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# Water Demineralization by Electrodeionization with Ion-Exchange Textiles. Comparison with Conventional Electrodialysis

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## ABSTRACT

An electrodeionization process has been developed using ion-exchange textiles (EDIT) instead of ion-exchange resins as currently used for the production of ultrapure water. The study was carried out with a laboratory pilot. Each membrane had an active area of 20 cm$^2$. Experiments were performed with NaCl solutions at low concentration (from 3 to 300 mg·L$^{-1}$) under various experimental conditions of flow rate and current density. Different configurations of textile were tested: the diluate compartment of the electrodialysis stack was filled by either alternating layers of cation or anion-exchange textiles, or one layer of amphoteric textile or bipolar textile. The best results were obtained with the latter configuration. The study allowed comparison of the performances of EDIT with conventional electrodialysis (ED) in terms of current efficiency, demineralization ratio, and energy losses. A significant improvement was obtained by introducing the textile. At the lowest concentration (3 mg·L$^{-1}$) and the highest flow rate (20.8 cm·s$^{-1}$), the current and demineralization efficiencies were 150% higher with EDIT than with ED.

*Key Words.* Electrodeionization; Ion-exchange textile; Low concentration; Demineralization; Current efficiency

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## INTRODUCTION

Water is the most used fluid not only for domestic but also for industrial applications. The required purity level of the water depends on the application, but increasingly demands are for a product with parts per billion of contaminants with expectations of parts per trillion to be more common in the future according to international norms. The major industrial areas for high purity water are boiler feedwater, electronics, metal finishing, medical and pharmaceuticals, and packaging. Several technologies can be used in order to produce pure water for these applications. They are effective to different degrees depending upon the application, and in many cases more than one method is employed to achieve the final desired quality.

In water desalination, several processes are in direct competition. Conventional processes like distillation and ion exchange (IE) have an economical advantage over membrane processes for very high and very low salt concentrations, respectively, while electrodialysis (ED) is more suitable to demineralize solutions in the 0.3 to 6 $g{\cdot}L^{-1}$ concentration range (1).

The use of a combined electrodialysis and ion-exchange system has been introduced on a commercial level as a means of producing high purity water from potable water sources (2). In this hybrid process an ion-exchange media, usually resin beads, fills the space between alternating anion and cation-exchange membranes in conjunction with a normal DC field. The use of ion-exchange resins in the diluate compartment of the ED stack has several advantages: on the one hand, it strongly reduces the electric resistance of the cell and consequently decreases the polarization phenomenon which occurs in low concentration ED applications; on the other hand, when this phenomenon takes place, protons ($H^+$) and hydroxyl ions ($OH^-$) produced from water splitting lead to an electrochemical regeneration of the resin. Under these conditions the system has the removal efficiency of a mixed-bed ion-exchange column without the need for chemical regeneration cycles, resulting in continuous deionization operation (CDI). The economics of CDI are particularly attractive at feed salt concentrations lower than 10 $g{\cdot}L^{-1}$ (3).

Ampholytic ion-conducting spacers and interwoven cation and anion-exchange spacers have also been used to reduce polarization in ED systems (4, 5).

The aim of this work is to substitute in a CDI system the ion-exchange resins by a porous ion-exchange material: ion-exchange textiles (IET) manufactured by the Institut Textile de France (6). These exchangers are nonwoven fibrous materials. Their nature and geometry are different from the previously mentioned spacers made of ion-exchange fibers.

Thanks to their structure, they have the following advantages in comparison with the beads of ion-exchange resins: hydrophilicity due to the cellulosic polymer network, faster ion-exchange kinetics due to the absence of crosslinking, lower poisoning by the hydrophobic macro-ions due to the macroporous structure, better contact with ion-exchange membranes due to the sheet shape, easier storage and possible burning.

A preliminary study has shown that the electrodeionization process using ion-exchange textiles (EDIT) can be used at flow rates significantly greater than the electrodeionization process using ion-exchange resins (EDIR) (7). The experiments have been performed with a laboratory cell. The solution treated was a NaCl solution of 300 $mg \cdot L^{-1}$. Below this salt concentration of the feed solution, ED is not economically competitive as compared to other demineralization processes (1).

The present study was carried out with more dilute NaCl solutions, varying from 3 to 300 $mg \cdot L^{-1}$, and circulating at higher circulation rates, up to 20.8 $cm \cdot s^{-1}$. The performances of EDIT and ED were compared in terms of current efficiency, demineralization ratio, and energy losses under similar experimental conditions.

## EXPERIMENTAL

### Ion-Exchange Materials

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

The ion-exchange textiles, produced by the Institut Textile de France, were obtained from copolymerization of cellulosic macromolecular chains which constitute a tridimensional network without crosslinking. The basic polymer is an artificial polymer (viscose) which is made from modified natural cellulose. The chemical structure consists of long chains of repeating units which are composed of two glucopyranoside rings (8).

The first step in the synthesis of ion-exchange textiles is the preparation of a nonwoven sheet. The fibers of viscose are separated, then mixed by carding to make a thick fibrous layer. Various layers are bound to form a sheet with a thickness varying from 2 to 8 mm.

The second step is the grafting. Before irradiation, the textile is imbibed with the monomer. An electron beam is used to create on the polymer chains free radicals able to react with a monomer. The activation is carried out continuously on moving textile sheets. An energy of 350 keV allowed a homogeneous penetration inside the material.

The monomer used was acrylamid-methyl-propane-sulfonic acid and quaternary ammonium methacrylate for cation and anion-exchange groups, respectively.

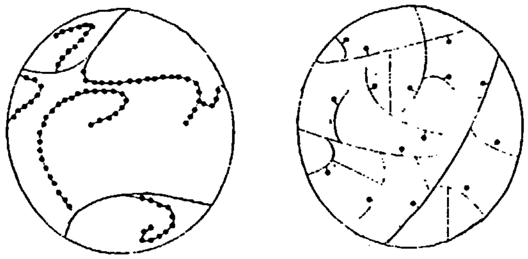

FIG. 1 Schematic representation of ion-exchange textiles and ion-exchange resins.

The ion-exchange sites in the textiles are distributed along the grafted polymer chains, while in the resins each ion-exchange site is fixed on the matrix, as schematically represented in Fig. 1.

Different kinds of textiles were used: anion-exchange textiles, cation-exchange textiles, amphoteric textiles where anion- and cation-exchange sites are mixed, and bipolar textiles where an anion-exchange layer and a cation-exchange layer are mechanically juxtaposed. The thickness can be adapted to different stacks. It was 8 mm in the earlier study (8) and 2 mm in the present study. The main characteristics of the textiles used are given in Table 1.

## Equipment Description

The flow diagram of ED/EDIT set-up used here is shown in Fig. 2.

Figure 3 is a schematic representation of the laboratory cell. It was composed of five compartments separated by ion-exchange membranes. Each separating membrane had an effective area of 20 cm$^2$. The thickness of each compartment was 2 mm.

The central compartment is the diluate compartment (D). It was constituted by a PVC frame which can be empty or filled with ion-exchange textiles compressed between the anion- and cation-exchange membranes. In this compartment the solution flowed continuously as a single pass system. A Masterflex DSCD pump was used.

On both sides of the central compartment are the concentrate compartments (C). The compartments E are the electrode rinse compartments. In the concentrate and electrode compartments, NaCl and $K_2SO_4$ solutions circulated, respectively, as a batch system by means of Asti 42/50W pumps.

ED and EDIT operations were carried out in the potentiostatic mode using a Lambda LQ 534W power supply.

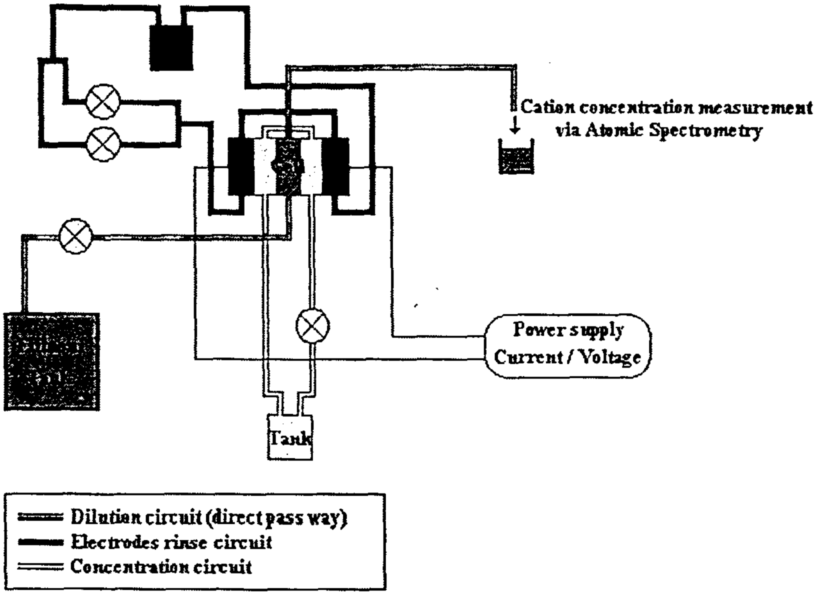

FIG. 2 Schematic flow diagram of ED/EDIT system.

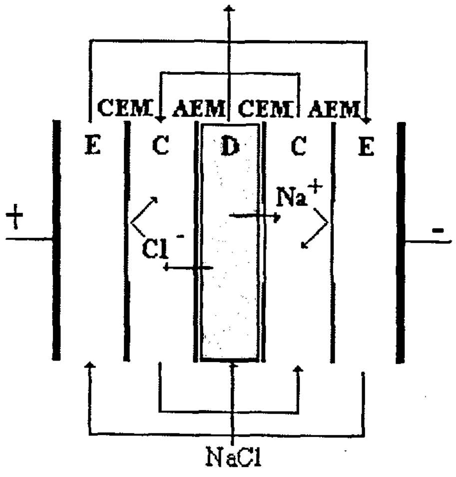

FIG. 3 Schematic design of the laboratory cell.

## Operating Conditions

Before all ED/EDIT operations, the ion-exchange membranes on both sides of the central compartment and the ion-exchange textiles introduced into this compartment were in the $Na^+$ and/or $Cl^-$ form. The ion-exchange membranes which separated the concentrate and electrode compartments were in the $K^+$ or $SO_4^{2-}$ form for the anodic and cathodic compartments, respectively.

The operating conditions were the following:

- The solutions treated were NaCl solutions. Four concentrations ($C_{NaCl}$) were used: 3, 30, 150, and 300 $mg{\cdot}L^{-1}$.
- These solutions flowed into the diluate compartment at four flow rates ($Q$) of 12, 30, 48, and 60 $L{\cdot}h^{-1}$, corresponding to circulation rates ($v$) of 4.2, 10.4, 16.6, and 20.8 $cm{\cdot}s^{-1}$.
- 2 liters of 0.5 M NaCl solution circulated in the concentrate compartments with a flow rate of 90 $L{\cdot}h^{-1}$;
- 2 liters of 0.5 M $K_2SO_4$ solution circulated in the electrode compartments with a flow rate of 130 $L{\cdot}h^{-1}$.
- The current densities ($i$) varied from 5 to 100 $mA{\cdot}cm^{-2}$.

The sodium concentration of solutions at the outlet of the dilution circuit was determined by atomic absorption spectrometry (Varian AA20) when steady-state was reached. The equilibrium time, which varied with the flow rate, was some 10 seconds.

## RESULTS

The results of the electrodeionization process with ion-exchange textiles and conventional electrodialysis without ion-exchange material in the diluate compartment were compared under various experimental conditions of current density, flow rate, and salt concentration of the feed solution.

The values of current efficiency ($R_F$), demineralization ratio ($R_D$), and energy losses ($W$) were calculated for each experiment.

Under a constant current, the ED current efficiency is given by the classical relation (9)

$$R_F = \frac{z_i J_i F}{i} \tag{1}$$

where $z_i$ is the electrochemical valence of Species $i$
$J_i$ is the flux of Species $i$
$F$ is Faraday's constant
$i$ is the current density

In the case of NaCl solution, Relation (1) can be written:

$$R_F = \frac{J_{Na}^{ext} F}{i} \tag{2}$$

where $J_{Na}^{ext}$ is the extraction flux of the sodium ions transferred under the electric field from the diluate to the concentrate compartment through the membrane area ($A$).

The cation loss at the outlet of the diluate compartment can be calculated from

$$J_{Na}^{ext} = (C_{Na}^0 - C_{Na}^t)\frac{Q}{A} \tag{3}$$

where $C_{Na}^0$ and $C_{Na}^t$ are the Na concentrations at the outlet of the diluate compartment at times 0 (before the electric current was applied) and $t$, respectively, and $Q$ is the flow rate. So the value of $R_F$ for sodium ion transport was calculated from the following relation:

$$R_F = (C_{Na}^0 - C_{Na}^t)\frac{QF}{I} \tag{4}$$

$I$, the current intensity, has a constant value under our experimental conditions.

The demineralization efficiency is the ratio of the number of cations extracted from the diluate over the total number of cations contained in the volume of solution affected by the electric current. It can be calculated from the following relation:

$$R_D = \frac{C_{Na}^0 - C_{Na}^t}{C_{Na}^0} \tag{5}$$

The current and demineralization efficiencies were expressed as percentages.

The expression of energy losses per volume of solution treated is given by Eq. (6):

$$\Delta W = \frac{UI(1 - R_F)}{Q(3.6 \times 10^6)} \tag{6}$$

where $\Delta W$ is expressed in kWh·m$^{-3}$

$U$ is the potential measured at the pilot terminals at time $t$

## Influence of Textile on Current Efficiency

Different textile configurations were used. The dilution compartment was filled by either alternate layers of cation- or anion-exchange textiles, or one layer of amphoteric textile or bipolar textile. Except with amphoteric textiles, where the cation- and anion-exchange sites are mixed, the textiles were introduced between the ion-exchange membranes with the anionic layer in contact with the anion-exchange membrane and the cationic layer in contact with the cation-exchange membrane. The thickness of the textiles used was equal to the thickness of the cell diluate compartment (2 or 8 mm).

Experiments were performed with a 300 mg·L$^{-1}$ NaCl solution and a flow rate of 60 L·h$^{-1}$. So the linear velocity was 5.2 and 20.8 cm·s$^{-1}$ when the diluate compartment thicknesses were 2 and 8 mm, respectively. The results of EDIT and ED were compared.

The current efficiencies are plotted versus current density in Figs. 4 and 5. When the linear velocity is 5.2 cm·s$^{-1}$, the current efficiency is higher in EDIT than in ED for all current densities (Fig. 4). It is slightly better with bipolar textiles than with the other textile configurations. These experimental conditions, which correspond to a diluate compartment thickness of 8 mm, are unfavorable to ED operations and, therefore, the use of ion-exchange textiles in the diluate compartment leads to substantial improvements in the performance of ED. As expected, when the linear velocity is 20.8 cm·s$^{-1}$, i.e., for a diluate compartment thickness of 2 mm, better current efficiencies are obtained (Fig. 5). In these experimental conditions of feed concentration and diluate compartment thickness, ED yields $R_F$ values of about 90%. Nevertheless, one notes that EDIT with bipolar textiles gives higher current efficien-

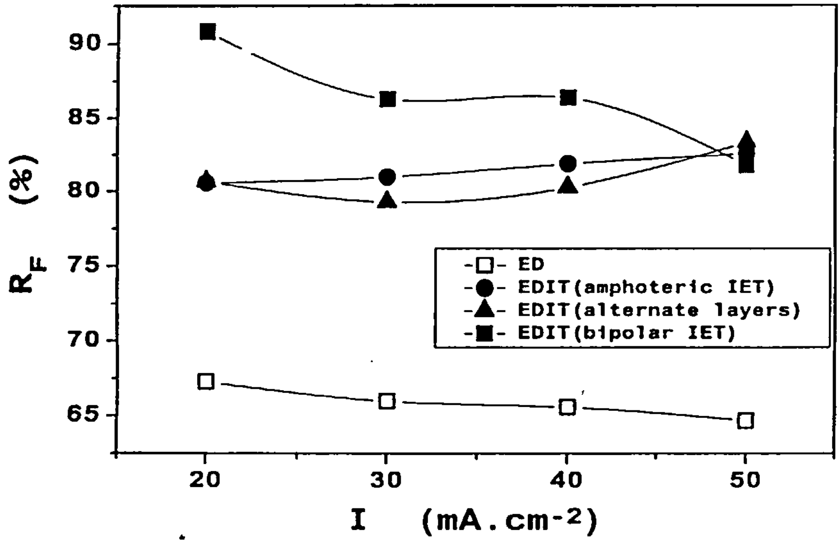

FIG. 4 Variation of the current efficiency with the current density at $Q$ = 60 L·h$^{-1}$ and $v$ = 5.2 cm·s$^{-1}$, [NaCl] = 300 mg·L$^{-1}$.

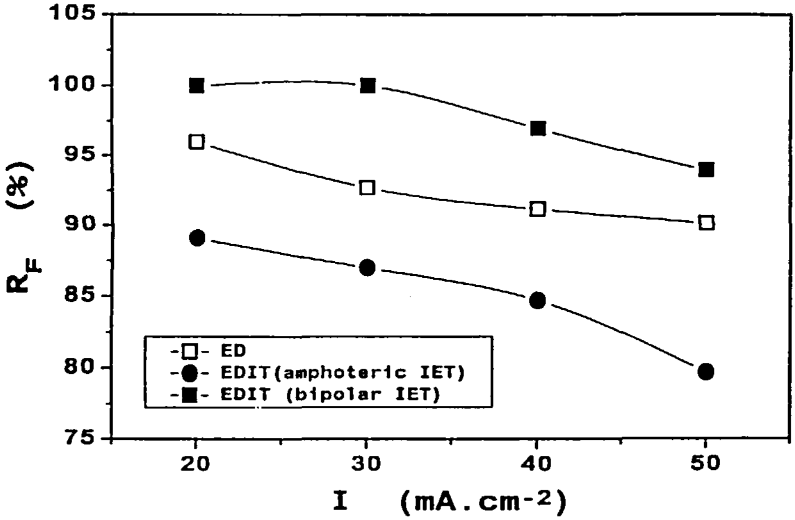

FIG. 5 Variation of the current efficiency with the current density at $Q = 60$ L·h$^{-1}$ and $v = 20.8$ cm·s$^{-1}$, [NaCl] = 300 mg·L$^{-1}$.

cies than ED, but EDIT with amphoteric textiles gives lower current efficiencies than ED.

The phenomena which are produced by an electric field acting on an ion-exchanger depend chiefly on the concentrations and mobilities of the mobile ions and on the properties of the ion-exchange material. Thus, if there are no gradients of concentration and pressure, the transport of mobile species is due solely to electric transference and convection (10):

$$J_i = (J_i)_{\mathrm{el}} + (J_i)_{\mathrm{con}} \tag{7}$$

$$J_i = -z_i\overline{C_i u_i}\,\mathrm{grad}\,U + \overline{C_i}\upsilon \tag{8}$$

where $\overline{C_i}$ and $\overline{u_i}$ are the concentration and mobility of Species $i$, respectively, in the ion-exchange material; $U$ is the electric potential; and $\upsilon$ is the rate at which the center of gravity of the pore liquid moves.

On the one hand, one can note that the concentration of ion-exchange sites is higher in bipolar textiles than in amphoteric textiles (Table 1). On the other hand, one can consider the counterion jump from one site to another which is the elementary step of ion transfer within the ion-exchange material. The mean distance of the jump is higher in the amphoteric textiles, where the cation- and anion-exchange sites are mixed, than in the bipolar textiles, where all the cation-exchange sites are concentrated near the cation-exchange mem-

TABLE 1
Main Characteristics of the Ion-Exchange Textiles

| Ion-exchange textiles | Cation-exchange capacity ($meq \cdot g^{-1}$) ($Na^+$ form) | Anion-exchange capacity ($meq \cdot g^{-1}$) ($Cl^-$ form) | Thickness (mm) | Surface weight ($g \cdot m^{-2}$) | Concentration of cation-exchange sites ($meq \cdot L^{-1}$) | Concentration of anion-exchange sites ($meq \cdot L^{-1}$) |
|---|---|---|---|---|---|---|
| CET | 0.6 | — | 2 | 390 | 117 | — |
| AET | — | 0.3 | 2 | 390 | — | 58.5 |
| Amphoteric | 0.3 | 0.3 | 8 | 800 | 15 | 15 |
|  | 0.67 | 0.6 | 2 | 190 | 32 | 28.5 |
| Bipolar | 0.75 | 0.7 | 8 | 1000 | 47 | 44 |
|  | 0.75 | 0.7 | 2 | 375 | 70.5 | 65.5 |

branes and all the anion-exchange sites are concentrated near the anion-exchange membranes. So the mobility of the counterions could be lowered in the amphoteric textiles. That could explain the gap observed between the current efficiencies obtained with the two kinds of textiles at very high flow rates.

Therefore, bipolar textiles corresponding to two ion-exchange layers mechanically juxtaposed were used for the following experiments.

## Influence of NaCl Concentration in the Concentrate Compartment

In order to minimize the ohmic drop, the electrolyte concentration must not be too low in the concentrate compartment. But, if it is too high, electrolyte diffusion across the membranes can occur from the concentrate to the diluate compartment, decreasing the demineralization ratio. Diffusion flux measurements were performed with NaCl concentrations varying from 0.03 to 30 $g \cdot L^{-1}$ and the solution, flowing in batch in the diluate compartment, was initially pure water. Very low fluxes of NaCl were obtained even at the highest NaCl concentration in the concentrate: $J_{NaCl} = 4.3 \times 10^{-10}$ $mol \cdot cm^{-2} \cdot s^{-1}$ for a NaCl concentration of 30 $g \cdot L^{-1}$. Therefore, because the salt diffusion flux was negligible, the latter NaCl concentration was used in all experiments.

## Influence of NaCl Concentration in the Diluate Compartment

The target was to test the efficiency of the EDIT process in dilute solutions under unfavorable conditions for ED. Experiments were performed with NaCl concentrations decreasing from 300 to 30 $mg \cdot L^{-1}$ and flowing in a single

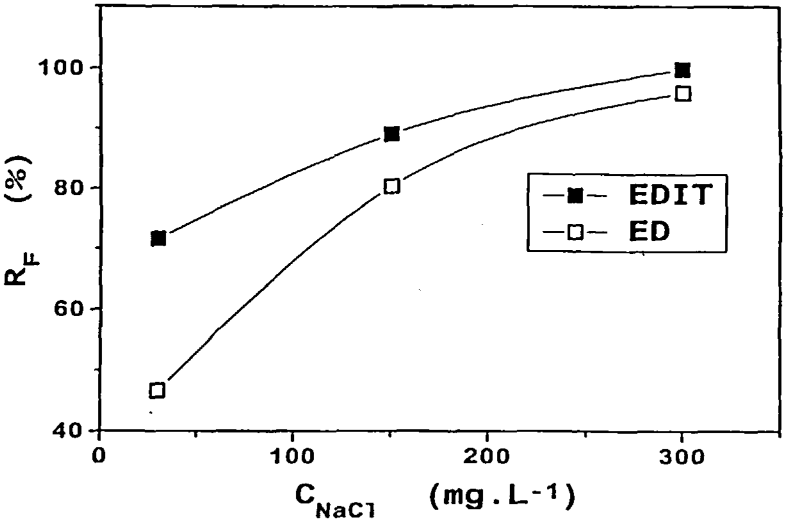

FIG. 6 Variation of the current efficiency with the feed concentration at $Q = 60$ L·h$^{-1}$ and $v = 20.8$ cm·s$^{-1}$, $i = 20$ mA·cm$^{-2}$.

pass in the diluate compartment at 60 L·h$^{-1}$ corresponding to 20.8 cm·s$^{-1}$. A current density of 20 mA·cm$^{-2}$ was applied. The results were compared to those obtained with conventional ED under the same experimental conditions.

Figures 6–8 show the variations of the current efficiency, demineralization ratio, and energy losses with the feed concentration. The current efficiency decreases when the electrolyte concentration decreases, despite the increase in the demineralization ratio. That can be due to the competition in electrotransport between the $Na^+$ ions and the protons generated by water splitting. Nevertheless, the current efficiency and demineralization ratio are higher in EDIT than in ED in all cases; inversely, the energy losses are lower in EDIT than in ED. One notes that the gap between the EDIT and ED results increases when the concentration of the feed solution is decreasing.

## Electrodeionization of 3 mg•L$^{-1}$ NaCl Solution

Experiments were performed with a NaCl concentration of 3 mg·L$^{-1}$, which was lower than the earlier concentrations studied. Various current densities and flow rates were used.

Figures 9–11 show the variations of the current efficiency, demineralization ratio, and energy losses with the current density at a flow rate of 60 L·h$^{-1}$, and Figs. 12–14 show the variations of the same parameters with the

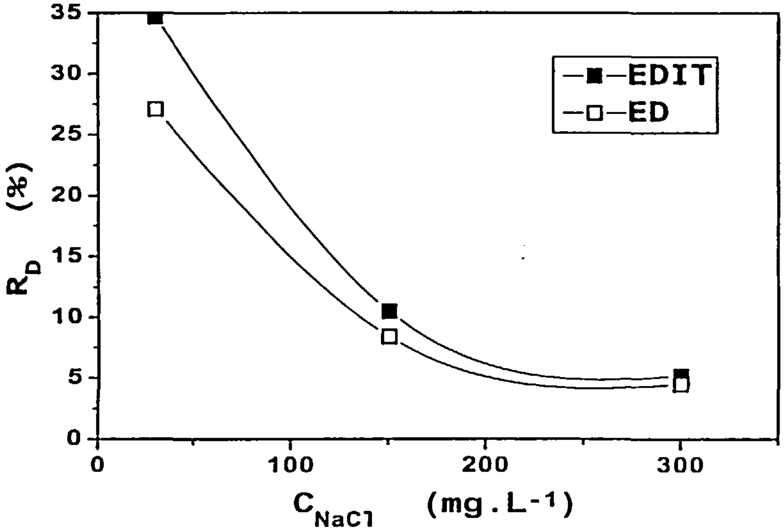

FIG. 7 Variation of the demineralization ratio with the feed concentration at $Q = 60\ \mathrm{L{\cdot}h^{-1}}$ and $v = 20.8\ \mathrm{cm{\cdot}s^{-1}}$, $i = 20\ \mathrm{mA{\cdot}cm^{-2}}$.

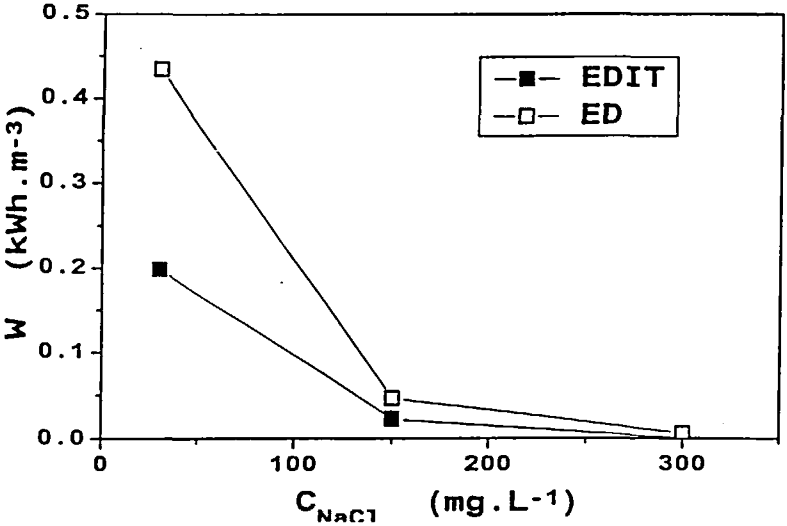

FIG. 8 Variation of the energy losses with the feed concentration at $Q = 60\ \mathrm{L{\cdot}h^{-1}}$ and $v = 20.8\ \mathrm{cm{\cdot}s^{-1}}$, $i = 20\ \mathrm{mA{\cdot}cm^{-2}}$.

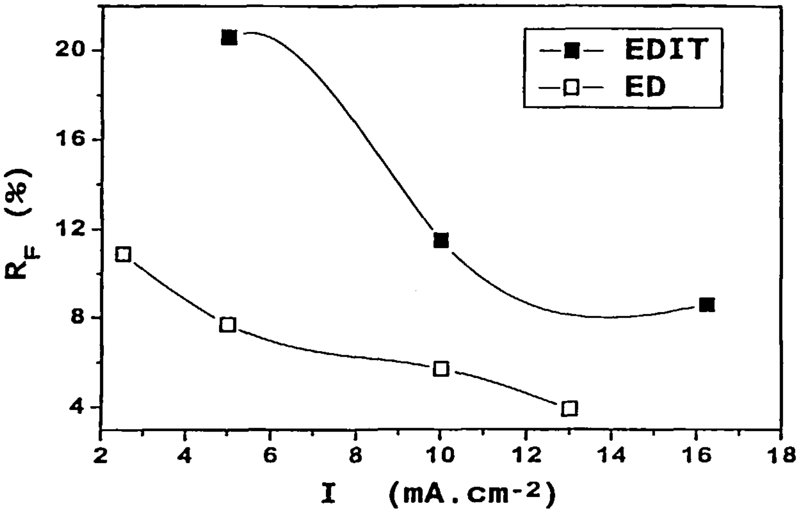

FIG. 9 Variation of the current efficiency with the current density at $Q = 60\ \text{L·h}^{-1}$ and $v = 20.8\ \text{cm·s}^{-1}$, [NaCl] = $3\ \text{mg·L}^{-1}$.

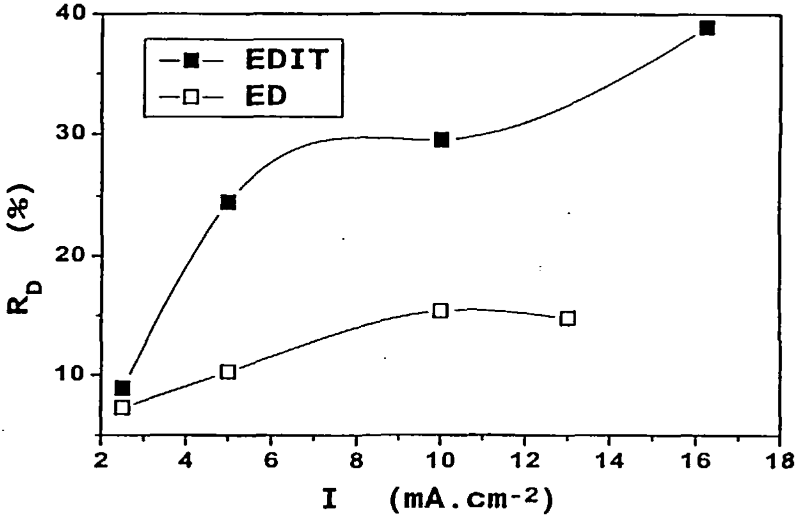

FIG. 10 Variation of the demineralization ratio with the current density at $Q = 60\ \text{L·h}^{-1}$ and $v = 20.8\ \text{cm·s}^{-1}$, [NaCl] = $3\ \text{mg·L}^{-1}$.

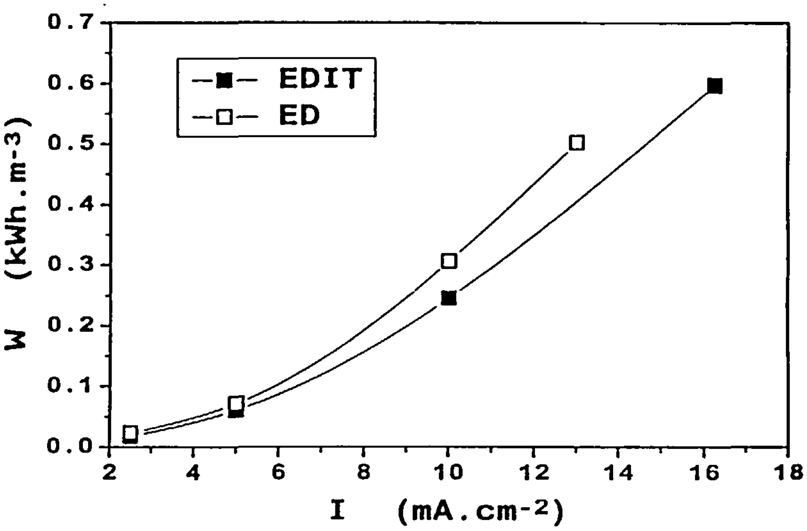

FIG. 11 Variation of the energy losses with the current density at $Q = 60\ L{\cdot}h^{-1}$ and $v = 20.8\ cm{\cdot}s^{-1}$, [NaCl] $= 3\ mg{\cdot}L^{-1}$.

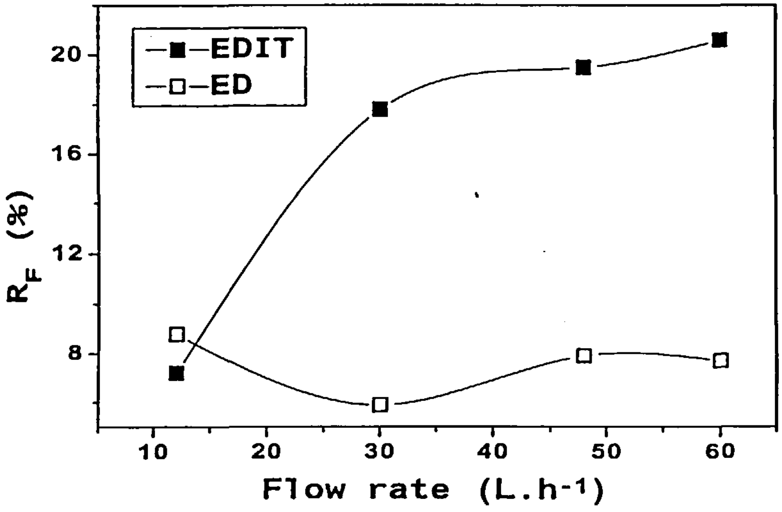

FIG. 12 Variation of the current efficiency with the flow rate at $i = 5\ mA{\cdot}cm^{-2}$, [NaCl] $= 3\ mg{\cdot}L^{-1}$.

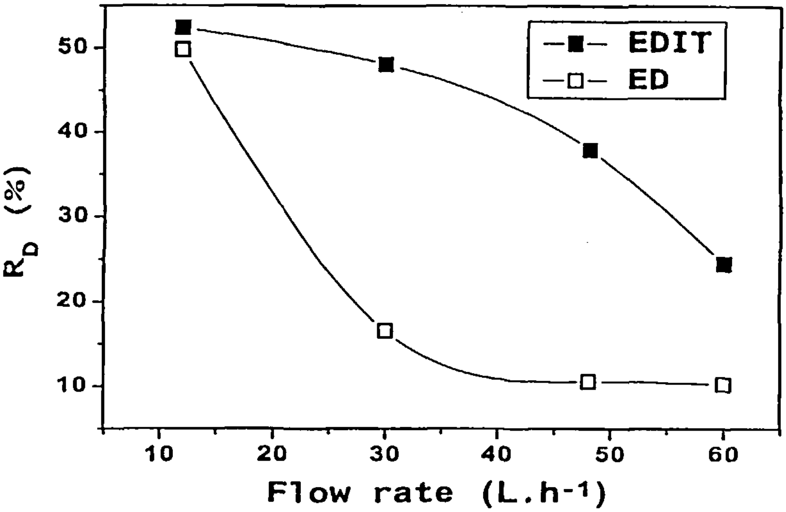

FIG. 13 Variation of the demineralization ratio with the flow rate at $i$ = 5 mA·cm$^{-2}$, [NaCl] = 3 mg·L$^{-1}$.

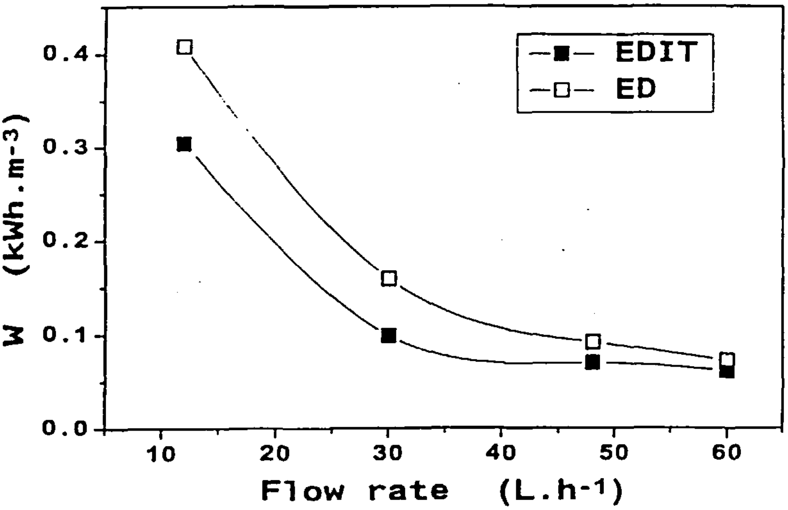

FIG. 14 Variation of the energy losses with the flow rate at $i$ = 5 mA·cm$^{-2}$, [NaCl] = 3 mg·L$^{-1}$.

flow rate at a current density of 5 mA·cm$^{-2}$. As expected, the current efficiency decreases with increasing current density and decreasing flow rate because of water splitting, as previously mentioned. Nevertheless, better results for $R_F$, $R_D$, and $W$ are always obtained with EDIT than with ED. Particularly in Figs. 12 and 13, one notes a significant gap between the EDIT and ED results for flow rates higher than 20 L·h$^{-1}$. These figures show large differences in the $R_F$ and $R_D$ variations between the two processes studied: on the one hand, the ED $R_F$ value keeps a constant value while the EDIT $R_F$ value increases significantly with the flow rate; on the other hand, the ED $R_D$ value decreases strongly while the EDIT $R_D$ value decreases slightly when the flow rate increases.

The EDIT and ED voltage variations are some 10% while $R_F$ variations can exceed 100%. So, the influence of voltage on the energy losses is significantly lower than those of the current efficiency (Eq. 6). At this low concentration where the polarization phenomenon is important, the current is partly carried by protons ($H^+$) and hydroxyl ions ($OH^-$) produced from water splitting, decreasing the ED current efficiency, while in EDIT these latter ions lead to a regeneration of the textile, increasing the demineralization of the solution.

All the results show that a decrease in the concentration of the diluate and an increase in the flow rate are particularly unfavorable for the conventional ED process. The EDIT process, where demineralization is due to both an electrodialysis process and a process of ion-exchange on textiles, which is enhanced by water dissociation, is more convenient in severe experimental conditions. Even if the study were to be carried out with a laboratory cell whose thickness was not optimized, it would allow comparison between electrodialysis performances without spacers and with spacers of different kinds.

## CONCLUSION

The use of ion-exchange textiles in the diluate compartment of the ED stack leads to substantial improvements in the performances of the process. The current and demineralization efficiencies are enhanced and the energy losses become weaker. At the lowest concentration tested (3 mg·L$^{-1}$) and the highest flow rate (20.8 cm·s$^{-1}$), the current and demineralization efficiencies were 150% higher with EDIT than with ED.

These materials are particularly well-adapted to be compressed between two plane ion-exchange membranes. They allow easy setting in an ED stack. (It has often been mentioned that one of the major difficulties of an electrodeionization system using ion-exchange resins is the problem of maintaining and operating resin-filled stacks.) Moreover, their porous and hydrophilic structure does not induce a significant pressure drop that would consequently allow high operating flow rates.

The EDIT process can be attractive not only in the demineralization of water but also in the recovery of valuable materials and/or the elimination of harmful ions in trace quantities in industrial fluid processing where large volumes of effluents must be treated.

## NOMENCLATURE

| | |
|---|---|
| $A$ | membrane area (cm$^2$) |
| AEM | anion-exchange membrane |
| AET | anion-exchange textile |
| CEM | cation-exchange membrane |
| CET | cation-exchange textile |
| $C_{\mathrm{Na}}$ | cation concentration (mol·L$^{-1}$) |
| $C_{\mathrm{NaCl}}$ | salt concentration (mg·L$^{-1}$) |
| ED | electrodialysis |
| EDIR | electrodeionization with ion-exchange resins |
| EDIT | electrodeionization with ion-exchange textiles |
| $F$ | Faraday's constant |
| $i$ | current density (mA·cm$^{-2}$) |
| $I$ | current intensity (A) |
| IET | ion-exchange textile |
| $J$ | flux (mol·cm$^{-2}$·s$^{-1}$) |
| $Q$ | flow rate (L·h$^{-1}$) |
| $R_{\mathrm{D}}$ | demineralization efficiency (%) |
| $R_{\mathrm{F}}$ | current efficiency (%) |
| $u$ | mobility |
| $U$ | potential (V) |
| $v$ | circulation rate (cm·s$^{-1}$) |
| $\Delta W$ | energy losses (kWh·m$^{-3}$) |
| $z$ | electrochemical valence |

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