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Separation Science and Technology

Publication details, including instructions for authors and subscription information:

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Evgueny Laktionov^a; Emmanuel Dejean^a; Jacqueline Sandeaux^a; Roger Sandeaux^a; Claude Gavach^a; Gerald Pourcelly^a

^a LABORATORY OF MATERIALS AND MEMBRANE PROCESSES, MONTPELLIER, CEDEX 5, FRANCE

Online publication date: 01 November 1999

To cite this Article Laktionov, Evgueny , Dejean, Emmanuel , Sandeaux, Jacqueline , Sandeaux, Roger , Gavach, Claude and Pourcelly, Gerald(1999) 'Production of High Resistivity Water by Electrodialysis. Influence of Ion-Exchange Textiles as Conducting Spacers', Separation Science and Technology, 34: 1, 69 – 84

To link to this Article: DOI: 10.1081/SS-100100637

URL: <http://dx.doi.org/10.1081/SS-100100637>

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Production of High Resistivity Water by Electrodialysis. Influence of Ion-Exchange Textiles as Conducting Spacers

EVGUENY LAKTIONOV, EMMANUEL DEJEAN,
JACQUELINE SANDEAUX, ROGER SANDEAUX,
CLAUDE GAVACH, and GERALD POURCELLY*

LABORATORY OF MATERIALS AND MEMBRANE PROCESSES
UMR 5635

CNRS 1919 ROUTE DE MENDE, 34293 MONTPELLIER CEDEX 5, FRANCE

ABSTRACT

Production of high resistivity water was investigated by electrodialysis (ED) using either inert or conducting spacers. Ion-exchange textiles were used as conducting spacers. Experiments were performed on a preindustrial scale with a pilot consisting of nine two-compartment cells, each membrane having an effective area of 176 cm². Three configurations of the ED stack were investigated for the dilution compartment: EDIT-(2) with a 2-mm thick ion-exchange textile, and ED-(2) or ED-(0.4), with a 0.4-mm thick inert spacer inserted between 2 or 0.4 mm thick dilution compartments, respectively. The textile induces a moderate increase in the pressure drop between the inlet and outlet of the stack. The performances of the different processes were compared under various experimental conditions of pH, nitrogen bubbling throughout the feed solution, flow rate, and current density. The results show that for an inlet conductivity of 10–15 $\mu\text{S}\cdot\text{cm}^{-1}$, a flow rate of $2.2 \times 10^{-5} \text{ m}^3\cdot\text{s}^{-1}$, and an applied voltage of 80 V, an outlet conductivity of 0.4 $\mu\text{S}\cdot\text{cm}^{-1}$ was obtained with the EDIT process, while no value lower than 5 $\mu\text{S}\cdot\text{cm}^{-1}$ was obtained with the ED process using both stacks.

Key Words. Electrodialysis; Continuous electrodeionization; Ion-exchange textile; Dilute solution; Demineralization; Mass transfer coefficient

* To whom correspondence should be addressed. FAX: (33) 467042820. E-mail: pourcell@admcnrs.cnrs-mop.fr

INTRODUCTION

For the treatment of low concentration solutions, electrodialysis (ED) performances are significantly improved by the introduction of ion-exchange materials instead of inert spacers between the ion-exchange membranes (1, 2). Electrodialysis has been used for the production of ultrapure water for the semiconductor industry. The combination of electrodialysis with mixed bed ion-exchange resins is attractive because completely deionized water is obtained without chemical regeneration of the ion-exchange resins (3). This process, named ionpure continuous deionization (4), was commercialized by Millipore in 1987. However, if a high level of water purity is to be obtained, some parameters will decrease the efficiency of resins within electrodialysis cells. One major problem involves conserving the resins between the membranes: these tend to be entrained by the liquid flow. Furthermore, it has been shown that in order to maintain maximum operating efficiency with minimum electrical resistance in the cell, the cation-exchange resins must be located near the cation-exchange membrane, and the anion-exchange resins near the anion-exchange membrane, which is difficult to achieved at large scale (5). The resins are also subject to irreversible surface degradation caused by friction between the spheres (erosion phenomenon); they must be both chemically and physically stable and resistant. In electrodeionization, using ion-exchange textiles rather than resins entails the same advantages as the latter but these textiles also have some characteristics which render them superior. For example, textiles have a much larger exchange surface with low resistance to solution flow, are easy to handle, have better distribution of exchange sites, and exhibit high electrical conductivity. In addition, they are easy to install in an electrodialysis cell. This results in an increased mass transfer rate compared to resins (6, 7).

Preliminary studies carried out with a laboratory cell have shown higher current and demineralization efficiencies and lower energy consumption when ion-exchange textiles are clamped between alternating anion- and cation-exchange membranes (8). In order to optimize the process, different kinds of textiles—amphoteric with mixed anion- and cation-exchange sites, or bipolar with various alternations of cation- and anion-exchange layers—were tested (9). The highest efficiency results from the highest desalination degree obtained with the lowest pressure drop and voltage.

In the present study, electrodeionization using ion-exchange textiles (EDIT) was carried out at a preindustrial pilot scale in order to obtain ultrapure water required for electronics and pharmaceuticals. Thus, the operating solutions were dilute NaCl solutions having low conductivities about $10\text{--}15\ \mu\text{S}\cdot\text{cm}^{-1}$. These



solutions were deionized by EDIT and classical ED using inert spacers under various experimental conditions of flow rate and current density. By using bipolar textiles which were previously selected for their efficiency in EDIT (9), the present paper reports on a comparison of the two processes. EDIT and ED performances are compared in terms of pressure drop, electric resistance of the cell, and conductivity of the outlet solution. In addition, the influence of pH and nitrogen bubbling throughout the operating solution on mass transfer was studied.

EXPERIMENTAL

Ion-Exchange Materials

The anion- and cation-exchange membranes were Selemion AMV and CMV membranes, produced by Asahi Glass.

The ion-exchange textile (IET), commercialized by the Institut Textile de France, was obtained from copolymerization of cellulose macromolecular chains which constitute a three-dimensional network without crosslinking (10). The matrix is an artificial polymer (viscose) made from modified natural cellulose. The chemical structure consists of long chains of repeating units which are composed of two glucopyranoside rings (11). The thickness of the textile was measured by means of a micrometer without applying any pressure.

A bipolar textile composed of anion- and a cation-exchange layers mechanically juxtaposed was used in the present study. The main characteristics of the textiles are shown in Table 1.

Experimental Scheme

Experiments were performed with preindustrial pilot-scale equipment previously described (12). The ED stack consisted of 9 two-compartment cells, i.e., 9 dilution compartments (DC) and 9 concentration compartments (CC).

TABLE 1
Main Characteristics of the Bipolar Textile

| | |
|--|-------|
| Thickness, mm | 2 |
| Porosity | 0.89 |
| Density of anionic sites: meq/dm ³ dry | 67 |
| meq/g dry | 0.7 |
| Density of cationic sites: meq/dm ³ dry | 70 |
| meq/g dry | 0.75 |
| Mass ratio A/C | 50/50 |

Narrow CCs of 0.4 mm thickness were formed by thin flow spacers made from a nylon web having 50 meshes per cm^2 . Either inert spacers or conducting spacers were introduced between the ion-exchange membranes to form the DCs. Bipolar textiles of 2 mm thickness were used as conducting spacers. They were compressed between the ion-exchange membranes with the anionic layer on the anion-exchange membrane and the cationic layer on the cation-exchange membrane. Thus, inert spacers with a thickness of 0.4 mm were used in the CCs and 2 mm thick textiles were used in the DCs. All spacers had the same length (32 cm) and width (5.5 cm), giving an effective membrane area of 176 cm^2 .

The hydraulic schemes of the process are presented in Fig. 1. In the so-called direct flow hydraulic mode (Fig. 1a), the same operating solution passed from the feed tank through the dilution and concentration compartments and then exited.

The electrode rinse solution circulated as a batch system in the electrode chambers (EC). Its concentration was controlled and maintained identical to the operating solution in order to minimize the counterdiffusion of ions from the electrode chambers to the CCs and DCs. The osmotic transference of water between the chambers can be neglected in this case.

In the recirculating hydraulic mode (Fig. 1b), the feed solution circulated as a batch system through the DCs. Nevertheless, a part of the solution after being deionized was directed to the CCs and ECs and then exited. In this case the NaCl concentration of the feed solution decreased with time from 4 mM to $2 \mu\text{M}$.

Both electrodes were made of dense graphite. Experiments were performed in the potentiostatic mode (constant applied voltage) by using a power supply.

The conductivity, pH, flow rate, temperature, and pressure were controlled at the DC's, CC's, and EC's inlet and outlet that corresponds to Points (2)–(6) of the schemes in Fig. 1.

RESULTS

In order to compare the performances of electrodialysis when inert spacers were substituted by conducting spacers, three configurations of stack were investigated. The corresponding electrodialysis operations were designed as follows:

EDIT-(2): the DCs (2 mm thick) were filled with 2 mm thick ion-exchange textiles

ED-(2): the DCs (2 mm thick) were filled with 0.4 mm thick inert spacers

ED-(0.4): the DCs (0.4 mm thick) were filled with 0.4 mm thick inert spacers



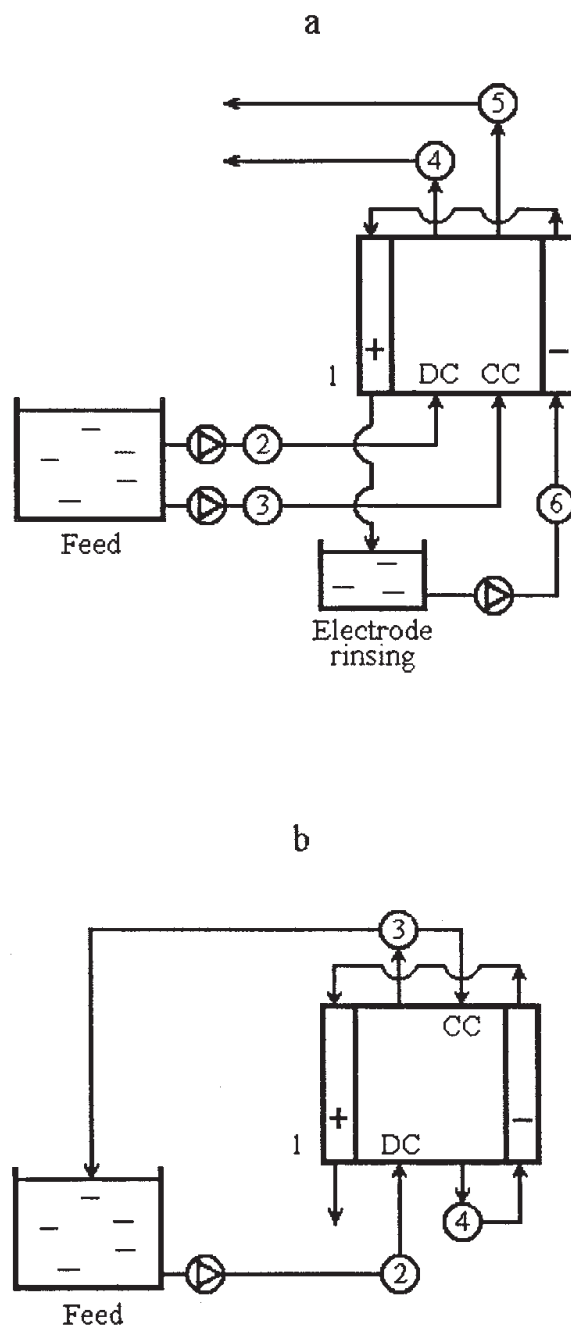


FIG. 1 Hydraulic schemes of the process for direct flow (a) and recirculating (b) modes. (1) Stack, (2)–(6) control points (conductivity, pH, flow rate, temperature, pressure).



The feed solutions had conductivities of about $10\text{--}15 \mu\text{S}\cdot\text{cm}^{-1}$. These values are usual for the outlets of reverse osmosis systems (13), and water of this quality is used for further treatment to obtain ultrapure water.

The operating conditions were determined in the recirculating hydraulic mode by studying the influence of pH and nitrogen bubbling throughout the feed solution on sodium transfer. For these experiments, NaCl solutions with higher conductivities were used ($500 \mu\text{S}\cdot\text{cm}^{-1}$).

Influence of pH and Nitrogen Bubbling

Keep in mind that the main goal of this work was the production of very low conductivity water. Therefore, it was necessary not only to remove the NaCl salt by electrodialysis but also to avoid the formation of parasite ions in the feed solution. On the one hand these ions are hydrogen carbonates which result from the dissolution of carbon dioxide from air, and on the other hand they are protons due to the water-splitting phenomenon occurring at the membrane–solution interface when anion-exchange membranes in contact with dilute media are subjected to an electric field (14). The mass transfer coefficient of i ions is determined as a flux of these ions normalized by their concentration in the bulk solution (15):

$$k_i = j_i/C_i \quad (1)$$

When applied to electrodialysis systems, this parameter is defined as the density of the flux resulting from the removal of salt ions from a DC, normalized by the DC's inlet concentration of the operating solution:

$$k_{\text{Na}} = j_{\text{Na}}/C_{\text{Na},\text{in}} \quad (2)$$

The physical meaning of this parameter is the mass transfer rate of a matter unit through a membrane surface unit. The flux density of sodium ions, j_{Na} , can be calculated from the salt cation loss at the outlet of the dilution compartment according to:

$$j_{\text{Na}} = (C_{\text{Na},\text{in}} - C_{\text{Na},\text{out}}) \frac{Q}{nA} \quad (3)$$

where Q is the flow rate, A is the membrane area, and n is the number of DCs.

$C_{\text{Na},\text{in}}$ and $C_{\text{Na},\text{out}}$, which are the Na^+ concentrations at the inlet and outlet of the dilution compartment, respectively, are calculated from experimental values of pH, temperature, and conductivity by using a computer program, taking into account values of the ionic mobilities. (See Appendix 1.)

The value of k_{Na} was calculated using



$$k_{\text{Na}} = \frac{(C_{\text{Na},\text{in}} - C_{\text{Na},\text{out}})}{C_{\text{Na},\text{in}}} \frac{Q}{nA} \quad (4)$$

In order to decrease both the formation of HCO_3^- ions and the amount of protons, an inert gas, such as nitrogen, was bubbled throughout the operating solution, and the pH of the solution was maintained close to the neutral value. Stabilization of the pH value was performed manually by the addition of small quantities of a concentrated solution of NaOH with intensive mixing as often as possible. Thus, different ED-(0.4) operations were carried out starting with a 4-mM NaCl solution ($500 \mu\text{S}\cdot\text{cm}^{-1}$) by using the recirculating hydraulic mode (Fig. 1b) under the following experimental conditions:

Without nitrogen bubbling at pH 5 for Experiment (1)

With nitrogen bubbling at pH 5 for Experiments (2) and (3)

With nitrogen bubbling at pH 7 for Experiment (4)

The NaCl concentration of the feed solution is decreasing with time, when operating in the recirculating hydraulic mode. Figure 2 shows the variations of k_{Na} with NaCl concentration. For Experiments (1)–(3), the curves show a maximum for a NaCl concentration of 10^{-5} M, identical to that of protons (pH 5).

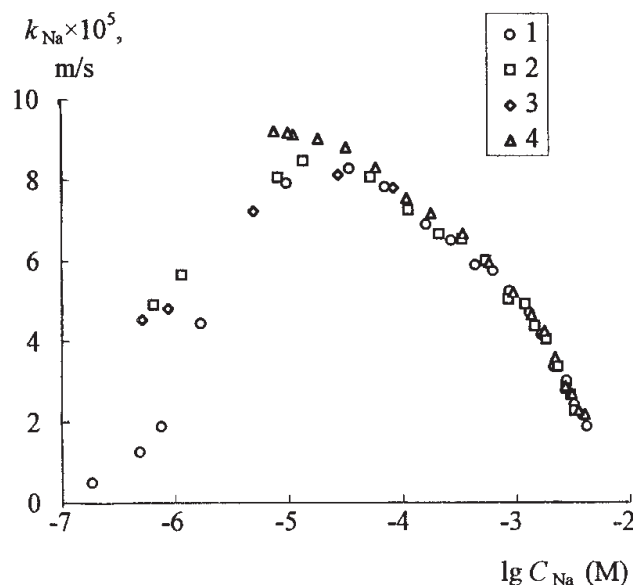


FIG. 2 Concentration dependence of the transfer rate of sodium ions for the ED-(0.4) process under the recirculating hydraulic mode. (1) Without nitrogen bubbling at pH 5, (2) and (3) with nitrogen bubbling at pH 5, (4) with nitrogen bubbling at pH 7. $Q = 1.7 \times 10^{-5} \text{ m}^3\cdot\text{s}^{-1}$, $U = 33 \text{ V}$.



In the 10^{-5} – 10^{-2} M NaCl range, the mass transfer rate, k_{Na} , increases as the sodium concentration decreases. This phenomenon is attributed to both the degradation of the diffusion layer close to the ion-exchange membrane surface due to the electroconvective mechanism (16) and the exaltation effect of the salt ions flux (17). No significant influence of the experimental conditions on the k_{Na} values was observed for a Na^+ concentration higher than 10^{-4} M. Nitrogen bubbling improves sodium transfer in the 10^{-5} – 10^{-4} M NaCl range.

In the 10^{-7} – 10^{-5} M NaCl range, k_{Na} decreases markedly with sodium concentration for Experiments (1)–(3) because the $[\text{H}^+]/[\text{Na}^+]$ concentration ratio is favourable to protons. Moreover, for Experiment (1) without nitrogen bubbling, the presence of HCO_3^- ions contributed to the electrical conductivity and introduced an error in the method of calculating the k_{Na} value. For Experiment (4) with nitrogen bubbling and a stabilized pH 7, the results exhibit a further growth of the mass transfer coefficient up to a NaCl concentration of 7×10^{-6} M. Below this concentration, the amount of Na^+ ions which needed to be added in the form of NaOH to the feed solution to maintain the pH at 7 is of the same order of magnitude as that transferred by the electrodialysis process. In these conditions, the experiment does not agree with the main goal of demineralization.

Therefore, for the demineralization of very dilute solutions, the pH value must be stabilized close to 7 in order to decrease the competitive transfer between Na^+ and H^+ ions. Under this experimental condition, nitrogen bubbling is necessary because 60% of the dissolved CO_2 is transformed into HCO_3^- ions instead of the 10% transformed at pH 5.5 (18).

Comparative EDIT-(2) operations were achieved by using the direct hydraulic mode (Fig. 1a) to study the influence of the inert gas on process performance, expressed by the degree of demineralization, γ , as follows:

$$\gamma = \frac{\kappa_{\text{in}} - \kappa_{\text{out}}}{\kappa_{\text{in}}} \quad (5)$$

Figure 3 clearly shows that bubbling nitrogen through the operating solution significantly increases the degree of demineralization. The conductivity of the inlet solution, κ_{in} , was about $10 \mu\text{S}\cdot\text{cm}^{-1}$, which corresponds to about a 10^{-4} M NaCl solution. For this concentration it is not necessary to monitor the pH of the feed solution, as shown in Fig. 2. A value of $7.78 \times 10^{-5} \text{ m}\cdot\text{s}^{-1}$ was found for the mass transfer rate, k_{Na} , which agrees with the curve of Fig. 2.

Therefore, all the following experiments were performed with both the direct flow hydraulic mode (single pass) and nitrogen bubbling throughout the feed solution.



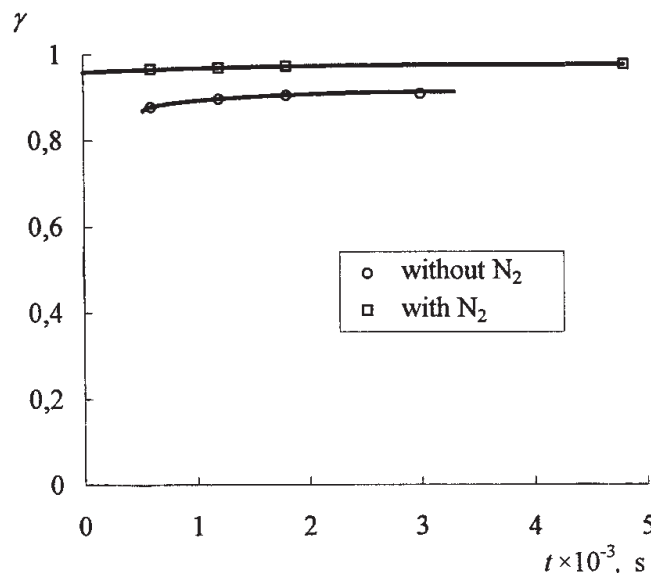


FIG. 3 Influence of nitrogen bubbling throughout the feed solution on the degree of demineralization for the EDIT-(2) process under the direct hydraulic mode. $\kappa_{in} = 10\text{--}15 \mu\text{S}\cdot\text{cm}^{-1}$, $Q = 2.2 \times 10^{-5} \text{ m}^3\cdot\text{s}^{-1}$, $U = 80 \text{ V}$.

Influence of the Textile on the Pressure Drop

Hydraulic characteristics of stacks containing either ion-exchange textiles (2 mm thickness) or spacers with a small intermembrane distance (0.4 mm thickness) such as that usually used in industrial application (8) are presented in Fig. 4. As shown, the textile obviously induces an increase in the pressure drop between the inlet and outlet of the stack despite hydrophilic properties and porosity close to those of the inert spacers that were used (19). The consequence is a slight increase in the economic cost of the process. Nevertheless, the order of magnitude of this increase is not very high when the high flow rates used (up to $5 \times 10^{-5} \text{ m}^3\cdot\text{s}^{-1}$) are taken into account, and it is not a major drawback for the EDIT process.

Influence of the Textile on the Electric Resistance

Different current intensities were measured in the ED and EDIT operations for an identical applied voltage of 80 V. The current density values used were 45.5, 11.4, and $24.4 \text{ A}\cdot\text{m}^{-2}$ for EDIT-(2), ED-(2), and ED-(0.4), respectively. The introduction of textiles in the DCs has a great influence on the electric resistance of the cell, and consequently results in a decrease in the economic



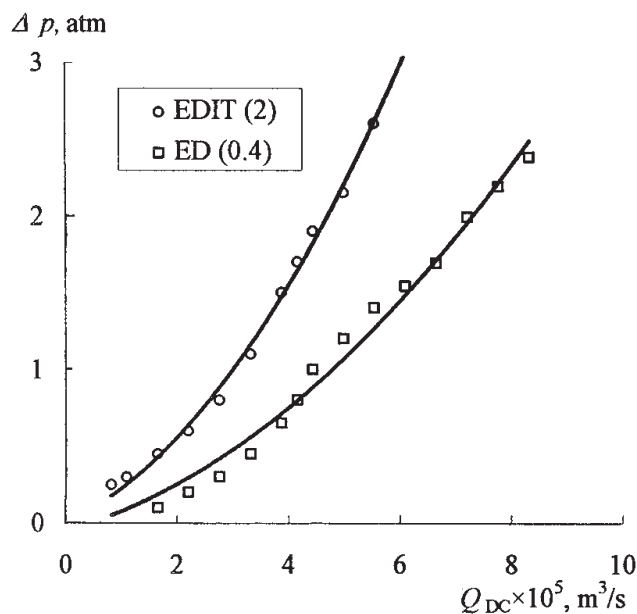


FIG. 4 Flow rate dependence of the pressure drop between inlet and outlet of the stack.

cost of the process. As expected, a decrease in the intermembrane distance significantly reduces the electric resistance of the stack used in classical ED. Nevertheless, even with narrow compartments, ED is not competitive compared with EDIT.

Influence of the Textile on the Degree of Demineralization

The three electrodeionization processes, EDIT-(2), ED-(2), and ED-(0.4), were also compared in terms of degree of demineralization, γ , expressed by Eq. (4). Experiments were performed under identical flow rate conditions ($2.2 \times 10^{-5} \text{ m}^3 \cdot \text{s}^{-1}$) and applied voltage (80 V). Figure 5 shows that the use of ion-exchange textiles as conducting spacers markedly increases the degree of demineralization which becomes close to unity, the maximum value. The tendencies [falling-off for ED-(2) and rising for ED-(0.4)] are not significant. For an inlet conductivity of $10\text{--}15 \mu\text{S} \cdot \text{cm}^{-1}$, an outlet conductivity of $0.4 \mu\text{S} \cdot \text{cm}^{-1}$ was obtained with the EDIT process, while no value lower than $5 \mu\text{S} \cdot \text{cm}^{-1}$ was obtained with the ED process using both stacks.



Influence of Flow Rate on the Outlet Conductivity

The operating solution was pumped through the stack with two constant flow rates (3.3×10^{-5} or $2.2 \times 10^{-5} \text{ m}^3 \cdot \text{s}^{-1}$) for the DCs and a varying flow rate for the CCs. Only two stack configurations were compared: EDIT-(2) and ED-(0.4), the latter corresponding to industrial applications.

Another parameter characterizing the demineralization performance is the ratio of inlet and outlet conductivity of the feed solution, $\kappa_{\text{in}}/\kappa_{\text{out}}$. Figure 6 shows the following points:

1. For a given inlet solution, the demineralization performance is significantly better for the EDIT-(2) process than for ED-(0.4): $\kappa_{\text{in}}/\kappa_{\text{out}}$ values are three and ten times higher for EDIT than for ED.
2. For the EDIT process, the best results were obtained for the lowest flow rates. Nevertheless, demineralization decreases slightly when the flow ratio, $Q_{\text{DC}}/Q_{\text{CC}}$, increases.
3. For the ED process, no significant influence of the flow rate and voltage on the demineralization performances was observed.

These results show that the mass transfer which takes place from site to site through the textile leads to substantial improvements in the deminer-

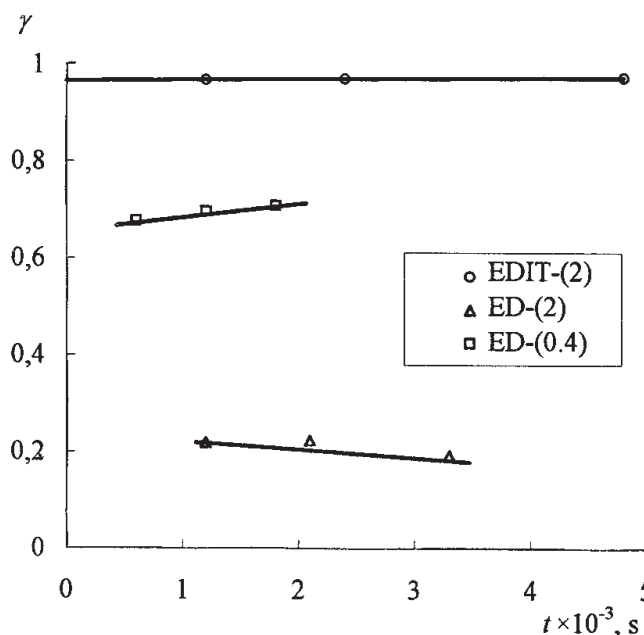


FIG. 5 Degree of demineralization for EDIT and ED processes under the direct hydraulic mode. $\kappa_{\text{in}} = 10\text{--}15 \text{ } \mu\text{S} \cdot \text{cm}^{-1}$, $Q = 2.2 \times 10^{-5} \text{ m}^3 \cdot \text{s}^{-1}$, $U = 80 \text{ V}$.



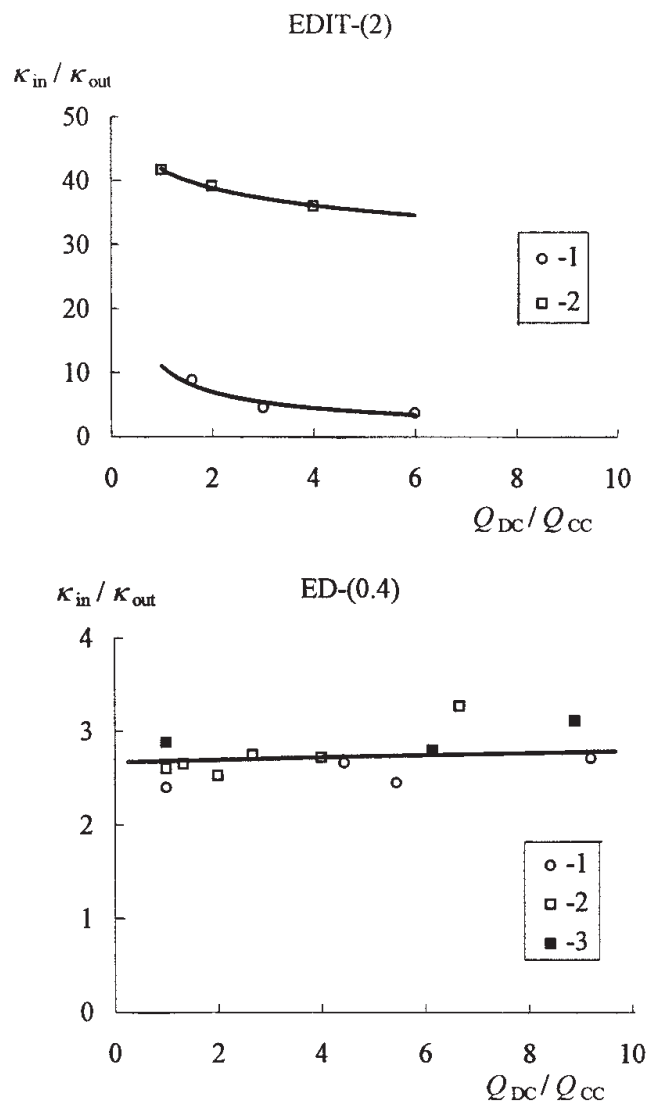


FIG. 6 Flow rate dependence of inlet and outlet conductivity of the feed solution. (1) $Q_{DC} = 3.3 \times 10^{-5} \text{ m}^3 \cdot \text{s}^{-1}$, $U = 80 \text{ V}$. (2) $Q_{DC} = 2.2 \times 10^{-5} \text{ m}^3 \cdot \text{s}^{-1}$, $U = 80 \text{ V}$. (3) $Q_{DC} = 2.2 \times 10^{-5} \text{ m}^3 \cdot \text{s}^{-1}$, $U = 40 \text{ V}$.

alization. An increase in the flow rate, which decreases the kinetics of transfer, consequently decreases the EDIT process performance. In classical ED where there is no mass transfer through inert spacers, variations of flow rate in the range studied do not induce significant variations of demineralization.



In the EDIT process the slight decrease observed when the gap between the Q_{DC} and Q_{CC} flow rates increases can be due to a difference of pressure between the DC and CC compartments. Since the DC flow rate remains at high values (2.2×10^{-5} or $3.3 \times 10^{-5} \text{ m}^3 \cdot \text{s}^{-1}$), the DC pressure increases when the CC flow rate decreases. In these conditions the intermembrane distance increases and contact between the textile and the membranes becomes lower, inducing preferential pathways into the DC compartments. Besides, the osmosis phenomenon occurring from the DC to the CC compartments becomes more favourable under a difference of pressure. Therefore, it is important to keep the same operating conditions in the EC, DC, and CC.

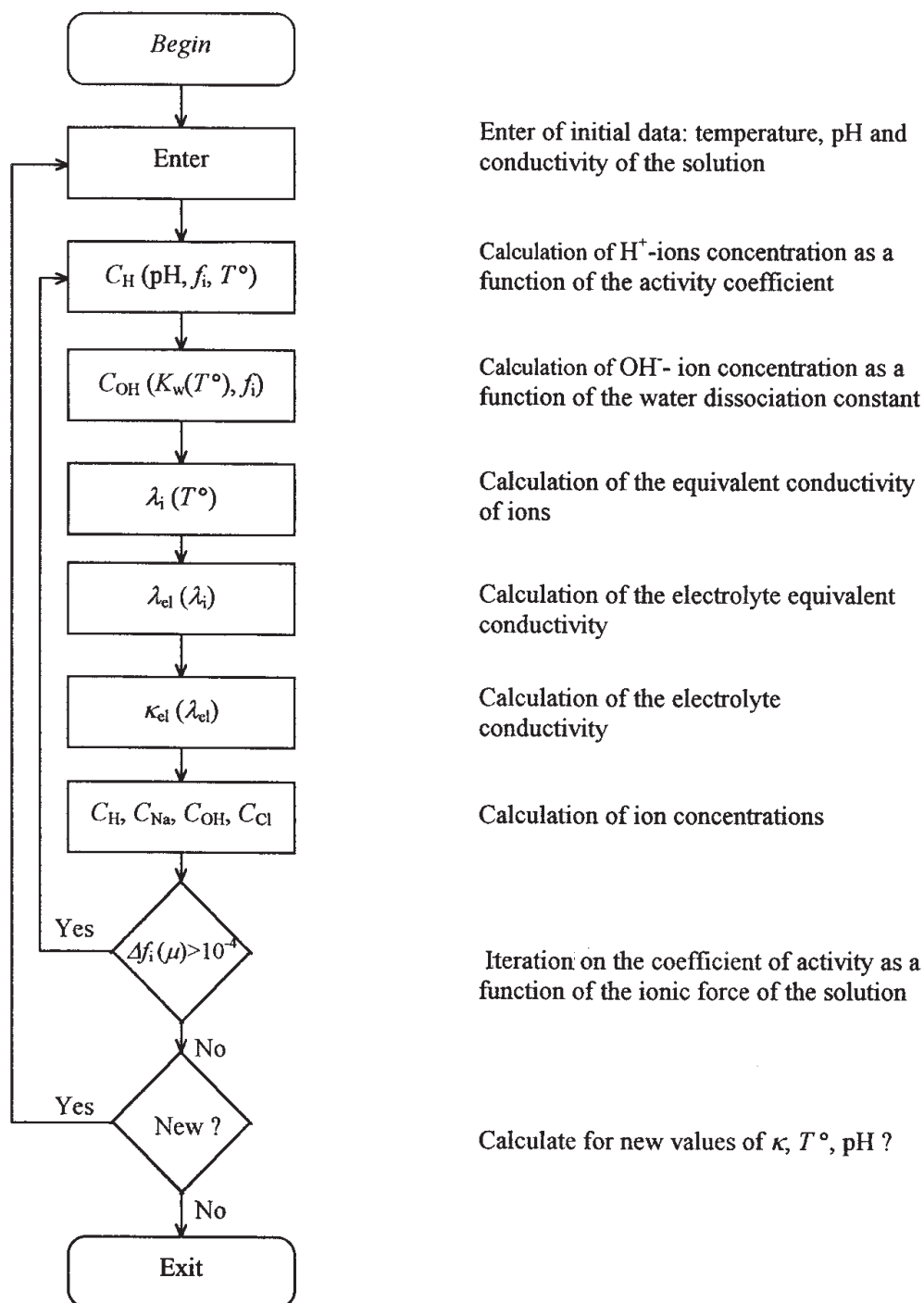
CONCLUSION

The EDIT process, which uses ion-exchange textiles as conducting spacers, is promising for the production of high quality water when the high degree of demineralization obtained in comparison with the classical ED process using inert spacers is taken into account. For a conductivity of the inlet solution of $10\text{--}15 \text{ }\mu\text{S} \cdot \text{cm}^{-1}$, a flow rate of $2.2 \times 10^{-5} \text{ m}^3 \cdot \text{s}^{-1}$, and an applied voltage of 80 V, an outlet conductivity of $0.4 \text{ }\mu\text{S} \cdot \text{cm}^{-1}$ was obtained with the EDIT process, while no value lower than $5 \text{ }\mu\text{S} \cdot \text{cm}^{-1}$ was obtained with the ED process. Results obtained from commercialized systems running with ion-exchange resins give a water conductivity twice as low as that of the EDIT process presently used. Nevertheless, the textiles used in this work were not optimized and had an ion-exchange density 15 times lower than those of ion-exchange resins. In previous studies carried out on a laboratory scale, bipolar textiles with an ion-exchange density twice as great than those used here gave very good results of deionization (9). Thus, the EDIT process using optimized textiles would give equivalent and even better results than electrodeionization on resins. Furthermore, ion-exchange textiles have some advantages compared to ion-exchange resins (6–9). Because of their shape, they are well-adapted for arrangement between ion-exchange membranes, and dismantling of stacks involving these materials is easier. Moreover, the porous and hydrophilic structure of textiles does not induce a significant pressure drop, and consequently they allow high operating flow rates. All these aspects have to be considered in further industrial applications, keeping in mind that the main development work to be done is the optimization of ion-exchange textiles.



APPENDIX 1

Organization Chart for Calculating the Concentrations of Na^+ , Cl^- , H^+ , and OH^- Ions in Solution Containing Their Mixture



NOMENCLATURE

| | |
|---|--|
| A | membrane area (m^2) |
| $C_{\text{Na,in}}, C_{\text{Na,out}}$ | concentrations of sodium ions at the inlet and outlet, respectively, of the desalination channel (M) |
| i | current density ($\text{A}\cdot\text{m}^{-2}$) |
| j_{Na} | flux density of sodium ions ($\text{mol}\cdot\text{m}^{-2}\cdot\text{s}^{-1}$) |
| k_{Na} | mass transfer coefficient of sodium ions ($\text{m}\cdot\text{s}^{-1}$) |
| n | number of cell pairs in the stack |
| p | pressure (bar) |
| Q | flow rate ($\text{m}^3\cdot\text{s}^{-1}$) |
| $Q_{\text{DC}}, Q_{\text{CC}}$ | flow rate through the diluate and concentrate compartment, respectively ($\text{m}^3\cdot\text{s}^{-1}$) |
| t | time (s) |
| U | potential difference across the cell (V) |
| γ | degree of demineralization |
| $\kappa_{\text{in}}, \kappa_{\text{out}}$ | conductivity at the inlet and outlet, respectively, of the desalination channel ($\mu\text{S}\cdot\text{cm}^{-1}$) |

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Received by editor December 31, 1997

Revision received April 1998



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