close to 11. To check the effectiveness of this process with the optimal operating parameters obtained, an application on underground water of Oued Bouthebane (Elfahs, Tunisia) was performed. Obtained results showed that the AG reverse osmosis membrane could be efficiently used (with 91.86% rejection) for the removal of boron from groundwater.

Keywords: Boron; Reverse osmosis membrane type AG; Operating parameters

1. Introduction

Water resources on our planet are rare, unevenly distributed, and highly threatened by overexploitation, pollution, and climate change. Indeed by 2050, the world will face a water crisis due to population increase and a decrease in the available water quantity

and quality [1]. To cope with this problem, we are led to the use of unconventional resources such as desalination of seawater and brackish water or even wastewater recycling. Recycling or desalination of water for human consumption requires increased elimination of micropollutants such as boron to minimize risks to human health and ecosystems.

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Boron is a natural element found everywhere in the environment, particularly in the seas and oceans [2]. It is an essential micronutrient for plant growth. At high doses, it is toxic and can cause the appearance of yellowish spots on fruits and vegetables, altering their taste and commercial qualities [3]. Similarly, it can cause infertility in humans and cause some nervous diseases [4]. Presence of boron in urban and industrial discharges is low as compared to natural inputs by erosion and dissolution of rocks; they are generally estimated at a quarter of the total contribution. Boron content in the industrial rejects comes mainly from the use of detergents, the nuclear industry, which uses boron as absorbing neutrons, and the metallurgical industries. Boron exists in mineral deposits and natural waters in different forms (calcium borate, boric acid, or boron hydride), but the main form in which it is present in water is boric acid. High levels of boron are found in seawater (between 4 and 5 mg/L), but in wastewater and groundwater, it can even reach up to 100 mg/L [5,6]. Recently, boron has been classified as a pollutant in drinking water (WHO Guidelines for Drinking Water Quality third edition: 0.5 mg/L, EU Directive 98/83/EC: 1 mg/L) [7,8]. Currently, in many situations, boron removal in water is necessary and has been the subject of numerous studies. But so far, no method has been proved to be quite satisfactory [9]. There are several conventional methods for the removal of boron in water based on ion exchange [10], adsorption [11], coagulation [12], electro-coagulation [13], and precipitation of the inorganic compounds [14]. These methods are not always able to meet environmental standards and often generate sludge that is difficult to manage. Our research team has therefore addressed the issue and has proposed a treatment strategy in order to have a quality of water which is in accordance with the environmental standards and is technically and economically efficient.

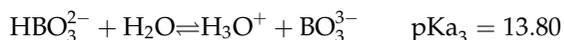
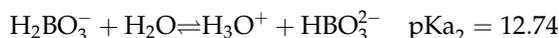
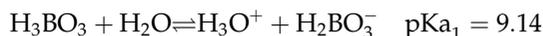
In this study, the polyamide thin-film composite reverse osmosis membrane denoted as AG-2514-TF is used. This membrane offered many advantages over traditional cellulose acetate (CA) RO membranes. The most important of these advantages were better rejection of dissolved solids and organics, increased productivity at lower operating pressures, great structural stability, and the ability to produce two to three times more purified water per unit area than CA membranes.

Moreover, we tried to test a reverse osmosis membrane of AG-2514 kind. The influence of different parameters such as concentration, ionic strength, pH, feed pressure, and temperature on membrane performance has been studied. In addition, for a better

understanding of the behavior of the membrane and in order to obtain more information about the retention of boron, this membrane was characterized by determination of permeability, charge, and roughness. Finally, an application on natural water containing boron was performed.

2. Chemistry of boron in water

Boron is usually present in water as boric acid, a weak acid which dissociates in ionic form (H_2BO_3^- , HBO_3^{2-} and BO_3^{3-}) according to:



The elimination of species by reverse osmosis membrane depends on their charges. In the case of boron, the ionic state is strongly dependent on pH, so its removal with reverse osmosis depends on pH. At $\text{pH} < 9.14$, boron is predominantly in the molecular form of boric acid. By increasing the pH, this species becomes increasingly negligible and the major species is boric acid in dissociated forms (H_2BO_3^- , then HBO_3^{2-} , then BO_3^{3-}) (Fig. 1).

3. Experimental and methods

3.1. Description of the reverse osmosis unit

Fig. 2 shows the schematic diagram of the reverse osmosis unit. The feed solution is pumped to the membrane by a high pressure pump. In the membrane, the feed stream is divided into permeate and concentrate. During operation, the permeate and the

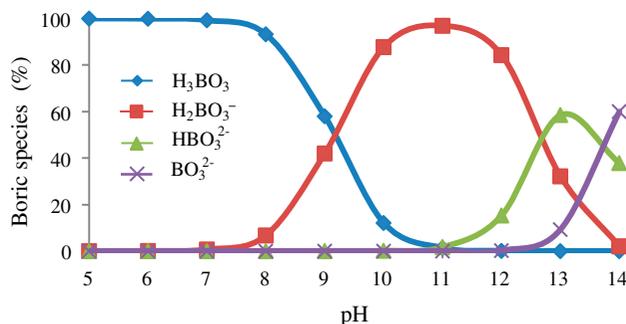


Fig. 1. Distribution of boric species at different pH (in normal operating condition).

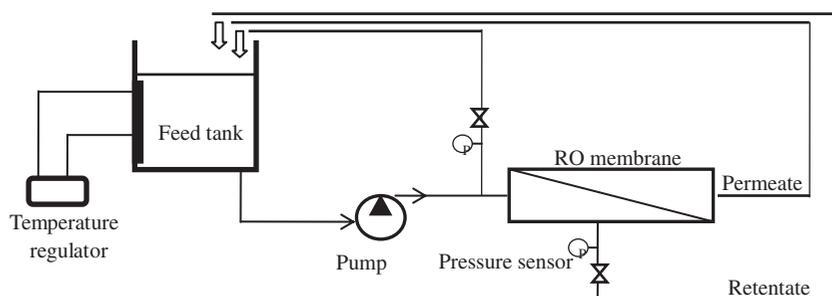


Fig. 2. Schematic of the reverse osmosis system.

concentrate streams were recirculated to the feed tank, simulating a closed-loop controlled pressure system.

The reverse osmosis membrane used in this study is a “thin-film-composite-”type AG, designed and manufactured by the company Osmonics. The manufacturer states that this membrane can operate at a maximum operating pressure of 30 bar (3.103 kPa), at a maximum temperature of 50°C, and a pH range of 4–13 in operating and cleaning. It has an active area of 0.6 m² and a nominal permeate flow rate of 0.68 m³/d at 3 × 10⁻² bar.

Membrane performance was measured in terms of membrane rejection (*R*) and permeate water flux (*J_v*). Rejection is a measure of solute separation by the membrane and is calculated as:

$$R = \left(1 - \frac{C_p}{C_f}\right) \cdot 100$$

where *C_p* and *C_f* are the solute concentration in the permeate and in the feed solution, respectively [15].

3.2. Analytical methods

Boron was analysed by molecular absorption spectrometry in the UV–visible range using Azomethine-H as a colorimetric reagent according to the procedure of López et al. [16]. Azomethine-H is not considered a standardized reagent for boron analysis in water. However, it has been selected owing to its simplicity and competitiveness over other spectrophotometric reagents, according to Fuente and Munoz [17].

Others parameters such as pH, conductivity, temperature, and salt concentrations were measured by: pH-meter Orion 2-Star, conductivity meter Metrohm 712, hand-held thermometer CTH 6200, and ionic chromatography Metrohm, respectively. Turbidimeter Lovibond TB250 IR was used for measuring turbidity. Potassium, sodium, calcium, and magnesium ions were analyzed by cation chromatography using a

Metrohm 792 compact IC with conductivity detector, an eluent of 7 mmol/L of nitric acid and 0.7 mmol/L of dipicolinic acid at 0.9 mL/min, an injection volume of 10 μL, and a pressure from 70 to 80 bar. Chloride, fluoride, nitrate, and sulfate ions were analyzed by anion chromatography using a Metrohm 761 compact IC with conductivity detector and chemical suppression, 4.6 × 250 mm Metrosep A Supp 1 (6.1005.300), an eluent of 3 mmol/L sodium carbonate at 1 mL/min, an injection volume of 20 μL, and a pressure from 80 to 90 bar.

4. Results and discussion

4.1. Membrane characterization

4.1.1. Pure water permeability

Pure water permeability, *L_p*, is one of the most studied characteristics of the membrane. It is obtained by measuring the permeate flow rate (*J_v*) vs. time and the transmembrane pressure (ΔP). The permeability of the membrane studied was measured under different operating pressures. The result obtained is shown in Fig. 3.

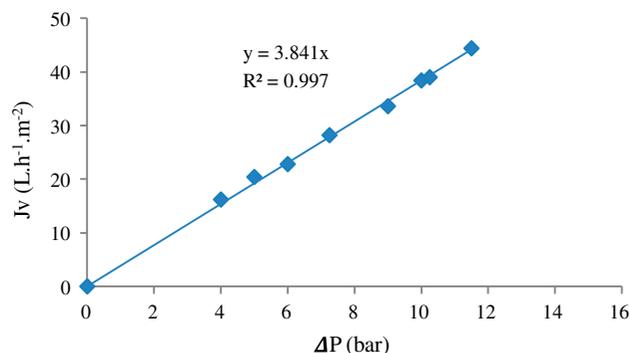


Fig. 3. The pure water permeability at 25°C.

The pure water flux through AG membrane increased linearly with increasing of the transmembrane pressure. Each data point in Fig. 3 represents an arithmetic mean value of three repeated experiments. The obtained value of the pure water permeability at 25°C is $L_p = 3.84 \text{ L h}^{-1} \text{ m}^{-2} \text{ bar}^{-1}$. This value will be considered as a reference in the remainder of our research. The systematic verification of this value can account for the effect of concentration polarization that may occur during testing.

4.1.2. Retention measurement with electrolytes solutions

To determine the charge on the membrane AG surface, we have prepared three salt solutions of NaCl, CaCl_2 , and Na_2SO_4 at a concentration of 10^{-3} mol/L . Then, we studied the retention of these three salts based on the permeate flux. The results are shown in Fig. 4.

The retention sequence for the membrane AG is: $R_{\text{Na}_2\text{SO}_4} > R_{\text{NaCl}} > R_{\text{CaCl}_2}$. This sequence shows that the membrane AG belongs to the category where the Donnan exclusion plays an important role in the retention mechanism of salts. We can deduce the following retention sequence: $R_{\text{SO}_4^{2-}} > R_{\text{Cl}^-}$ and $R_{\text{Na}^+} > R_{\text{Ca}^{2+}}$. Thus, we find that the retention of divalent anions is higher than that of monovalent anions and the retention of monovalent cations is higher than divalent cations. This is typically a negatively charged membrane behavior [18].

4.1.3. AFM images of reverse osmosis membrane

The atomic force microscopy is an excellent method for studying the topography of the skin layer of the membrane. AFM images were obtained by using NanoScope III from Digital Instruments, USA.

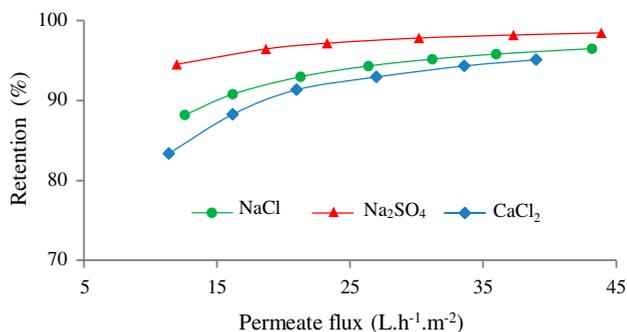


Fig. 4. The variation in the salts retention as a function of the permeate flux.

Non-contact mode of AFM in air was used to investigate the membrane pore sizes and roughness parameters. These analyses were performed on coupons from the flat membrane. AFM images of AG membrane are shown in Fig. 5.

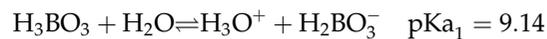
The AG membrane has a moderately smooth surface and presents a roughness of about 209 nm.

4.2. Effect of various operative factors on boron removal

4.2.1. Effect of operating pressure and pH on rejection

Results of boron removal by AG-RO membrane at different pH and pressure values are shown in Fig. 6. This part of the experiment was realized with distilled water doped with boric acid to obtain a concentration of boron equal to 5 mg/L and sodium hydroxide added for pH adjustment. It is seen that pressure has no significant effect on the rejection contrary to pH.

The RO membrane (AG-2514) exhibited boron rejection of 36.85 and 93.01 at pH values of 7 and 11, respectively. The effect of pH can be illustrated by the equilibrium dissociation of boric acid in water according the following equation:



According to pKa values of boric acid (pKa = 9.14), the dissociation of boric acid at pH < 9.14 is negligible. This explains the low percentage of boron rejection. At pH levels above 9.14, borate ion dominates, and the rejection of boron becomes increasingly higher. Generally, charged species are better retained by the RO membranes due to repulsive forces between the surface of the membrane and the anionic species. This result is similar to those found by several authors [19,20] where they mention that boron is effectively eliminated at pH 11.

4.2.2. Effect of the temperature on rejection

This part of the experiment was realized by varying the temperature from 25 to 42°C. To achieve this, additional immersion heater was used. The feed water is a 5 mg/L boron solution at pH 6.8. Fig. 7 shows the effect of the temperature on boron rejection.

The results show that the retention of boron decreases while increasing the temperature [7]. The increase in temperature causes a reduction in the viscosity of the solution, and consequently an increase of the permeate flux density. The retention rate decreases. Various authors explain this decrease by increasing the diffusivity of water and solutes with the temperature [21].

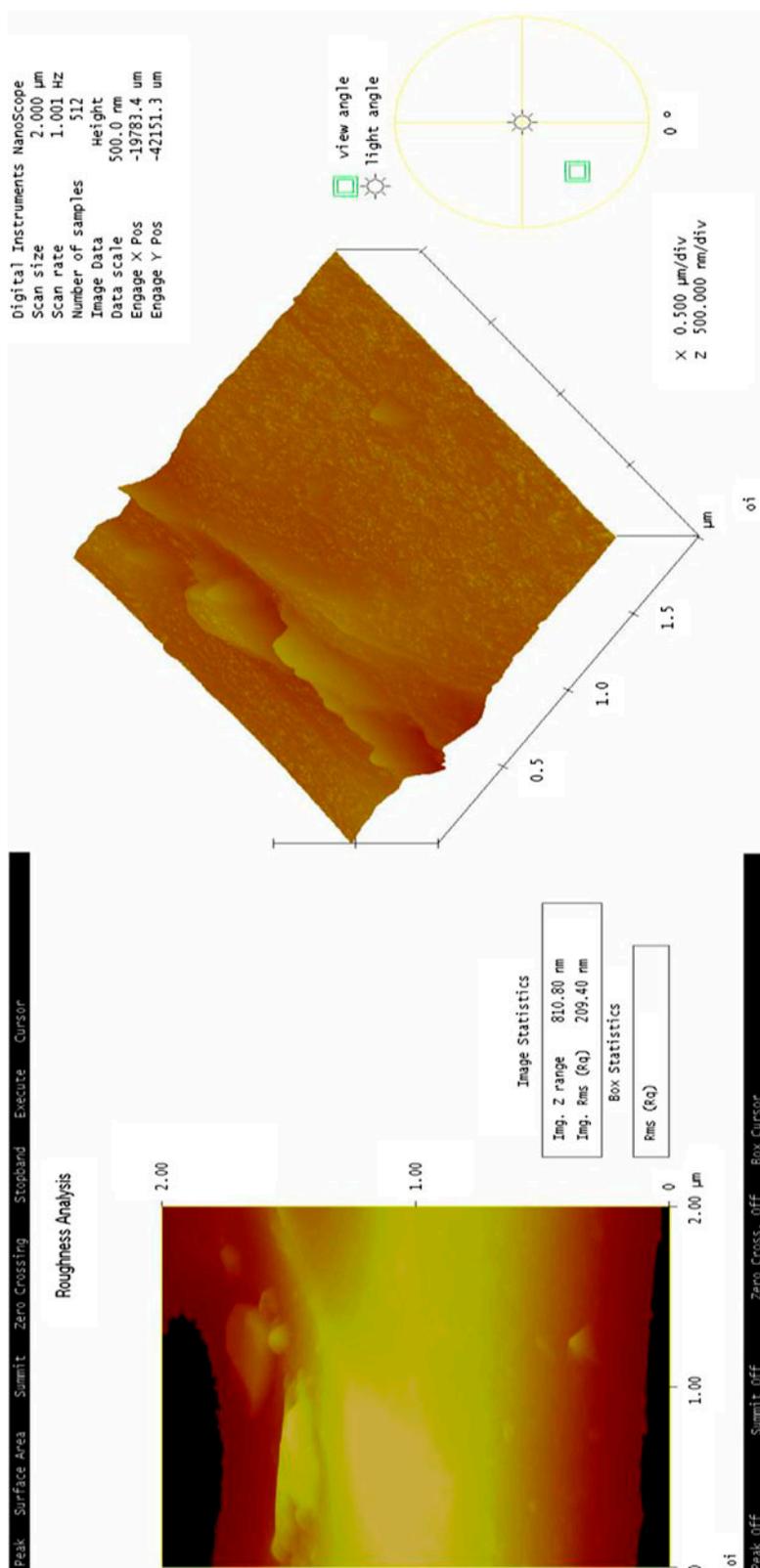


Fig. 5. AFM 3D and plane images of AG-2514 membrane.

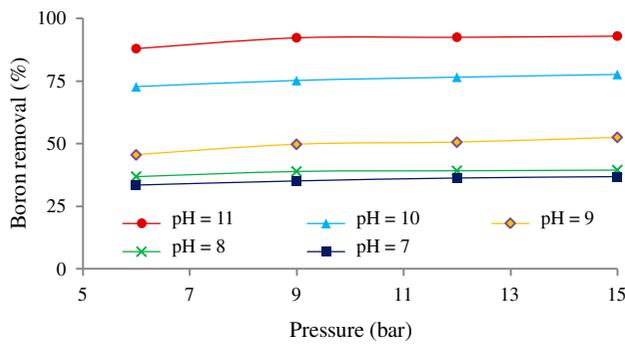


Fig. 6. Effect of pH on boron removal by the RO membrane AG-2514 at different pressures.

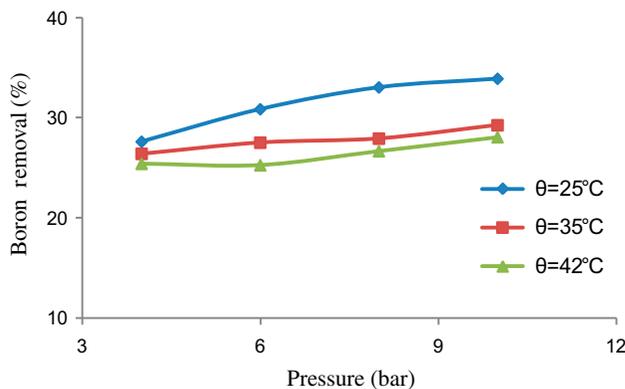


Fig. 7. Effect of temperature on boron removal by the RO membrane AG-2514 at different pressures.

4.2.3. Effect of the feed concentration on rejection

The effect of the initial concentration of boron in the feed solution was studied to determine the scope of the membranes AG. Various tests were performed maintaining the temperature (25°C) as a constant. The boron

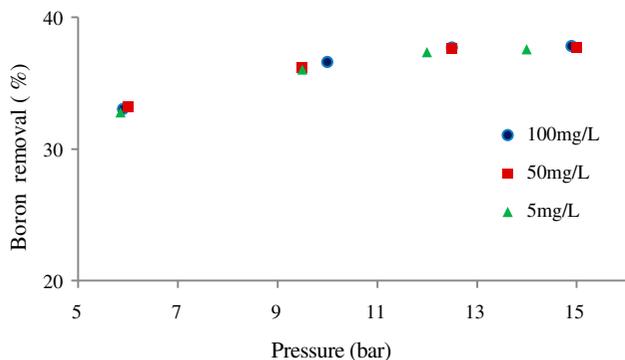


Fig. 8. Effect of feed concentration on boron removal by the RO membrane AG-2514 at different pressures.

concentrations studied were 5, 50, and 100 mg/L. The results are shown in Fig. 8.

These results indicate that the boron concentration of the feed solution does not have a significant effect on boron removal. In other words, boron rejection is not dependent on the concentration of the feed (in the concentration range 0–100 mg/L). This is explained by the fact that the increasing of feed concentration causes an increase in the boron amount in the permeate in such a way that the retention rate remains constant [22].

4.2.4. Boron removal at different ionic strength

The feed water is a 5 mg/L of boron solution at pH 6.8, $\theta = 25^\circ\text{C}$, $p = 15$ bar, and different ionic strengths (10^{-3} , 10^{-2} , 5×10^{-2} , and 10^{-1} mol/L). The ionic strength of the solution is fixed by adding NaCl at different concentrations. The results of boron removal at different ionic strengths are shown in Fig. 9. We can see that the elimination of boron decreases when the ionic strength increases. This result is explained by the fact that the increase of ionic strength of the solution decreases the dissociation constant of boric acid. Therefore, boric acid becomes less sensitive to the effect of repulsion exerted by the membrane and will therefore be less rejected [23].

4.2.5. Effect of ions on rejection

The natural water containing boron is characterized by the presence of several major ions such as calcium, magnesium, potassium, sodium, chlorides, fluorides, nitrates, and sulfates. These ions may have an influence on the rejection of boron. Sodium salts Na_2SO_4 , NaF, NaNO_3 , and NaCl with a concentration of 10^{-1} mol/L were used to study the influence of anions (SO_4^{2-} , F^- , NO_3^- ; and Cl^-) on the boron

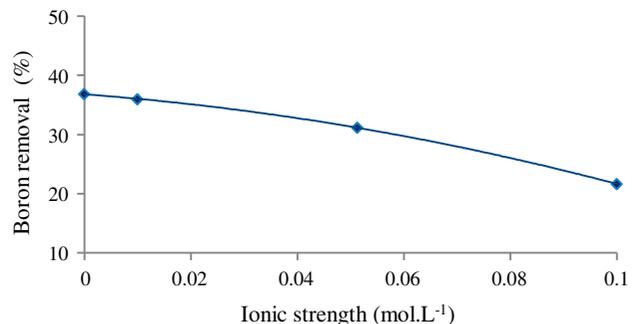


Fig. 9. Effect of ionic strength on boron removal by the RO membrane AG-2514.

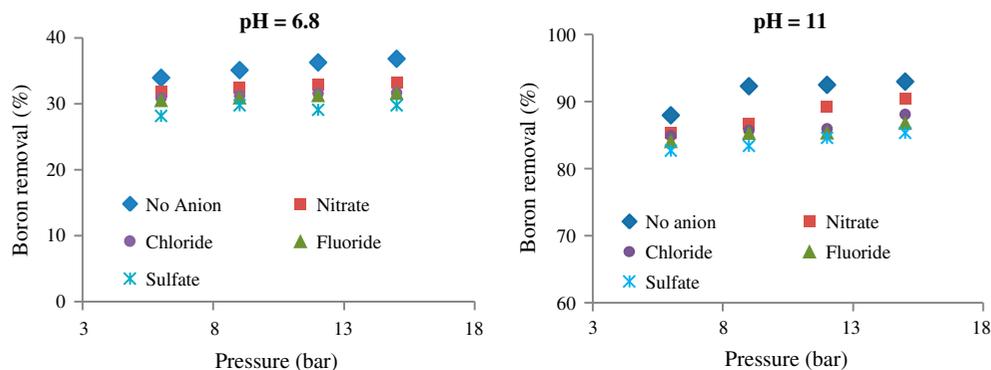


Fig. 10. Effect of anions on boron removal by the RO membrane AG-2514 at different pressures.

Table 1
Hydration energy of some anions [25]

Anions	NO ₃ ⁻	Cl ⁻	F ⁻	SO ₄ ²⁻
Hydration energy (kJ/mol)	310	325	449	1,047

rejection. Results obtained from the anion effect experiments with an amount of 5 mg/L boron, at two pH conditions (6.8 and 11), and $\theta = 25^\circ\text{C}$ are shown in Fig. 10.

At pH 6.8 and at a pressure of 15 bar, the rejection of boron changes from 36.85% in the absence of anions to 33.21, 31.7, 31.2, and 29.8% in the presence of NO₃⁻, Cl⁻, F⁻ and SO₄²⁻, respectively. At pH 11, the order of rejection in the presence of each of the ions remains the same. The rejection of boron changes from 93% in the absence of anions to 90.47, 88.13, 86.89, and 85.37% in the presence of NO₃⁻, Cl⁻, F⁻ and SO₄²⁻, respectively. Therefore, we have: $R_B(\text{SO}_4^{2-}) < R_B(\text{F}^-) < R_B(\text{Cl}^-) < R_B(\text{NO}_3^-)$. This order is closely related to ion charges and hydration energy (Table 1). Transport of boron became harder with increasing hydration energy of ions and ion charges. In the presence of the

bivalent anions (sulfate), the boron is less retained (compared to monovalent ions). This can be explained by the higher repulsion between the membrane surface and multivalent anions. When the hydration energy of anions were compared, it was seen that the order of hydration energy followed $\text{NO}_3^- < \text{Cl}^- < \text{F}^- < \text{SO}_4^{2-}$. In addition to this, Mnif et al. have shown that the transport of the SO₄²⁻ ion was lower than F⁻, Cl⁻, and NO₃⁻, because of its ionic charge while the transport of the F⁻ ion was more than the NO₃⁻ and Cl⁻ ions because of its hydration energy [24].

The influence of the cations K⁺, Na⁺, Mg²⁺, and Ca²⁺ on the rejection of boron is studied for solutions of these ions in the form of chloride salts (10⁻¹ mol/L). The effect of these cations on the boron rejection with an amount of 5 mg/L boron, at two pH conditions (6.8 and 11) and $\theta = 25^\circ\text{C}$ is represented in Fig. 11.

It was the same for cations, the rejection of boron decreases in the presence of cations in the solution. In the presence of divalent cations, it is seen that the rejection of boron is lower than that in the presence of monovalent cations. On the other hand, we note that in the presence of two cations of the same charge the

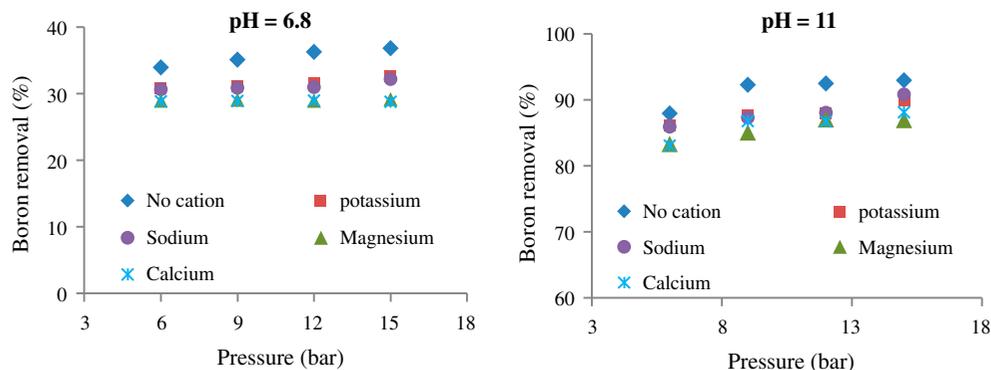


Fig. 11. Effect of cations on boron removal by the RO membrane AG-2514 at different pressures.

Table 2
Changes of water qualities through treatment by reverse osmosis unit

Parameters	Feed water	First treatment	Second treatment
Salinité (mg/L)	3,716	170	8.4
pH	7.62	7.57	7.6
Turbidity (NTU)	5.54	0.09	<0.01
Sodium (mg/L)	1,087	64.33	1.82
Potassium (mg/L)	33.2	1.8	0.06
Calcium (mg/L)	88	2.34	0.07
Magnesium (mg/L)	125	3.65	0.1
Chloride (mg/L)	1,406	83.36	2.98
Nitrate (mg/L)	33	1.87	0.07
Fluoride (mg/L)	0.51	<0.05	<0.05
Sulfate (mg/L)	367	24.87	0.77
Boron (mg/L)	3	2.18	0.244

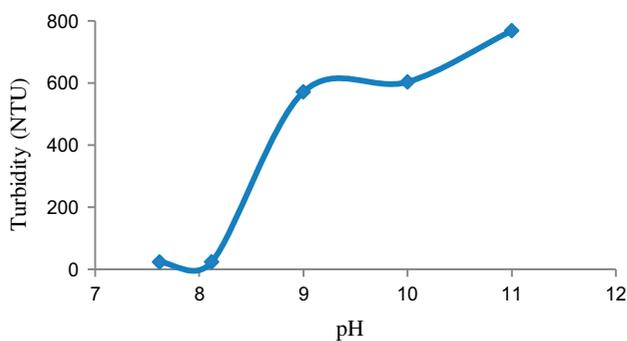


Fig. 12. The turbidity of the Oued Bouthebane water at different pH.

rejection rate is practically the same. Indeed, the presence of cations in the solution partially neutralizes the negative membrane surface charge and therefore, causes a decrease in the retention of boric species in the solution.

4.3. Natural water application

In this study, we propose to apply the reverse osmosis process for boron removal from brackish

water of the slick Oued Bouthebane in Tunisia. The variation in brackish water characteristics, including pH, conductivity, turbidity, salt composition, and boron concentration, is given in Table 2.

Boron is found in the natural waters in the form of boric acid. However, it is weakly dissociated, and its rejection depends on the pH of the feed solution. This concentration exceeds the limits set by the World Health Organization and the European Community Directive. The characteristic of the water after passage through the RO membrane is presented in Table 2. Our study proved that the boron concentration in the permeate is reduced to 2.18 mg/L, a retention rate of 27.33%, and the percentage of salts removed by the membrane exceeds 95%. In these circumstances, the percentage of boron removal is low, and the boron concentration in the permeate remains high compared with the standards. However, it is proposed that we increase the pH of the feed solution and monitor the percentage removal of boron. For the given composition of the matrix of water studied, an increase in the pH can result in the precipitation of some salts such as calcium hydroxide and magnesium hydroxide. To check the existence of this problem, we have followed the turbidity change of the treated water according to the pH. The results are shown in Fig. 12.

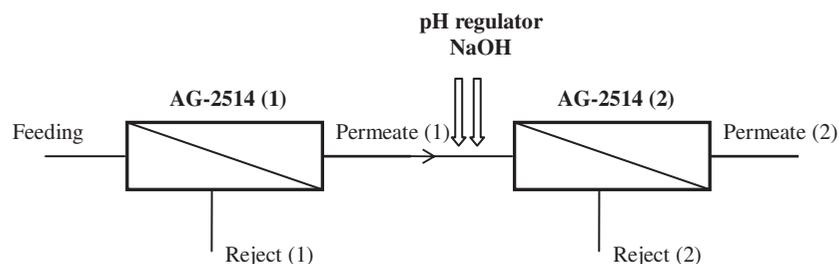


Fig. 13. The experimental design used in this application.

It is noteworthy that for pH values <8.2, the turbidity is very low. In this pH range, the addition of the sodium hydroxide (NaOH) does not spark the formation of the precipitates, and thus, this water can be treated by the reverse osmosis membrane. As from pH 8.2, it is noticeable that the turbidity of the water increases to 770 NTU at pH 11. This increase is explained by the formation of precipitates during the addition of NaOH. It therefore is followed by cake layer formation on the RO membrane surface.

To address this problem, we propose to pass the production of the reverse osmosis membrane through another RO module of the same reference [7]. The experimental setup used in this application is given in Fig. 13.

The experimental device consists of two reverse osmosis modules. The first can reduce the amount of salts in the water (Table 2). At the output of this module, we added in sufficient quantities of NaOH in order to obtain the optimum pH for the removal of boron by the reverse osmosis membrane. This solution is sent to the second reverse osmosis module to eliminate the remaining quantity of boron. The result indicates that the boron concentration in the permeate reached 0.244 mg/L and the total boron retention reached 91.86%.

5. Conclusion

The use of reverse osmosis for boron removal from natural water was studied. Characterization of reverse osmosis membrane AG-2514-TF showed that pure water permeability is equal to $3.8 \text{ L h}^{-1} \text{ m}^{-2} \text{ bar}^{-1}$, the membrane is negatively charged, and it has a fairly smooth surface with a roughness of about 209.4 nm.

The determination of the effect of operating parameters on the removal of boron was done as follows. Boron rejection depends largely on the pH. Indeed, at pH 7, the boron removal percentage does not exceed 35%. Moreover, at pH 11, this percentage attains 93%. The feed concentration and pressure have a little effect on the retention. However, boron removal depends on the temperature and on the ionic strength. The presence of other ions in the solution affected the decrease of the boron removal percentage, which is related to the nature and the charge of these ions.

Finally, elimination studies of boron from groundwater of the slick Oued Bouthebane in Tunisia was carried out. It was shown that an increase of pH results in a cake layer formation on the RO membrane surface. This problem can be resolved by realizing a coupling between two reverse osmosis modules of the same reference. The first reduces the amount of salts

present in water. The other module can eliminate boron after the addition of NaOH to reach the optimum pH. Thus, by this process the boron concentration is reduced to 0.244 mg/L, which is a removal percentage of 91.86%.

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