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ORIGINAL PAPER

Electroremediation of air pollution control residues in a continuous reactor

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Abstract Air pollution control (APC) residue from municipal solid waste incineration is considered hazardous waste due to its alkalinity and high content of salts and mobile heavy metals. Various solutions for the handling of APC-residue exist, however most commercial solutions involve landfilling. A demand for environmental sustainable alternatives exists and electrodialysis could be such an alternative. The potential of electrodialysis for treating APC-residue is explored in this work by designing and testing a continuous-flow bench-scale reactor that can work with a high solids content feed solution. Experiments were made with raw residue, water-washed residue, acid washed residue and acid-treated residue with emphasis on reduction of heavy metal mobility. Main results indicate that the reactor successfully removes toxic elements lead, copper, cadmium and zinc from the feed stream, suggesting that the electrodialytic technique is an alternative to land deposition of APC-residues.

Keywords Fly ash · Electrodialytic treatment · Heavy metals · Incineration

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1 Introduction

During combustion of municipal solid waste in incinerators a waste by-product is generated, air pollution control residue (APC), which results from the treatment of the flue gas coming out of the combustion chamber. APC is considered hazardous waste due to its alkalinity and high content of salts, mobile heavy metals and organics [1]. In Denmark all non-reusable waste is incinerated for energy production, generating around 90,000 tons of APC every year. As the waste production in Denmark is around 1% of the total EU-waste generation, and EU waste strategies for the future resemble Danish practice, the future potential APC generation in EU may approach 9 million tons a year. Various solutions for the handling of APC-residue exist in different regions; however, most commercial solutions involve deposition after inertization, in special waste landfills. Inertization requires additional raw materials and increases overall mass and volume of waste. Several applications have been mentioned for reusing APC residues [1] but if they are landfilled the materials in them are permanently lost. A demand for more environmentally sustainable alternatives exists. Electrodialysis (ED) could be such an alternative. A DC field is applied, forcing the contaminants out of the waste, while ion-exchange membranes strategically placed in the path of the contaminants control the flow of contaminants into specific chambers, from where they can be removed. ED has been investigated for treatment of not only APC residues [2, 3] but also other types of heavymetal contaminated waste such as soil [4–8], biomass ash [9], timber waste [10] and mine tailings [11, 12]. ED remediation of waste is still at research level and has been conducted in reactors ranging in size from small laboratory acrylic cells of 10 by 4 cm to larger pilot reactors the size of a room. In 2002 the stirred cell system was introduced by Pedersen [13], replacing the earlier static cells and allowing for reduced remediation time. For ED of waste to be able to go full scale at industrial facilities another evolution is still necessary. Most industrial processes generate residues continuously, while most of the treatment systems developed so far are of batch-type. REFA, a municipal solid waste incinerator in Denmark has shown interest in implementing a pilot plant for ED treatment of its APC-residue, making it imperative to develop a new treatment system that can handle waste in a continuous way and which will serve as a base for the future pilot plant. The aim of this work was therefore to develop a functional continuous-flow bench-scale reactor and test its applicability for ED treatment of APC-residues. The primary focus of this work is on reduction of heavy metal mobility and the influence on this of various pretreatments (washing, acid washing and acidification) of the APC.

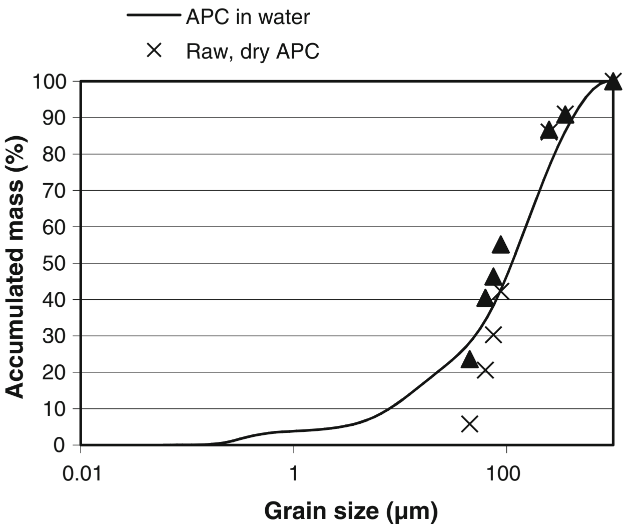


Fig. 1 Cumulative grain size distributions of APC residue

2 Materials and methods

2.1 APC residue

The APC-residues (REFA APC) were collected on the 2nd of May 2006 from a municipal waste incinerator in Denmark (Faelleskommunal Affaldsbehandling for Lolland-Falster—I/S REFA) with a dry flue-gas cleaning system. The APC-residue includes by-products added for the flue-gas-cleaning. APC residues were stored dry in a 10 L bucket with lid at the laboratory. Table 1 shows the heavy metal content of the APC residue as well its solubility and pH. Figure 1 shows the grain size distribution.

2.2 Analysis

To determine heavy metal and sodium content three subsambles were taken from the bucket for analysis by ICP-OES (Varian 720-ES ICP-OES, Software version: 1.1.0) after digestion using a microwave (Multiwave 3000, Microwave Sample Preparation, Firmware Version: v.14, software version v.1.27). Microwave digestion was in nitric acid (0.25 g of sample in 10 mL concentrated nitric acid) at 800 W power for 1 h (ramping time 30 min + hold time 30 min), followed by an additional holding time of 15 min at 0 W power.

Table 1 Chemical and physical characterization of APC residue

Parameter	Value
Pb (mg/kg)	5,783 ± 421
Cd (mg/kg)	167 ± 5
Zn (mg/kg)	24,581 ± 4,481
Cu (mg/kg)	568 ± 17
pH	12
Solubility (%)	35.2 ± 0.4

Solubility was determined by weight loss after mixing 10 g of sample with 1 L distilled water, shaking for 72 h at 150 rpm (shaking table model Gerhardt Laboshake LS 500/RO 500), filtering through 0.45 µm cellulose nitrate filter (Sartorius AG), washing with distilled water and centrifuging at 3,000 rpm for 15 min until water Cl-deficient and transferring undissolved residual into a glass beaker and drying at 105 °C.

Electrical conductivity was measured after mixing 5 g of of APC residue in 12.5 mL of distilled water, shaking for 30 min and settling for 20 min.

pH was measured after mixing 5 g of APC in 12.5 mL 1 M KCl solution, shaking for 1 h and settling for 10 min. All analyses were made in triplicate. The grain size in water was analyzed by laser diffraction. Grain size distribution of raw, dry APC and water washed, dried APC was analyzed with sieves.

2.3 Experimental set-up

Conventional electrodialysis consists of a stack of alternating ion-exchange membranes, separated by spacers. This set-up results in a succession of alternating feed and concentration chambers [14, 15], as schematically depicted in Fig. 2.

Conventional electrodialysis is made on liquid solutions, while the APC suspension has a high solids content. Development of a continuous-flow bench-scale reactor for the APC was done by adapting an electrodialysis cell previously developed by Jurag Separation A/S for

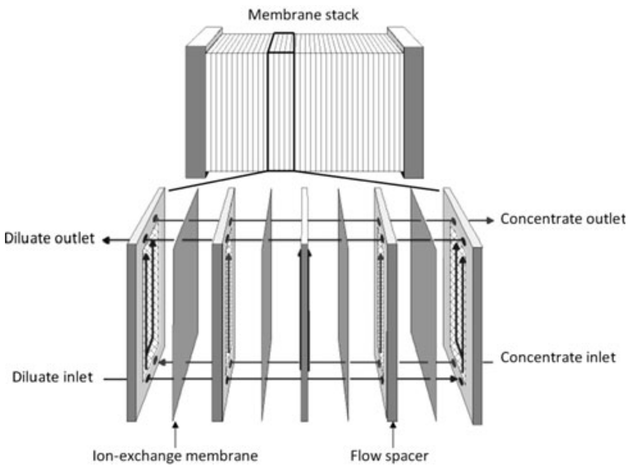
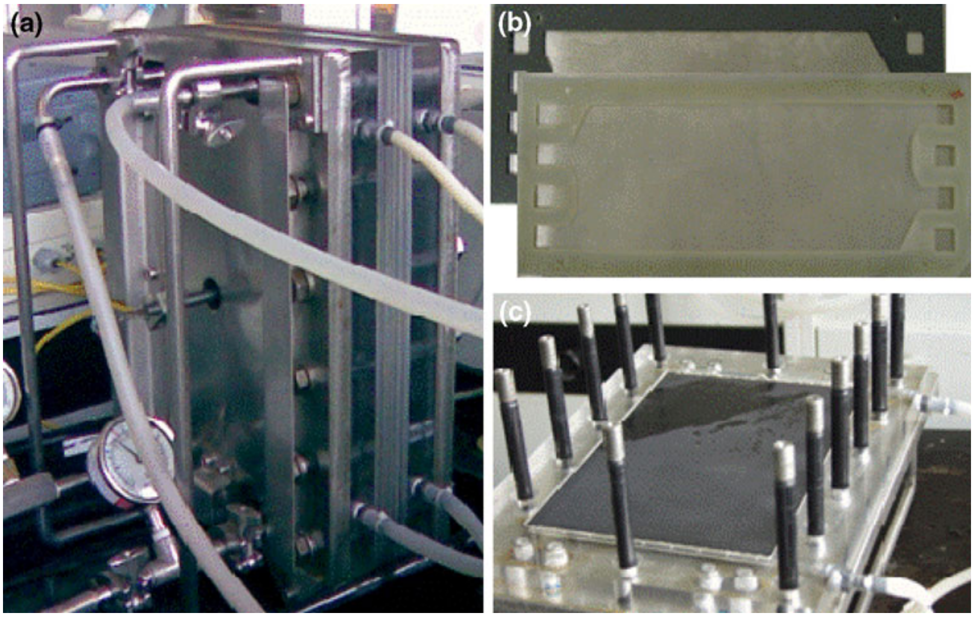


Fig. 2 Schematic representation of conventional electrodialysis

treatment of fermentation products. Process solutions from fermentation processes have a high solid content and thereby resemble APC residues in suspension.

Preliminary testing showed that the original Jurag ED cell would clog after a short time when fed with a 10% suspension of APC-residue. To accommodate the larger particles present in APC-residue and allow for higher suspension concentrations, a better ‘sheet flow’ version of the feed spacer was developed, in which access holes are 3 mm in diameter and the spacer is 5 mm wide. A stack with two cell pairs (two concentrate spacers and two diluate spacers) and a membrane surface area of 530 cm^2 was used. This spacer design was found able to handle 30% APC suspension without clogging, and was therefore used in the ED experiments reported here. Conventional 0.6 mm wide spacers were used for the concentrate solution. The bench-scale electrodialytic cell used is shown in Fig. 3. The process solution was introduced by a hole in the bottom of each feed chamber and left by a hole in the top. Concentration chambers also had an inlet in the bottom and an outlet, allowing the circulation of the concentrate solution. As the feed solution was pumped through the feed chambers, metals migrated into adjacent chambers under the electric field (concentration chambers) through the ion-exchange membranes. The electrodes were plates of titanium covered by a layer of metal oxides placed at both ends of the stack. The electrodes were separated from the concentrate/diluate by ion-exchange membranes and an electrode rinse solution was circulated in the electrode compartment. The spacers, membranes and electrodes were assembled into a stack using screws and bolts, which tightly bond each item of the system and prevent leakages. The whole set-up is schematically depicted in Fig. 4. The solid black circuit represents the diluate (APC) circuit. The grey circuit represents the concentrate circuit, and the dotted black, the electrode rinse. The PC collected data on voltage and current. The APC was kept in suspension by constant stirring with plastic-flaps attached to a glass-stick and connected to an overhead stirrer (RW11 basic from IKA). Nitric acid (HNO_3) was used for both electrode rinse (0.25 M) and for initial concentration solution (0.01 M). The APC suspension and concentrate solution (2 L each) were circulated through the ED stack using mechanical pumps (Totton Pumps Class E BS5000 Pt 11) with constant electrical current (5 mA/cm^2), while voltage increased or decreased according to the changing electrical resistance (as the conductivity of the process solutions changed). The

Fig. 3 Bench scale electrodialytic cell used for the treatment of APC-residues. From *left, clockwise*: **a** assembled cell; **b** spacers; **c** opened cell, with the electrode plate showing



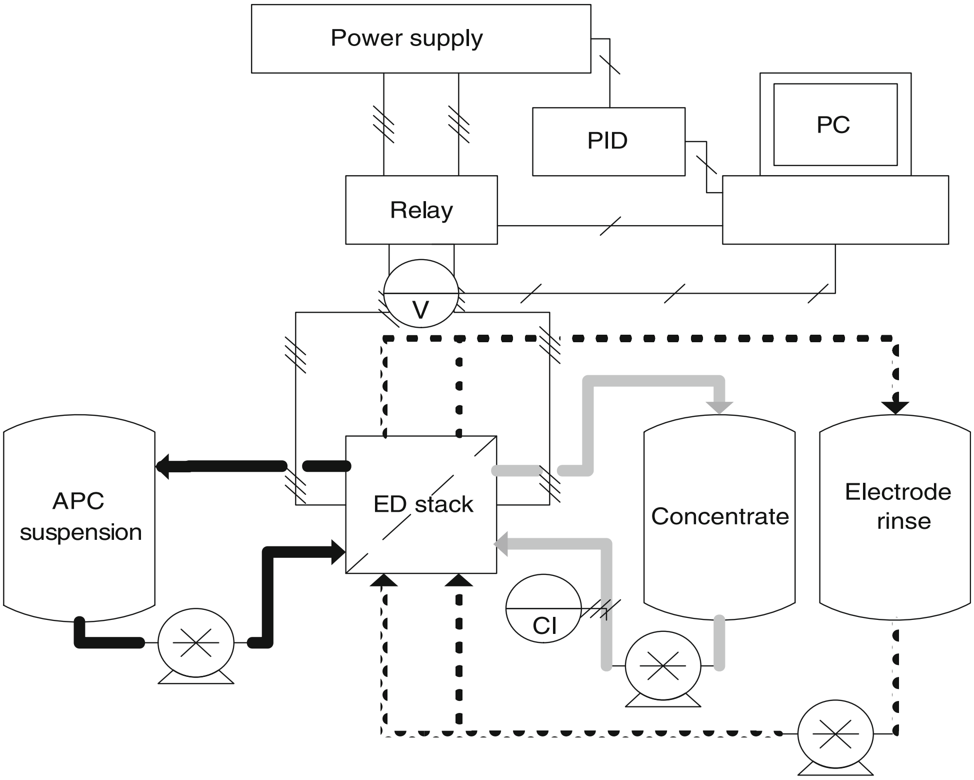


Fig. 4 Schematic presentation of the experimental setup. The solid blackcircuit represents the diluate (APC) circuit. The grey circuit represents the concentrate circuit, and the dotted black, the electrode rinse

electrolyte solutions from cathode and anode end were mixed in a plastic container in a closed system through a rubber cork through which a third tube vented gasses (e.g. chloride gasses) out. The membranes used were Neosepta CMX (cation-exchange membrane) and Neosepta ASM (anion-exchange membrane) from Astom (Japan).

2.4 Experiments

Four experimental ED-treatments of APC residue were made (Table 2). In all experiments, the APC was sieved through a 0.710 mm sieve prior to treatment and only the finegrained fraction (representing 97.4%, by mass) was treated. The current density was set to 5 mA/cm^2, and the maximum voltage was 15 V. In experiment 1, 200 g of untreated (Raw) APC was mixed with 2 L of distilled water in a plastic beaker and treated directly by ED. In experiment 2, the APC was pre-washed before electrodialysis by mixing 200 g of APC with 2 L of distilled water in a plastic beaker, stirring for 18 h and centrifugation for 15 min at 3,000 rpm. The supernatant was decanted and stored for analysis. Before ED 2 L of distilled water were added. In experiment 3, the APC was acid pre-washed by mixing 200 g of APC with 2 L of 0.2 M HNO_3 in a plastic beaker, stirring for 18 h and centrifugation for 15 min at 3,000 rpm. The supernatant was decanted and stored for analysis. Before ED 2 L of distilled water were added. In experiment 4 the APC was treated like in experiment 3, however subsequently titrated with nitric acid prior to the ED-treatment to pH 7 and during the first 5 h of the ED-treatment to pH 2. All experiments lasted for 27 h which is equivalent to a retention time of the APC solution inside the ED-stack of approximately 16 h.

After the experiments, the APC residue was separated from the diluate by filtering the suspension through 45 μm filters. The APC residue was dried at 40 °C prior to digestions for analysis of the total heavy metal content and pH, and for heavy metal leaching experiments.

Table 2 Electrodialytic experiments

Experiment	APC	Pretreatment of APC	Treatment of APC during ED
1	R	None (raw APC)	None
2	WPW	Water pre-washed	None
3	APW	Acid pre-washed	None
4	AT	Acid pre-washed and acid titrated	Acid titrated

2.5 Leaching tests

Leaching tests were conducted according to the Danish act no. 1480 of 12/12/2007 on raw and treated APC-residue by mixing APC and distilled water at a liquid to solid ratio (L/S) of 2. The suspension was then shaken using a shaking table (Gerhardt Laboshake LS 500/RO 500) for 6 h, and filtered through 0.45 cellulose nitrate filter (Sartorius AG). The liquid fraction was analyzed by ICP-OES (Varian 720-ES ICP-OES, Software version: 1.1.0) to reveal the leaching of target elements Cd, Cu, Zn and Pb.

3 Results and discussion

3.1 Removal of heavy metals

Experiments were made according to the procedure described in the experimental set-up section. Table 3 shows the concentration of the selected elements Pb, Cu, Cd and Zn in the washing liquids, the diluate (prior to and after ED-treatment) and in the concentrate (after ED-treatment). The primary goal of pre-washing was to remove soluble non-toxic salts like chloride and calcium which constitute a major part of common APC-residues [16] to prevent a high energy consumption for removal of these highly soluble salts during ED-treatment. In exp. 2, however, the water pre-washing extracted not only non-toxic salts but also high levels of zinc and in particular lead, which is highly mobile at the natural alkaline pH of the APC due to the formation of soluble lead hydroxides [17]. Our aim was to concentrate the mobile and toxic elements by ED-treatment, and thus hinder dissolution of the toxic elements in large volumes of liquid, further we believe that handling or possible reuse of the produced solutions would be simplified by separation of the nontoxic salt from the toxic metals. Therefore washing at pH 10 was tested in experiments 3 and 4, as solubility of zinc and in particular lead is lower at this pH than at pH 12 [18]. As assumed concentrations of lead and zinc in the washing liquids from these experiment were much lower and thus our goal was obtained.

The initial concentrations in the diluate similarly reflect the pre-treatment: in exp. 1 the lead and zinc concentrations were high while in exp. 2, from which the fraction mobile in water was already washed out, the initial concentrations were much lower. In exp. 3 and 4 in which pH was 10 and thereby the mobility of the investigated elements was reduced, the initial diluate concentrations were equally low. The final diluate concentration was reduced for all elements in exp. 1, however a relatively large amount of dissolved lead remained in the solution. In exp. 2 the lead concentration was substantially reduced in the diluate while the concentration of the remainder elements increased. In experiment 3 the diluate concentration of all elements and in particular zinc increased, and finally in experiment 4 the lead and cupper concentrations increased while the cadmium and zinc concentrations decreased.

Table 3 HM concentrations in liquids (ppm) after washing, before EDR (start) and after EDR (end)

Liquid/experiment	Experiment 1	Experiment 2	Experiment 3	Experiment 4
Washing liquid				
Pb	–	30.4	1.66	2.07
Cu	–	0.21	0.24	0.19
Cd	–	0.09	0.14	0.23
Zn	–	9.41	0.11	0.59
Diluate (APC-suspension) start				
Pb	30.1	4.16	0.20	0.42
Cu	0.27	0.04	0.03	0.05
Cd	0.09	0.02	0.03	0.19
Zn	21.3	3.68	0.01	3.18
Diluate (APC-suspension) end				
Pb	8.75	0.14	0.28	1.63
Cu	0.17	0.05	0.08	0.46
Cd	0.01	0.14	0.36	0.05
Zn	0.02	6.41	24.3	0.79
Concentrate end				
Pb	6.36	3.36	3.26	20.2
Cu	9.12	6.64	4.59	15.7
Cd	0.21	0.31	0.83	1.72
Zn	18.5	25.4	61.8	236

Concentration of the elements in the concentrate was successfully obtained, with significantly higher values in experiment 4 (2 to 4 times higher than second best), indicating that acid washing APC residue at pH10 and adjusting pH during ED results in higher metal removal from the APC residue. However for Pb in exp. 1 and 2 and zinc in exp. 1 the concentrations obtained were lower than those of the initial diluate solutions.

3.2 Concentration of heavy metals

Figure 5 shows the pH and build-up of heavy metals in the concentration chambers as a result of the transport through the membranes from the feed chambers. pH dropped from alkaline to nearly neutral with time in exp. 1–3, whereas in exp. 4 pH dropped from 7 to 2 within the first 5 h of the experiment due to the acid titration. Compared to the remainder elements, zinc obtained a higher concentration in the concentrate solution in all experiments, due to the higher total concentration (Table 1) and leachability (Fig. 6) of this element. In the acid -treated APC the concentration of zinc reached 236 mg/L after 27 h—demonstrating that it was very mobile at the low pH. Figure 5 indicates that further ED-treatment in all experiments would increase the transfer of zinc into the concentrate solution. Copper and lead are released to the concentrate solution to a similar extent in all experiments, reaching a level of 3–10 mg/L in experiments 1–3 and 15–20 mg/L in experiment 4 after 27 h. This occurs even though both total concentration (Table 1) and leachability (Fig. 6) of lead is an order of magnitude higher than that of copper. In exp. 2–4 this is explained by the washing procedure, which for lead removes the mobile fraction in exp. 2 and immobilises the mobile fraction in exp. 3 and 4. In exp. 1, however, the lead concentration in the diluate solution was still high after the experiment finalized, and the relatively bad removal of the soluble lead fractions is likely to reflect the presence of lead as a neutral species not transferable by the

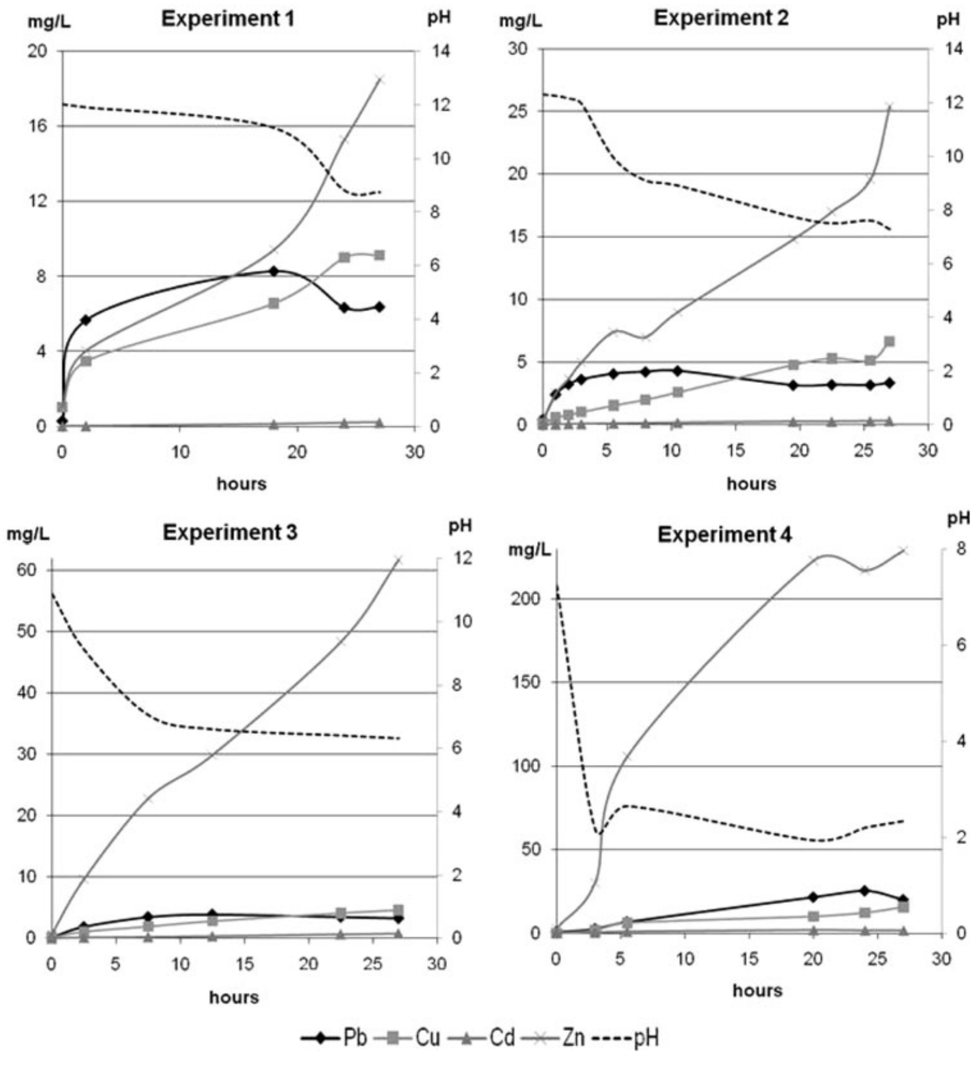


Fig. 5 Evolution with time (hours) of heavy metals and pH in the concentrate stream in ED treatment: experiment 1—raw APC residue; experiment 2—water pre-washed APC; experiment 3—acid pre-washed APC; experiment 4—acid treated APC

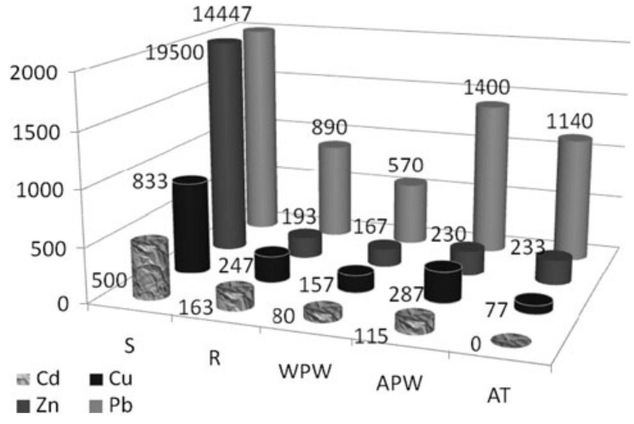


Fig. 6 Leaching of Cd, Cu, Zn and Pb before (S) and after treatment (µg/L). (Note that initial concentrations of Pb and Zn are out of scale). R—raw ED treated; WPW—water pre-washed ED-treated; APW—acid pre-washed (pH 10) ED treated; and AT—acid (pH 2) and ED treated residue

electrical current. These mechanisms are also illustrated from the graphs in Fig. 5, which indicate that further treatment would only transfer additional lead very slowly into the concentrate in exp. 1–3 and not at all in exp. 4. Like for zinc the graphs in Fig. 5 indicate that further ED treatment would transfer more cupper into the concentrate solution, although the trend is less pronounced and probably only valid in exp. 2–4. Cadmium obtains concentrations between 0.2 mg/L in experiment 1 and 1.72 mg/L in experiment 4, reflecting its lower total concentration (Table 1) but in contrast to its relatively high leachability (Fig. 6) which is almost as high as that of copper. Cadmium also does not seem to be as soluble/mobile as expected from other ash types such as straw combustion ashes [19]. On the other hand, cadmium is not released easily from wood ashes [20]. Like for lead cadmium removal seems to have seized in exp. 4 and be very slow in the remaining experiments.

3.3 Leaching of toxic metals

The mobility of metals present in APC-residue at the beginning and at the end of ED treatment was evaluated by a leaching test. The results show that the leachable metals in the APC-residue was substantially reduced after ED treatment (Fig. 6). Leaching of Zn was reduced by two size-orders of magnitude, while the leaching of Pb was reduced by more than one size order of magnitude. The leachability of the metals seems to be dependent of the pre-washing prior to the ED treatment. Considering lead and zinc, the water pre-washed APC (WPW) seems to form the most chemical stable residue after the ED treatment. On the other hand, no more cadmium could be leached from the acid pre-treated sample (AT), and residual copper seemed also to be more stable after this pre- and ED treatment. According to Danish legislation [21], the limiting value for possible reuse of waste materials in the construction industry, and thus our target value, is leachate concentrations of 40 µg/L of Cd, 100 µg/L of Pb, 1,500 µg/L of Zn and 2,000 µg/L of Cu obtained from the leaching test used in the present work. For Cd the target value is only met in exp. 4, while for Pb it is not met at all. For Zn the target value is met after all treatments. Likewise for cupper which meet the target value even in the untreated APC. Thus emphasis should be put on optimisation of reduction of the Pb leaching, and thus the water pre-washing seems to be the most promising treatment method.

3.4 Conductivity and pH

The initial conductivity of the APC suspension in experiment 1 (raw APC) was substantially higher (52 mS/sm) than in other experiments (9–13 mS/cm) because the washing operation removed readily soluble salts. During the electrodialytic treatment, conductivity and pH of the APC suspension changed. As ions were transferred from the diluate (the APC suspension) into the concentrate, the conductivity of the APC suspension decreased (Fig. 7), except for experiment 4, where the acid-driven dissolution of APC residue resulted in an increase of conductivity during the first 5 h. After ending the acid titration this dissolution seized and the conductivity of the APC suspension decreased during the rest of experiment 4. By the end of ED treatment conductivities ranged from 5.0 mS/cm (experiment 2) to 1.7 mS/cm (in experiment 4), reflecting that most charged species in solution had been removed.

Also pH decreased during treatment in all experiments (Fig. 8). The initial pH of the raw and the water

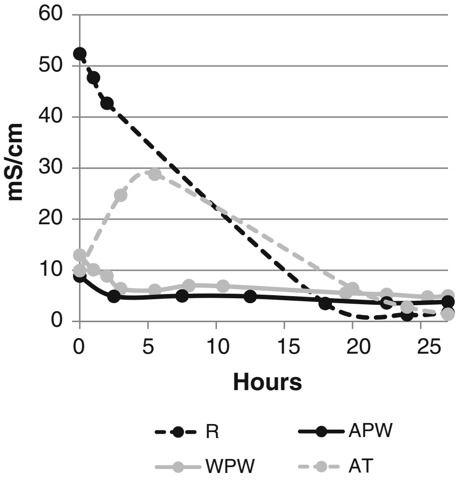


Fig. 7 Conductivity development in the APC suspension during ED treatment. R = Raw APC, WPW = Water Pre-Washed APC, APW = Acid Pre-Washed APC, AT = Acid Treated APC

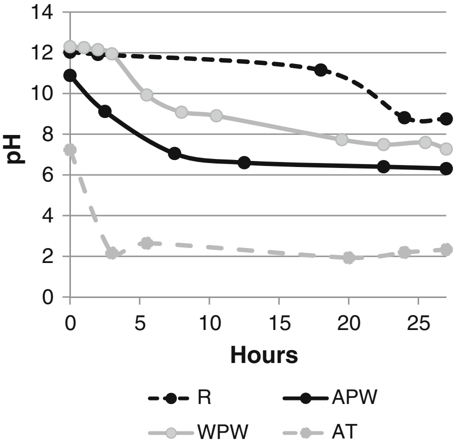


Fig. 8 Development of pH in the APC suspension during ED treatment. R = Raw APC, WPW = Water Pre-Washed APC, APW = Acid Pre-Washed APC, AT = Acid Treated APC

pre-washed APC was 12.0 and 12.3, respectively, while the initial pH of the acid pre-washed APC was 10.9 and the initial pH of the acid treated APC was 7.2. In the first three experiments pH dropped as hydroxide ions were removed from the suspension, and the final pH in experiments 1, 2 and 3, respectively was 8.8, 7.3 and 6.3, while the acid treated APC achieved a pH as low as 2.3 due to the acid titration.

3.5 Dissolution of APC

During the wet treatment of the APC, soluble compounds in the APC were dissolved and removed from the APC suspension. Electrodialytic treatment of the raw APC decreased mass by 62.5% from 200 g to 75 g. By washing, the mass was reduced by 33.5% to 133 g while washing and subsequent electrodialysis reduced the mass of APC by 57–86 g (Fig. 9). The higher dissolution of the raw APC during electrodialysis compared to the water pre-washed APC is in accordance with the lower conductivity obtained by the end of electrodialysis of the raw APC (Fig. 7), and may reflect that the water pre-washing induces an immobilization of some of the APC constituents.

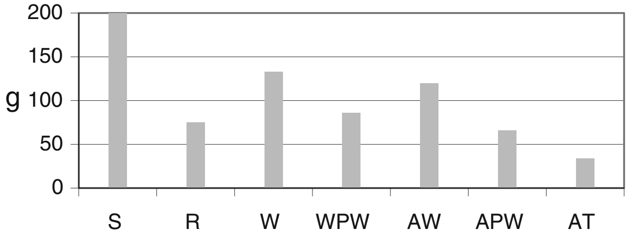


Fig. 9 Mass of solid material at the start and the end of treatments of APC. S = Start amount, R = Raw ED treated APC, W = Water Washed APC, WPW = Water Pre-Washed ED treated APC, AW = Acid Washed APC, APW = Acid Pre-Washed ED treated APC, AT = Acid Treated ED treated APC

Acid washing resulted in slightly higer dissolution than water washing with dissolution of 40% and 120 g of dry mass left. Even higher dissolution (57%) was found after acid wahing followed by electrodialysis (66 g left), while the highest dissolution tof the APC was found after both acid and electrodialysis treatment (83%, 34 g left). In the optimal treatment as little APC as possible should be dissolved while simultaneously obtaining sufficient reduction in the leachability of all harmfull substances. Paying attention to only dissolution of the APC, water pre-washing is the optimal treatment.

4 Conclusions

ED-treatment of APC residue in suspension in a stack resembling conventional ED treatment and suitable for continuous treatment, developed for the high solids content solution successfully reduced leaching of the target heavy metals zinc, lead, cadmium and copper. As the only element lead did not meet the target value of Danish legislation after any of the experiments, however, leaching was still reduced by more than one size order of magnitude. Treatment of raw APC was hampered by the likely presence of uncharged species, while treatment of water prewashed APC was a good alternative minimizing the dissolution of the bulk APC and providing the highest reduction in leachability of zinc and lead. Acid pre-washing also provides a good solution with the possibility of selectively removing soluble salts while preventing the dissolution of large amounts of lead during the pre-treatment. Acid treatment during ED provides the best reduction in Cd leaching while also the highest dissolution of the bulk APC and further seems non-optimal concerning the reduction of the leaching of the remainder elements.

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