

Qualification of an electro-deionization module via experimental design and ion-chromatographic studies

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Abstract

To meet the needs of the laboratory-water market, a modified electro-deionization (EDI) module has been developed to produce Type 2 purified water. An EDI module consists of desalting and concentrating fluidic compartments that are both filled with anion and cation ion-exchange resins; an anode and a cathode electrode are at opposite ends. In the design in this research, the anode electrode is segmented into three parts and individual dc amperages are applied to each segment; the cathode electrode is a single common electrode. Critical to the performance and longevity of this type of EDI module are: (1) the optimization of the applied dc amperages and (2) the ionic mass balance (i.e., the concentrations of specific and total ions of the RO feedwater to the module compared to the concentrations in the water exiting the module via the desalting and concentrating compartments). To determine a suitable current for each electrode pair, a full-factorial experimental design was developed and employed. For the application of this combination of amperages, the critical parameter of specific-ion mass balance was determined using ion-chromatographic measurements.

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1. Introduction

The purification of an aqueous solution by reducing the ion concentration has been an area of substantial technological interest. Numerous techniques have been used to purify water. The most well known processes include distillation [1], electrodialysis [2,3], reverse osmosis [4], membrane filtration [5] and ion-exchange [6], as well as electro-deionization (EDI) [7,8].

EDI technology is a combination of electrodialysis and ion-exchange technologies, whereby purified water is produced continuously in combination with reverse-osmosis (RO) pretreatment. The RO pretreatment uses a semi-permeable membrane to remove salts and other impurities

to purify water [4], and the resulting permeate is used as the feed water for an EDI module. All EDI modules further purify the RO permeate by passing it through a mixed bed of anion and cation ion-exchange media (such as beads) that are sandwiched in between a series of anion- and cation-selective membranes. Thus, desalting and concentrating compartments are formed; at each end of this “sandwich” is a pair of electrodes, to which a constant dc electrical field is applied (see Fig. 1). Electrolysis of water produces hydroxyl and hydrogen ions, which allow the continuous regeneration of the ion-exchange resins [8–10]. The water quality typically produced by this type of EDI module is Type 2 water, as defined by the American Society for Testing and Materials (ASTM) International [11].

The first known apparatus and method for treating liquids by EDI was described by Walters et al. [12] in 1955. In 1957, a US patent was granted on an electrodialysis device filled with ion-exchange resins between the anion- and cation-exchange membranes [13]. These resins increased the electrical conductivity between the membranes and acted as a path for ion transfer.

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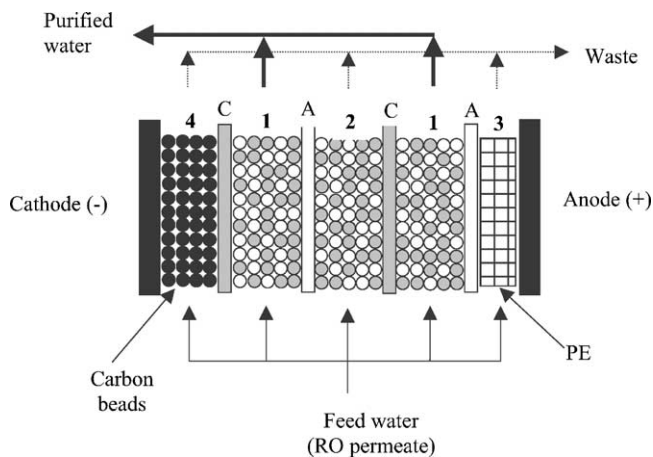


Fig. 1. Cross-sectional schematic diagram of an EDI module producing purified water [9]; carbon beads are in the cathode-electrode compartment (4) and a polyethylene (PE) screen fills the anode-electrode compartment (3); the desalting compartments (1) are producing purified water; the concentrating (2) and electrode compartments (3 and 4) are contributing to the waste; adjacent compartments are separated by an anion (A) or cation (C) membrane; anode and cathode electrodes are at opposite ends of the EDI stack, with the carbon beads' being in the cathode compartment; mixed-bed resin (made up of anion- and cation-resin beads) is used in desalting (1) and concentrating (2) compartments.

A given EDI module must be kinetically balanced (i.e., the total concentration of ions exiting a module is at least equal to the total concentrations of ions entering, meaning that ions are not retained by the module) [14]. The only source of ions entering a module is the RO feed water; the sources of ions exiting the unit are the product (purified) water and the concentrating (waste) water.

An important measurement of this kinetic balance is the ionic mass balance (IMB). The IMB is the concentration ratio of ions retained by the EDI module to ions entering the module, and can be calculated as shown in Eq. (1):

$$\text{IMB} = \frac{[\text{ion}]_f - ([\text{ion}]_p + [\text{ion}]_r)}{[\text{ion}]_f} \quad (1)$$

where, $[\text{ion}]_f$ is the specific-ionic concentration of RO water, $[\text{ion}]_p$ is the specific-ionic concentration of product water and $[\text{ion}]_r$ is the specific-ionic concentration of concentrating water. In a properly operating system, the numerator should not be positive (i.e., the concentration of ions exiting is equal to or greater than the concentration of ions entering).

The IMB is an excellent indicator of how well a given EDI module is regenerating its ion-exchange resins, and whether there is a continuous release of ions from the concentrating (waste) compartment. If there is a disruption in the regeneration of the ion-exchange resin, (e.g., insufficient amount of dc amperage applied), then there will be a retention of ions by the EDI module. Once saturated, the module will cease to produce purified water. Thus, to evaluate and qualify the expected performance of a new EDI module, it is important that reliable IMB measurements (via ion chromatography) be obtained, when the EDI module is operating under normal conditions.

Traditionally, the amount of voltage or amperage necessary for this type of EDI module to function correctly has been highly dependant on the concentration of dissolved CO_2 in the feed water; the feed water (which has already been pretreated by RO) should have a total ionic conductivity of no more than $20 \mu\text{S}/\text{cm}$ [14]. The equilibrium shifts among the dominant species (H_2CO_3 , carbonate and hydrogen carbonate) in RO-type water are largely dependant on the pH. These ions need to be removed in a given EDI module for the production of purified water.

Additionally, the RO permeate feedwater to the EDI module should not be high in concentration of alkaline-earth ions. Otherwise, significant CaCO_3 precipitation will be deposited onto the anion-membrane side of the EDI concentrating compartment (refer to Fig. 1). Also, the cathode-electrode surface will become coated. Consequently, the electron flow will be inhibited; eventually, the regeneration of the ion-exchange material and the production of purified water will be halted.

Recently, a US patent described the elimination of CaCO_3 formation at the cathode surface [9]. The inclusion of synthetic carbon beads in the cathode compartment inhibited the precipitation.

This publication describes the qualification and validation of a newly designed electro-deionization module for the purpose of producing purified water ($<0.2 \mu\text{S}/\text{cm}$) as a commercial product. This new EDI module includes the use of a segmented anode (divided into three parts) to deliver a constant dc amperage for the deionization of water and the regeneration of ion-exchange resins. The three-segment anode-electrode design was chosen based on initial laboratory results and on the size restrictions of the development of this EDI design. It was observed that this new EDI design is capable of purifying RO feed water that contains very high concentrations of dissolved carbon dioxide; however, the mechanism(s) is not understood. To determine a suitable amperage for each segment of the anode, a full-factorial experimental design was applied, based on a previously published strategy [15]. The suitable dc amperage for each of the three segments was determined via statistical software, using two measured outputs: (1) the quality of the purified water (in $\text{M}\Omega \text{cm}$), and (2) the measured voltage at each electrode. Once determined, the suitable amperages were applied to EDI modules for at least 3 months. To evaluate the performance of the modules at these amperages, the critical parameter of IMB was monitored via ion-chromatography measurements.

2. Experimental

2.1. Materials

Stock standards (1000 mg/l each) of sodium, calcium and chloride were purchased from Spex (Metuchen, NJ, USA). For eluent preparation and dilution of standards, deionized

Table 1
Typical composition of the RO feed water

| Parameters | Typical ranges |
|---------------------|-----------------------------------|
| [CO ₂] | 40–63 mg/l |
| [Ca ²⁺] | 2.5–3.0 mg/l as CaCO ₃ |
| Silica | 0.1–0.2 mg/l |
| Conductivity | 11–20 μS/cm |

(DI) water (18.2 MΩ cm) was delivered by a Milli-Q element (Millipore, Billerica, MA, USA). The typical composition of RO feed water that was used in this research is presented in Table 1.

2.2. Instrumentation and chromatography conditions

Ion analysis was performed using a Dionex DX 500 ion chromatography system (Dionex, Sunnyvale, CA, USA) with a GP50 Gradient Pump, an AS40 Automated Sampler and a CD20 Conductivity Detector. A sample volume of 25 μl was used throughout the study. For the anion chromatography, an IonPac AG17 Guard (50 mm × 4 mm) and AS17 Analytical column (250 mm × 4 mm) was used with an Anion Self-Regenerating Suppressor (ASRS-Ultra, 4 mm), with an eluent flow rate of 1.5 ml/min. The eluent, KOH, was generated by an EG-40 Eluent Generator; a concentration gradient was applied as shown in Table 2. For the cation chromatography, an IonPac CG12 Guard (50 mm × 4 mm) and CS12 Analytical column (250 mm × 4 mm) was used with a Cation Self-Regenerating Suppressor (CSRS-Ultra, 4 mm), with an eluent flow rate of 1.0 ml/min; the eluent was 20 mM methanesulfonic acid (Fluka, Buchs, Switzerland). Both suppressors were operated in the recycle mode. All the tubing in the chromatography paths was PEEK (polyether ether ketone) with an i.d. of 0.125 mm.

Instrument control and data collection were accomplished using a personal computer and PeakNet 5.1 software. JMP 4.0 software (SAS Institute, Cary, NC, USA) was used to design experiments and carry out statistical calculations [16].

The fabrication of the EDI modules in this study was based on the procedures previously reported [9,17]. Included were the fabrication of the anion- and cation-exchange membranes, the preparation of the ion-exchange resins used in the desalting and concentrating compartments, and the overall assembly of the EDI module. A total of eight EDI modules

was used in the course of this research, whereby four had a nominal flow rate of 100 l/h (EDI100), two had a nominal flow rate of 70 l/h (EDI70) and two had a nominal flow rate of 35 l/h (EDI35). Only the EDI100 units were used in the experimental-design work; all eight modules were used in the mass-balance study.

2.3. Calibration standards

All solutions were prepared in new polypropylene flasks (100-ml capacity) and stored in new high-density polyethylene (HDPE) narrow-mouth bottles (125-ml capacity) (both from Nalge Nunc, Rochester, NY, USA). Gilson (Middleton, WI, USA) pipettes P5000, P1000 and P100 (each with the appropriate tip) were used to deliver standard solutions for dilution. All new plasticware was rinsed thoroughly with 18.2 MΩ cm water prior to initial use.

For the anion calibration study, a standard (100 ml, in DI water) containing 10 mg/l of chloride was prepared from the corresponding 1000 mg/l stock standard. For the cation calibration work, a mixed standard (100 ml, in water) containing 10 mg/l each of sodium and calcium was prepared from the corresponding 1000 mg/l stock standards. Six working standards (blank; 0.05, 0.1, 0.5, 1.5 and 2.5 mg/l) were prepared from each of the 10-mg/l standards and injected onto the appropriate ion-chromatographic column to generate calibration data.

For each calibration study, the set of working standards was prepared and analyzed (in random order) on each of five separate days. Peak areas (PAs) were used to measure each analyte response.

All masses were determined using a Mettler AJ100 analytical balance (Mettler-Toledo, Columbus, OH, USA) and were recorded to 0.0001 g. Dilution errors in the daily working standards were estimated by conducting a Monte Carlo simulation [18]. This exercise was based on the upper bound (0.0001 g) on the magnitude of weighing error for the balance. In the simulation, weighing errors were randomly drawn from a Normal distribution with mean equal to zero and standard deviation equal to the upper bound. The distribution of these relative concentration errors was found never to exceed 0.1% relative error, which was considered negligible.

3. Results and discussion

3.1. The newly designed EDI module

Typically, a commercially available EDI module consists of only one electrode each for the anode and cathode compartments (refer to Fig. 1), whereby a dc amperage is applied to promote the deionization of the flowing water and the regeneration of the resins. In the EDI module in this study, there is a segmented (into three parts) electrode for the anode compartment and a common electrode in the

Table 2
Gradient program used for the anion-chromatographic analyses

| Time (min) | KOH concentration (mM) |
|------------|------------------------|
| 0 | 1 |
| 2 | 1 |
| 6.5 | 1 |
| 9.5 | 20 |
| 12.5 | 40 |
| 12.5 | 1 |
| 30 | 1 |

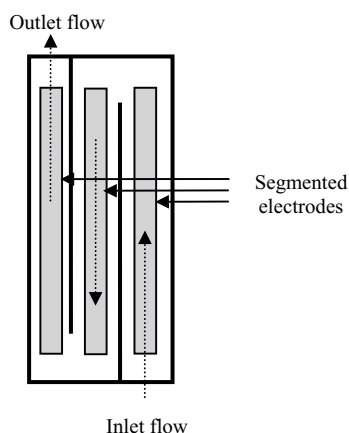


Fig. 2. End-on schematic diagram of the segmented anode overlapping the flow path in the fluidic compartments.

cathode compartment. Fig. 1 illustrates the cross-sectional view of this EDI module; Fig. 2 illustrates the end-on view of the same module. Each third of the anode electrode covers a corresponding portion of the desalting and concentrating compartments that are visible in Fig. 1. This arrangement allows for a suitable dc amperage to be applied to the appropriate portions of each compartment, and for an appropriate amount of total deionization and total regeneration to occur within the module. This design results in a more efficient EDI module overall.

The various desalting and concentrating compartments are connected in parallel; the feed water enters from the bottom of the compartment and exits from the top. A full cell is defined as one desalting compartment that is surrounded by two concentrating compartments, as shown in Fig. 1. With each cell, the nominal flow rate is fixed at 5 l/h. Because of the parallel arrangement, cells can be combined to facilitate the manufacture of EDI modules with different nominal flow rates. For example, a unit that will produce 100 l/h will require 20 cells. The amperages that are applied to the corresponding portions of the segmented anode will be the same throughout the various cells.

A significant improvement observed with this new EDI design was its ability to operate with very high concentrations of dissolved carbon dioxide present in the RO feed water (>40 mg/l), compared to previous EDI designs available on the market [14]. The dissolved-CO₂ concentrations were naturally as high as 63 mg/l in the feed water (refer to Table 1), yet all the modules used in this study still produced high purity of product water. Normally, with this type of EDI module, high amperages would need to be applied if there was a significant concentration of carbon dioxide present in the feed water (refer to Section 3.2); however, increased currents were not required for this new design, demonstrating the advantage of the segmented-electrode concept. It is thought that the dedicated dc amperage for each segmented anode deionizes and regenerates the appropriate amount at each of the three flow compartments, compared to a single

electrode. With just a single anode, the amperage would be self-distributed among the same three compartments. Such a distribution would change continually and would be highly dependant on the conditions in each compartment at any particular time. This variability causes the entire deionization/regeneration process to be inefficient. For example, if an air bubble were to become trapped in one compartment, the impedance would rise and the current would fall below what is actually needed. The advantage of the segmented-anode concept is that constant amperage is continuously delivered to each compartment; the deionization and regeneration processes are far more efficient kinetically and require a lower overall electrical power compared to a single anode.

3.2. Theoretical electrical current required for purifying water

For a given EDI module, the dc amperage required to purify water is largely dependant on the concentration of dissolved CO₂ in the RO feed water and the ionic load that must be removed from the desalting compartments [14]. Other anions, such as sulfates, are negligible, since: (1) the solubility products of their calcium salts are higher compared to those of the calcium carbonates, and (2) their concentrations are negligible compared to those of the carbonates. The theoretical amperage is determined by ionic-mass-balance considerations. One Faraday of electrical charge is necessary to remove one equivalent of ions from the desalting compartments. The minimum amperage needed is directly proportional to the ionic load present and can be determined using Eq. (2) [14] as follows:

$$I' = \frac{Q \times C \times F}{3600 \times N} \quad (2)$$

where, I' is the theoretical amperage, Q is the flow rate of the desalting compartment (l/h), C is the total ion concentration (equiv./l), F is the Faraday's constant (96,489 coulombs/equiv.), and N is the number of cells in the EDI stack.

A sample calculation that shows the theoretical amperage (I') required to purify RO feed water at a flow rate of 50 l/h (based on 10 cells with each cell's contributing 5 l/h) with a total conductivity = 20 μ S/cm and [CO₂] = 40 mg/l is described below.

The contribution of carbon dioxide (as a dissolved gas) to the ionic conductivity of pure water is:

$$[\text{CO}_2] = 40 \text{ mg/l} = 8 \mu\text{S/cm} \quad [19]$$

Thus, the remaining ionic conductivity is:

$$20 \mu\text{S/cm} - 8 \mu\text{S/cm} = 12 \mu\text{S/cm}.$$

This net conductivity can be converted to mg/l as NaCl (equivalent) via a conversion factor of 0.5 (mg cm)/(μ S l):

$$\begin{aligned} 12 \mu\text{S/cm} \times 0.5 \text{ (mg cm)} / (\mu\text{S l}) \\ = 6.0 \text{ mg/l as NaCl (equivalent)} \end{aligned}$$

Also, the CO₂ concentration can be converted to NaCl (equivalent) via a unitless conversion factor of 1.3 (i.e., the atomic-mass-unit ratio between CO₂ and NaCl):

$$40 \text{ mg/l} \times 1.3 = 52 \text{ mg/l as NaCl (equivalent)}$$

The total mg/l as NaCl (equivalent) is the sum:

$$\begin{aligned} C &= (6.0 + 52) \text{ mg/l as NaCl (equivalent)} \\ &= 58 \text{ mg/l as NaCl (equivalent)} \end{aligned}$$

This number is converted to equivalents/l by dividing by the molecular weight of NaCl:

$$\frac{58 \text{ mg/l as NaCl (equivalent)}}{58 \times 10^{-3} \text{ equiv./mg}} = 1.0 \times 10^{-3} \text{ equiv./l}$$

Finally, I' can be calculated from Eq. (2):

$$\begin{aligned} &\frac{(50 \text{ l/h} \times 10^{-3} \text{ equiv./l} \times 96489 \text{ coulombs/equiv.})}{(3600 \times 5)} \\ &= 134.01 \text{ mA.} \end{aligned}$$

Based on the above calculations, an EDI module producing 50 l/h of Type 2 purified water will require a minimum of 134 mA to remove the required concentration of ions from the desalting compartments. Repeating the same calculation with 100 l/h with 20 cells and 35 l/h with 7 cells, the identical minimum amperage (I') of 134 mA.

In practice, it is necessary to multiply the minimum amperage (I'), so that the regeneration of the ion-exchange resins will take place [14]. Previous work in the author's laboratory has shown that a minimum theoretical amperage (I') should be multiplied by 3. Therefore, a total of about

400 mA must be used for this EDI module to produce purified water continuously at the nominal flow rate. However, the distribution of this total amperage among the three electrodes had not been addressed prior to this research.

3.3. Determination of suitable dc amperages via experimental design

Statistical design of experiments was utilized to determine a suitable combination of output amperages for the three electrodes in the anode compartment; these amperages were the control factors in the design. A full-factorial design was chosen, whereby a maximum and minimum were chosen for each control factor; all possible combinations (plus a center point) were calculated using the JMP software. Trial runs were conducted to determine realistic maximum and minimum values for these dc amperages. Results were:

- (1) electrodes 1 and 2: minimum = 100 mA and maximum = 175 mA;
- (2) electrode 3: minimum = 50 mA and maximum = 100 mA; and
- (3) total applied current \leq 400 mA.

The experiments that were performed in this study were determined by the JMP software and are presented in Table 3. These experiments were applied randomly to the four individual EDI100 modules described earlier in Section 2.2, operating under an alternating 2 h on–2 h off regime throughout this study and typifies a routine operation in a given laboratory. Each experiment was allowed to run for 17–18 days. From the experimental data, a suitable combination was proposed via JMP software; the calculated

Table 3
Full-factorial design for currents applied to each of the three anode segments in a given EDI module

| Experiment number | mA 1 ^a | mA 2 ^a | mA 3 ^a | Total mA ^a | Round experiment was used on EDI100 module number | | | |
|-------------------|-------------------|-------------------|-------------------|-----------------------|---|---|---|---|
| | | | | | 1 | 2 | 3 | 4 |
| 1 | 100 | 100 | 100 | 300 | 3 | | | |
| 2 | 100 | 150 | 50 | 300 ^b | 2 | | | 2 |
| 3 | 100 | 175 | 50 | 325 | | | 2 | |
| 4 | 100 | 175 | 100 | 375 | | 2 | | |
| 5 | 137 | 137 | 75 | 349 ^c | 1 | 1 | 1 | 1 |
| 6 | 150 | 150 | 50 | 350 ^d | | 3 | | |
| 7 | 150 | 150 | 100 | 400 ^e | | | 3 | 3 |
| 8 | 175 | 100 | 100 | 375 ^d | | | | |
| 9 | 175 | 175 | 50 | 400 ^d | | | | |

See Section 3.3 for additional discussion of these experiments.

^a The “mA” designations refer to the current (in milliamps) applied to anode segments 1, 2 and 3. “Total mA” is the sum of these three applied currents.

^b Experiment 2 was designed to be 100, 100 and 50 mA, respectively. However, it was known that the total of 250 mA was too low to work successfully; earlier work indicated that increasing current 2 to 150 mA would be a logical revision.

^c Experiment 5 was rounded (from 137.5, 137.5, 75 mA) because amperage settings are only made to the nearest whole amp.

^d Experiment 6 was designed to be 175, 100, 50 mA. However, the application of 137 mA to the first electrode (in the first round) for one of the modules resulted in voltages that were double the corresponding values on the first electrode of all other modules, which also received 137 mA. For this reason, it was decided not to conduct any of the three experiments calling for 175 mA on electrode 1; experiments 8 and 9 were dropped altogether; experiment 6 was scaled to 150, 150, 50 mA to provide a comparison with experiment 7.

^e Experiment 7, as designed from the software, was 175, 175, 100 mA. However, the total current of 450 mA exceeds the maximum allowed (i.e., 400 mA). Thus, the values were scaled to 155, 155, 90 mA to reduce the total to 400 mA. For practical considerations, these values were rounded to 150, 150, 100 mA.

combination was 150, 130 and 100 mA, to be applied to the first, second and third electrodes, respectively. This theoretical set of amperages was applied to all eight of the modules (i.e., four EDI100, two EDI70 and two EDI35 units) for approximately three months, to see if these settings resulted in acceptable mass-balance data.

3.4. Ion-chromatographic calibration study

Before any mass-balance work was undertaken, thorough calibration studies were conducted on both ion-chromatographic systems (see Section 2.3 for calibration-design details). A straight-line model with ordinary least squares (OLS) as the fitting technique was proposed to fit the data for each ion. The chosen calibration design allowed for modeling of the standard deviation of the responses (i.e., peak areas), as well as for testing of the model.

Calibration diagnostics were performed on the study data. Standard-deviation modeling of the PAs showed that OLS was appropriate only for chloride; weighted least squares (WLS) was needed calcium and sodium. Residual patterns indicated that a straight line was appropriate only for chloride; a quadratic fit was chosen for calcium and sodium. Prediction-interval widths at 1.5 ppb and Hubaux-Vos detection limits [20] (all at the 95% confidence level) are given in Table 4. These statistics were deemed acceptable for the precision needed in this research.

3.5. Validation of the suitable amperages via ion chromatography

The EDI modules were evaluated for approximately three months at the amperage combination calculated via JMP software. During this time, ion chromatography was used to monitor the critical chloride, sodium and calcium levels in the water that entered and exited the modules. Representative chromatograms of the product water are shown in Fig. 3. These data were used to construct mass-balance graphs, which were used to evaluate the performance of the modules.

As can be seen from Figs. 4 and 5, the cumulative mass-balance data behaved properly during a typical 2 h operation. The results shown in Figs. 4 and 5 are the analyses of both the product and waste outlets combined for each of the modules. It is important to note that the specific-ionic

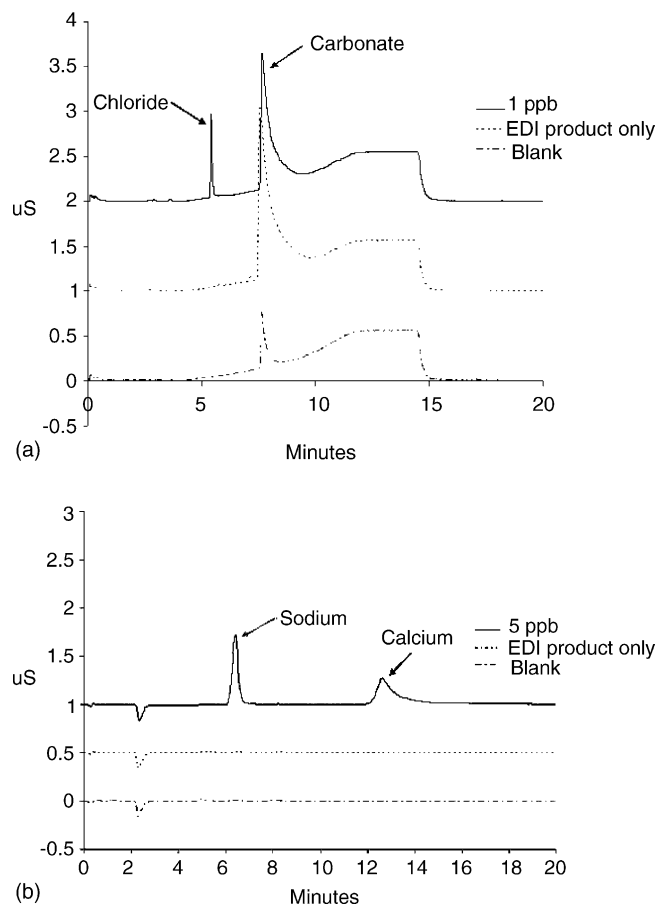


Fig. 3. Representative (a) anion chromatogram comparing: (1) the product water from an EDI35, (2) the 1 ppb Cl^- standard solution and (3) the blank solution; (b) cation chromatogram comparing: (1) the product water from an EDI35, (2) the 5 ppb Li^+ , Na^+ and Ca^{2+} mixed standard and (3) the blank solution. *Note:* the baselines have been offset for ease of comparison.

contributions from the product outlets were always less than 0.5% for the ions analyzed and the quality of the product water produced remained at $10 \text{ M}\Omega \text{ cm}$ or better throughout this cumulative ion mass-balance study. As shown in Figs. 4 and 5, there is an initial retention of the specific ions as indicated by the positive slope for all the modules analyzed. This retention is the result of ions already present inside the modules while they were not operating. Once the regeneration of the ion-exchange resins (via electrolysis of water) is equal to the incoming ion concentrations, there is

Table 4
Calibration-curve data for the three analytes investigated in the mass-balance study

| Analyte | Model | Fitting technique ^a | Hubaux-Vos detection limit (mg/l) | \pm Prediction interval (mg/l) at 1.5 mg/l |
|----------|---------------|--------------------------------|-----------------------------------|--|
| Calcium | Quadratic | WLS | 0.08 | 0.34 |
| Sodium | Quadratic | WLS | 0.03 | 0.06 |
| Chloride | Straight line | OLS | 0.03 | 0.01 |

The concentrations in each IMB sample were calculated via these calibration plots. Data are reported at the 95% confidence level. See Sections 2.3 and 3.4 for discussion of the calibration design and diagnostics.

^a WLS: weighted least squares; OLS: ordinary least squares.

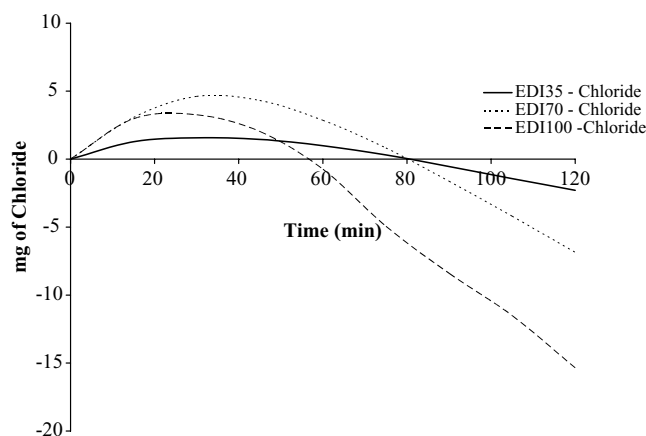


Fig. 4. Cumulative chloride-ion mass balance observed for each of the EDI modules (EDI35, EDI70 and EDI100). See Section 3.5 for additional details.

a typical negative slope observed with the cumulative ion mass balance. This phenomenon was observed for all the modules after about 30–40 min of operation; none exhibited retention of chloride, sodium or calcium ions, as indicated by the observed negative slopes. Thus, ion-chromatographic results indicated that the combination of 150, 130 and

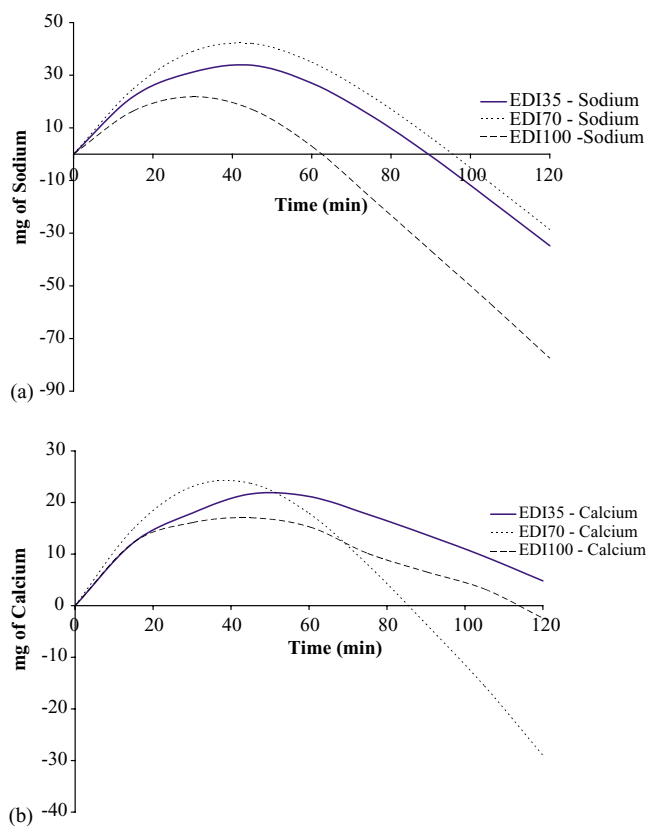


Fig. 5. Cumulative sodium- (a) and calcium-ion (b) mass balances observed for each of the EDI modules (EDI35, EDI70 and EDI100). See Section 3.5 for additional details.

100 mA was indeed suitable for routine operation of this design type of EDI module.

4. Conclusions

Via statistical design of experiments and ion chromatography, a new type of EDI module was successfully qualified for the production of Type 2 purified water. The required amperages to be applied to the segmented anode of the new EDI design were determined via a full-factorial experimental design. Statistical analysis of the data indicated that these currents should be 150, 130 and 100 mA for the first, second and third electrodes, respectively.

To verify these proposed amperages, the currents were applied to the segmented anodes of eight EDI modules for approximately three months. During that time, specific-ion mass-balance studies were conducted on three critical ions: chloride, sodium and calcium. Calibration studies indicated that ion chromatography was an appropriate technique for obtaining IMB data. IMB calculations demonstrated that: (1) there was no retention of ions by these EDI modules and (2) the regeneration of the ion-exchange resins via the electrolysis of water was occurring. In addition, it was also observed that the segmented-electrode concept was highly efficient in producing purified water, even when there was a significantly high concentration of dissolved carbon dioxide present in the RO feed water.

5. Nomenclature

Terms and abbreviations used

| | |
|---------------------|---|
| ASTM | American Society for Testing and Materials |
| <i>C</i> | total ion concentration, in equiv./l |
| EDI | electro-deionization. A process whereby ion-exchange resins are continuously regenerated by constant dc amperage and Type 2 purified water is produced. |
| <i>F</i> | Faraday's constant = 96489 coulombs/equiv. |
| <i>I'</i> | theoretical amperage |
| IMB | ionic mass balance. The concentration ratio of ions retained by the EDI module to ions entering the module. $\text{IMB} = \frac{([\text{ion}]_f - ([\text{ion}]_p + [\text{ion}]_r))}{[\text{ion}]_f}$ |
| <i>N</i> | number of cells in an EDI stack. |
| OLS | ordinary least squares. A fitting technique that minimizes the sum of squares of the residuals |
| Prediction interval | a range of values defined by a pair of limits that bracket the uncertainty in one future measurement |
| PA | peak area |

| | |
|--------------|--|
| Q | flow rate of the desalting compartment, in l/h |
| RO | reverse osmosis. A process that uses a semi-permeable membrane to remove salts and other impurities to purify water. |
| Type 2 water | water whose quality is at least $1.0\text{ M}\Omega\text{ cm}$ ($0.1\ \mu\text{S/cm}$), as defined by ASTM. |
| WLS | same as OLS, except observations are numerically weighted to reflect non-uniform levels of variation in the responses. |

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