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# Electrodialytic remediation of municipal solid waste incineration residues using different membranes

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## Abstract

In the present work, three different commercial membrane brands were used in an identical electro dialytic cell setup and operating conditions, in order to reduce the leaching of metals and salt anions of two types of municipal solid waste incineration residues: air pollution control residues of a semi-dry flue-gas cleaning system and fly ashes from a plant with wet flue-gas cleaning system. The results showed a general reduction of the leaching in both residues after ED remediation. For the following elements, the leaching was found to be different after ED treatment depending on the membrane used, with statistical significance:

- Air pollution control residues of the semi-dry flue-gas cleaning system: Cr, Cu, Ni, Pb, Zn;
- Fly ashes from a plant with wet flue-gas cleaning system: Al, Ba, Cu, Ni, Zn, Cl, SO<sub>4</sub>.

Final leaching values for some elements and membranes, but not the majority, were below than those of certified coal fly ash (e.g. Al or Cr), a material which is commonly used in construction materials; at the same time, some of these values were reduced to below the Danish law thresholds on the use of contaminated soil in constructions. These results show the potential of ED as a technology to upgrade municipal solid waste incineration residues.

**Keywords:** MSWI; APC residues; Fly ashes; Heavy metals; Electrodialysis

21 **1. Introduction**

22 Worldwide municipal solid waste generation is expected to double by 2025 [1]. Improving solid waste management is  
23 therefore an urgent priority. In many developed countries, a common practice is the incineration of solid waste, as it  
24 reduces the volume of waste by up to 90% [1] and allows energy recovery . Worldwide, approximately 120 million  
25 tonnes of waste are incinerated every year [1]. This practice generates fly ashes (FA) and air pollution control (APC)  
26 residues, originating from the treatment of the flue gas coming out of the combustion chamber, and constituting 1-5% of  
27 the original waste by mass [2]. FA and APC residues are hazardous and contain several pollutants; among them, salts  
28 and heavy metals [3].

29 Both residues can have different potential applications as substitution of other raw materials in production of  
30 construction materials [4]. However, such use is not allowed in some countries like Denmark, where (as in many other  
31 western countries) FA and APC residues are normally deposited after treatment [3]. At the same time, huge amounts of  
32 virgin resources are used in the construction sector. An upgrade to a higher environmental standard (like a reduced  
33 metal and salts leaching), could make them suitable for substitution of raw materials in e.g. concrete production.

34 Electrodialysis (ED) has been proven a promising technology for this purpose [5, 6]. ED is based on application of an  
35 electrical current to a solution or a solid suspension containing ions, forcing cations to move towards the cathode and  
36 anions towards the anode. Ion exchange membranes (IEMs), which are able to let chemical species pass depending on  
37 their charge, are strategically placed to separate cations and anions from an ion-rich compartment into different  
38 compartments. The ED setup used in this work can be seen in Fig. 1.

39 Previous research has focused on optimising different processing parameters for ED treatment of different materials  
40 such as contaminated soils and harbour sediments, municipal solid waste incineration (MSWI) FA/APC residues or  
41 sewage sludge ashes; important parameters being the liquid-to-solid ratio (L/S), duration of treatment, current intensity,  
42 pH as well as the number of cell compartments or the use of chemicals like  $H_2SO_4$  [7-10]. The performance of different  
43 commercial IEMs has been compared in several ED processes like the denitrification of an aqueous solution [11], the  
44 removal of organic salts from a fermentation broth [12], as well as the production of acid from waste pickle solutions  
45 [13]. Their results show that the transport of the same ion through the different IEMs can vary substantially among the  
46 different brands: up to a threefold for  $Na^+$  and  $NO_3^-$ , up to a 1.3-fold for organic salts, and up to a fourfold for  $Cl^-$ . Two  
47 different ion exchange membrane (IEM) brands have been used in different studies of ED upgrading of MSWI APC

48 residues and FA: Ionics [9, 14, 15] and Neosepta [5, 6, 16]. The influence of the IEM brand used for ED treatment of  
49 MSWI residues has, however, never been investigated.

50 To better understand the influence of the IEM type on upgrading of APC residues and other particular materials by ED,  
51 the present work assesses the performance of four IEM trademarks on the ED treatment of MSWI residues. All IEMs  
52 were tested for treatment of two common and different waste materials (MSWI FA and APC residues) in order to  
53 evaluate if the IEMs differently affect the MSWI residue characteristics after the ED treatment. The overall aim of the  
54 investigation was to compare the leachability changes of these residues, induced by the ED treatment, among the four  
55 different IEM brands.

## 56 2. Materials and methods

### 57 2.1 MSWI residues

58 Two different Danish MSWI residues were studied:

- 59 I. APC residues collected from a semi-dry (**SD**) flue-gas cleaning system from the MSWI plant REFA I/S. REFA  
60 I/S incinerates approximately 120,000 tonnes of household and industrial waste annually. SD were collected  
61 on 28<sup>th</sup> of June 2011 from line 3 after APC additives (slaked lime and activated carbon).
- 62 II. **FA** collected from the electrostatic precipitator in a wet flue-gas cleaning system from the MSWI plant  
63 Vestforbrænding I/S, which is the largest plant in Denmark, incinerating approximately 520,000 tonnes of  
64 household and industrial waste annually. FA was collected in week 50 in 2011 from the ash silo 5 prior to  
65 mixing with the wet scrubber sludge.

66 After sampling, both residues were dried at 40°C. They were subsequently dry sieved through a 1 mm sieve, as  
67 performed in previous pilot experiments [5, 6, 16], to prevent clogging in the flow spacers when scaling up. The  
68 removed fraction represented a 1% of the total initial mass [16].

### 69 2.2 Analytical methods

70 Mineralogy, the metal and water soluble salt concentrations and leaching were investigated for both treated and raw  
71 MSWI residues after drying them at 105 °C. Water solubility was measured only for the untreated residues. The  
72 targeted metals were: Al, As, Ba, Ca, Cd, Cu, Cr (total Cr and Cr (VI)), Mn, Mo, Na, Ni, Pb, Zn, V, Zn; whereas the  
73 target anions were: Cl<sup>-</sup>, SO<sub>4</sub><sup>2-</sup>.

74 The mineralogy of each residue sample was examined using X-ray powder diffraction (XRPD) with a PANalytical  
75 X'Pert PRO. Water solubility was calculated by measuring the weight loss after washing the untreated residues: 100 g  
76 of each residue was shaken with 500 mL of distilled water for 5 minutes, and thereafter the supernatant was separated  
77 and filtered at atmospheric pressure. This procedure was repeated two more times for the same residue before all the  
78 suspension was filtered, and dried overnight at 105°C. The metal content was measured by ICP-OES (Varian 720-ES  
79 ICP-OES) in triplicates after pre-treatment by Danish Standard DS259 [17]: 1 g of each residue and 20 mL 7.3 M HNO<sub>3</sub>  
80 were heated at 200 kPa (120°C) for 30 min. The liquid was thereafter separated by vacuum filtration through a 45 µm  
81 filter and diluted to 100 mL by de-ionised (DI) water.

82 The water soluble content of SO<sub>4</sub><sup>2-</sup> and Cl<sup>-</sup> was determined on IC (DIONEX DX-120 IC) in triplicates according to DS /  
83 EN ISO 10304-2 [18] after water extraction. This was performed as follows: 2.5 g of each residue and 25 ml distilled  
84 water were mixed, shaken for 16 hours on a horizontal shaker operating at 150 rpm, and the extractants were vacuum  
85 filtered through 45 µm filters.

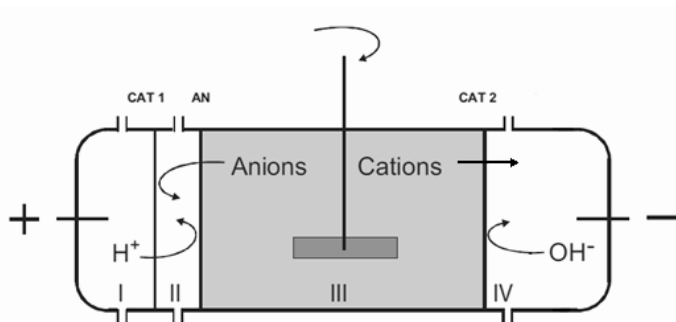
86 Leaching experiments were made according to DS/EN 12457-1 [19] in triplicates. The L/S was 2, mixing 40 g of each  
87 residue with 80 mL DI water. The resulting suspension was shaken at 150 rpm for 24 h before vacuum filtration through  
88 a 45 µm filter and the filtrate was divided into two subsamples after measuring the pH by a Radiometer Analytical pH  
89 electrode. One subsample was used to measure sulphate (SO<sub>4</sub><sup>2-</sup>) and chloride (Cl<sup>-</sup>) concentrations on ionic  
90 chromatograph (IC). The other was diluted at a ratio of 1.25 with concentrated HNO<sub>3</sub>, and heated at 200 kPa (120°C)  
91 for 30 min, before the metal content analysis on ICP-OES (Varian 720-ES ICP-OES).

## 92 2.3 Experimental set-up

93 The Electrodialytic (ED) cell was similar to the ones used in previous experiments [14, 20]. It consisted of four (I, II, III,  
94 IV) compartments (Fig. 1) made of cylindrical Plexiglas® with an internal diameter of 8 cm. Compartment III was 10  
95 cm long and contained in each experiment 35 g ash and 350 ml of DI water. An electrolyte solution (0.01 M NaNO<sub>3</sub>, pH  
96 < 2 adjusted with HNO<sub>3</sub>) was circulated in each of the electrolyte compartments (I, II and IV) using Totton Pumps NDP  
97 10/2. The electrolyte solution was selected based on positive results from previous researches [14, 20], where no  
98 reactions were observed between the electrolyte solution and the transported species (for example, precipitate formation)  
99 or the electrodes (for example, gas formation) was observed. This can also allow a more accurate calculation of the  
100 mass balances explained in section 2.6. The total volumes used were 500 mL for I and IV, and 350 mL for II. A plastic

101 strip attached to a glass-stick connected to an overhead stirrer (IKA RW11) was used to stir the ash suspension during  
102 remediation. The electrodes were made of platinum coated titanium wire (diameter 3 mm) obtained from Permascand®.  
103 An Agilent E3612A DC power supply was used to maintain a constant electric dc current. Details on the IEMs used can  
104 be found in section 2.4.

105 Compartment II was included because a high concentration of soluble salts containing Cl<sup>-</sup> was expected [3]; otherwise,  
106 these anions would enter compartment I, enabling the formation of toxic Cl<sub>2</sub> gas from the electrode reaction at the  
107 anode:  $2\text{Cl}^- \rightarrow \text{Cl}_2 + 2\text{e}^-$



108

109 **Fig. 1** Schematic view of a cell used for the ED treatment of both APC residues. AN: anion-exchange membrane; CAT1  
110 /CAT2: cation-exchange membranes.

## 111 2.4 Membranes

112 Four IEM pairs were selected according to several criteria. The first two trademarks have been used previously in  
113 experimental work with ED of MSWI residues:

- 114 - Ionics (cationic: CR67-HMR, anionic: AR204-SZRA from GE), from GE Water & Process Technologies  
115 (United States), used in laboratory scale ED cell investigations to reduce the content [14, 21] or the leachability  
116 [15] of heavy metals in MSWI residues.
- 117 - Neosepta (cationic: CMX, anionic: AMX), from ASTOM Corporation (Japan), used in two pilot scale ED  
118 stack investigations to reduce the leachability of heavy metals, chloride and sulphate in MSWI residues [5, 6,  
119 16].

120 The other two based on their pH range tolerance (0-14):

- 121 - Ralex® (cationic: CM-PP, anionic: AM-PP), from MEGA a.s. (Czech Republic).

122 - Excellion™ (cationic: I-100, anionic: I-200), from SnowPure, LLC (United States).

123 The pH range is a crucial criterion because of the pH differences between the compartments. MSWI residues, found in  
124 compartment III, had a considerable alkalinity [3, 15, 16]. Contrarily, the solutions in the other compartments were kept  
125 in an acidic range as explained in the section 2.5.

126 Common commercial applications of these IEMs are the deionisation of food products like juices or whey, water and  
127 wastewater desalination, as well as electro-coating of automotive parts. Moreover, Ionics membranes have been used in  
128 research fields like phosphorus recovery from sewage sludge ashes [10] or heavy metal removal from contaminated  
129 soils [22, 23].

## 130 2.5 Experiments

131 Combining the two MSWI residues studied and the four IEMs selected, eight different combinations of ED experiments  
132 were replicated until enough ED treated material was produced to carry out the analyses described in section 2.2. The  
133 duration of the ED treatments was 70 minutes at a current density of 4.67 mA/cm<sup>2</sup>. Voltage and current between the two  
134 electrodes were monitored during the whole experiment. The pH (for all compartments) and electrical conductivity (for  
135 compartment III) were measured at the beginning, at the end, and every 20 min during the ED treatment. During the  
136 experiments, the pH of the compartment IV (Fig.1 ) was adjusted to <2.5 with HNO<sub>3</sub> 1M, to avoid precipitations in the  
137 electrolyte due to the production of OH<sup>-</sup> from electrolysis at the cathode.

138 Electrolyte samples were taken at the beginning and at the end of all experiments for each compartment in order to  
139 study the metal ion mobility among them. At the end of the experiments, the suspension in the central compartment  
140 was filtered at atmospheric pressure and dried during 48 hours at 50 °C. The resulting residue was loosened by hand in  
141 a mortar and stored in plastic bags under dry conditions. The volumes of the filtered liquid from the middle  
142 compartment and the electrolytes were measured. The electrodes were rinsed in 5 mol·L<sup>-1</sup> HNO<sub>3</sub>, whereas the IEMs and  
143 the stirrer in 1 mol·L<sup>-1</sup> HNO<sub>3</sub>. The samples of compartments III and IV were diluted at a ratio of 1.25 with concentrated  
144 HNO<sub>3</sub>, and heated at 200 kPa (120°C) for 30 min, prior to the metal content analyses in all liquids on ICP-OES (Varian  
145 720-ES ICP-OES).

146

## 147 2.6 Calculations

148 Mass balances for each studied metal, defined as the relation between the sum of mass found in the different ED cell  
149 compartments at the end of the experiments and the amount initially found in the mass of untreated MSWI residue  
150 placed in the ED cell, were made to control the quality of the experiments.

151 An analysis of variances (ANOVA) was run for each element and each treated MSWI residue, so as to evaluate whether  
152 the leachability changes after the ED treatment was influenced by the IEM used.

## 153 3. Results and discussion

### 154 3.1 ED experiments performance

155 During the ED remediation with one IEM brand (Excellion), liquid flows between the compartments were observed in  
156 all experiments. After contacting the manufacturer, it was determined that the membranes were degraded during the ED  
157 experiments due to the use of nitrate solutions in the electrolyte compartments (I, II and IV). Specifically, it was  
158 detected that a water permeability as high as  $0.01327 \text{ mL}\cdot\text{min}^{-1}\cdot\text{cm}^{-2}\cdot\text{psi}^{-1}$  (the normal value would be below  $0.0005$   
159  $\text{mL}\cdot\text{min}^{-1}\cdot\text{cm}^{-2}\cdot\text{psi}^{-1}$ ). Thus, the transport mechanisms of ions between compartments might not be dominated by  
160 electromigration, as it can be assumed for the other three IEMs. In consequence, this IEM brand was proven not to be  
161 suitable for the present ED remediation of these MSWI residues, and its results are not further analysed. The other IEMs  
162 did not present major issues during the experiments.

163 The mass balances of the studied metals, defined as the recovered amount of an element in percentage of the initial  
164 amount, was mainly found in a range of 80-120%. This indicates an acceptable quality of the experiments and the  
165 validity of the results presented.

166 The conductivity in compartment III was stable during the ED experiments (results not shown). For the SD, the values  
167 oscillated between 40 and 50 mS/cm, whereas for FA the values oscillated between 25 and 30 mS/cm, with a slight  
168 tendency for decrease over the 70-minute experiments for both residues. The higher conductivity in SD than in FA  
169 experiments is probably due to a higher presence of leached elements from the residues: water solubility was found to  
170 be higher for SD (45.6%) than FA (12.5%). Furthermore, water soluble chloride content was almost three times higher  
171 in SD than in FA before ED (Table 1). Thus, solubilised anions and cations are likely to be higher in the ED  
172 experiments with SD residues. The decrease in the electrical conductivity during the experiments is due to the migration  
173 of ions from the suspension into the cathode and the anode compartments. Similar trends were found in previous works



174 with APC residues from REFA [5, 6]. As a consequence of the electrical conductivity decrease in compartment III,  
175 voltage increased during all experiments by up to 30%. pH in compartment III was stable and between 11 and 12 during  
176 ED remediation for both residues, which is due to the high alkalinity of the MSWI residues.

### 177 3.2 MSWI residues characteristics before and after ED treatment

178 All XRPD diffractograms showing the mineralogy of the treated and untreated residues had a high background due to  
179 the presence of amorphous material in the MSWI residues, as previously seen in other studies [24, 25]. The identified  
180 minerals were calcite ( $\text{CaCO}_3$ ), anhydrite ( $\text{CaSO}_4$ ), portlandite ( $\text{Ca(OH)}_2$ ) and Quartz ( $\text{SiO}_2$ ) for untreated and treated  
181 SD. The same minerals phases, except for  $\text{SiO}_2$ , and together with Calcium chloride hydroxide ( $\text{CaOHCl}$ ), Halite ( $\text{NaCl}$ )  
182 and Sylvite ( $\text{KCl}$ ) were identified for untreated FA. All these phases are consistent with the results from the literature  
183 for these two kinds of MSWI residues [24-29]. After ED treatment of FA, two new main phases, (gypsum ( $\text{CaSO}_4 \cdot \text{H}_2\text{O}$ )  
184 and  $\text{SiO}_2$ ) were found, whereas soluble phases like  $\text{NaCl}$  and  $\text{KCl}$  were not identified. Also, XRD characteristic peaks  
185 for  $\text{Ca(OH)}_2$  had lower intensity for SD and were not detected for FA after the ED experiments; similarly,  $\text{CaOHCl}$   
186 peaks disappeared after ED treatment of FA. These results indicate that a considerable part of  $\text{NaCl}$ ,  $\text{KCl}$ ,  $\text{CaOHCl}$  and  
187  $\text{Ca(OH)}_2$  dissolved during ED. Anhydrite intensity peak increased after all ED treatments, which can be attributed to a  
188 higher proportion due to its low solubility and the removal of other compounds.

189 Table 1 show the metal and the soluble salts content of both MSWI residues before and after ED. Overall, and as seen  
190 in a previous study [5], the total concentration of all metals except Na increased slightly, whereas Cl and  $\text{SO}_4$  content  
191 were considerably reduced after the ED experiments (Table 1). These facts confirm that water-soluble salts, like  $\text{NaCl}$ ,  
192 and  $\text{KCl}$  for both residues, and possibly  $\text{Na}_2\text{SO}_4$  for FA, in the untreated residues, were dissolved and removed during  
193 ED. Despite their dissolution, Na as well as the water-soluble part of Cl and  $\text{SO}_4$  were, together with Al, Ca, Pb and Zn,  
194 the major compounds found for both MSWI residues before and after ED treatment.

195

196

**Table 1** Metal and water-soluble salt anion content of SD and FA (mean value±standard deviation). The results after

197

ED are calculated as an average of the results of the three IEMs.

| Compound (Units)       | SD        |           | FA        |           |
|------------------------|-----------|-----------|-----------|-----------|
|                        | Before ED | After ED  | Before ED | After ED  |
| Al (g/kg)              | 15.5±0.09 | 21.4±1.11 | 29.4±0.36 | 35±0.74   |
| As (mg/kg)             | 57.7±2.13 | 68.4±5.98 | 86±2.19   | 95.5±5.02 |
| Ba (mg/kg)             | 374±6.35  | 409±24.2  | 692±19    | 797±49.6  |
| Ca (g/kg)              | 296±3.52  | 301±11.7  | 186±2.98  | 198±5.84  |
| Cd (mg/kg)             | 134±0.78  | 162±16.6  | 95.3±1.37 | 107±4.99  |
| Cr (mg/kg)             | 104±0.87  | 125±7.54  | 97.2±5.17 | 109±4.15  |
| Cu (mg/kg)             | 519±3.94  | 662±31.2  | 712±5.87  | 879±134   |
| Mn (mg/kg)             | 352±10.4  | 480±13    | 649±10.2  | 739±14.2  |
| Mo (mg/kg)             | 35.5±0.49 | 43.1±2.05 | 55.4±0.87 | 63.2±3.05 |
| Na (g/kg)              | 32.5±0.68 | 5.38±0.37 | 34.6±0.32 | 5.88±0.11 |
| Ni (mg/kg)             | 27.2±1.13 | 33.3±4.23 | 48.5±10.3 | 47±3.6    |
| Pb (g/kg)              | 2.36±0.03 | 2.38±0.14 | 2.90±0.05 | 2.70±0.09 |
| V (mg/kg)              | 35.1±0.78 | 40.7±2.61 | 46.2±3.01 | 56.7±6.04 |
| Zn (g/kg)              | 15.8±0.26 | 21.1±1.21 | 20.7±0.08 | 23.1±1.02 |
| Cl (g/kg)              | 160±2.15  | 15.5±1.10 | 60.7±1.63 | 2.41±0.11 |
| SO <sub>4</sub> (g/kg) | 14.9±0.03 | 10.7±0.37 | 34.3±0.98 | 14.7±0.35 |

198

199

## 200 3.3 Leaching of elements

### 201 3.3.1 General behaviour before and after ED treatment

202 Tables 2 and 3 show the results from the leaching tests. Category 3 values, the Danish guideline levels for water  
203 leachable metals and salts for the reuse of residues as in construction materials [30], are also included. MSWI FA/APC  
204 residues are generally classified as hazardous and thus these legislation limits would not be applicable, but they can be  
205 used as reference to understand the upgrade made after ED experiments.

206 Ba, Pb and Zn leaching was considerably lower in FA than in SD (Tables 2 and 3), even though their content was  
207 similar (Table 1). One reason can be a higher concentration of more alkaline soluble compounds containing either of  
208 these elements in SD, such as  $\text{PbCl}_2$ ,  $\text{PbO}$  [31],  $\text{ZnCl}_2$  (previously identified in MSWI residues [26]) or  $\text{BaCl}_2$ . Another  
209 explanation can be the lower pH at the end of the leaching test in FA than in SD (Tables 2 and 3): previous researches  
210 showed a lower leaching of Ba, Pb and Zn at pH 11 than at pH 12 in MSWI residues [15, 32-34].

211 Overall, average leaching for metals and salts was reduced during ED treatment, a tendency also observed in previous  
212 researches on ED remediation of FA and APC residues [5, 6, 15, 16]. The explanation for this trend can be the  
213 dissolution and consequent removal to the electrolyte compartments (I, II and IV in Fig. 1) of the leachable part during  
214 the ED treatment. Moreover, ions migration through the IEMs can enhance the dissolution of some compounds by  
215 equilibrium displacement. Leaching was reduced, on average, from above to below Category 3 limits for Ba and Na in  
216 SD and for As, Na, Zn, Cl,  $\text{SO}_4$  in FA. Na and Cl leaching reduction are consistent with the decrease in total  
217 concentrations (Table 1). Average leaching values were also reduced from above to below Category 3 limits for As in  
218 SD regardless the IEM used, as well as for Pb in FA treated with Ralex IEMs.

219 Notable exceptions were Cr and Ni for both SD and FA, as the leaching increased after the ED experiments, in some  
220 cases from below to above Category 3 limits (Table 2 and 3). The other exception was  $\text{SO}_4$  in SD, with a leaching  
221 slightly higher after ED treatment. For each element, a separate discussion is needed:

222 - The increase of Cr leaching after the experiments was also observed in previous researches for ED remediation  
223 of similar FA and APC residues [5, 15, 16]. Cr-solubility in SD and FA has been suggested in other researches  
224 to be controlled rather by the presence of sparingly-soluble Cr(VI) phases, like  $\text{BaCrO}_4$  and  $\text{PbCrO}_4$ , than by  
225 Cr(III) phases [26, 35]. In fact, the solubility of these Cr(VI) compounds at a high pH is higher than for Cr(III).  
226 For instance, an alkaline extraction of  $\text{PbCrO}_4$ -spiked soils at L/S 20 released up to 50,000  $\mu\text{g Cr/L}$  [23]; in

227 contrast, Cr(III) solubility at pH~12 is limited to around 10 µg/L[36]. The increase in Cr solubilisation after  
228 ED can be due to the removal of soluble Ba and Pb compounds, other than chromates: leaching values of Pb  
229 for both residues, and Ba in SD, were noticeably reduced after ED treatment (Tables 2 and 3). Ba and Pb ions  
230 scarcity in the leaching experiments after ED treatment can favour Cr dissolution. On the contrary, the higher  
231 concentration of Ba and/or Pb ions in the untreated residues leaching experiments may prevent the dissolution,  
232 or re-precipitate in case of release, of Ba- and Pb- chromates.

233 - Ni leaching increase was not generally observed before in others researches using ED to remediate similar  
234 MSWI FA and APC residues [5, 16]. The reason can be the enhanced dissolution of Al-Ni layered double  
235 hydroxide (LDH), which was found to limit Ni solubility in cement materials [37], during ED: mobilized Al  
236 during the experiments is around 20 and 50 times higher than in the leaching tests of untreated SD and FA,  
237 respectively. Thus, a higher dissolution of Al could imply a partial dissolution of Ni hydroxides, which have a  
238 higher solubility than in Al-Ni LDH.

239 - SO<sub>4</sub> leaching increase after ED treatment of SD, which was previously observed in another study for the same  
240 APC residues ([16] and Table 2), can be due to the removal of soluble Ca compounds during ED: Ca leaching  
241 in SD was considerably reduced (Table 2) after ED. The higher presence of Ca cations in the untreated than  
242 ED-treated SD leaching test can decrease CaSO<sub>4</sub> solubility in the former. After ED treatment, the lower  
243 content of Ca in the leaching tests can favour CaSO<sub>4</sub> dissolution.

### 244 3.3.2 Comparison with previous results, Category 3 guidelines and certified coal fly ash values

245 Compared with a previous study on the same MSWI residues [16], the leaching values before the ED treatment were  
246 within two standard deviations for all elements, except for Pb in FA which were still in the same order of magnitude  
247 (Tables 2 and 3). Leaching values for untreated SD and FA were also in the same order of magnitude for Cr, Cu, Pb and  
248 Zn as another investigation about the same residues [15]. After ED, leaching of most elements were in the range of the  
249 results in [16] (in which Neosepta was used) for SD (Table 2), and within two standard deviations for FA (Table 3).  
250 Moreover, they had the same order of magnitude for Cr, Cu, Pb and Zn than the values obtained in [15] with a 3-  
251 compartment ED cell using Ionics. Therefore, the leaching values before and after ED were in line with previous  
252 investigations.

253 For SD, Category 3 limits were exceeded on average for Cr, Pb, Zn and Cl after treatment regardless the IEM used and  
254 for Ni after treatment using Ionics and Neosepta IEMs (Table 2). The variability of As values was high enough for

255 Ionics and Neosepta IEMs, so that Category 3 limits could be exceeded or not within one standard deviation (Table 2).  
256 For FA, Category 3 limits were exceeded on average for Pb after treatment with Ionics and Neosepta IEMs; remarkably,  
257 Category 3 limits were within one standard deviation below the mean value for both IEM (Table 3). Category 3 limits  
258 were also slightly exceeded on average for Ni after ED treatment with the three IEMs. It should be noted that there was  
259 variability enough for Cr for all IEMs, for Ni with Ralex and for As with Ionics and Ralex to exceed or not Category 3  
260 limits within one or two standard deviations (Table 3). Leaching values exceeding Category 3 for Pb, Zn and Cl were  
261 observed in a previous work with APC residues from the REFA plant [5]; in that research, the combination of the ED  
262 treatment with carbonation was successful in order to reduce the leachability for most of heavy metals except for Cr. A  
263 similar treatment could have a potential beneficial effect for the two MSWI residues of this study, although it requires  
264 further study.

265 Certified coal fly ash is commonly used in cement production, in contrast to MSWI APC residues, despite that its Cr  
266 leaching values normally exceeds Category 3 limits leaching values [38]. In general, considering a range of two  
267 standard deviations, ED treated SD and FA have a higher metal leaching than certified coal fly ashes. Exceptions for  
268 treated FA were Al, Ba, Ca and Cr, as their leaching in coal fly ashes is higher: 2,600 µg/L, 0.7 mg/L, 1,200 g/L and  
269 1,000 µg/L [38], respectively (see Table 3 for comparison). For treated SD, the exceptions were Al and Cr for some  
270 IEMs (see Table 2 for comparison).

271

**Table 2** Metal and salt leaching results from SD according to DS/EN 12457-1 (mean value±standard

272

deviation). Average values exceeding Category 3 limits are shown **in bold**. \*Below ICP limit of detection

273

(0.02 mg/L, LOD) \*\* Range of average values.

|                       | Before ED     | After ED        |                 |                 | Previous research [16] |                | Category 3 [28] |
|-----------------------|---------------|-----------------|-----------------|-----------------|------------------------|----------------|-----------------|
|                       |               | Ionics          | Neosepta        | Ralex           | Before ED              | After ED **    |                 |
| pH                    | 12.3±0.04     | 12.1±0.01       | 12.1±0.01       | 12.1±0.08       | 11.9                   | 10.2-12.2      | -               |
| As (µg/L)             | <b>91±15</b>  | 43±16           | 36±4.2          | 48±25           | <91                    | <20-650        | 50              |
| Ba (mg/L)             | <b>21±4.4</b> | 1.7±0.14        | 1.7±0.08        | 1.6±0.14        | <b>17±5</b>            | 1.6-3.0        | 4               |
| Cd (µg/L)             | <20*          | <20*            | <20*            | <20*            | <20                    | <20            | 40              |
| Cr (µg/L)             | 64±15         | <b>1,200±45</b> | <b>760±40</b>   | <b>880±77</b>   | 61±2                   | 114-1,015      | 500             |
| Cu (µg/L)             | 1,200±1,500   | 61±4.0          | 55±5.7          | 240±15          | 889±397                | 32-141         | 2,000           |
| Mn (µg/L)             | 100±160       | <20*            | <20*            | <20*            | <100                   | <20            | 1,000           |
| Na (g/L)              | <b>14±2.1</b> | 1.1±0.04        | 1.2±0.05        | 1.1±0.09        | <b>13±2</b>            | <b>1.6-7.8</b> | 1.5             |
| Ni (µg/L)             | <20*          | <b>110±13</b>   | <b>86±6.9</b>   | 56±5.1          | <20                    | <20-72         | 70              |
| Pb (mg/L)             | <b>570±77</b> | <b>4±0.03</b>   | <b>3.5±0.81</b> | <b>14±1.3</b>   | <b>453±168</b>         | <b>1-21</b>    | 0.1             |
| Zn (mg/L)             | <b>39±3.9</b> | <b>4.5±0.13</b> | <b>4.4±0.45</b> | <b>6±0.41</b>   | <b>32±10</b>           | <b>0.1-3.7</b> | 1.5             |
| Al (µg/L)             | 930±120       | 360±71          | 270±14          | 220±280         | -                      | -              | -               |
| Ca (g/L)              | 32±4.7        | 4.8±0.13        | 4.9±0.2         | 4.7±0.43        | -                      | -              | -               |
| Mo (µg/L)             | 770±57        | 600±23          | 600±16          | 550±50          | 770                    | 211-708        | -               |
| V (µg/L)              | <20*          | <20*            | <20*            | <20*            | <20                    | 311-662        | -               |
| Cl (g/L)              | <b>85±1.7</b> | <b>8.3±0.25</b> | <b>8.9±0.26</b> | <b>8.7±0.36</b> | <b>87±3</b>            | <b>5.1-35</b>  | 3               |
| SO <sub>4</sub> (g/L) | 0.87±0.03     | 1.3±0.03        | 1.3±0.01        | 1.3±0.03        | 0.9±0                  | 0.9-1.2        | 4               |

274 **Table 3** Metal and salt leaching results from FA according to DS/EN 12457-1 (mean value±standard  
 275 deviation). Average values exceeding Category 3 limits are shown **in bold**. \*Below ICP LOD. \*\* One of the  
 276 triplicate measurements value below LOD, so the result is shown as an average of the other two triplicates and  
 277 LOD/2.

|                       | Before treatment | After treatment  |                  |               | Previous research [16] |                |                | Category 3 [28] |
|-----------------------|------------------|------------------|------------------|---------------|------------------------|----------------|----------------|-----------------|
|                       |                  | Ionics           | Neosepta         | Ralex         | Before ED              | After ED       |                |                 |
| pH                    | 11.3±0.24        | 11.1±0.01        | 11.1±0.01        | 11.0±0.03     | 12.6                   | 10.1±0.1       | 11.9±0.4       | -               |
| As (µg/L)             | <b>59±20</b>     | 40±14            | 36±2.8           | 48±22         | <20                    | <20            | <29            | 50              |
| Ba (mg/L)             | 0.46±0.05        | 0.29±0.02        | 0.3±0.003        | 0.22±0.02     | 0.4±0.03               | 0.8±0.9        | 2.6±0.1        | 4               |
| Cd (µg/L)             | <20*             | <20*             | <20*             | <20*          | <20                    | <20            | <20            | 40              |
| Cr (µg/L)             | 18±7.5**         | 430±44           | 480±49           | 460±46        | 20±0                   | <b>991±234</b> | 341±146        | 500             |
| Cu (µg/L)             | 30±6.4           | 26±1.7           | 20±3             | 16±3.5        | 23±11                  | 37±14          | 28±25          | 2,000           |
| Mn (µg/L)             | <20*             | <20*             | <20*             | <20*          | <20                    | <20            | <20            | 1,000           |
| Na (g/L)              | <b>12±0.8</b>    | 0.43±0.02        | 0.46±0.02        | 0.5±0.04      | <b>14±2</b>            | <b>3.6±1.1</b> | <b>3.1±0.6</b> | 1.5             |
| Ni (µg/L)             | <20*             | <b>130±21</b>    | <b>140±7.7</b>   | <b>75±8.4</b> | <20                    | < <b>193</b>   | <47            | 70              |
| Pb (mg/L)             | <b>18±2.4</b>    | <b>0.47±0.74</b> | <b>0.13±0.13</b> | 0.03±0.01     | <b>40±2</b>            | <b>1.8±2.2</b> | <b>18±11</b>   | 0.1             |
| Zn (mg/L)             | <b>4.5±0.67</b>  | 0.57±0.26        | 0.46±0.13        | 0.27±0.02     | <b>3.3±1.1</b>         | 0.2±0.0        | <b>3.5±2.8</b> | 1.5             |
| Al (µg/L)             | 610±44           | 1,100±130        | 1,000±96         | 150±14        | -                      | -              | -              | -               |
| Ca (g/L)              | 1.4±0.07         | 0.97±0.01        | 0.98±0.01        | 0.93±0.06     | -                      | -              | -              | -               |
| Mo (µg/L)             | 2,000±150        | 640±27           | 660±14           | 630±48        | 2,024±48               | 704±76         | 497±16         | -               |
| V (µg/L)              | <20*             | <20*             | <20*             | <20*          | 4±55                   | <b>135±177</b> | <b>264±339</b> | -               |
| Cl (g/L)              | <b>31±0.21</b>   | 1.4±0.03         | 1.4±0.02         | 1.6±0.06      | 31±1                   | <b>14±11</b>   | <b>20±7</b>    | 3               |
| SO <sub>4</sub> (g/L) | <b>8.4±0.30</b>  | 1.6±0.004        | 1.7±0.04         | 1.8±0.04      | 9±0.5                  | 1.9±0.4        | 1.0±0.4        | 4               |

### 278 3.3.3 Difference in leaching values among the IEMs

279 The result of the ANOVA among the different ED treatments for each residue with a P-value>0.05 showed that mean  
280 leaching values were found not to be the same for some of the elements among the three IEMs studied. More  
281 specifically, mean leaching values of Cr, Cu, Ni, Pb and Zn for the treated SD, and Ba, Cu, Ni, Al, Cl and SO<sub>4</sub> for the  
282 treated FA were found to be different depending on the IEM used. It is worth mentioning that Pb and Zn leaching  
283 values had a high variability for FA treated with Ionics and Neosepta IEMs (Table 3), which requires further study. The  
284 reason could not be the pH of the different leachates, since they were almost identical regardless the membrane used  
285 (Tables 2 and 3).

286 Main differences were found for Pb in SD: leaching after ED treatment with Ralex was around 3 to 4 times higher than  
287 with the other two IEMs (Table 2). The reason can be a different transportation rate of chloride anions through the IEMs.  
288 Ralex IEMs are likely to transport Cl<sup>-</sup> at a slower rate than Ionics and Neosepta. This can be seen in the water soluble Cl  
289 content for both residues, which was slightly higher after ED treatment with Ralex than with Ionics or Neosepta: 17.0,  
290 15.6 and 15.0 mg/kg for SD and 2.54, 2.36 and 2.32 mg/kg for FA, respectively. A slower migration of Cl through  
291 Ralex IEMs can entail a lower dissolution of PbCl<sub>2</sub> by equilibrium displacement, and, consequently, the content of  
292 sparingly soluble PbCl<sub>2</sub> in SD after ED treatment with Ralex can be higher than when using Ionics or Neosepta.  
293 However, the transportation of Cl<sup>-</sup> and the other species through the different IEM brands, and its connection to  
294 differences in leachability, require specific research for proper confirmation.

## 295 4. Conclusions

296 The leaching of some heavy metals and salts in two MSWI residues was found to be different after ED treatment  
297 depending on the IEM brand used, without achieving a better overall result for any of them. Further investigation is  
298 required in order to understand the unlike transportation of chloride and other ions among different IEMs during ED,  
299 and its influence in the leaching behaviour of the hazardous elements from MSWI residues.

300 ED remediation resulted in the reduction of the leachability for most heavy metals and salts, probably due to the  
301 removal of the leachable part. For some metals, like Al or Cr, the leaching was reduced to below the values found in  
302 certified coal fly ash. At the same time, leaching was reduced for some elements like As from above to below the  
303 Danish guideline levels for the use of residues as construction materials. However, none of the resulting ED-treated



304 MSWI residues had all the leaching values below these levels, and for most elements the final values were higher than  
305 for certified coal fly ash.

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