



## Clogging of the electrodeionization chamber

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### ABSTRACT

To achieve the limits given by power engineering standards (e.g. VGB-R-450 Le, EPRI) of make-up water for high pressure boilers and steam generators, the power industry has been utilizing mixed bed units in the majority of cases (fed by the water treated with the demineralization units). But, nowadays, the mixed bed units are more often substituted by electrodeionization (EDI) units. The basic advantage of EDI units is the reduction of operating expenses (OPEX). The OPEX decreases because of the regeneration by electric current instead of using regenerating chemicals. Unlike classical mixed bed units, these EDI units require better quality of feed water (quality of the reverse osmotic permeate and better). Operational experience has shown irreversible clogging of EDI chamber during continuous operation, in several days, in case of using feed water without reverse osmosis treatment. The aim of this work was to simulate clogging (also fouling of membranes and clumping of mixed bed) of EDI chambers on the laboratory scale to prove the presumption that the origin of clogging is represented by colloidal substances (which can not be removed from surface water without the use of reverse osmosis or nanofiltration unit). However, the work shows that the model solution of colloidal silica nanoparticles could pass through the EDI chamber without any clogging, regardless the diameter of the particles. Thus, these results refuted the hypothesis about the key influence of colloidal particles on so-called clogging of EDI chambers. To get a deeper understanding of the general clogging process in EDI chambers, a series of experiments using very low quality feed water was carried out. The qualitative analysis of clogged components proved that the clogging is principally caused by dissociated ions (e.g. iron, calcium, magnesium, etc.) The experiment also revealed that the sorption of silica in EDI unit is given by the steady state of the dissociated and colloidal form.

*Keywords:* Electrodeionization; Colloidal silica; Clogging; Ion exchangers

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## 1. Introduction

Efficiency and lifetime of electrodeionization (EDI) module is related to the quality of incoming water [1]. The generally known fact is based on the hypothesis [2] that high concentration of colloidal substances is related to the clogging of EDI chamber (resulting in decreasing desalination efficiency of EDI followed by decommissioning of the unit). The aim of this work was to dispute the hypothesis according to industrial experience.

The experimental results are confronted with the data from industrial operation. The laboratory experiments were carried out using model colloidal aqueous solutions [3] represented by the silica particles of different diameter and different concentrations. The water used for preparation of model solution was demineralized water made by different EDI devices. Before and after exposure to this solution, the demineralizing efficiency of EDI was measured.

To compare the resistance of different filling, the EDI chambers were filled with different ion exchangers [4]. The gel and macroporous ion exchangers were compared. The macroporous ion exchangers were chosen as they are supposed to be more resistant to colloidal solutions when utilized in the standard columns.

## 2. Experimental Part

### 2.1. The type of EDI module

For the measurement, the laboratory EDI module with resin-filled compartments MemBrain EDI Z2-AF plate type with 5 membrane pairs was chosen. The active surface of each membrane was 114.8 cm<sup>2</sup> (28.7 × 4 cm) and the thickness of each chamber (for the resin filling) was 0.27 cm. For the first series of measurements chosen [5], the mixture of gel resins: strongly basic anion exchanger Amberjet 4400 Cl and strongly acidic cation exchanger Amberjet 1200 Na. In a second series of tests, a mixture of macroporous resins: strongly basic anion exchanger Amberlite IRA-900 Cl and strongly acidic cation exchanger Purolite C-160 Na were used [6]. These are commonly used ion exchange resins in water treatment for power plants.

### 2.2. Characteristics of test

Firstly, the reference test was done for the determination of the degree of desalination by EDI. The reference test was done again before each exposure. The reverse osmosis permeate was used for this reference test because it is the only suitable feed for EDI in

industrial scale [7]. This experiment also revealed the demineralizing efficiency for each type of resin in the EDI module.

The efficiency was approximated by the grade LD (“level of demineralization”). LD was calculated according to eq. (1), where  $\kappa_0$  and  $\kappa_1$  represents conductivity of feed solution and product, respectively. Measurement was carried out at a constant voltage of 3 V/(membrane pair) for gel resins and 5 V/(membrane pair) for macroporous resins. The flow rate of the diluate and concentrate cells was maintained at 30 and 20 L/h, respectively.

$$LD = \frac{\kappa_0 - \kappa_1}{\kappa_0} \cdot 100 \quad (1)$$

### 2.3. Solution of colloidal silica

The SDI<sub>15</sub> (silt density index measured in 15 min interval) in the feed of EDI should be below 1. However, this parameter cannot guarantee the concentration of colloidal particles lower than 450 nm (i.e. size of pores in cellulose filter for determination of SDI<sub>15</sub>). The presence of colloidal particles below this size is still supposed to be sufficient for irreversible clogging of EDI in tens of operation hours.

The concentration and composition of colloidal solution in industrial operation is mostly random [8]. Therefore, the preparation of reproducible colloidal solution of exact concentration and defined size of particles was not simple and the variety of solutions was prepared [9]. Finally, the experiments were based on the preparation of colloidal solution from commercial products Ludox<sup>®</sup> and Aerodisp<sup>®</sup> W7330 N containing colloidal silica particles of size 22 nm and 120 nm, respectively.

To determine the level of clogging, the experiments were performed with colloidal solution of an input concentration of 10 and 100 mg/L of SiO<sub>2</sub> in demineralized water. The time of exposure to colloidal solution was 20 h because the complete irreversible clogging of EDI chamber in industrial scale is observed to be less than 160 h [10].

Compared to previous experiments, the process efficiency could not be estimated by grade LD because the demineralized water and nonionic forms of silica had very low conductivity (under 0.3 μs/cm). Thus, the performance of exposed EDI was evaluated by the following demineralization test. The results were compared to the reference tests.

Simultaneously, the influence of the ion exchange resin for the removal of colloidal silica was verified by analytical methods i.e. optical emission spectroscopy

ICP–OES using reaction of dissolved silica with molybdate [11].

### 3. Results

#### 3.1. Reference tests with RO permeate

The conductivity of RO permeate ranged between 7 and 9  $\mu\text{S}/\text{cm}$ , the average concentration of dissolved  $\text{SiO}_2$  in the permeate was 0.343 mg/L  $\text{SiO}_2$  and the concentration of  $\text{SiO}_2$  colloidal form in the permeate was under the limit of determination. The grade LD calculated due to Eq. (1) was 97.9% for the gel resin and 95.7% for the macroporous ion exchange resins. Comparing the data shows that the stable values of conductivity has been achieved for both types of ion exchange resin during the same operation time. On the other hand, the values of current densities for the macroporous ion exchange resin were 1.5 times lower in average.

#### 3.2. Tests with solution of colloidal $\text{SiO}_2$

As described above, reference test was done before each exposure to the colloidal solution for 20 h, then the demineralization test was carried out and the results were compared with the reference tests.

The results in Figs. 1–4 represent the demineralization tests after exposure of EDI module to 10 and 100 mg/L of colloidal silica (in the reverse osmosis permeate) for both types of ion exchange resin. Conductivity of the diluate in the exposed module is compared with the results of reference test.

For gel type ion exchange beads, we can notice identical conductivity values before and after processing of a colloidal solution, as seen in Figs. 1 and 2. The results show that no clogging or ion exchange

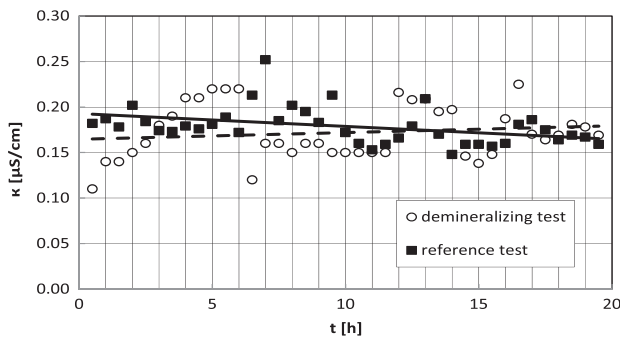


Fig. 1. Conductivity of the diluate: Electrodeionization membrane module filled with gel type ion exchange resin exposed to colloidal silica particles of  $d = 22$  nm and concentration of  $c_0 = 10$  mg/L for 20 h.

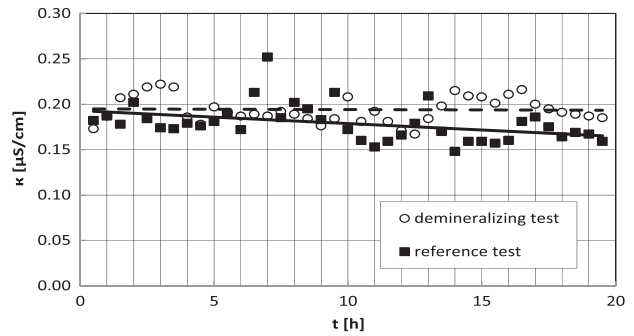


Fig. 2. Conductivity of the diluate: Electrodeionization membrane module filled with gel type resin exposed to colloidal silica particles of  $d = 22$  nm and concentration of  $c_0 = 100$  mg/L for 20 h.

membrane scaling occurred and the module is fully operational. This conclusion is confirmed by the unchanged concentration of the colloids in the diluate and concentrate streams indicating that the colloid passed through the module without any interactions: the concentration of dissociated silica was determined by the ICP–OES as 0.026 mg/L (out of 10 mg/L input colloidal silica).

The membrane module filled with macroporous ion exchangers [12], which was exposed to the solution of 10 ppm colloidal silica, also during the demineralization tests did not exhibit any clogging. Moreover, as seen in Fig. 3, the demineralization test results are even better when compared to reference test. The explanation of this phenomenon is in the equilibration of the EDI module during operation.

Most significant difference was achieved for demineralization test with electrodeionization membrane module filled by macroporous ion exchange beads which was exposed to 100 mg/L colloidal solution (Fig. 4). Even though the conductivity values

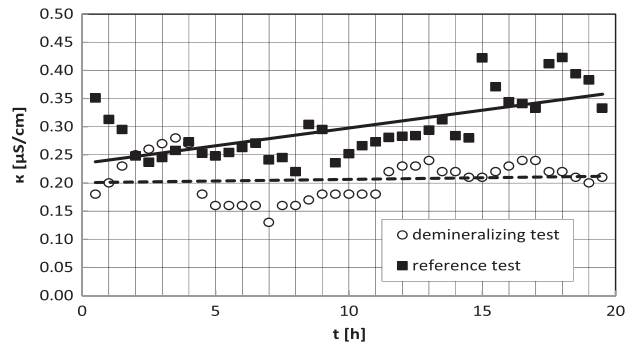


Fig. 3. Conductivity of the diluate: Electrodeionization membrane module filled with macroporous type resin exposed to the colloidal silica particles of  $d = 22$  nm and concentration  $c_0 = 10$  mg/L for 20 h.

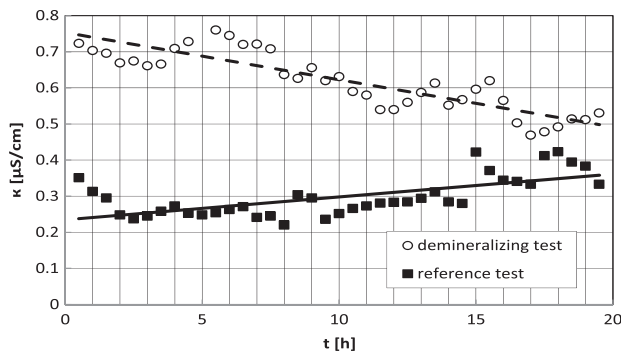


Fig. 4. Conductivity of the diluate: Electrodeionization membrane module filled with macroporous type resin exposed to the colloidal silica particles of  $d = 22$  nm and concentration  $c_0 = 100$  mg/L for 20 h.

approach the values of the reference test slightly in comparison with other tests, there is a noticeable decrease in the efficiency of demineralization which may indicate an interaction of the ion exchange resin with colloidal particles or their concentration at the membrane surface. However, this interaction was not irreversible as clearly seen in Fig. 4.

The industrial experience of the electrodeionization operation without the reverse osmosis pretreatment showed that the clogging of EDI appears during processing of distillate from evaporator which contained colloidal particles of calcium carbonate down to 30 nm diameter. Therefore, it was assumed that the colloidal particles bigger than 30 nm can clog the EDI chamber.

Despite this assumption, the performance of the EDI membrane module was not affected irreversibly even by the processing of colloidal solution with a particle size of 120 nm as seen in Figs. 5 and 6. The concentration of silica in the output stream is approaching the value of concentration in the feed solution. This indicates that the colloidal particles

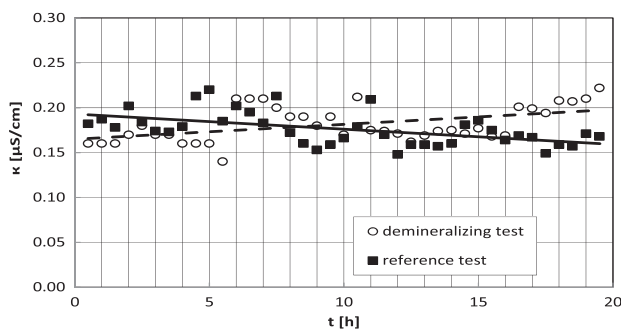


Fig. 5. Conductivity of the diluate: Electrodeionization membrane module filled with gel type resin exposed to the colloidal silica particles of  $d = 120$  nm and concentration  $c_0 = 100$  mg/L for 20 h.

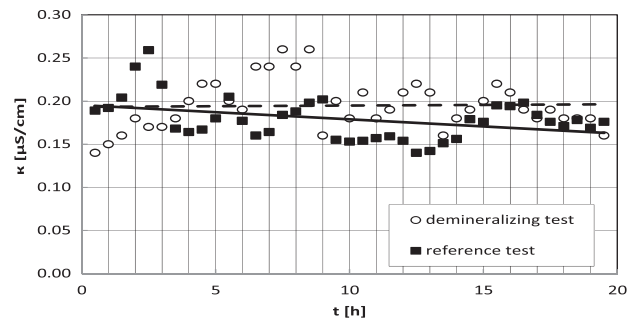


Fig. 6. Conductivity of the diluate: Electrodeionization membrane module filled with macroporous type resin exposed to the colloidal silica particles of  $d = 120$  nm and concentration  $c_0 = 100$  mg/L for 20 h.

more than five times larger (compared with the first tests) passes through the filling of EDI membrane module without the capture. Even in this case, there was no significant difference between the used type of ion exchange resins, gel and macroporous. The explanation can be found in too short processing time. But, on the other hand, the time is substituted by much higher concentration of colloidal solution and the concentration of silica in feed solution will never be as high in the real industrial operation.

All these results clearly show the lack of the interaction between colloidal particles and compartments of the EDI chamber. The deviation in results for different filling of EDI chambers and different colloidal solutions are negligible.

The chemical analysis of clogged compartments of EDI from industrial application exhibited increased content of metals, especially iron ions [13–15]. However, the capacity of the resins in this analysis showed unchanged sorption capacity of the ion exchange resin filling.

The explanation of clogging phenomena due to laboratory and industrial results tends to be the synergic effect of few phenomena resulting in start of clogging by clumping effect of ion exchange resins in the EDI chambers as described previously in the work of Matejka [16].

#### 4. Conclusion

Results of demineralization tests designed to reflect a degree of electrodeionization membrane module clogging did not confirm decreased performance neither for gel nor for macroporous ion exchange resin type. The colloidal particles (both diameters 22 and 120 nm) passed through the electrodeionization membrane module without the interaction.

The problem of irreversibly clogged and clumped EDI during less than 7 d in industrial application was clarified and approved by the chemical qualitative and quantitative analysis. The clogged chambers exhibited higher concentration of metal ions (represented especially by the iron) and colloidal particles with diameter greater than 120 nm (especially, aluminosilicate compounds).

The industrial experience described in this work showed that the clogging of EDI might be dedicated to synergic effect of dissociated multivalent metals and colloidal particles starting by clumping effect in mixed bed.

This work also revealed that there is not significant difference in treatment of water containing colloidal particles using gel or macroporous ion exchange resin filling of electrodeionization chambers.

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