# ЭЛЕКТРИЧЕСКАЯ ОБРАБОТКА БИОЛОГИЧЕСКИХ ОБЪЕКТОВ И ПИЩЕВЫХ ПРОДУКТОВ

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# DENITRIFICATION OF BRACKISH WATER BY ELECTRODIALYSIS: EFFECT OF PROCESS PARAMETERS AND WATER CHARACTERISTICS

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## 1. Introduction

Tunisia is characterized by semiarid climate and by limited conventional water resources especially groundwater resources. These underground waters represents an important resource exploited for human consumption, for agricultural and for industrial use [1-2]. Owing to these activities, the nitrate concentration in surface and groundwater have increased to such an extent that the admitted standards in drinking water have been largely exceeded in many regions [3]. In fact Nitrate pollution is caused by the intensive use of nitrogen fertilizers, crop irrigation with domestic wastewater and use of manure. Therefore, it is concern of diffuse pollution. A maximum limit of 50 mg $L^{-1}$ $NO_3^-$ in drinking water is permitted [4]. Nitrate contaminated water above these limit is considered as toxic to human health, specifically to children [4–6].

A survey of literature yielded an abundance of information on the technical treatment to remove nitrate from water. Different approaches have been proposed and proven. The typical process can be grouped into chemical, physical and biological denitrification processes [5–17]. Biological and chemical denitrification needs a continuous monitoring, such as addition of a carbon source, pH control, temperature maintenance, and also requires the removal of by-products such as nitrite [5, 7, 8]. Extractive methods like reverse osmosis and ion exchange resins are also largely used to remove nitrates. However, these techniques produce a large amount of effluents, which must be treated later and therefore increase the overall cost of the process [7, 8, 11]. The interest in using of electrodialysis processes to remove excess inorganic contaminants such as boron ($B(OH)_4^-$), fluoride ($F^-$) and nitrate ($NO_3^-$) from drinking water has increased worldwide principally because it is a simple process and does not have many of the defects of chemical processes [18–24]. Some studies were conducted and demonstrated that electrodialysis is a reasonable process for removing these inorganic contaminants from brackish water [14–24].

The electrodialysis is a widely used electro-membrane process especially for desalination of brackish water and sodium chloride recovery from sea water. The removal of ionic components from aqueous solution through ion exchange membranes is carried out under the driving force of an electrical field [25–27].The mandatory condition for an electrodialysis process to be executed is an alternating order of cation and anion membranes and electric field applied across the entire assembly (Fig. 1).

When a direct current is applied between two electrodes, the charged cations will move to the cathode passing through the cation exchange membrane but they will be retained by the anion exchange membrane. Conversely, charged anions will move to the anode through the anion exchange membrane and they will be retained by the cation exchange membrane. While the negatively charged anions move to the anode, passing through the anion exchange membrane and retained by the cation exchange membrane. The two types of membranes create alternately two compartments. These compartments will accumulate the ions because the ion exchange membrane between them would prevent ions from moving further. Therefore the solution in the even compartments (1) will be demineralised and solution in the odd compartments (2) will be concentrated. As a result the entire flow incoming to the electrodialysis apparatus can be separated into desalinated and concentrated streams. These flows are called respectively dilute and concentrate.

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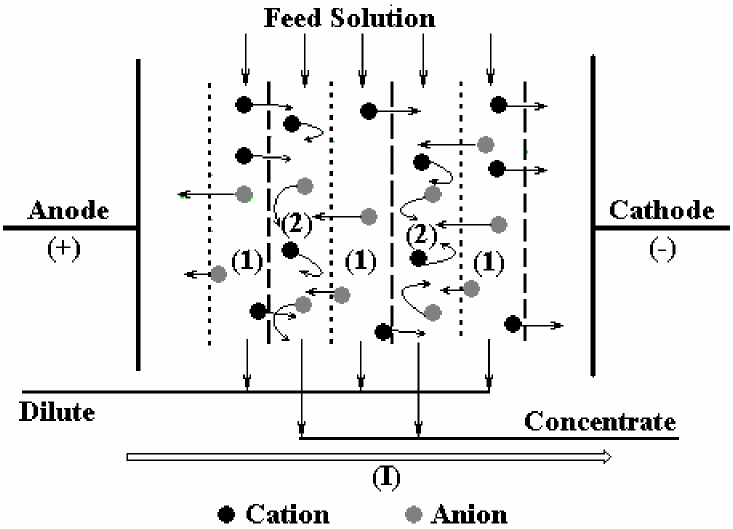


*Fig. 1. Principle of electrodialysis*

The aim of this work is to study the removal of nitrate from polluted water using electrodialysis. The influence of several parameters, such as flow rates, initial feed concentration, co-existing anions and initial pH on process efficiency were studied. This efficiency is expressed by the removal rate, demineralization rate and power consumption. At the end, the denitrification of a contaminated groundwater real sample also was investigated so as to improve the efficiency of denitrification process.

## 2. Materials and methods

### 2.1. Electrodialysis equipment and membranes

The ED setup consists of: a power DC, a brine tank, a feed tank, an electrode rinse tank, three centrifugal pumps ($P$ = 84W, total head = 4.2 m) equipped each with a flowmeter and three valves to control feed flow rate in the compartments of ED cell. Fig. 2 shows a simplified diagram of the ED setup working in batch recirculation mode.

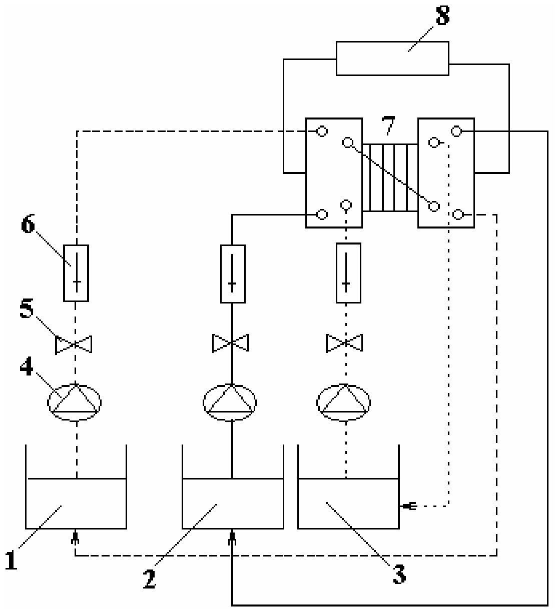


*Fig. 2. Schematic of the ED system used in this study*

The electrodialysis operation was carried out on a laboratory stack “PCCell ED 64 002” supplied by PCA-Polymerchemie Altmeier GmbH and PCCell GmbH, Heusweiler, Germany. ED cell is packed with ion exchange membranes (cation and anion), spacers and a pair of electrodes (anode and cathode). Both electrodes are made of Pt/Ir- coated Titanium. The membranes and spacers are stacked between the two electrode-end blocks. Plastic separators are placed between the membranes to form the flow paths of the dilute and concentrate streams. These spacers are designed to minimize boundary layer effects and are arranged in the stack so that all the dilute and concentrate streams are manifolded separately. In this way a repeating section called a cell pair is formed; it consists of a cation exchange membrane, a dilute flow spacer, an anion-exchange membrane, and a concentrate flow spacer. In this work, experiments were carried out by this stack equipped with three cation exchange membranes (CEM) and two anion exchange membranes (AEM). For each membrane, the active surface area is 0.0064 $m^2$ and the flow channel width between two membranes is 0.5 mm. PCA-SK standard cation exchange membranes and PCA-SA standard anion exchange membranes are used in the stack. Furnished informations by the manufacturer about the membranes are given in Table 1. The ED system is designed to handle 2–5 L laboratory batches.

*Table 1. Information on PCA standard cation and anion exchange membranes*

| Membrane | Thickness (µm) | Ion exchange capacity (meq $g^{-1}$) | Chemical stability (pH) | Permselectivity | Functional groups | Surface potential (Ω $cm^2$) |
|---|---|---|---|---|---|---|
| PC-SK | 130 | ~1 | 0-11 | > 0.93 | $-SO_3^-$ | 0.75-3 |
| PC-SA | 90-130 | ~1.5 | 0-9 | > 0.96 | $-NR_4^-$ | 1-1.5 |

### 2.2. Reagents

Analytical grade sodium chloride, sodium nitrate, magnesium chloride, calcium chloride and sodium sulphate salts are used in all experiments to produce solutions with known amount of salts and electrode rinse solution. Distilled water was used throughout.

### 2.3. Real water sample

Brackish water sample with total dissolved salts TDS around 15 g $L^{-1}$ was taken from underground water in the region of Borj-Cédria (Tunis City). Since an electrode overheating can occur in the ED system for the treatment of solution with relatively high salinity (TDS over 5 g $L^{-1}$), this sample was diluted by mixing it with raw water to reduce the total salinity until 5 g $L^{-1}$. And because the measured concentration of nitrate, 25 mg $L^{-1}$, was below the maximum recommended concentration by WHO, small amount of sodium nitrate salt was added to this sample to simulate a contaminated one. The physico-chemistry characteristics of obtained sample are given in Table 2.

*Table 2. Composition, pH and conductivity of brackish water Sample*

| Physico-chemistry parameter | |
|---|---|
| Conductivity at 25 °C (µS $cm^{-1}$) | 5 008 |
| pH | 7.2 |
| $F^-$ (mg $L^{-1}$) | 2.9 |
| $Cl^-$ (mg $L^{-1}$) | 2 674 |
| $HCO_3^-$ (mg $L^{-1}$) | 202 |
| $NO_3^-$ (mg $L^{-1}$) | 225 |
| $SO_4^{2-}$ (mg $L^{-1}$) | 707 |
| $Na^+$ (mg $L^{-1}$) | 1041 |
| $K^+$ (mg $L^{-1}$) | 300 |
| $Ca^{2+}$ (mg $L^{-1}$) | 235 |
| $Mg^{2+}$ (mg $L^{-1}$) | 127.6 |
| TDS (mg $L^{-1}$) | 5 424 |

### 2.4. Experiment and analysis methods

#### 2.4.1. Experimental procedure

During the experiments, the volume of dilute, concentrate and electrode rinse solution was 2 L. As initial concentrate and dilute, the same solution was used. Prior to the experiments, pH was adjusted by the addition of 1 M HCl and/or NaOH. 0.1 M $Na_2SO_4$ was used as electrode rinse solution circulating in electrode compartments, in order to prevent the generation of chlorine or hypochlorite, which could be hazardous for the electrodes. Flow rate of electrode rinse solution was fixed to 80 L $h^{-1}$ for all experiments. Although the flow rate of other solutions (dilute and concentrate) were fixed at the beginning of experiment. Dilute and concentrate were recirculated through the ED cell at a these flow rates until the desired product concentration or total dissolved salts (~ 0.5 g $L^{-1}$) was achieved in the dilute.

The applied current density across the ED cell was fixed at a value below the limiting current ($I_{\mathrm{lim}}$) which is determined by primary tests. Total voltage drop, including voltage drop in the membrane stack as well as on the electrodes, was measured in the experiments.

Samples from dilute and concentrate were collected at the beginning and at 5 min intervals for analysis.

After the completion of each experiment, cleaning solutions of 0.1 M HCl, 0.1 M NaOH and ultrapure water were circulated through the ED cell for 30 min each in order to remove any deposits.

#### 2.4.2. Analytical methods

The electrical conductivity (EC) of samples taken from the dilute and concentrate during each experiment was measured using a 712 Conductometer (Metrohm AG, Switzerland). $Ca^{2+}$ and $Mg^{2+}$ amounts were determined by atomic absorption spectroscopy using an analytical AAS Vario 6 spectrometer. $Na^+$ and $K^+$ were analyzed by atomic emission spectroscopy using a Jenway PFP 7 spectrometer. Sulphate and chloride anions were determined by ion chromatography using a Metrohm 761 Compact IC with conductivity detection (Metrohm AG, Switzerland). The anion chromatography measurements with chemical suppression were made with a Metrosep anion dual 2 column (4.6×75 mm) with a particle diameter of 6 µm.

654 pH-metre (Metrohm AG, Switzerland) equipped with a glass electrode was used for measuring pH solutions

$NO_3^-$ concentrations were determined using ion-selective electrodes (ISE) in conjunction with a standard reference electrode connected to a Metrohm 781 Ion Meter (Metrohm AG, Switzerland). The ISEs were regularly calibrated and cleaned using standard methods. Standards and samples were mixed with a total ionic strength adjustment buffer (TISAB) to avoid possible interferences resulting from changes in solution pH and conductivity.

#### 2.4.3. Data analysis

The purpose of the experiments was to study the effects of several parameters on ED cell performance. All figures and tables refer to concentration changes in the dilute. The mass balance of the ions present in the feed solution was verified for dilute, concentrate and electrode rinse solution. Thus, data for concentrate and electrode rinse solution will not be presented in this article.

##### 2.4.3.1. Determination of the demineralization rate

The conductivity allowed following the demineralization rate [28, 29]. The demineralization rate can be calculated as:

$$DR(\%) = 100.\left(1 - \frac{EC_t}{EC_0}\right)$$

Where *DR* is the demineralization rate expressed in percentage, $EC_0$ and $EC_t$ are, respectively, the initial and conductivity of the dilute, expressed in µS $cm^{-1}$.

##### 2.4.3.2. Determination of the flux and removal of nitrate

The flux values were evaluated for all experimental conditions in order to compare to transport of nitrate ions from feed to receiver phase. The flux of nitrate ion (J) was determined by using the following equation [20]:

$$J\,(mol\,cm^{-2}\,s^{-1}) = \left(\frac{V}{A}\right)\left(\frac{\Delta C}{T}\right)$$

Where V is the volume of receiver phase (L), A is the effective membrane area ($cm^2$), $\Delta C$ is the transported amount of nitrate at a time (mol $L^{-1}$) and T is the period time (s).

The removal of nitrate was defined as [20]:

$$R(\%) = 100\left(1 - \frac{C_t}{C_0}\right)$$

Where $C_t$ is nitrate concentration (mol $L^{-1}$) in dilute compartment, and $C_0$ is the initial concentration of nitrate in the feed phase (mol $L^{-1}$). To compare the removal of nitrate ions, the percent removal was evaluated for all experiments.

### 2.4.3.3. Determination of the limiting current ($I_{\text{lim}}$)

In electrodialysis, concentration polarization takes place at the membrane surface. The magnitude of the concentration polarization is a function of various parameters including the applied current density, the feed flow velocity parallel to the membrane surface, the cell design, and the membrane properties [30–34]. The transport of charged species to the anode or cathode through a set of ion exchange membranes leads to a concentration decrease of counter-ions in the laminar boundary layer at the membrane surface facing the dilute cell and an increase at the surface facing the concentrate cell. When the ion concentration at the surface of the cation and/or anion exchange membranes in the cells with the depleted solution, the dilute, will become zero, the current density will approach to the maximum value in the process, which is defined as the limiting current density (LCD) [34].

The limiting current density in the electrodialysis process is an important parameter which determines the electrical resistance and the current utilization. It is generally accepted that an electrodialysis process shows higher electrical resistance or lower current utilization when operated at above LCD. It also can give rise to problems such as water dissociation or salt precipitation. It is necessary, therefore, to determine the LCD to prevent problems and operate the electrodialyzer successfully [32].

The limiting current can be determined experimentally by plotting the electrical resistance across the membrane stack ($E\ I^{-1}$) or the pH value in the dilute cell as a function of the reciprocal electric current ($I^{-1}$). This is called a Cowan–Brown plot after its original developers [33–34].

Usually, the limiting current depends on membrane and solution properties as well as on the electrodialysis stack construction and various operational parameters such as the flow velocity (flow rate) of the dilute solution. The set of primary tests was done to determine the limiting current density for the operating conditions to be adopted in this study.

### 2.4.3.4. Specific power consumptions

Specific power consumption (SPC) can be described as the energy needed to treat unit volume of solution. SPC was calculated using the following equation [23]:

$$SPC = \frac{E\int_0^t I(t)\,dt}{V}$$

Where $E$ is the applied potential, I the current, $V$ the dilute stream volume and t is the time.

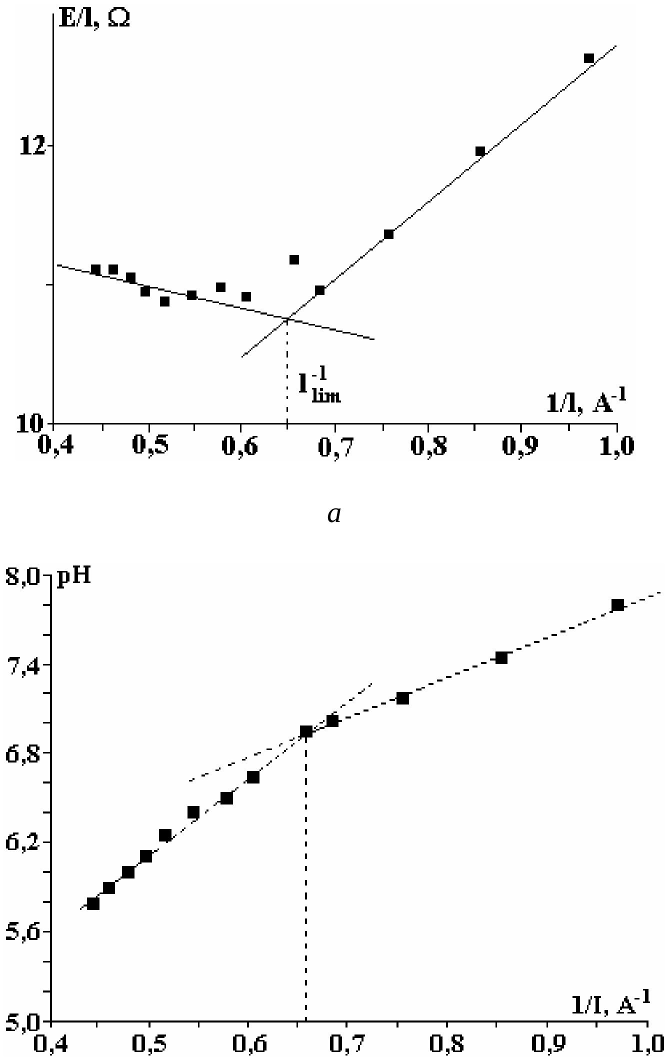
The SPC values were calculated for each experimental condition and 200 mg $NO_3^-$ $L^{-1}$ removed.

## 3. Results and discussions

### 3.1. Determination of the limiting current ($I_{\text{lim}}$)

In this study the limiting current ($I_{\text{lim}}$) was determined by measuring the potential and the cell resistance as a function of the applied current. Figs. 3(*a*) and (*b*) shows the curves illustrating the determination of the limiting current from the experimental result obtained with a 0.05 M NaCl solution and a linear feed flow velocity of 0.104 m $s^{-1}$ (15 L $h^{-1}$ flow rate). In the relationship between the dilute pH and the current in Fig. 3 (*a*), the limiting current was determined where the slope was changed due to water dissociation on the surface of ion exchange membranes [33, 34]. In addition, the limiting current was determined from the graph showing the cell resistance versus the reciprocal of the current (Fig. 3 (*b*)).

NaCl Solutions with different concentrations were used to determine these values at different flow rates. The obtained results were summarized in Table 3.



*a*

*b*

*Fig. 3. (a) Variation of cell resistance versus the reciprocal of applied current (b) Variation of pH in the dilute compartment versus the reciprocal of applied current*

*Table 3. Limiting current values as function of flow rate and feed concentration*

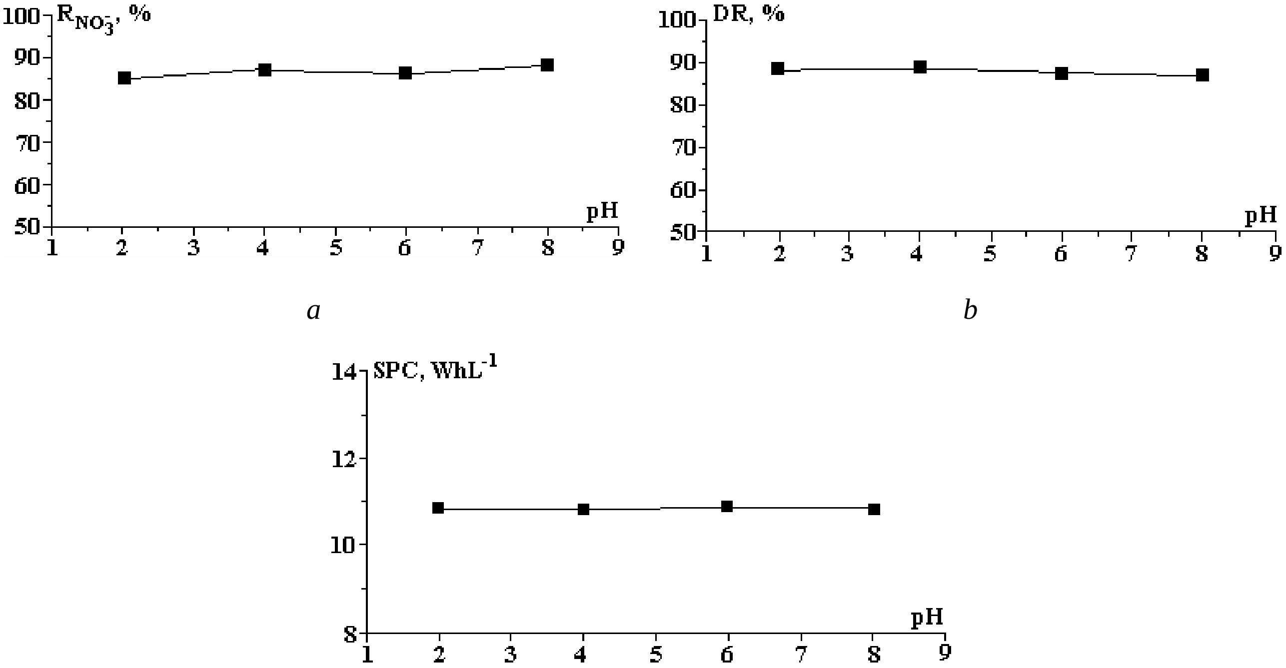
| Flow rate (L h$^{-1}$) | Feed solution concentration (g L$^{-1}$) | | | |
|---|---|---|---|---|
| | 0.5 | 1 | 1.5 | 2.5 |
| 5 | 0.2 | 0.4 | 0.82 | 1.16 |
| 15 | 0.34 | 0.62 | 0.98 | 1.52 |
| 25 | 0.46 | 0.86 | 1.15 | 1.96 |
| 30 | 0.52 | 1 | 1.26 | 2.2 |

The applied current in all experiment was fixed from the beginning to a value below the limiting current ($I$ = 0.8 $I_{lim}$)[35].

### 3.2. Denitrification of synthetic solution

### 3.2.1. Effect of pH of the feed solution

Effect of pH of the feed solution on the removal of nitrate, demineralization rate and specific power consumption are showed in Figs. 4 (*a*), (*b*) and (*c*).



*a* *b*

*c*

*Fig. 4. pH of feed solution effect (a) on the removal of nitrate, (b) on the DR and (c) on the SPC ($NO_3^-$ 250 mg $L^{-1}$, applied current 1.72 A, initial feed solution 2.5 g $L^{-1}$ and flow rate 30 L $h^{-1}$)*

The removal of $NO_3^-$ was independent of pH, which is due to the pH independence of $NO_3^-$ speciation [21].

### 3.2.2. Effect of feed flow rate

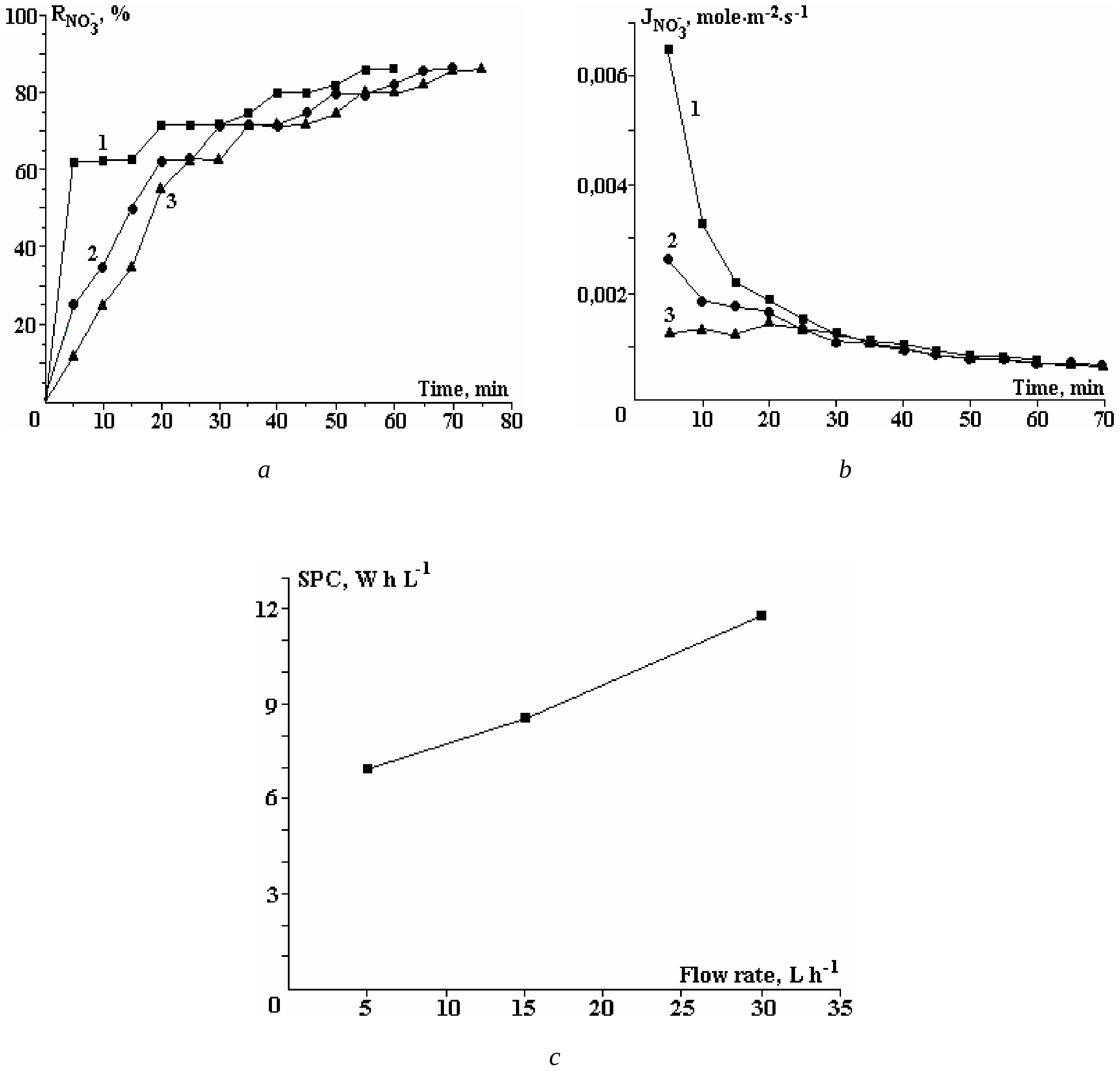
The operation was carried out using different feed flow rates (5, 15, 30 L $h^{-1}$) and with a nitrate solution of 250 mg $L^{-1}$ and 1.5 g $L^{-1}$ as initial feed concentration.

As seen in Figs. 5 (*a*) and (*b*), flow rate have a significant effect on nitrate removal only in the beginning of the experiment (before 20 min) in the range of flow rates studied. The initial flux of nitrate is higher in the beginning of experiment mainly for low rates. It decreases quickly and become constant for all flow rates.

The nitrate ions transferred from the diluted to concentrated compartments are higher in the beginning of experiment because the amount of nitrate contained in the initial compartments is at higher level and high variations can be calculated. For low flow rate these ions still longer time in the dilute compartments and can be transferred from one to another compartments. After short experiment time the concentration of nitrate in dilute is reduced and the variations during time becomes low and constant. The opposite observations were obtained in the removal rates as function of time [29].

In the other hand, as depicted in Fig. 5 (*c*), the specific power consumption depends also on this parameter. The SPC is lower for low flow rate. This can be explained by two propositions. Effectively the SPC depends mainly on two parameters: the applied current and the time of experiment. Firstly the applied current (the limiting current) is depending on the flow rate as shown in Table 3. It is lower for low flow rate. Secondly the experiment time to obtain the same results (removal of 200 mg $L^{-1}$ of $NO_3^-$) is increasing when the flow rates increases.

It was not possible to increase the flow rate above 30 L $h^{-1}$ since the maximum capacity of the pumps in the present system is defined as 30 L $h^{-1}$.



*a* *b*

*c*

*Fig. 5. Effect of feed flow rate (a) on the removal of nitrate, (b) on the nitrate flux and (c) on the SPC ($NO_3^-$ 250 mg $L^{-1}$, initial feed solution 1.5 g $L^{-1}$)*

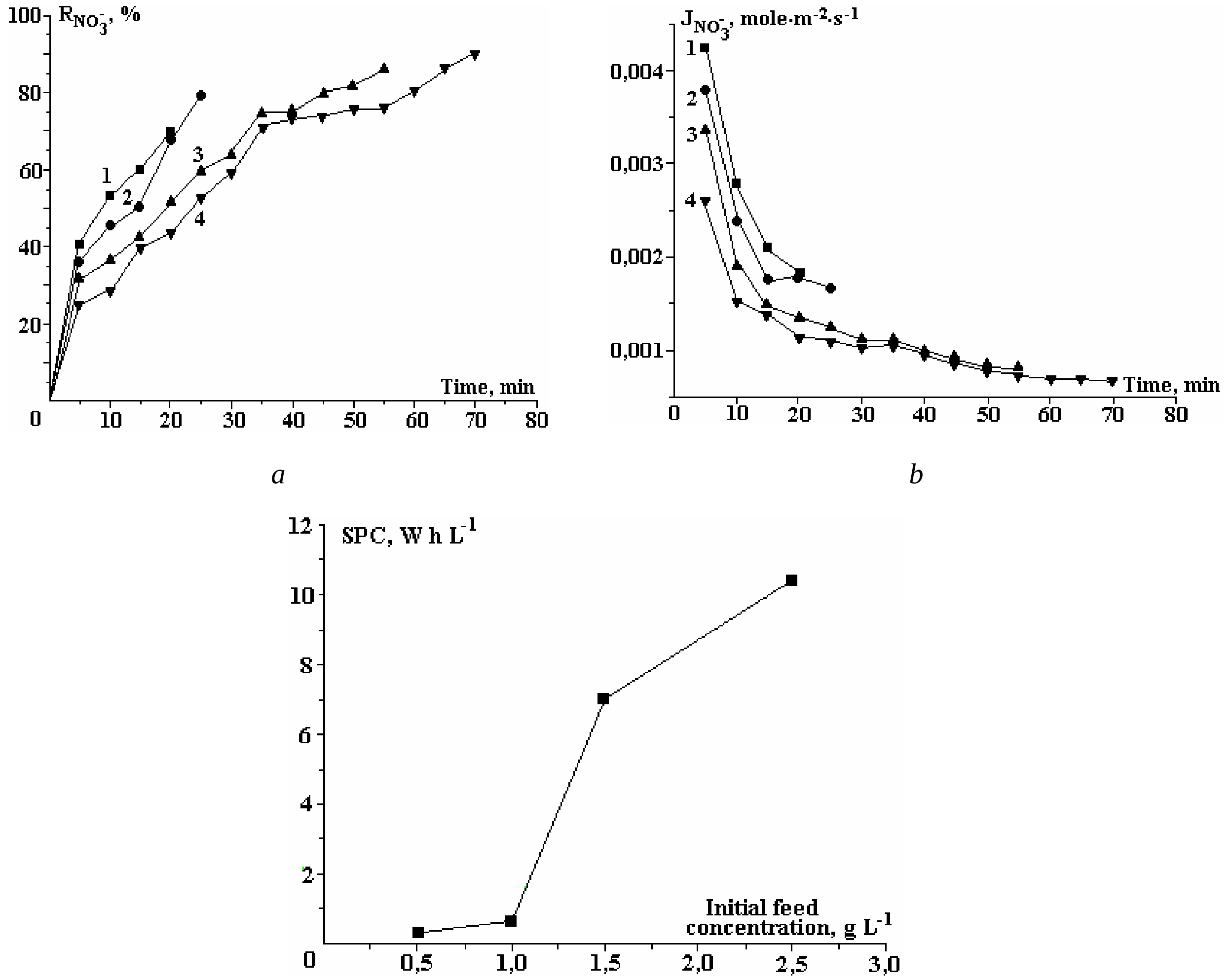
### 3.2.3. Effect of initial feed concentration

The effect of initial feed concentration on the removal of nitrate was investigated using aqueous solutions at various concentrations of NaCl salt (0.5, 1, 1.5 and 2.5 g $L^{-1}$) and fixed nitrate solution of 250 mg $L^{-1}$ .

As shown in Figs. 6 (*a*) and (*b*), the initial salt concentration has a significant effect on the nitrate removal. The most important observation is that total process time increased with increasing initial concentration in the feed. Consequently the specific power consumption is closely depending on it as seen in Fig. 6 (*c*).

These results can be explained by the increase of number of ions in solutions when the concentration of salts increases. As a result a competitive transfer can appear between nitrate ions and other ions (chloride ions in this case).

It was not possible to increase the concentration of initial feed solution above 3 g $L^{-1}$ since an electrode overheating appeared in the present system as a result of the increase of cell conductivity.



*a* *b*

*c*

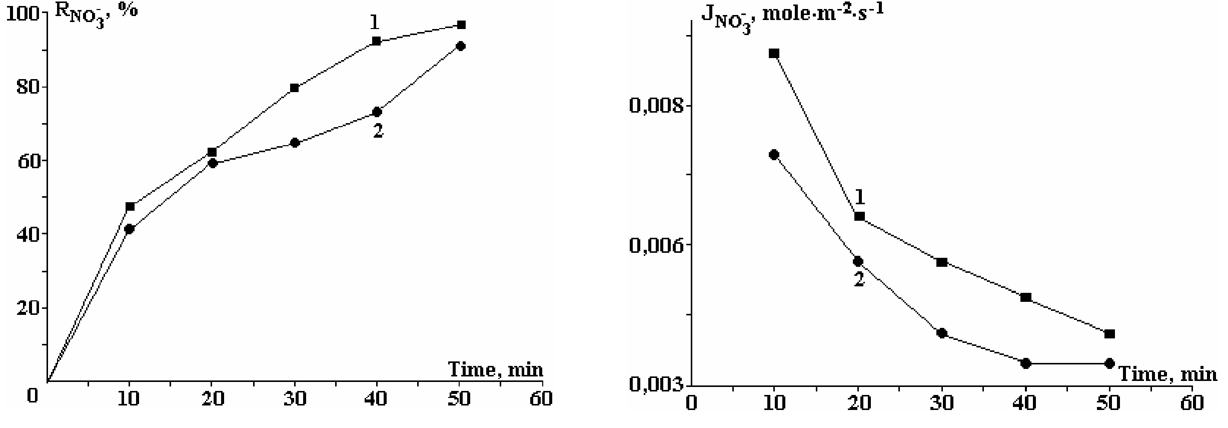
*Fig. 6. Effect of initial feed concentration (a) on the removal of nitrate, (b) on the nitrate flux and (c) on the SPC ($NO_3^-$ 250 mg $L^{-1}$, flow rate 5 L $h^{-1}$).*

### 3.2.4. Effect of coexisting ions

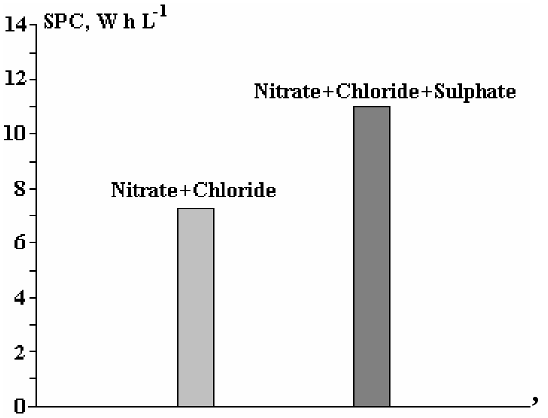
In the presence of chloride and sulphate ions, removal of nitrate by electrodialysis was investigated. The experiments were conducted with $NO_3^-$ (250 mg $L^{-1}$ solution) paired only with chloride and mixtures containing both chloride and sulphate ions.

It was obtained that the competition between nitrate and chloride ions was certain (Figs. 6 (*a*) and 6 (*b*)). The increase of chloride ions on the feed solution leads to the decrease of the removal rate and flux of nitrate ions. These phenomena are also clearly observed in the presence of sulphate ions. Effectively and as depicted in Figs. 7 (*a*) and (*b*) nitrate ions flux and retention rate decreases more in these conditions.

As shown in Fig. 7 (*c*), the specific power consumption increases more and more in the presence of the other species. This is due mainly to the augmentation of the total experiment time.



*a* *b*



*c*

*Fig. 7. Effect of accompanying ions (a) on the removal of nitrate, (b) on the nitrate flux and (c) on the SPC ($NO_3^-$ 250 mg $L^{-1}$, applied current 1.72 A, initial feed concentration 3g $L^{-1}$ and flow rate 30 L $h^{-1}$)*

### 3.3. Application of denitrification of the real water sample

Finally, the application of electrodialysis was performed on the real brackish water. As seen in Table 4, denitrification of real water was achieved with nitrate concentration (25.5 mg $L^{-1}$) lower than the maximum recommended by WHO (50 mg $L^{-1}$ ) despite to the presence of chloride and sulphate ions in sample. The concentrations of different species in the obtained treated water are below the amounts recommended by WHO for drinking water.

In other words, nitrate removal from real water sample was 88% at the end of 80 min of electrodialysis (Fig. 8) and with 13.2 W h $L^{-1}$ as specific power consumption.

*Table 4. Composition of the treated brackish water*

| Physico-chemistry parameter | Treated water | Recommendations of WHO [4] |
|---|---|---|
| Conductivity at 25 °C (µS $cm^{-1}$) | 0.808 | - |
| pH | 5.1 | 6.5-8.5 |
| $F^-$ (mg $L^{-1}$) | 0.22 | 1.5 |
| $Cl^-$ (mg $L^{-1}$) | 182 | 250 |
| $HCO_3^-$ (mg $L^{-1}$) | 202 | - |
| $NO_3^-$ (mg $L^{-1}$) | **25.5** | 50 |
| $SO_4^{2-}$ (mg $L^{-1}$) | 24.67 | 400 |
| $Na^+$ (mg $L^{-1}$) | 49.92 | 250 |
| $K^+$ (mg $L^{-1}$) | 24.23 | - |
| $Ca^{2+}$ (mg $L^{-1}$) | 14.03 | - |
| $Mg^{2+}$ (mg $L^{-1}$) | 12.72 | - |
| TDS (mg $L^{-1}$) | 350 | 500 |

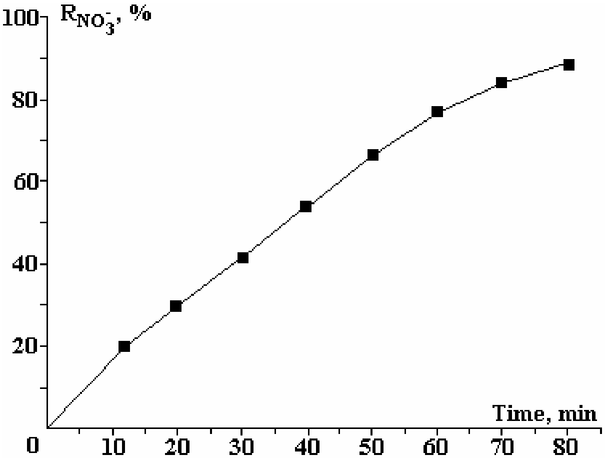


*Fig. 8. Removal of nitrate from real water sample by electrodialysis (nitrate concentration of real water: 225 mg $L^{-1}$; flow rate: 25 L $h^{-1}$, applied current: 1.92 A)*

## Conclusion

The results from the presented study can be concluded as follows:

- The removal of $NO_3^-$ was independent of pH.
- The feed flow rate has a significant effect on the denitrification efficiency and mainly on the specific power consumption. The decrease of this parameter induces a decrease on the energy needed to perform required operation.
- The initial salt concentration of feed solution has a significant effect on the nitrate removal. An increase of this concentration leads to the increase of total process time and consequently the augmentation of the specific power consumption to obtain required denitrification efficiency.
- The removal of nitrate decreased in the presence of chloride and sulphate ions in the feed phase. In other words, the transport of the nitrate ions is more effective in the absence of co-existent anions.

As a result of the application of electrodialysis on the real water sample, nitrate concentration could be reduced from 225 to 25.5 mg $L^{-1}$ (88% removal), which was lower than WHO standard (50 mg $L^{-1}$). Moreover the concentrations of different species in the obtained treated water are below the amounts recommended by WHO for drinking water.

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## Summary

The aim of this work is to study the removal of nitrate from brackish polluted water using electrodialysis. The influence of several parameters, such as flow rates, initial feed concentration, co-existing anions and initial pH on process efficiency were studied. This efficiency is evaluated by the removal rate, demineralization rate and power consumption. The denitrification process showed to be independent of pH of feed solution. Although The flow rate as well as the initial salt concentration and also the coexisting anions on the feed solution play a significant role on the denitrification efficiency and mainly on the specific power consumption. The decrease of this parameter induces a decrease on the total energy needed to perform required operation. The denitrification of a contaminated real water sample was investigated so as to improve the efficiency of denitrification process. The nitrate concentration could be reduced from 225 to 25.5 mg L-1 (88% removal) which was lower than World Health Organization (WHO) standard (50 mg L-1). Moreover the concentrations of different species in the obtained treated water are below the amounts recommended by WHO for drinking water.