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Study of nitrate removal from aqueous solution by electrodialysis

Thouraya Turki¹ · Abdelkader Hamdouni²

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Abstract

Electrodialysis (ED) is one of the membrane processes that proved reliable and efficient in many applications, especially in desalination of drinking water. In this paper, the influence of initial nitrate concentration, the cell voltage, and the flow rate have been investigated. The effects of sulfate, chloride, and bicarbonate on the nitrate removal by electrodialysis have been systematically studied. An important reduction in concentration of nitrate by the use of the electrodialysis process has been obtained (from initial 400–800 mg/L to 4–8 mg/L), with a removal rate of 99%.

Keywords Electrodialysis · Ion-exchange membrane · Nitrate · Drinking water

Introduction

The high concentration of nitrates in drinking waters is mainly due to the intensive use of nitrogen fertilizers in agriculture and to the increase in domestic waste amounts (Liu et al. 2012). Excessive concentrations of nitrate in water can be hazardous to human health,

especially for infants and pregnant women. These excesses can make the asphyxiation in infants (methemoglobinemia) and risk of birth defects. Several conventional nitrate removal methods such as ion exchange, electrodialysis, membrane, and biological processes were used. Electrodialysis (ED) is one of the most commonly used techniques for the removal of specific ions such as nitrates even at high levels. However, they produce large amounts of brines which are extremely concentrated not only in nitrate but also in the various ions present in water (sulfate, hydrogen carbonate, chloride). In addition to that, they achieve only a transfer

of the pollution to the sewers (Liu et al. 2012; Sadrzadeha et al. 2009).

Various methods have been tested for the denitration of water among others: biological denitrification (Blecon et al. 1997; Hanus and Bernard 1988), exchange ion (Deguin, 1988), and membrane techniques (Choi et al. 2001). A remarkable efficiency has been showed for the reverse osmosis and electrodialysis in the removal of inorganic pollutant from potable and waste water (Clifford et al. 1986).

The recovery of sodium chloride from sea water, as well as the desalination of brackish water, was done by the use of an electro membrane process which is electrodialysis (ED). Under the gradient of an electric field, ion exchange membranes have allowed the elimination of certain pollutants from aqueous solutions (Hell et al. 1998). The objective of electrodialysis is to combine the phenomenon of ion transport under the impulse from an electric field to that of dialysis through a polar selective membrane. We use homopolar ion exchange membranes that do not allow only one type of ion: cations (cationic membranes) or anions (membranes anionic). The membranes are in the form of a sheet. If we have parallel to the electrodes (and orthogonally to the electric field) of such membranes inside an electrolysis stack, it is thus possible to extract the ionic species. Figure 1 illustrates the most common current configuration.

The apparatus consists of an alternation of anionic and cationic membranes delimiting dilution (D) and concentration (C) compartments. MX saline solution is fed (M^+ , X^-) the central compartment. Under the effect of an electric field, the cations migrate to the cathode: they leave compartments

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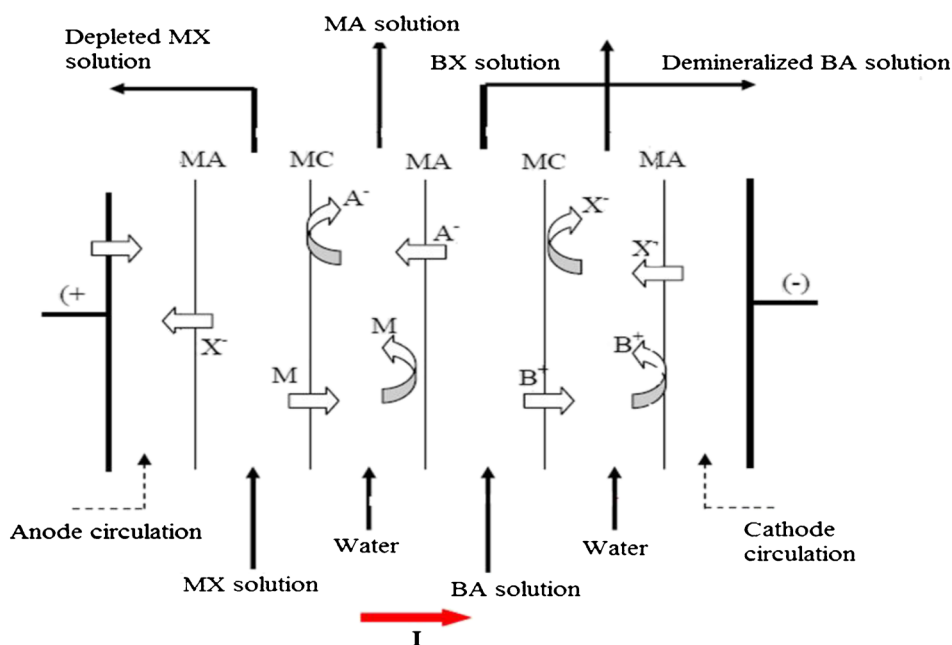
“This article is part of the Topical Collection on *Water Quality, Global Changes and Groundwater Responses*.”

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Fig. 1 Schematic diagram of two-compartment electrodi-
alysis



D by crossing a cationic membrane MC, but they cannot leave compartments C because they are blocked by the anionic membrane MA. Simultaneously, the anions migrate towards the anode: they leave the D compartments in crossing an anionic MA membrane, but they cannot leave the C compartments because they are blocked by a cationic membrane MC. As a result, compartments D become depleted in salts, called compartments diluted, and compartments C enrich with salts, they are called “compartments” brine or “concentrates” (Strathmann, 1992; Lonsdale and Pinho 1986).

Zeni et al. have reported the use of electrodi-alysis process for the elimination of fluoride from potable water (Zeni et al. 2005). The results of the research have shown the high efficiency of electrodi-alysis in the removal of fluorides and nitrates from brackish waters and waters with high NaCl concentrations in the order of 35 g/L (Banasiak et al. 2007; Tahaik et al. 2006). The impact of operative and water quality parameters on brackish water decontamination by electrodi-alysis process was reported by Aliaskari and Schäfer (2021). In fact, optimal parameters obtained for the removal of nitrate and fluoride has been 5 V (ddp) and 50L/h (Flow rate), respectively.

D. Yvana et al. (Ahdab et al. 2021) have studied the selectivity of the widely used Neosepta MSED and the Fujifilm MSED membranes to treat wastewater in greenhouses for reuse or discharge.

Removal of nitrate and fluoride ions from photovoltaic cells by electrodi-alysis process was been studied by Belkada F. D. et al. (Belkada et al. 2018). They obtain a nitrate removal rate of 98% using 0.1 A and 1000 ppm as current intensity and nitrate initial concentration, respectively.

Drinking water contains several other ions, particularly anions. These anions will tend just like nitrates to pass from one compartment to another by modifying the flow of nitrates. We were therefore interested in studying their influence on the rate of denitration by electrodi-alysis. The effects of accompanying ionic species (chloride, sulfate, and carbonate) and process parameters on nitrate removal by electrodi-alysis were studied.

Materials and methods

Work solutions

Reactive solutions of 400 to 800 mg-NO₃/L simulating a nitrate-rich aqueous medium are prepared from potassium nitrate crystals (KNO₃) diluted in deionized water and using various potentials (5, 10, and 20 V) and flow rates (30, 40, 60, and 70 L/h). The concentration of each ion was determined analytically. The solution ionic strength was fixed at 2 g/L by adding NaCl. All reagents are from analytical grade.

Electrodi-alysis

Electrodi-alysis cell description

The electrodi-alysis cell consists of three components: electrodes, ion exchange membranes, and separator frames. In this work, the cell used is “PCCell ED 64,004” (PCCell GmbH, Germany). The anode and cathode are made of Pt/Ir alloy coated with titanium and V4A steel. These electrodes

are located in propylene cavities. The latter are designed in such a way that a solution can circulate independently of the other compartments of the electrodialysis cell (Fig. 2). This solution is called electrode rinsing solution. The separator frame consists of a seal and a separator-distributor, allowing the flow of fluids through the various compartments of the cell through the sinks and collectors while preventing leakage of these fluids. The separators used in this work are based on silicone/polyester with a thickness of 0.5 mm, the membranes used, total number of 21, ten anionic membranes and eleven cationic (2 cationic membranes in contact with the electrode compartment). The active surface of each membrane is 64cm^2 . The general procedure for assembling a cell with electrodialysis is described as follows: the membranes are separated by separating frames, consisting of a watertight frame (attached) and a star, in the active area filled with electrolytes. This fabric prevents contact between two membranes. The specific stacking of the separator frames forms two distinct channels allowing the flow of the diluent and the concentrate without them being in contact with each other.

Mounting and experimental protocol (Fig. 3)

Cationic membranes must be kept in a solution of sodium chloride (NaCl) at a concentration of 0.1 mol/L whereas anionic membranes must be kept in a solution of hydrochloric acid (HCl) 0.1 mol/L, at a rate of about 100 mL per 10cm^2 of membrane.

The membrane stacking shall be cleaned and rinsed after each test to restore the initial conditions and to allow reproducibility of the tests. A first rinse consists of circulating distilled water in the three compartments for 15 min to remove chemical impurities that can be stuck to the surface of the membranes on the side of the concentrate and a second rinse with the working solution is necessary to avoid the variation of pH which is a determining parameter during the course of the test of part of the excessive dilution of the working solution by the dead volume in the electrodialysis, mainly in the circulation pipes. The pilot is formed by three circuits, each of which is filled with one litre of solution:

Fig. 2 Arrangement of the different constituents of the electrodialysis cell

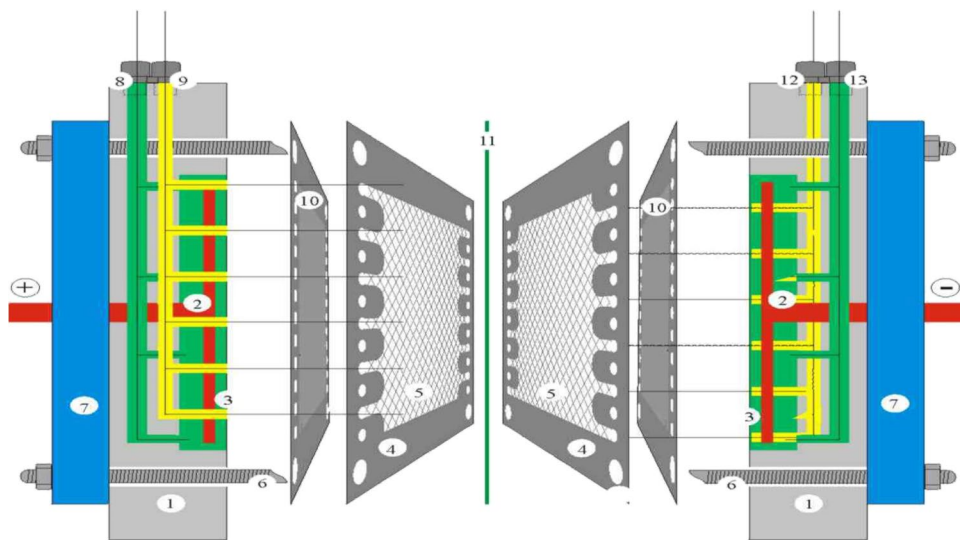
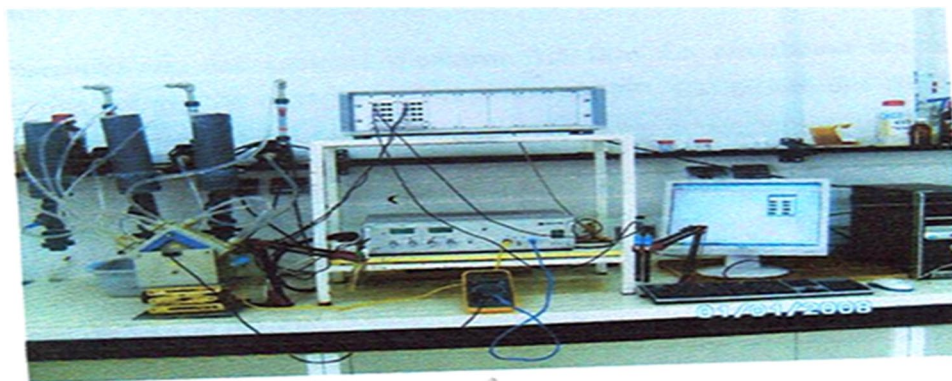


Fig. 3 Mounting of electrodialysis



-A circuit contains the solution to be treated. This solution is called a diluent whose ion concentration decreases with the time of electrodialysis.

-A concentration circuit containing the concentrate whose ion concentration decreases with time.

-A circuit for rinsing electrodes.

The electrodialysis cell is concentrated in centrifugal pumps equipped with a flow meter to ensure the flow of fluids between the tanks and the electrodialysis cell through Tygon pipes. These pipes are relatively transparent in order to allow us to follow the flow of fluids. The flow rates in these different compartments are adjusted by means of manual pliers.

The power supply is provided by a DC generator, Statron-3254.1, which provides a constant potential difference (ddp) between the two electrode terminals of the electrodialysis cell. The total current passing through the circuit is tracked using a "Digital MY64" type multimeter. The change in the conductivity and pH of the diluent and concentrate is followed throughout the treatment cycle with two conductometers and two pH meters connected to a consort D292/D291 acquisition chain. All experiments are performed in batch mode by recycling each of the feed fluids. The temperature is maintained at 25 °C.

Experimental procedure

We propose to treat these waters using an efficient and clean process: denitration by electrodialysis. At the laboratory level, we tried to optimize the physico-chemical parameters that can influence the process. For this, we prepared saline solutions ($[\text{NaCl}] = 2 \text{ g/L}$) with a nitrate content higher than the WHO allowed. We first studied the influence of the initial concentration of the nitrate ions (400, 600, 800 mg NO_3/L) and then the influence of the ddp applied between the electrodes (5, 10, and 20 V) and also the flow rates effect of the solutions (30, 40, 60, and 70 L/h). A study of the pH of the solution and the effect of the accompanying ions of nitrate ions was also carried out. For each electrodialysis operation, we followed the variation of the nitrate ion elimination rate over time while following the conductivity variation of the two compartments.

During all experiments, the volume of diluate, concentrate and rinsing electrode solution was 1 L each. Rinse solution was 0.1 M Na_2SO_4 in order to prevent generation of toxic gas.

Before the onset of the desalination test, the same solution of brackish water was introduced in diluate and concentrate compartments. Flow rate of electrode rinse solution was fixed at 100 L h^{-1} throughout the test and for all experiments.

Kinetic study of conductivity, flow rate, and pH has been explored in this study. Conductivity and pH value have

measured respectively, by conductivity meter (model LF320/SET, WTW, Arles, France) and pH meter (model pH 320/SET, WTW, Arles, France). Electrodes of pH and conductivity meter have been immersed in the concentrate and the diluate compartment.

The solution's pH, conductivity, and flow rate were recorded in time. pH measured with a samples from concentrate and diluate tanks were collected during test progress and analyzed according to Rodier protocol (Rodier, 1996) for the determination of nitrate.

After every experiment, ED cell was cleaned with circulation of 0.1 M HCl solution during 30 min followed by three successive rinsing of 15 min each with ultra-pure water.

Results and discussions

Influence of initial nitrate concentration

Solutions of NaCl with a concentration of 2 g/L containing different concentrations of nitrate ions ranging from 400 to 800 mg/L were used to study the effect of the initial concentration of the solution to be treated on the denitration rate. Experiments are performed with the system of the PC-SA/PC-SK membranes and a voltage of 10 V. The results obtained are illustrated in Fig. 4.

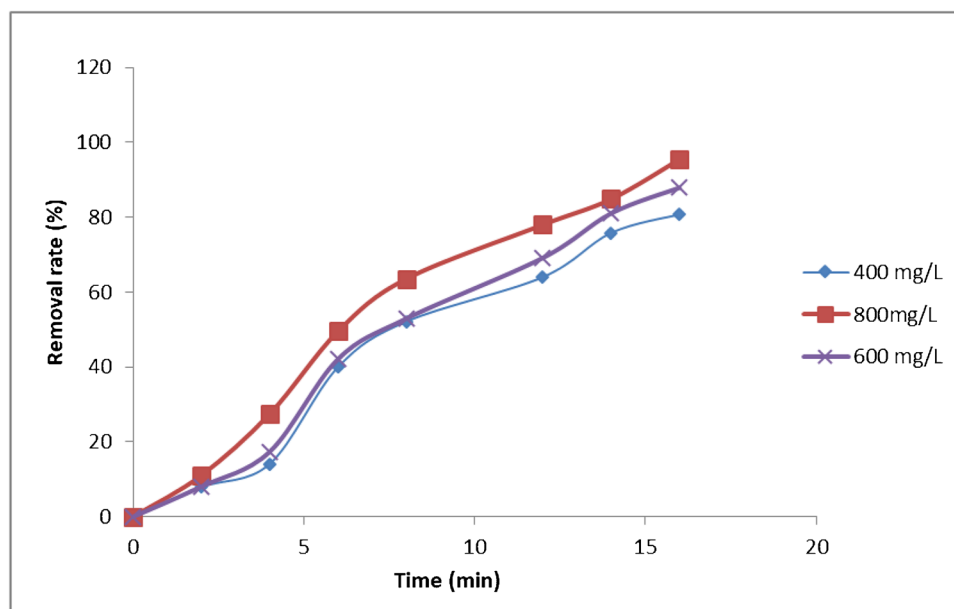
As shown in Fig. 4, the rate of removal of nitrate ions depends on their initial concentration in the solutions to be treated. Indeed, we note that by increasing the initial concentration of nitrate ions, the denitration rate increases. These results can be explained by the increase of the potential difference at the interface of the membrane due to the increase of the concentration of ions in solution, and therefore, the ionic fluxes increase to reach equilibrium and ensure equal potential. The total process duration does not increase with the increase in the initial concentration of nitrate ions in the solution. The same result was found in previous studies (Trathmann 1985).

Voltage influence

The driving force for ion transport in electrodialysis is the difference of the applied voltage across the electrodes, so the change in the value of this applied voltage could influence the different parameters involved in the electrodialysis process. To study the effect of the applied voltage between the electrodes on the removal of nitrate, the concentration of the solution to be treated and its volumetric flow rate were fixed respectively to 2 g/L NaCl, 800 mg/L NO_3^- , and 70 L/h.

On the other hand, the applied voltage across the electrodes E was varied from 5 to 20 V.

Fig. 4 The effect of nitrate initial concentration on the removal rate



The membranes used are PCCell brand PC-SA model for anionic membranes (ten in number) and PC-SK for cationic membranes (nine in number).

Current variation

Figure 3 shows the rate of change in the current intensity measured in the system as a function of time for the different values of the potential applied between the electrodes. It was seen that the charged current in the system depends on the applied voltage. By further increasing the value of the applied voltage, a significant increase in the current is noticed. We reached 1.8 A for a voltage of 20 V and 0.2 A by applying a voltage of 5 V.

As shown in Fig. 5, the time required for the operation to reach 0.1 A (limit current) is all the more important since the value of ddp applied is low (5 V). Indeed, the duration of

electrodialysis processes is equal to 27, 19, 24 min respectively for a ddp equal to 5, 10, and 20 V. In addition, it is noted that the current flow in the system depends on the applied potential. By further increasing the value of the applied d.d.p, a significant increase in current is noticed. We reached 1.8 A for a voltage of 20 V; then, by applying only a ddp of 5 V, we reached a current value equal to 0.2 A.

Conductivity variation

Variation in conductivities of the dilute and concentrate compartment was followed as a function of time and the applied voltage. The curves are illustrated in Fig. 4.

When analyzing Fig. 6, we find that the applied voltage has a direct influence on the evolution of the conductivity in both compartments. Indeed, an increase in this applied voltage causes an increase in conductivity of the concentrate and conversely a decrease of this parameter in the dilute compartment. This can be explained by the increase or reduction of salts in each compartment upon transfer of these entities between these two compartments.

There will be an ion migration of dilute to the concentrate compartment.

Nitrate removal rate.

The variation in the denitration rate versus time for various applied voltage is shown in Fig. 7.

Figure 7 illustrates the significant influence of applied voltage on the removal rate of nitrate. In fact, an increase in this potential leads to an increase in the percentage of ions transferred to the concentration compartment and consequently an improvement in the efficiency of the process. Indeed, by increasing from a potential of 5 to 20 V, the yield increases by 20.25% and the duration of treatment decreases

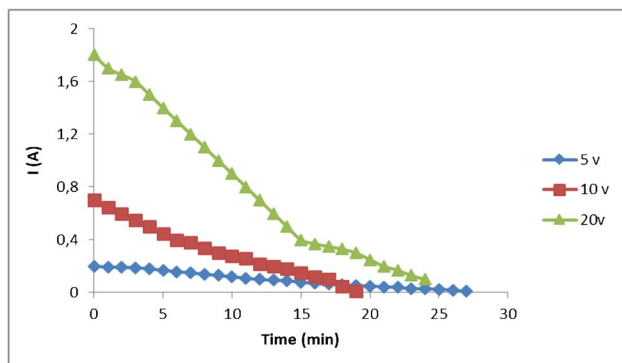


Fig. 5 Variation of the current as a function of the time and the applied potential

Fig. 6 Conductivity vs. time plots as a function of applied voltage

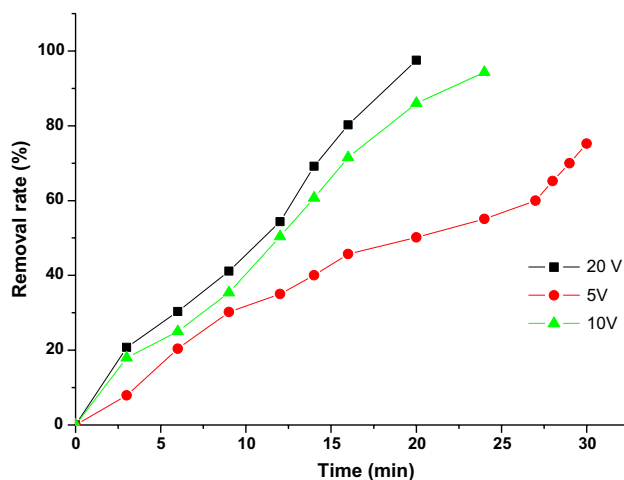
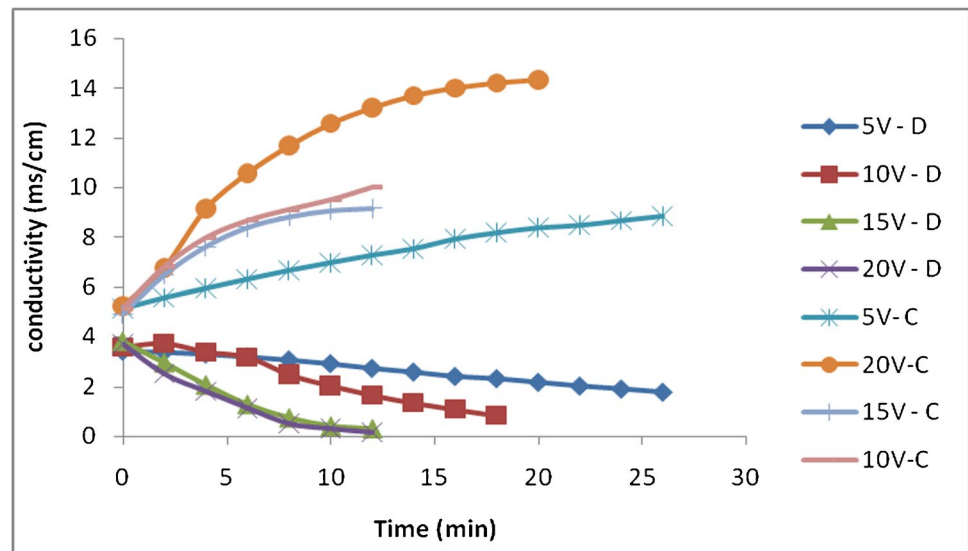


Fig. 7 Nitrate removal rate as a function of applied voltage

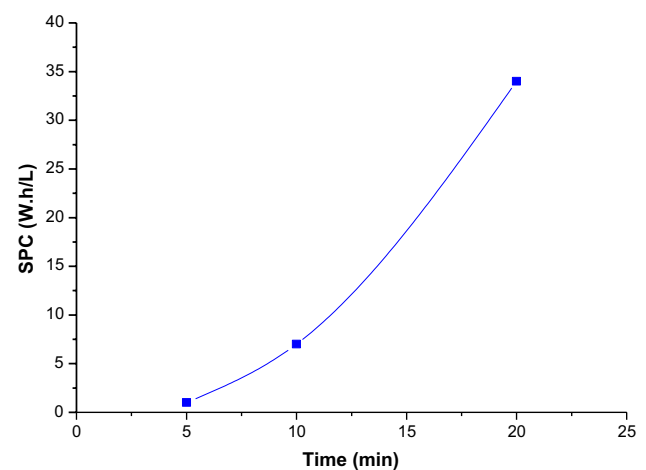


Fig. 8 Effect of cell voltage on energy consumption

by 14 min. This result is closer to this reported by Tishchenko (1991).

Specific power consumption (SPC) can be described as the energy needed to treat unit volume of solution. SPC was calculated using the following equation:

$$SPC(WhL^{-1}) = E \frac{\int_0^T I(t) dt}{V} \quad (1)$$

where E is the applied voltage, I the current, V the dilute stream volume, and t is the time.

Figure 8 shows that energy consumption is closely dependent on the difference in potential applied. This dependency is expected since the mathematical expression of this parameter is defined according to the ddp applied to the stacking terminals.

This increase is mainly due to the rapid increase of the current following the application of a significant ddp to the terminals of the electrodialysis cell. The difference in potential applied to the electrodialysis cell terminals has a direct influence on the efficiency of the denitratation process. In the course of this study, we found that the increase in potential leads to an increase in the current, the conductivity of the solutions, and the elimination rate of the nitrates. However, the use of high voltages results in higher energy consumption.

Effect of flow rate

We used a solution of NaCl with a concentration of 2 g/L containing 800 mg/L nitrates. In order to study the flow effect of the solution to be treated on the denitratation

rate, we used the PC-SA/PC-SK membrane stacking and imposed a 10 V ddp. The flow rate of this solution was adjusted before the start of each experiment (30, 40, 60, 70/h). The results are shown in Fig. 9.

According to this figure, it can be seen that the flow has a fairly significant effect on nitrate removal in the range studied. An increase in the flow rate conduct to an increase in the removal rate of nitrate. Indeed, a flow rate of 70L/h allowed us to achieve an elimination rate of 97.5% corresponding to a nitrate concentration of 20 mg/L whereas with a flow rate of 30L/h, the final nitrate concentration is 139.2 mg/L. This result is closer to others reported in previous researches (Bi et al. 2011; Savas and Ogutver 2002). This can be explained by the fact that the treated solution could stay longer in the electrodialysis device and the ions could be transferred freely into the membrane.

Influence of co-existing anions on nitrate removal rate

In previous experiments, we have worked with synthetic solutions containing only nitrates and chloride ions. However, drinking water contains several other ions, especially anions. These anions will tend just like nitrates to pass from one compartment to another by modifying the flow of nitrates. We therefore looked at their influence on denitrata-tion rates.

Here, elimination of nitrate by electrodialysis was studied in the presence of carbonate and sulfate ions. The samples were done with nitrate (800 mg NO_3^-/L) accompanied only with either sulfate or carbonate ions. Effects of these ionic species on the removal of nitrate were shown in Fig. 10.

The results presented in the figure above show that denitrata-tion during the treatment of a mixture is less important than that in the case of a simple solution. Indeed, with an aqueous solution of NO_3^- , we were able to reduce the

Fig. 9 Effect of flow rate on nitrate removal

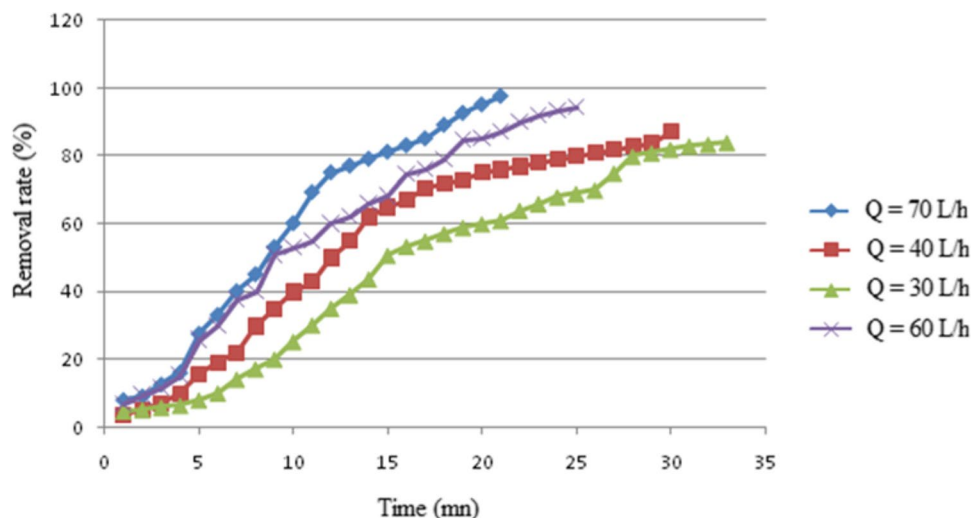
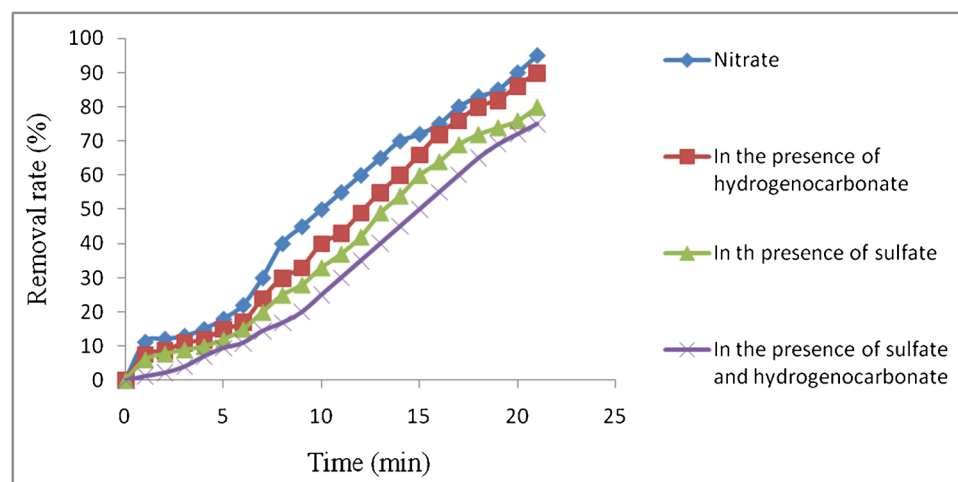


Fig. 10 Effect of hydrogenocarbonate and sulfate on nitrate removal rate



concentration of these ions to 10.4 mg/L which corresponds to a rate of elimination of 98.7%. On the other hand, with solutions containing other ions such as HCO_3^- and SO_4^{2-} , the denitration rate decreases and the movement of nitrate ions is prevented by increasing the number of coexisting ions (chloride, bicarbonate, and sulfate). This is due to the competition of anions to occupy fixed membrane sites.

In fact, the mobility of the mono- and bivalent ions in an ion exchange membrane was investigated by Miyoshi (Miyoshi, 1998), who reported that a monovalent ion makes an ion pair with a fixed ion in the membrane and it is transferred following several steps between neighboring fixed ions. In case of bivalent ions, two fixed ions were used by bivalent ions for transporting. Namely monovalent ions needs only one fixed charged ion that is corresponding of a half-set of a bivalent ion. The results achieved reflect the explanation.

Effect of pH on removal of nitrate

The effect of pH on the removal rate of nitrate is shown in Fig. 11. However, a pH value of 7 was seen to be the best. This is probably due to the absence of foreign ions at a pH of 7 (at other pH values, other ions are added to the working solution to adjust the pH) which would otherwise interfere with the transport of nitrate across the membranes.

Since most of water sources containing excess nitrates have pH values of 7, there is no need to adjust the pH and hence to add any chemicals before and after treatment.

Nitrate removal from Soliman natural water

For this study, a natural water of the region of Soliman (situated in Nabul, Region 2, Tunisia) which contains a high

nitrate concentration (818 mg/L) was selected for testing. Electrodialysis of this natural water from Soliman was undertaken, of which the results for nitrate removal are outlined in Fig. 12.

The nitrate removal efficiency obtained with the drinking water of Soliman is off 96%.

This study showed that desired drinking water can be obtained by the electrodialysis of this kind of water with nitrate concentration above Drinking Water Guidelines levels.

Conclusion

In the present study, batch ED was studied for the removal of highly polluting and toxic NO_3^- ions from aqueous solutions using PC-SA and PC-SK ion exchange membranes. The effect of operating parameters on the performance of the ED process was investigated. The optimum operating conditions for the ED desalination were thus selected by considering the following criteria: (i) achievement of high removal of NO_3^- ions in order to obtain non-toxic natural waters or even suitable to be discharged into the environment, and (ii) the efficiency of the ED process, which was assessed by evaluating the process parameters like CE and EC.

In this work, it was found that the removal of nitrate from water can be carried out effectively by applying electrodialysis. This method has several advantages such as not using chemicals and not giving rise to any sludge problem. Moreover, it eliminates the need of adjusting the pH. In addition to all these advantages, nitrate can be concentrated in the anode compartment and fed to any related process. Thus, the ED process investigated here showed very good performances in efficient removal and concentration of strongly polluting

Fig. 11 Denitration rate as a function of time and pH of the solution (flow rate = 70 L/h and ddp = 10 V)

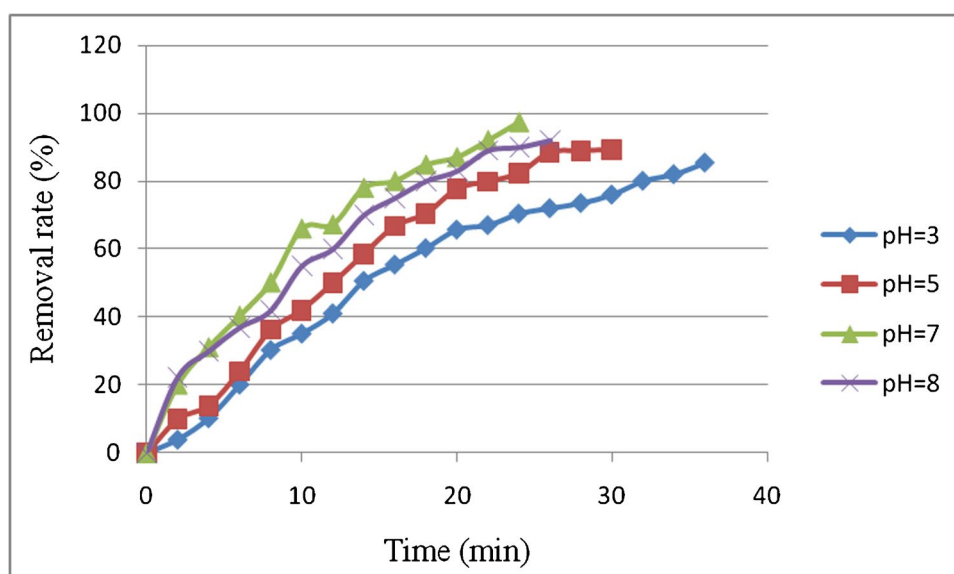
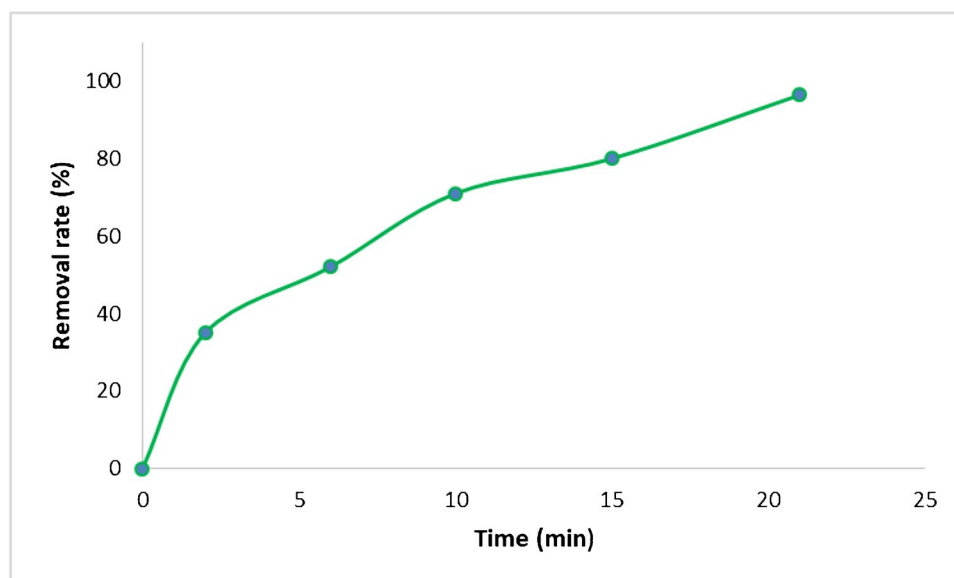


Fig. 12 Nitrate removal from Soliman drinking water (ddp=10 V, flow rate=70L/h)



nitrate ions under mild and economically advantageous optimal conditions, namely: applied voltage of 10 V, flow rate and process temperature of 70 L/h and 25 °C, and initial nitrate concentration of 800 mg/L. The nitrate concentration can reach the final concentration of about 8 mg/L, which is below the OMS level (50 mg/L).

Declarations

Conflict of interest The authors declare no competing interests.

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