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JACEK WIŚNIEWSKI*

ION EXCHANGE BY MEANS OF DONNAN DIALYSIS AS A PRETREATMENT PROCESS BEFORE ELECTRODIALYSIS

The author applied Donnan dialysis in order to remove troublesome anions (SO_4^{2-} and HCO_3^-) or cations (Ca^{2+}, Mg^{2+}) from water solutions before their electrodialytic desalination. As the result of Donnan dialysis (with the Neosepta AFN membrane), molar share of troublesome anions (SO_4^{2-} and HCO_3^-) decreased to 4.3% of all anions. The cation exchange was responsible for a decrease in the molar share of Ca^{2+} and Mg^{2+} ions to 5.5% of all cations in the solution. Due to the change of solution ionic composition, a number of advantageous phenomena were observed during electrodialytic desalination: the increase in the limiting current density, higher rate of the salt removal (up to 20%) and lower consumption of energy necessary for the transport of a given amount of salt (up to three times). It was concluded that Donnan dialysis with the anion-exchange membrane (Neosepta AFN) was an advantageous pretreatment process before electrodialysis because the fluxes of the removed anions were even two times as high as those observed with the cation-exchange membrane for cation removal.

1. INTRODUCTION

Donnan dialysis consists in the exchange of ions of the same charge between two solutions separated from each other by ion-exchange membrane. As a result of relatively high salt concentration in the receiving solution, the transport of counter-ions to the feeding solution takes place. Because the ion-exchange membrane disables flux of co-ions in the same direction, for the electroneutrality of both solutions to be obtained, equal amount of counter-ions is transported from the feed to the receiver. The process of ion exchange between the solutions lasts until the so-called Donnan equilibrium is achieved. It is described by the following equation [1]:

$$\left(\frac{C_{ir}}{C_{if}}\right)^{1/Zi} = K,$$

* Institute of Environmental Protection, Wrocław University of Technology, Wybrzeże Wyspiańskiego 27, 50-377 Wrocław, Poland. E-mail:

where: C_{ir} and C_{if} are the respective activities of an *i*-ion in the receiver and in the feed, Z_i stands for the valence of the ion, and K is constant for all counter-ions in the system. From the above equation one can see that with adequately high concentration of the counter-ions in the receiver, efficient removal of troublesome counter-ion from the feed is possible, and – at the same time – its concentration in the receiver can be observed.

Donnan dialysis with the anion-exchange membrane offers the most interesting application possibilities. The process is used mostly for the removal of harmful anions, such as fluorides and nitrates, from drinking water. It was proved that with an appropriate anion-exchange membrane (Selemion DSV) fluoride concentration can be decreased below the limiting value for drinking water, i.e., 1.5 mg/dm^3 [2]–[4]. In paper [5], the authors described the effect of Donnan dialysis on the nitrate removal from water. The concentration of this ion was reduced from 90 to 16 mg NO_3^-/dm^3, which is below the limiting value for drinking water (50 mg NO_3^-/dm^3). An interesting solution was also proposed by the authors of paper [6]: nitrates were removed with mono-anion-selective membrane from treated water to bioreactor, where their biological reduction took place. Such a combination of the processes made it possible to increase the nitrate flux to the receiver and to obtain significant reduction in the NO_3^- concentration in the treated water (from 150 to 20 mg NO_3^-/dm^3).

Donnan dialysis with cation-exchange membrane is used for the recovery of valuable metals from industrial waste streams, e.g., aluminium, iron and titanium from the red mud after aluminium production [7], [8], chromium from the solutions of metal salts [9] and aluminium from the sediments after coagulation in the water treatment plant [10].

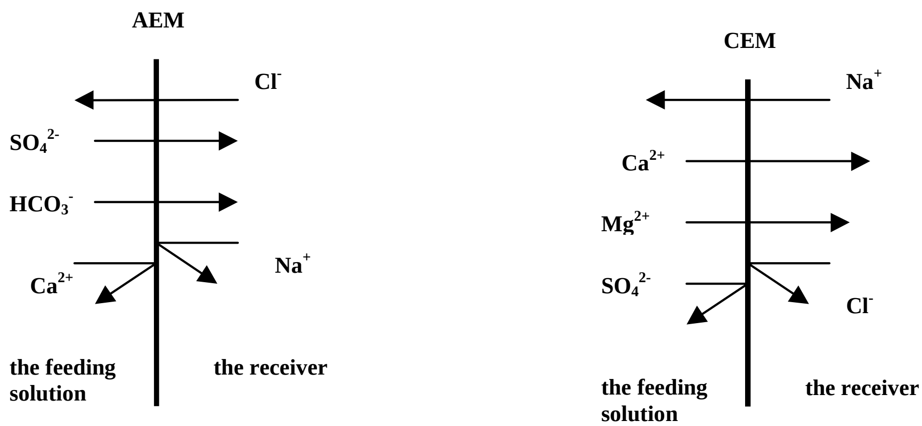


Fig. 1. Ion exchange in the Donnan dialysis with anion-exchange membrane (AEM) and cation-exchange membrane (CEM)

In this paper, Donnan dialysis is applied in order to change the ionic composition of the solution before its electrodialytic desalination. During the process with the anion-exchange membrane, SO_4^{2-} and HCO_3^- ions are being replaced with the Cl^- ions, while with the use of cation-exchange membrane – Ca^{2+} and Mg^{2+} ions are exchanged for the Na^+ ions (figure 1).

In both processes, readily-soluble chloride or sodium salts are formed. As a result of the change of the solution ionic composition, the troublesome sediments in the concentrate compartments of the electrodialyzer will not push out, and the rate and the efficiency of the electrodialytic desalination should be higher than those observed during the desalination of the raw solution.

2. EXPERIMENTAL

2.1. DONNAN DIALYSIS

The process was conducted in the laboratory dialytic set equipped with 20 cell pairs with anion-exchange membrane (Neosepta AMX, Neosepta AFN) or cation-exchange one (Neosepta CMX, Selemion CMV). The working area of the membrane was 0.140 m^2. Characteristics of the membranes tested is shown in table 1. Dialysis was conducted with the recirculation of both solutions (batch system) until the equilibrium concentration of the exchanged ions in the feed was obtained. The volume ratio of the feed and the receiver was 4:1 (10 dm^3 : 2.5 dm^3).

Table 1

Characteristics of ion-exchange membranes used in the Donnan dialysis

Characteristics	AEM		CEM	
	AMX	AFN	CMX	CMV
Ion-exchange capacity, mmol/g	1.30	3.15	2.46	4.31
Water content, %	26.1	64.8	26.5	39.9
Thickness, mm	0.13–0.14	0.12	0.18	0.14

Ion exchange took place in the solution consisting of $NaNO_3$, Na_2SO_4, $NaHCO_3$ and NaCl (3 mmol/dm^3 of each component). NaCl solution of the concentration of 300 mmol/dm^3 was used as the receiving solution (the value has been chosen according to the previous experiments [11]).

During the process anion concentrations in the feed were measured. The concentration of NO_3^- and SO_4^{2-} was determined with the DREL2000 spectrophotometer; the Cl^-

and HCO_3^- concentration – by the titration with the $AgNO_3$ and HCl, respectively. On the basis of the measurements, the removal efficiency and the ionic flux from the feed were calculated as well as the flux of chlorides from the receiver to the feed.

The solution of $CaCl_2$ (5 mmol/dm^3) and $MgCl_2$ (5 mmol/dm^3) was used for the cation exchange. The receiver was the NaCl solution of the concentration of 200 mmol/dm^3 [12].

Concentration of Ca^{2+} and Mg^{2+} ions was measured in the feed (by means of titration with the sodium versenate) as well as the chloride concentration. On this basis, the removal efficiency and the ionic flux from the feed were calculated as well as the sodium flux entering the feed.

2.2. ELECTRODIALYSIS

Electrodialytic desalination of the solutions was conducted in the laboratory electrodialyzer equipped with 15 cell pairs with the Neosepta AMX and the Neosepta CMX membranes. The working area of the membrane was 0.108 m^2. The process was carried out at the volume ratio of the diluate to the concentrate of 5:1 (10 dm^3 : 2 dm^3).

The following solutions were examined:

• Raw solution of the concentration of 12 meq/dm^3; the concentration of each anion (NO_3^-, SO_4^{2-}, HCO_3^- and Cl^-) was 3 meq/dm^3; the counter-ions were Ca^{2+} (6 meq/dm^3), Mg^{2+} (3 meq/dm^3) and Na^+ (3 meq/dm^3).

• Solution after Donnan dialysis with the Neosepta AFN membrane (with the main anion in the solution being Cl^-).

• Solution after Donnan dialysis with the Selemion CMV membrane (with the main cation in the solution being Na^+).

Based on the ion concentration measurements in the diluate, the amount of removed salt and the salt flux from this solution were calculated. Moreover, the energy consumption for the transport of ions was estimated.

3. RESULTS AND DISCUSSION

3.1. DONNAN DIALYSIS WITH ANION-EXCHANGE MEMBRANE

It was observed that anionic species has significant influence on the rate and the efficiency of the anion removal from the solution during Donnan dialysis. Figures 2 and 3 present the anion exchange with Neosepta AMX and Neosepta AFN membranes.

One can notice that the process of anion exchange is much faster with NO_3^- and SO_4^{2-} ions than with HCO_3^- for Cl^- ions. This is caused mainly by the differences in the ionic size: hydrated ionic radius of NO_3^- ion is 0.349 nm, ionic radius of SO_4^{2-} equals 0.380 nm, and HCO_3^- is the longest as its radius exceeds 0.394 nm [13].

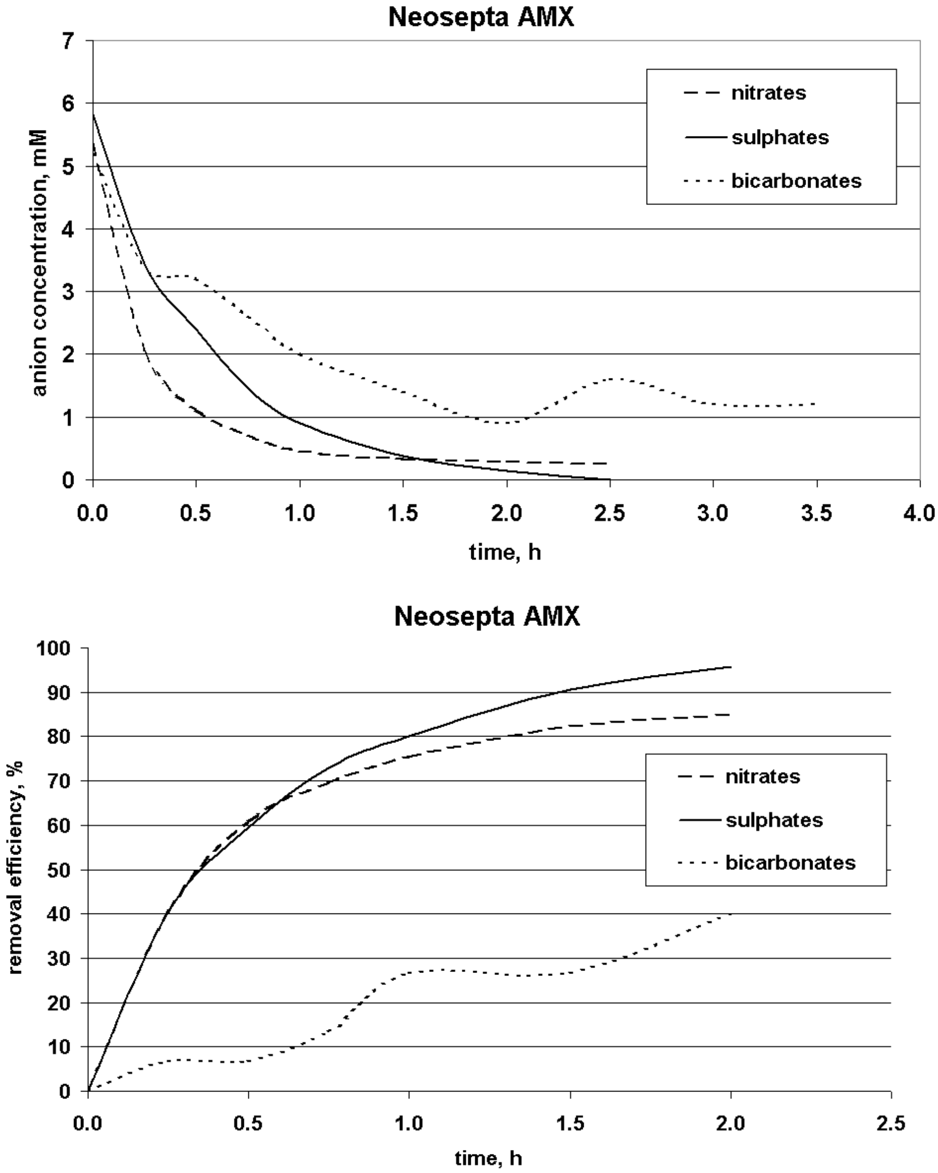


Fig. 2. Concentration decrease and removal efficiency of anions during Donnan dialysis with the Neosepta AMX membrane

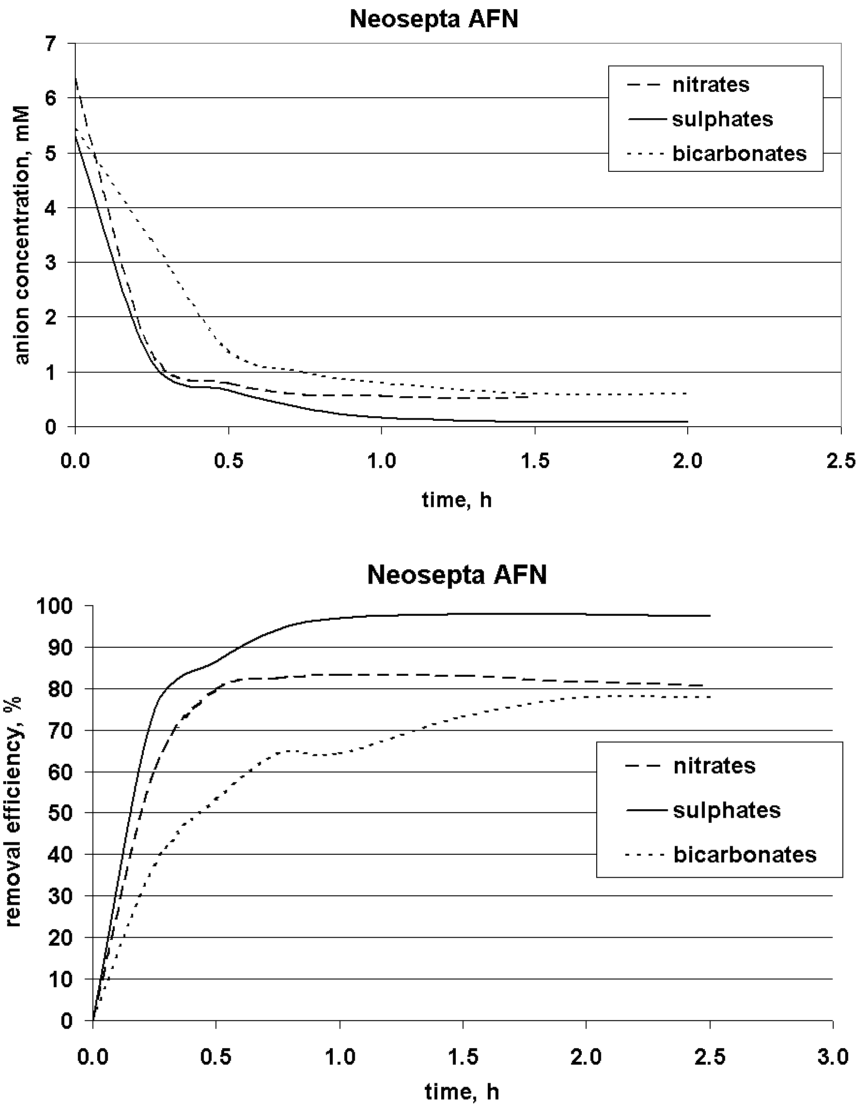


Fig. 3. Concentration decrease and removal efficiency of anions during Donnan dialysis with the Neosepta AFN membrane

It should also be stressed that the lowest equilibrium concentration (and at the same time – the highest removal efficiency) is obtained for the SO_4^{2-} ions. This can be linked to the interactions of the ions whose ionic charge is higher with ion-exchange groups inside the membrane matrix. As a result, sulphates are removed with higher efficiency than nitrates – despite the fact that the SO_4^{2-} ion is bigger than the NO_3^- ion.

Comparing the results of the anion exchange for the two membranes, it can be seen that the Neosepta AFN allows us to obtain higher rate and efficiency of the process than the Neosepta AMX membrane. This relates especially to the removal of troublesome ions, e.g., SO_4^{2-} and HCO_3^- (figure 4).

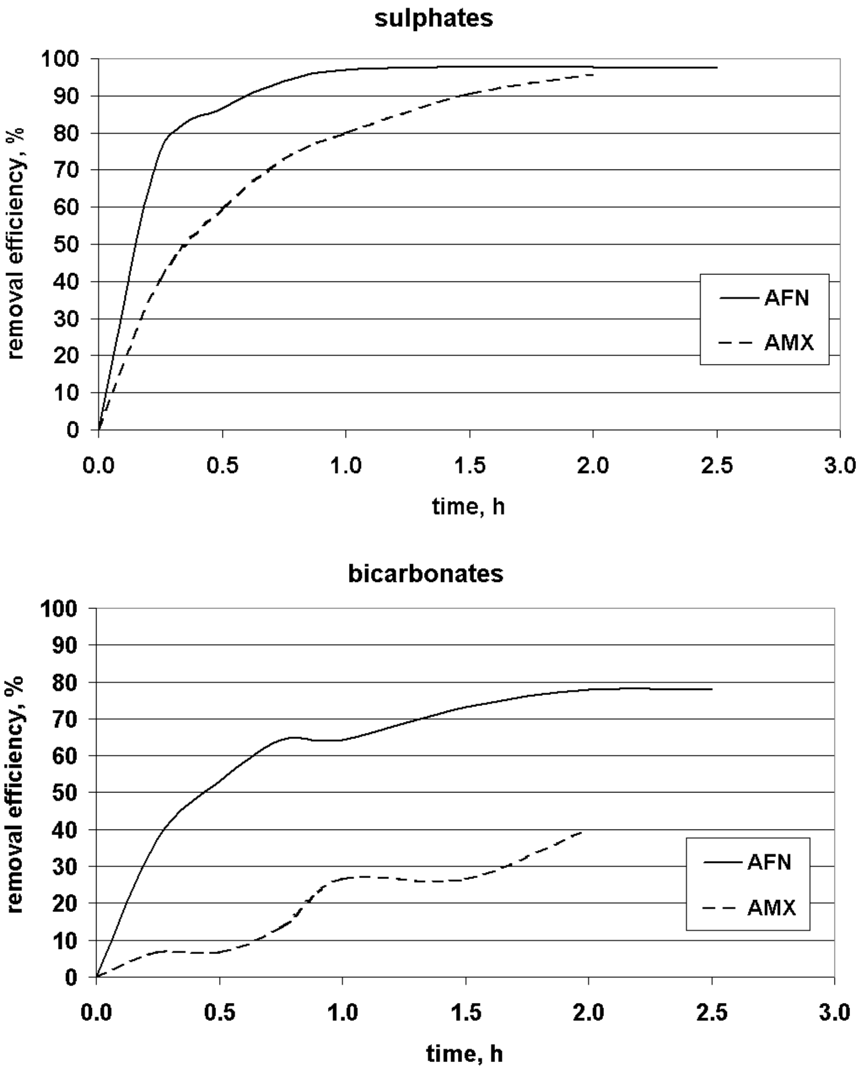


Fig. 4. Comparison of sulphate and bicarbonate removal with different anion-exchange membranes

The mean sulphate flux through the Neosepta AFN membrane is 0.154 mol/m^2·h (for the AMX membrane, 0.131 mol/m^2·h). Definitely greater differences are observed for bicarbonates: the mean flux of this anion through the AFN membrane reaches 0.090 mol/m^2·h, and through the AMX membrane, 0.046 mol/m^2·h. Also the removal effciency of the HCO_3^- ion is significantly higher for the Neosepta AFN membrane (78%) than for the Neosepta AMX (40%).

Table 2 presents the rate and the efficiency of anion exchange with the anion-exchange membranes examined.

Table 2

Mean ionic fluxes (J_{av}, mol/m^2·h) and anion removal efficiency (R, %) in the Donnan dialysis with the anion-exchange membrane

Ion		Membrane	
		Neosepta AMX	Neosepta AFN
NO_3^-	J_{av}	0.115	0.210
	R	85.1	83.2
SO_4^{2-}	J_{av}	0.131	0.154
	R	95.7	97.8
HCO_3^-	J_{av}	0.046	0.090
	R	40.0	77.8

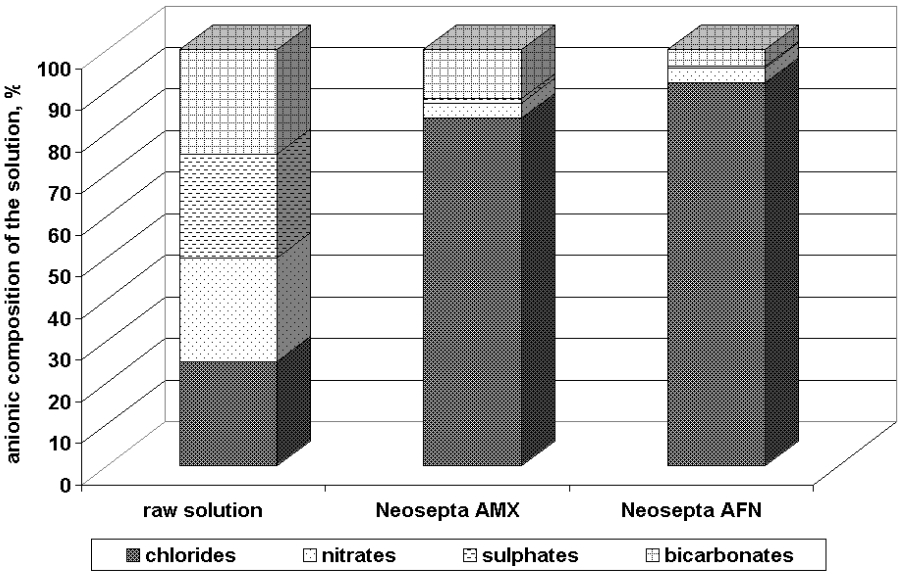


Fig. 5. Ionic composition of the raw solution and the solution after Donnan dialysis with the Neosepta AMX and the Neosepta AFN membranes

Advantageous properties of the Neosepta AFN membrane in the anion exchange are connected with high ion-exchange capacity and high water content of this membrane (table 1). These parameters are over two times as high as those of the Neosepta AMX membrane. Such values of these parameters, especially of water content in the membrane (that is the result of reduced internal cross-linking of the polymeric matrix), accelerate and facilitate the transport of counter-ions from the feeding solution – even large HCO_3^- ions [14].

High water content in the Neosepta AFN membrane facilitates not only the counter-ion transport but also the salt leakage from the receiver to the feed. As a result, the salt concentration in the solution after Donnan dialysis increases. Despite this phenomenon, one should stress advantageous changes in the ionic compsition of the solution after dialysis with anion-exchange membrane: molar share of troublesome ions (SO_4^{2-} and HCO_3^-) decreased from 50% in the raw solution to 12.8% after the process with the Neosepta AMX and to 4.3% after the process with the Neosepta AFN membrane (figure 5).

3.2. DONNAN DIALYSIS WITH CATION-EXCHANGE MEMBRANE

Figure 6 presents the efficiency of the cation exchange with the Neosepta CMX membrane, and figure 7 – with the Selemion CMV membrane. One can notice that the removal efficiency of Ca^{2+} and Mg^{2+} is similar, while the removal rate of the Ca^{2+} ions in the initial phase of their exchange is higher than that of the Mg^{2+} ions. Only when the calcium ions are in the main part (over 70%) exchanged for the sodium ions, does the process of magnesium ion exchange accelerate. This phenomenon is connected not only with the difference in ionic size (hydrated ionic radius of Ca^{2+} (0.412 nm) is slightly smaller than that of Mg^{2+} (0.429 nm)), but also with different affinity of both ions for the ion-exchange groups inside the cation-exchange membrane. According to Pauling, electronegativity of calcium is 1.04, while that of magnesium, 1.23 [16]. Due to the difference in electronegativity Ca^{2+} ions are more strongly attracted to the negatively charged ion-exchange groups of the membrane than Mg^{2+} ions.

Comparing the process of cation exchange with different membranes, it is obvious that the transport of Ca^{2+} and Mg^{2+} ions by the Selemion CMV membrane is more advantageous than that by the Neosepta CMX membrane (figure 8).

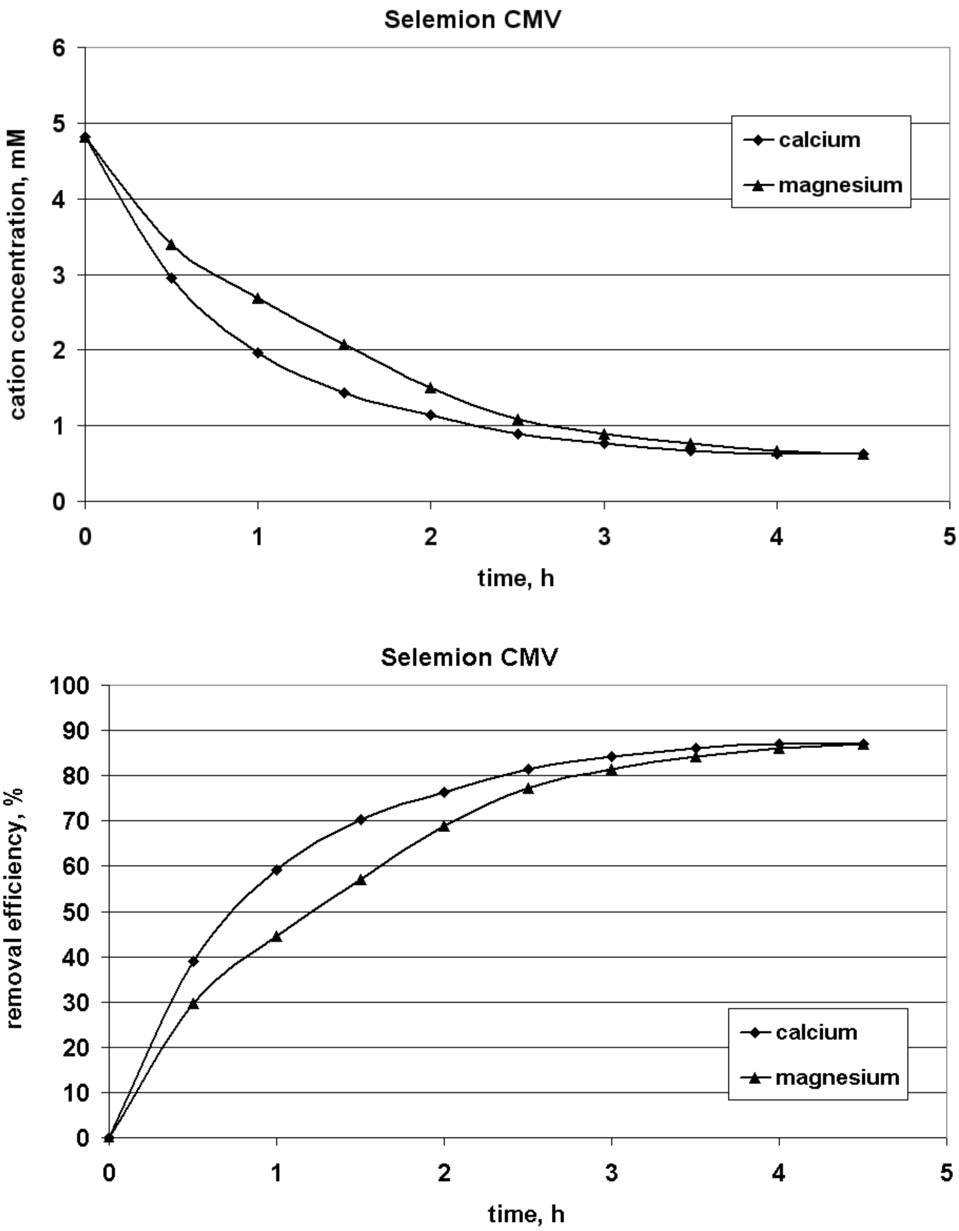


Fig. 6. Concentration decrease and removal efficiency of Ca^{2+} and Mg^{2+} in the Donnan dialysis with the Selemion CMV membrane

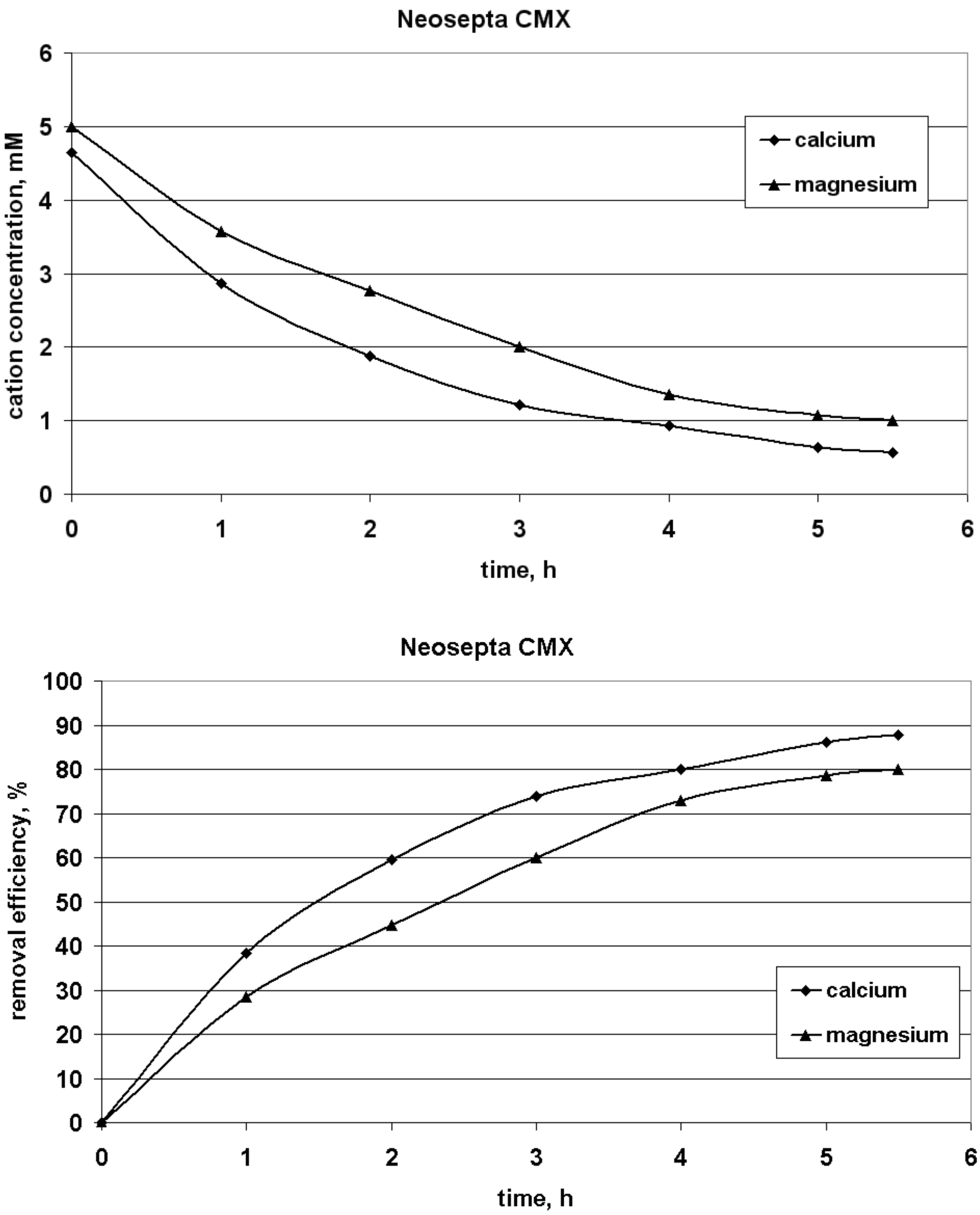


Fig. 7. Concentration decrease and removal efficiency of Ca^{2+} and Mg^{2+} in the Donnan dialysis with the Neosepta CMX membrane

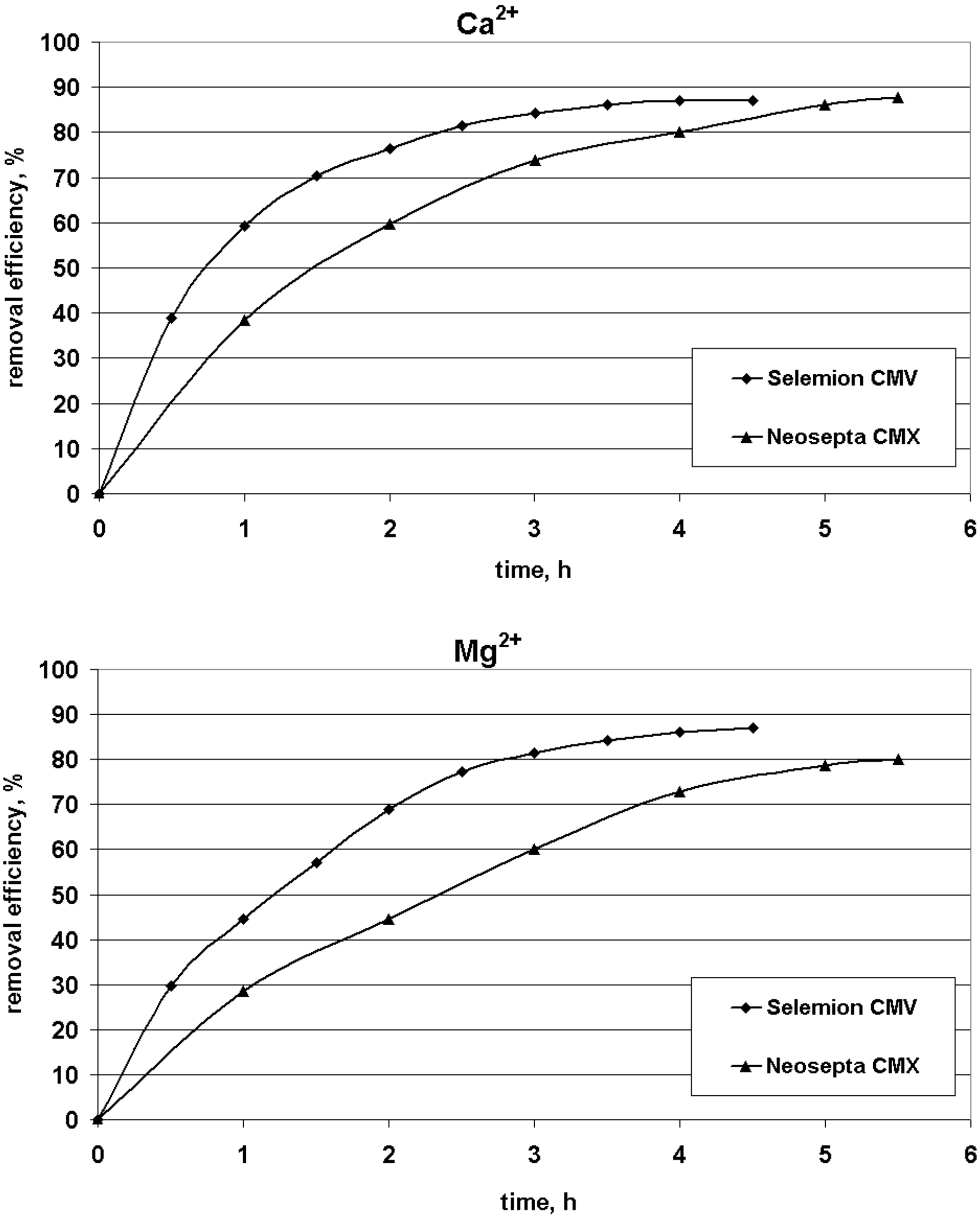


Fig. 8. Removal of Ca^{2+} and Mg^{2+} ions in the Donnan dialysis with the Selemion CMV and the Neosepta CMX membranes

The mean fluxes of Ca^{2+} ions equal 0.067 mol/m^2·h (CMV) and 0.053 mol/m^2·h (CMX), while the mean fluxes of Mg^{2+} ions are, respectively, 0.067 mol/m^2·h and

0.052 mol/m^2·h. As a result, the cation exchange with the Selemion CMV membrane is faster, and the time of the process – shorter. This is connected, as in the case of the anion-exchange Neosepta AFN membrane, with high ion-exchange capacity and increased water content of the Selemion CMV membrane (table 1). These properties facilitate and accelerate the transport of counter-ions (Ca^{2+} and Mg^{2+}) from the feed to the receiver.

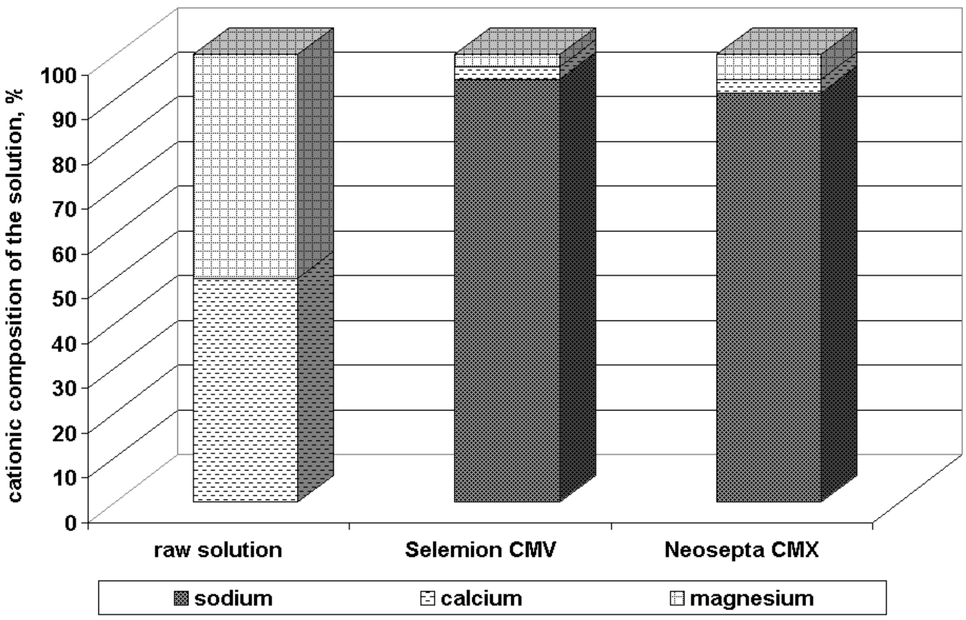


Fig. 9. Ionic composition of the raw solution and the solution after Donnan dialysis with the Neosepta CMX and the Selemion CMV membranes

Despite the increase of the salt concentration in the feed (the result of the salt leakage from the receiver), Donnan dialysis with the cation-exchange membrane promoted advantageous changes of ionic composition of the solution (figure 9). The molar share of Ca^{2+} and Mg^{2+} ions was reduced from 100% (in the raw solution) to 8.6% (after the process with the Neosepta CMX membrane) and to 5.5% (after the process with the Selemion CMV membrane).

3.3. ELECTRODIALYSIS OF THE RAW SOLUTION AND OF THE SOLUTIONS AFTER DONNAN DIALYSIS

Before electrodialytic desalination the limiting current density of the solutions was determined according to the Cowan and Brown method [16]. Figure 10 presents limiting current densities of the raw solution and the solutions after Donnan dialysis with the Neosepta AFN and with the Selemion CMV membranes.

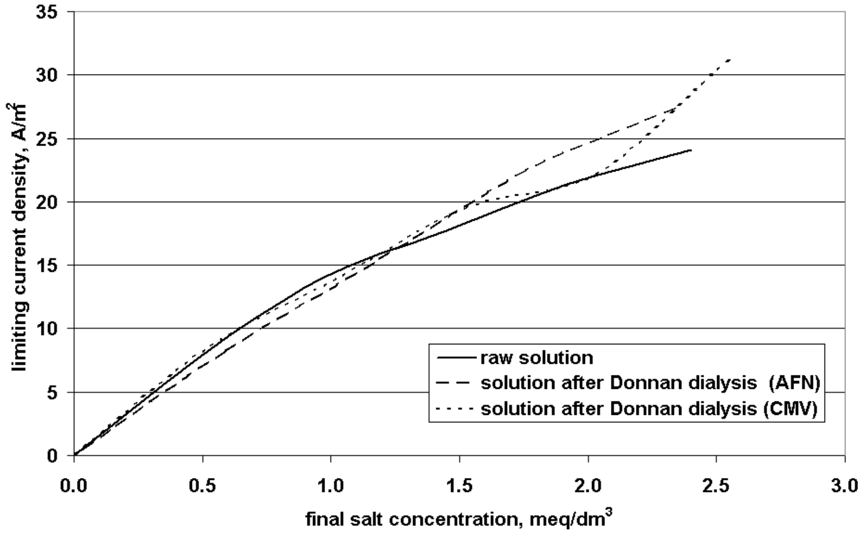
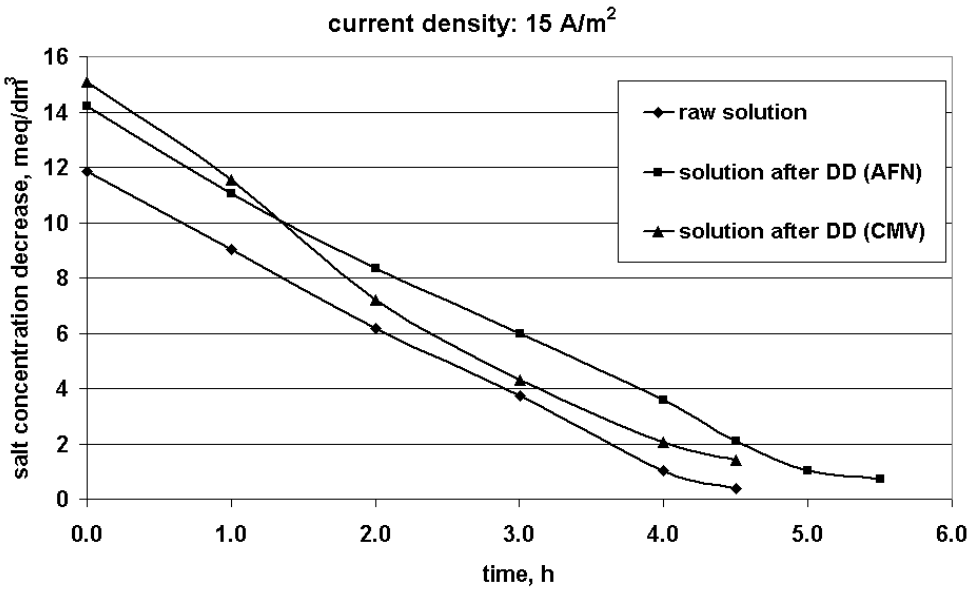


Fig. 10. Limiting current densities of the raw solution, the solution after Donnan dialysis with the Neosepta AFN and for the solution after Donnan dialysis with the Selemion CMV membrane



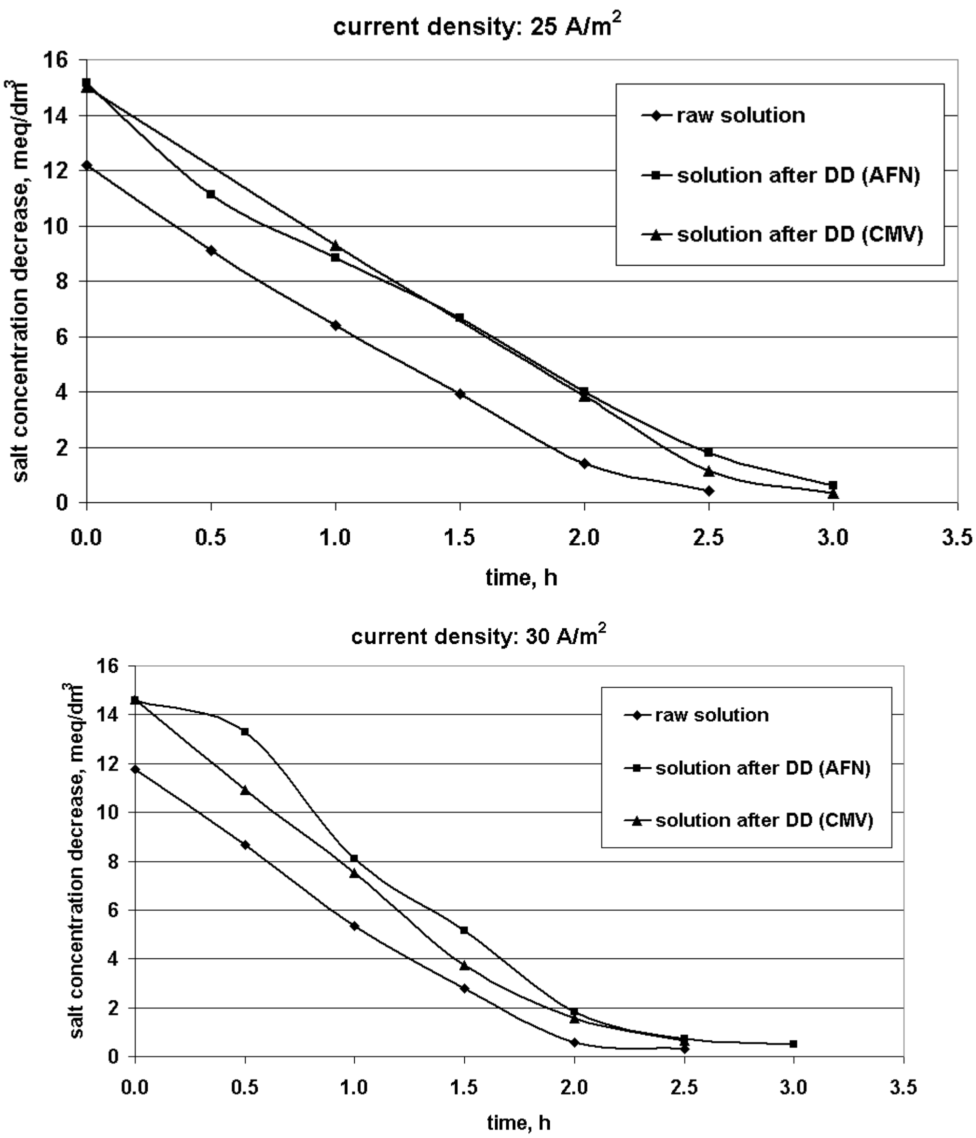


Fig. 11. Decrease in the salt concentration during electrodialysis of the raw solution and of the solution after Donnan dialysis with the anion- and cation-exchange membranes (AFN and CMV, respectively) at different current densities

One can notice that the limiting current densities of the solutions after Donnan dialysis (at the concentrations exceeding 1 meq/dm^3) are slightly higher than those of the raw solution because of the exchange of large ions for smaller ones (Cl^- or Na^+). Ionic radius of the Cl^- anion of 0.332 nm is smaller than the radii of NO_3^- (0.349 nm),

SO_4^{2-} (0.380 nm) and HCO_3^- (0.394 nm) ions present in the raw solution [13]. At the same time, ionic radius of the Na^+ ion (0.358 nm) is smaller than the radii of Ca^{2+} (0.412 nm) and Mg^{2+} (0.429 nm) ions. Due to their smaller size, Cl^- and Na^+ ions achieve higher mobility and their transport rate in the solution increases [17]. This means that in the boundary layers of the anion- and cation-exchange membranes, respectively, one should expect higher ion concentration than in the case of the raw solution. As a result, the limiting current density increases.

The limiting current densities for the raw solution and the solutions after Donnan dialysis are equal to 22 and 25 A/m^2, respectively, (at the salt concentration of 2 meq/dm^3). Because of that fact, electrodialysis of the solutions was conducted at the following current densities: 15, 25 and 30 A/m^2. Figure 11 presents the decrease of the salt concentration during electrodialysis of the solutions testes.

One can see that the rate of the salt removal from the solutions after dialysis is higher compared to that of the raw solution due to the change in ionic composition of the solutions. In the solution, after Donnan dialysis with the anion-exchange membrane, the equivalent share of troublesome anions (SO_4^{2-} and HCO_3^-) does not exceed 5%, whereas in the raw solution these anions make up 50% of all anions. At the same time, in the solution after Donnan dialysis with the cation-exchange membrane, the equivalent share of large Ca^{2+} and Mg^{2+} cations is equal to 3.3%, and in the raw solution they make up 75% of all cations. Because large SO_4^{2-} and HCO_3^- anions are replaced with smaller Cl^- ions, and Ca^{2+} and Mg^{2+} cations – with smaller Na^+ ions, the ionic mobility in the membrane increases [17], which results in the increase of the salt fluxes during electrodialysis (table 3).

Table 3

Mean salt fluxes from the raw solution and from the solutions after Donnan dialysis with the anion-exchange membrane (Neosepta AFN) and the cation-exchange membrane (Selemion CMV)

Current density, A/m^2	Mean salt flux (eq/m^2·h) from the solution		
	Without pretreatment	After dialysis with the AFN membrane	After dialysis with the CMV membrane
15	0.233	0.249	0.279
25	0.433	0.493	0.507
30	0.421	0.506	0.512

The data presented show that the type of ion-exchange membrane used in Donnan dialysis only slightly affects the salt flux during electrodialysis. However, it should be stressed that the desalination rate of the solutions after ion exchange is significantly higher than that of the raw solution (up to 20%).

The comparison of the energy consumption during desalination of the solutions tested versus the amount of removed salt also shows that Donnan dialysis (figure 12) is advantageous to the solution pretreatment.

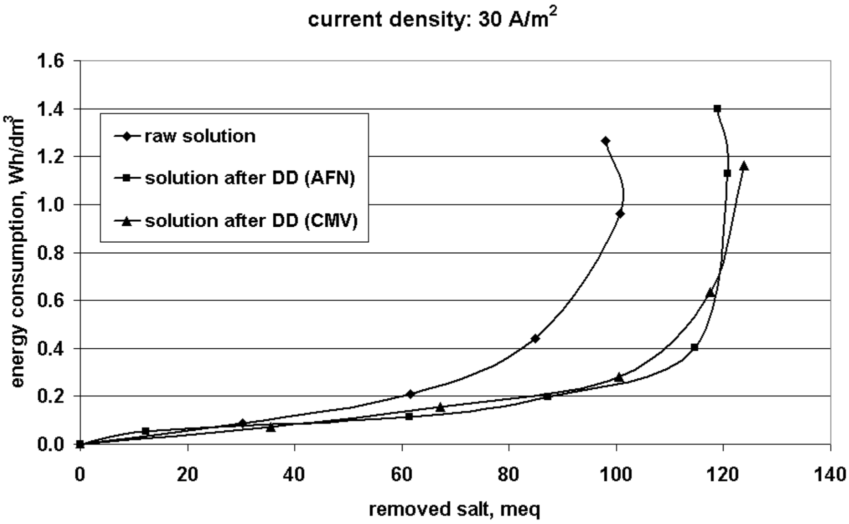


Fig. 12. Energy consumption vs. the amount of salt removed during desalination of the raw solution and the solutions after Donnan dialysis with the anion- and the cation-exchange membranes (AFN and CMV, respectively); $i = 30$ A/m^2

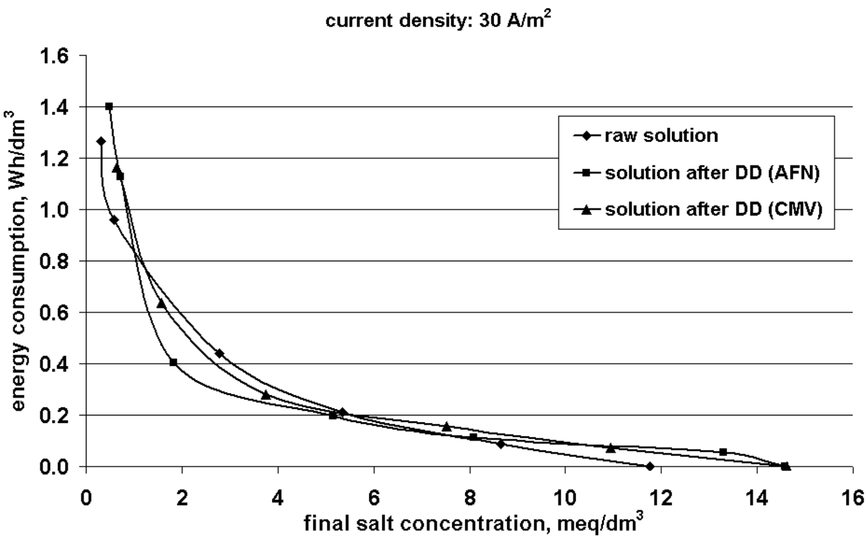


Fig. 13. Energy consumption during desalination of the raw solution and the solutions after Donnan dialysis with the anion- and the cation-exchange membranes (AFN and CMV, respectively); $i = 30$ A/m^2

The energy consumption for the transport of a given amount of salt in the solutions after dialysis is up to three times smaller than that for the raw solution. Comparing the consumption of energy necessary for achieving a given final salt concentration, one can see that its values are similar for the raw solution and for the solution after cation exchange (figure 13). On the other hand, desalination of the solution after anion exchange requires slightly less energy (at the final salt concentration of about 2 meq/dm^3).

Taking into account that after ion exchange the chloride or sodium salts predominate in the solutions and that they do not produce troublesome sediments in the concentrate compartments of electrodialyzer, one should positively assess Donnan dialysis as a pretreatment process before electrodialysis.

4. CONCLUSIONS

1. Donnan dialysis with the Neosepta AFN membrane allows one to obtain higher (nearly twice) removal rate of NO_3^-, SO_4^{2-} and HCO_3^- anions from the solution, compared to the process with the Neosepta AMX membrane. The removal efficiency of HCO_3^- anions is significantly higher as well (78 and 40%, respectively).

2. The exchange of Ca^{2+} and Mg^{2+} cations for Na^+ ions is faster with the Selemion CMV membrane. Cation fluxes are by about 30% higher than those in the process with the Neosepta CMX membrane.

3. Donnan dialysis with the anion-exchange membrane (Neosepta AFN) is an advantageous pretreatment process before electrodialysis. In this process, the fluxes of troublesome anions (SO_4^{2-} and HCO_3^-) are about two times as high as the fluxes of cations (Ca^{2+} and Mg^{2+}) transported in Donnan dialysis with the cation-exchange membrane (Selemion CMV).

4. As a result of the exchange of NO_3^-, SO_4^{2-} and HCO_3^- for Cl^- ions, or the exchange of Ca^{2+} and Mg^{2+} cations for Na^+ ions, the limiting current density in electrodialysis increases. This enables desalination of the solution with the higher current density, which in turn reduces the process costs.

5. Electrodialysis of the solution after anion or cation exchange enables higher rate of the salt removal (up to 20%) and reduction of energy consumption (up to three times) for the transport of a given amount of salt compared to electrodialysis of the raw solution.

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WYMIANA JONOWA METODĄ DIALIZY DONNANA JAKO PROCES WSTĘPNEGO OCZYSZCZANIA PRZED ELEKTROLIZĄ

Dializę Donnana zastosowano do usuwania uciążliwych anionów (SO_4^{2-} i HCO_3^-) lub kationów (Ca^{2+}, Mg^{2+}) z roztworów wodnych przed ich odsalaniem metodą elektrodializy. W wyniku procesu z membraną anionowymienną (Neosepta AFN) udział molowy uciążliwych anionów (SO_4^{2-} i HCO_3^-) zmniejszył się do 4,3% wszystkich anionów. Dzięki wymianie kationów (z membraną Selemion CMV)

zmniejszył się udział molowy kationów Ca^{2+}, Mg^{2+} do 5,5% wszystkich kationów. Wskutek zmiany składu jonowego roztworu zaobserwowano wiele korzystnych zjawisk podczas elektrodialitycznego odsalania takich jak: wzrost granicznej gęstości prądu, większą szybkość usuwania soli (do 20%) oraz mniejsze zużycie energii potrzebnej do transportu określonej ilości soli (do trzech razy). Stwierdzono, że dializa Donnana z membraną anionowymienną (Neosepta AFN) jest korzystniejszym procesem wstępnego przygotowania wody przed elektrodializą niż ten sam proces z membraną kationowymienną, ponieważ strumienie usuwanych anionów są nawet 2 razy większe od strumieni kationów przenoszonych przez membranę kationowymienną.