Electrodesalination of sandstones with irregular shapes and uneven distribution of salts

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Abstract

Electrochemical desalination of stone is based on application of an electric potential gradient. In case the electrodes are placed in a poultice, which buffer the acid from oxidation of water at the anode, the methods has shown very effective. Most investigations have been carried out in laboratory scale on stones with an even salt distribution. This investigation was designed to answer different questions arising if the method is to be applied to sculptures and monuments with an uneven salt distribution and irregular shapes. Laboratory experiments were made with stones samples from a historic warehouse. The sampling was in connection to renovation. Before the renovation an on-site experiment was made as a part of the investigation.

The initial concentration in the stone segments for the different laboratory experiments varied reflecting the inhomogeneous distribution in the wall. Whether the electrodes were placed on the same side of the stone or on two opposite sides did not influence the removal rate of Cl^- and NO_3. A stone part, which was not placed directly between the electrodes, was desalinated, but with a slower rate than the other parts. Two stone parts were isolated from each other during the desalination, and the current mainly passed through the part with high salt concentration. The on-site experiment was successful. Chloride was removed to a very low level in all depth of the investigation (10 cm) and from cathode end to anode end, though the desalination had not finished as the concentration was still little too high in the anode end. All together 131 g Cl^- was removed during 5½ month. All results showed that the method had potential for desalination of monuments with irregular surfaces and salt distribution.

Keywords: electrodesalination, sandstone, electrode placement

1   Introduction

Electrochemical desalination is a method for desalination of salt infected monuments and buildings. An electric DC field is applied from electrodes, which are placed on the surface. In the applied electric field the main transport mechanism for the salt ions is electromigration. Positive ions in the pore solution will move towards the negative electrode and the negative ions will move towards the positive electrode. During the process the concentration of ions from the salts will thus decrease in the salt infected material as the ions concentrate around the electrodes.

Electrochemical desalination has shown large potential treatment of different matrices both through laboratory scale experiments: baked clay bricks [1-5], sandstones [6-8], granite [9] and glazed tiles [10-11] and pilot scale tests [12-13]. Also a numerical model over the process has been developed [14].

At the electrodes there are pH changes due to electrolysis reactions:

At the anode: $H_2O \rightarrow 2H^+ + ½ O_2 (g) + 2e^-$ (1)

At the cathode: $2 H_2O + 2e^- \rightarrow 2OH^- + H_2 (g)$ (2)
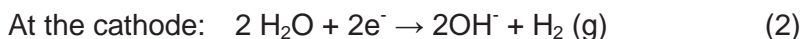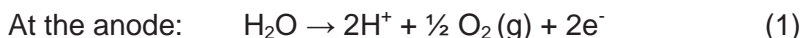
As seen from (1) and (2) pH decreases at the anode and increases at the cathode. It is necessary to neutralize acidification at the anode in order to prevent decay of the material to be desalinated. Hendrix et al. 2011 [7] underlined how important it is to avoid the acidification, as in experiments without pH neutralization the stones were severely damaged close to the anode. Also in order to obtain sufficient desalination pH neutralization can be crucial. Kamran et al. 2012 [4] showed that without neutralization at the electrodes, the desalination process in bricks stopped due to formation of a sharp transition zone between the acidic and alkaline region. This zone resulted in a large electrical potential gradient due to a local depletion of ions here. So for different reasons, the metallic electrodes should not be placed directly on the material to be desalinated.

Carbonate rich clay poultice can be placed between the electrodes and the stone for neutralization of pH changes. Calcareous clay for brick production or a mixture of kaolinite and calcite were tested both efficiently neutralized the acid from electrolysis at the anode [8]. The acid was neutralized by the $CaCO_3$ in the poultices. When using the $CaCO_3$ rich poultices, the transition zone between low and high pH is moved from the stone to the anode poultice [14]. The increased concentration of OH^- ions in the material must be counterbalanced by cations (electro-neutrality) and in [14] it is suggested from numerical-chemical simulations that these are mainly Ca^{2+} from dissolution of calcium-carbonate in the anode poultice. There may thus be precipitation of $Ca(OH)_2$ in the material. Over time $Ca(OH)_2$ reacts with CO_2 from air and form $CaCO_3$, but neither $Ca(OH)_2$

nor $CaCO_3$ are considered damaging, because aqueous solutions of calcium hydroxide (limewater) have been used for many centuries to protect and consolidate limestone. In addition to neutralizing the acid, the poultice also gives good electrical contact between metallic electrode and stone, and it serves as sink for removed ions. When the clay poultices are removed after the desalination, the ions of the salts are removed with it.

The present work is conducted with salt infected sandstones removed from a historic warehouse during renovation. The previously published laboratory work on electrodesalination has been conducted with samples contaminated with salts in the laboratory. The present investigation is focused on the influence from electrode placement on the desalination process in relation to both the stone shape and salt distribution.

## 2 Experimental section

2.1 Stones of the investigation

The experiments were conducted with Obernkirchner sandstones (Bremersandstones) from a historic warehouse, Eigtveds Pakhus, in the harbor area in Copenhagen, Denmark. The sandstones were from the outer surface of the building, and they were removed during a renovation action due to salt decay (scaling and delamination of the surface). Figure 1 shows how the sandstones were cut from the wall in long pieces. The laboratory experiments are made with such pieces after further segmentation. A small on-site experiment was conducted at another section of the masonry before the renovation and removal of sandstone.

The stone segments were brought to the laboratory where they were placed in a closed bucket over (but not in contact with) water in order to keep the stones moist. Figure 2 shows one of the stone pieces after it was segmented (into a reference segment and segments for experiments 1 and 2). The stone segment for experiment 2 is a corner, so two sides from this segment has been exposed to free air.


Figure 1: Removal of experimental stone pieces from wall
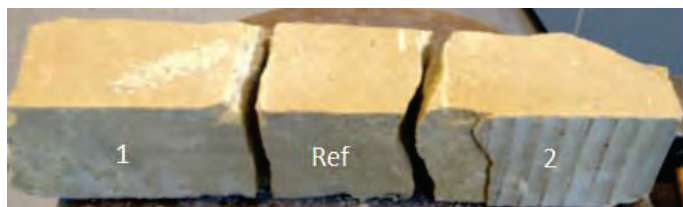
Figure 2: Segmentation of a stone piece into a reference segment (Ref) and segment 1 and 2 for desalination experiments.

2.2 Electrochemical desalination experiments

Four electrochemical electrodesalination experiments were conducted in the laboratory. The stones were cut in different shapes, and the electrodes placed in different positions on the stones. Figure 3 is an overview of the different setups. The salt distribution was investigated in a reference stone. In addition an on-site experiment was made on the site (before the stones were replaced during the renovation).
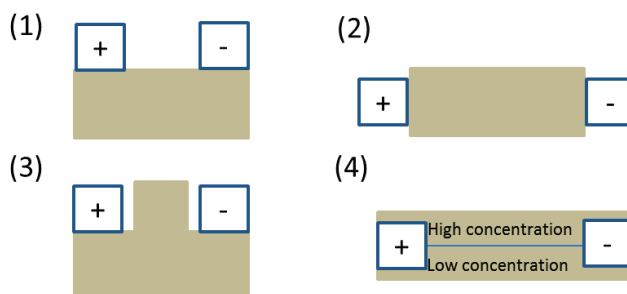
Figure 3: Summary of the four different laboratory setups for electrochemical desalination in this investigation

The laboratory experiments were designed to investigate: (a) the influence on the desalination from the placement of electrodes on opposite side or same side of the stone (comparison of lab experiments 1 and 2); (b) desalination of a part of the stone which is not placed directly in the electric field (lab experiment 3), (c) simultaneous desalination of volumes with low and high salt concentration (lab experiment 4), and (d) removal on-site compared to in the lab (on-site experiment)

Plastic frames with clay poultice (one for each electrode) were placed on the surface of the sandstone segments in positions shown in figure 1. The clay poultice was a mixture of kaolinite and $CaCO_3$ [15]. Inert electrode meshes were placed on the top of the poultice. Finally the electrodes were connected to a power supply (Hewlett Packard E3612A), which supplied a constant current. The whole setup with stone segment and electrode units were wrapped in plastic film to hinder evaporation.

During the experiments, the poultices were replaced regularly and the contents of Cl, NO_3 and SO_4 were measured in the removed poultice to follow the progress in the desalination. At the end of the experiments the stone segments were sliced using hammer and chisel and the concentrations of the same anions were measured.

2.2.1 Reference segment

Concentration profiles of Cl^-, NO_3^- and SO_4^{2-} in depth for a reference segment (the middle segment figure 2) were measured. The reference segment was analysed directly and had not been subjected to placement of electrode units or an applied electric field.The reference segment was sliced into 6 slices starting from the original outer surface of the wall. The slices were approximately 1-1.5 cm thick.

2.2.2 Experiments 1 and 2. Poultice at same or opposite side of stone

The stone segments for experiment 1 and 2 were taken from the same long stone piece (figure 2) on both sides of the reference segment.

Experiment 1: electrodes were placed on the same side of the stone segment. The electrodes were placed on the original outer surface (figure 4(a). At the end of the desalination experiment the stone segment was segmented into 5 slices as shown in figure 4(b).
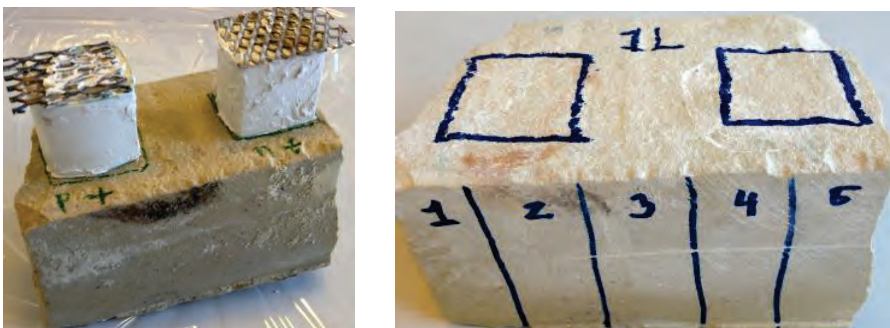
Figure 4: Experiment 1. (a) Placement of the electrode units and (b) Segmentation of the stone segment into slices at the end of the experiment

Experiment 2: The electrode units were placed in each end of the stone segment opposite to each other. The cathode unit was placed on an original outer surface (the stone segment had an original corner from the building). At the end of the experiment, the stone segment was sliced into 5 slices of approximately same sizes running from anode to cathode.

Experiments 1 and 2 were run simultaneously. During the first 24 hours 5 mA was applied, but as the voltage increased the current was decreased to 2 mA in the remaining 77 days of the experiments.

2.2.3 Experiment 3. Part of stone outside the electric field

The shape of the stone for experiment 3 is seen in figure 5. The electrodes were placed horizontally on parts 4.1 and 4.3. There was no physical contact between electrodes and part 4.4. The original outer surface was represented in all four parts to have the most similar salt concentration and distribution. Anion content was measured in the cutoffs over 4.1 and 4.3. At the end the segment was parted in four parts (figure 5).

Figure 5:   Stone segment for experiment 3

2.2.4 Experiment 4. High and low initial concentration

The stone segment was cut in two pieces of equal size. One piece was submerged one week in distilled water and the other in 79.6 g/l NaCl. This was in order to have one piece with high concentration and one with low. An initial sample was taken from both parts. For the desalination experiment the two pieces were placed together again, but with plastic between, to hinder transport of ions between the two parts.

Figure 6:   Stone segments for experiment 4

The two pieces were kept together by tape during the desalination experiment. The electrodes were placed on the same side as indicated by the markings for the electrode units at figure 6. The electrode units covered approximately the same area from the two stone pieces. At the end of the experiment five slices were made from each of the two stones. This experiment is focused on Cl removal, as the stone with the high concentration was submerged in NaCl.

2.2.5   On-site experiment

A set of electrodes were placed on the surface of the building (see figure 7a) before renovation. The electrode units were designed as the units described in [12-13]. The electrode units were placed in the height of 45 cm (to lower edge). The distance between the electrodes was 20 cm.

The electrode casings (50 cm long) were made from plastic and consisted of two parts; a box and a movable bottom (see figure 7b). The purpose of the movable bottom was to ensure mechanical pressure between poultice and masonry. This is crucial to passage of current. As the poultice dries slightly during desalination it loose volume and the bottom is moved closer to the masonry due to pressure from springs. It is further possible to tighten the springs by tightening the three buttons at the back of the casing. An inert metallic electrode mesh was placed on top of the bottom and the casing was filled with poultice. The experiment lasted 5½ month (July-Dec. 2010). Poultices were changed four times. A constant current of 50 mA was applied.
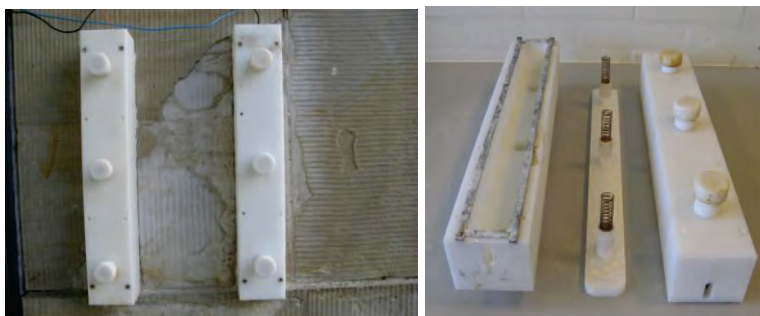
Figure 7: (a) the electrode units mounted on the wall, (b) The design of the electrode casings. To the left the hole in the box is shown, in which the movable bottom in the middle is placed with the springs down.

Samples from the masonry for determination of salt concentrations were taken during mounting of electrodes. The drilling powder from the holes made to fasten each electrode was collected (in the heights 45 and 95 cm). The holes were 6 mm in diameter and about 5 cm deep. By the end of the test drilling samples from masonry were taken to the depth 10 cm. The samples were divided in intervals: 0-1 cm; 1-2 cm; 2-5 cm and 5-10 cm. Samples were taken in the height of 70 cm (middle of electrode placement). The sampling positions were where the electrodes had been placed and one in between. Sampling from the poultice was done each time it was changed. Three sub-samples of the same size were taken from each unit (from top, middle and bottom) and these sub- samples were mixed before analysis to have a representative sample.

2.2.6 Overview of experimental conditions

An overview of the experiments is given in table 1.

Table 1: Conditions for the conducted experiments.

| Experiment	Stone weight (g)	Current (mA)	Duration (d)
Ref. No current	0.59	0	-
1 Electrodes same side	1.05	2	77
2 Electrodes on opposite sides	0.83	2	77
3 Stone part outside electric field	0.99	2	42
4 High-low part concentrations	0.61/0.57	2	29
On-site	-	50	164

2.3   Analytical

Extractions were made with 10 g dry, powdered sandstone (powdered by hand in a mortar), drilling powder or poultice and 25 mL distilled water. The suspensions were agitated 24 hour. The suspension filtered and Cl^-, NO_3^- and SO_4^{2-} concentrations of were measured by ion chromatography.

3 Results and discussion

3.1 Concentration profiles in the reference segment

Concentration profiles of Cl^-, NO_3^- and SO_4^{2-} in the reference segment are in figure 8. To evaluate the concentrations the ONORM [16] is used. The norm is debated, but the only one of its kind. The Cl^- conc. is rather constant throughout the depth of the stone at a level of about 0.4 wt% exceeding the upper level from [16] (0.1 wt%). The SO_4^{2-} concentration is also about constant and at 0.04 wt%, which is less than the lower limit from [16] (0.1 wt%).The pattern for NO_3^- is very different from the two others, as the concentration decreases almost linearly from the outer surface (0.3 wt%) to 0.1 wt% in the depth of the sample (7-8 cm).
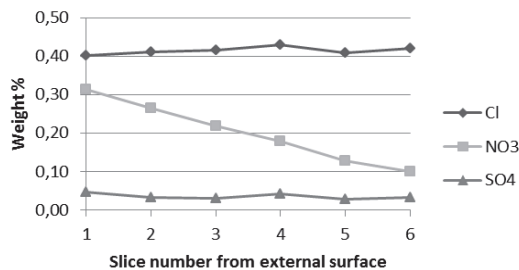
Figure 8: Concentration profiles of Cl^- , NO_3^- and SO_4^{2-} in the reference stone running from the original outer surface over the depth of 7-8 cm

3.2 Desalination with electrodes on the same side of the stone or on opposite sides

Experiments 1 and 2 are discussed together. The removal of the anions into the poultices as a function of the applied charge (Q) is shown in figure 9 ($Q = I \cdot t$, where I is current [A] and t is time [s]). Figure 10 shows the concentration profiles of the anions in the stone at the end of the experiments. Finally the average initial concentrations in the reference segment and calculated initial concentrations for experiments 1 and 2 are in table 2. The average concentrations in the reference segment are calculated on basis of dry weight and concentration in the six different slices. The average concentrations from experiments 1 and 2 are calculated on basis of final concentrations in the stone pieces and mass removed into the poultices (and dry weight of the corresponding matrices). The calculated average Cl^- concentration is underestimated as an unknown amount of Cl^- is lost from the anode process $2Cl^- \rightarrow Cl_2$ (g) + 2e$^-$ (this reaction did take place as some of the removed poultices smelled of chlorine). It is seen from the table that even within one stone piece there

are significant concentration variations and thus this parameter is differing between all experiments of this investigation.

The most important transport mechanism involved in removal of ions from the salts into the poultice is electromigration. Diffusion may also be significant, due to the concentration gradient between the high concentration in the pore water of the sandstone and the extremely low concentration in the pore water of the poultice. This means that the anions removed into the anode poultice is a sum of the ions transported by electromigration and diffusion, whereas the concentration in the cathode poultice is due to diffusion alone. The difference is clearly seen from the differences in the level of mass of removed anions into the two poultices (figure 9 a and b).

A higher mass of the anions is transported into both anode and cathode poultice in experiment 1 than 2. The graphs for each of the anions in the anode poultice from the two experiments are almost parallel (figure 9a). The major difference between the levels of the two graphs is related to the different level in transport from diffusion. This can be seen from the removal into the cathode poultice (figure 9b), where the level is significantly higher in experiment 1 than 2. The reason is the placement of electrodes in relation to the original outer stone surface. Both electrodes were placed on the original outer surface in experiment 1, whereas the electrodes were placed on original internal parts in experiment 2. For NO_3^- this makes a major difference, as it was found that the NO_3^- concentration was highest at the surface (figure 8). The higher diffusion of NO_3^- than Cl^- (experiment 1) is though not at first sight explained by the difference in concentration gradient, as the concentration of Cl^- was found highest in the first slice from the original surface (figure 8), however as figure 8 shows average concentrations for the slices, it is not known if a fraction of NO_3^- in the first slice was actually soluble salt crystals at the surface and if so the surface concentration of NO_3^- could have been higher than Cl^-.
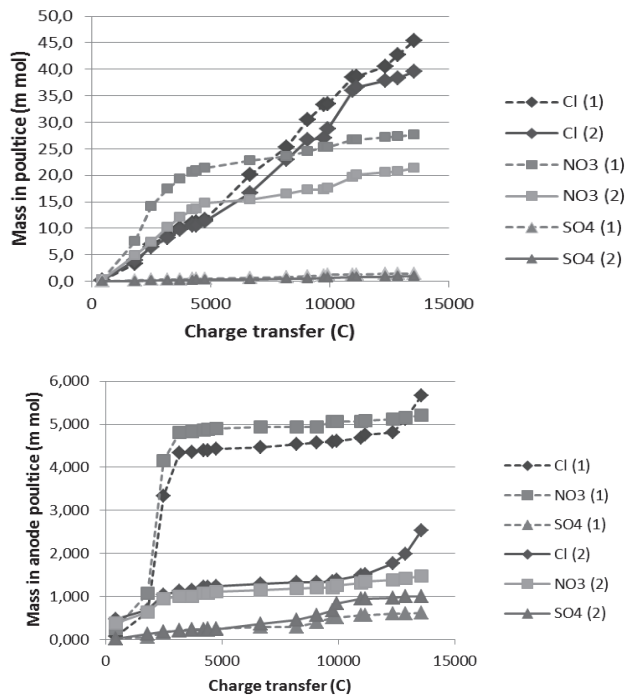
Figure 9: Accumulated mass of Cl^-, NO_3^- and SO_4^{2-} in the poultices as a function of the charge transfer in experiments 1 and 2 (top) anode poultice and (bottom) cathode poultice
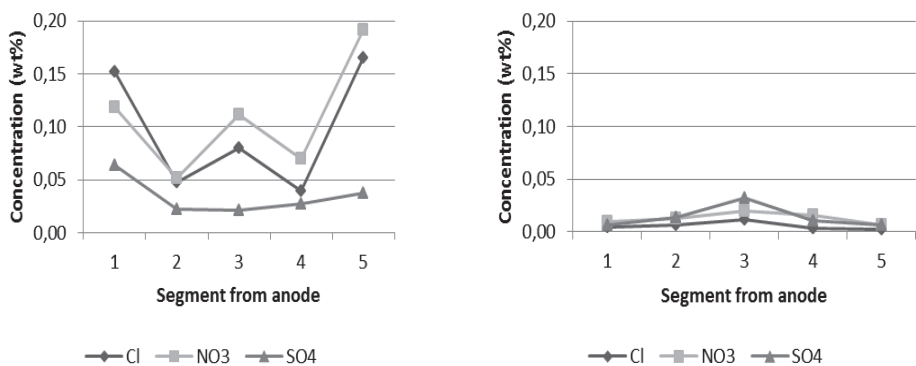
Figure 10: Concentrations of Cl^-, NO_3^- and SO_4^{2-} in the stone segments at the end of experiment 1 (left) and experiment 2 (right)

Table 2: Calculated average initial concentrations in the stone segments of the reference and desalination experiments 1 and 2. (*Chloride concentration is underestimated as an unknown amount is lost in the anode reaction)

Experiment	Cl^- (mg/kg)	NO_3^- (mg/kg)	SO_4^{2-} (mg/kg)
Reference	4110	2190	350
Experiment 1	2540*	2900	470
Experiment 2	1910*	1890	370

Final Cl^-, NO_3^- and SO_4^{2-} concentrations in the stone from experiment 2 are very low, and much lower than in experiment 1. So even if a higher mass was removed in experiment 1, the lowest concentrations were obtained in experiment 2. This is because both stone mass and initial concentrations were highest in experiment 1. Unfortunately this means that direct comparison of the electrode placements is not possible. The removal rates from electromigration are though very similar (figure 9a), which implies that there is no major difference. The Cl^- transport numbers for the two experiments are 0.32 and 0.28, respectively.

3.3 Experiment 3. Part of stone outside the electric field

Figure 11 shows the overall results from experiment 3. The Cl^-, NO_3^- and SO_4^{2-} concentrations are shown for each of the stone parts and the initial concentrations (reflected by the concentrations in the parts, which were cut off before the desalination), are shown as well. The concentrations in each piece are considered relatively similar before desalination as the stone was cut so each part contained the original outer surface. The Cl^- and NO_3^- concentrations are also in the same level in the two initial samples, but the SO_4^{2-} concentration was double as high in one sample as in the other. Compared to the reference stone (figure 8) the concentration of Cl^- was only half in this stone, the NO_3^- concentration in the same level and the SO_4^{2-} was less in the left sample and in the same level in the right.
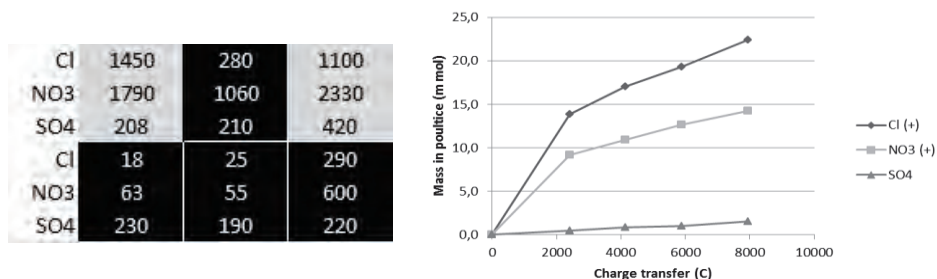

Figure 11: left: Concentrations (mg/kg) Cl^-, NO_3^- and SO_4^{2-} in the different parts of the stone in experiment 3 (black). Light grey parts show initial concentrations found from cutoffs; right: Removal to anode poultice as function of charge transfer.

After desalination the Cl⁻ and NO_3^- concentrations are lowest in the parts placed directly between the electrodes (three lower parts). Concentrations in the first two parts here are very low and also concentrations in the part closest to the cathode are decreased significantly. The removal into the poultice shows good and stable removal (figure 11). The piece, which had not been directly in the electric field (the central upper piece), has decreased Cl⁻ and NO_3^- concentrations compared to the initial. Especially the Cl⁻ concentration was lowered. The result indicates that also salt infected volumes, which are not placed directly between the electrodes, can be desalinated, though with a slower rate. This is important if the method is applied to sculptures with different attributes. Electrodes need not to be placed on top of these often very fragile parts.

3.4 Experiment 4. High and low concentration

Figure 12 shows the Cl⁻ concentration in the two initial samples and the profiles in the samples after electrodesalination. The concentration was lowered in both samples, compared to the initial concentrations. In respect to the removed moles from the two sides it can be concluded, that the major part of the current passed through the part with high initial concentration. Initially pH was 7.5 and 8.0. At the end of the experiment pH was 7.6-8.5 in the sample with low concentration and 8.3–9.1 in the one with high concentration. This supports that the major part of the current passed through the stone with high concentration. The result shows that the electric field is strongest where the conductivity is highest i.e. parts of e.g. a wall with highest salt contamination.
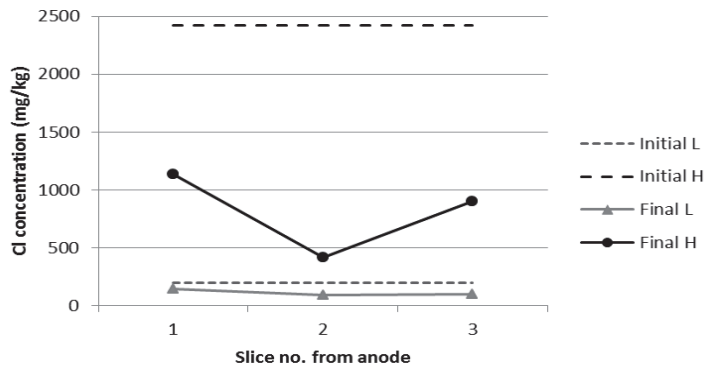

Figure 12: Initial and final concentrations in the two parts (L part with low initial Cl⁻ concentration and H part with high initial Cl⁻ concentration)

3.5   On-side experiment

The concentrations measured in the drilling powder when mounting the electrodes were 0.40 – 1.38 wt% Cl^-; 0.03 – 0.33 wt% NO_3^-; 0.01 – 0.03 wt% SO_4^{2-}. The Cl^- concentration was much higher than the highest level from the ÖNORM (0.1 wt%) [16], whereas the SO_4^{2-} concentration was much lower and only one of the initial samples did exceed the limit for NO_3^-. The initial Cl^- concentration was much higher than in the reference segment for the lab experiments (table 2).

Figure 13 shows the Cl^- concentration profiles in the wall from anode to cathode in four different depths. It is clearly seen that in every depth the concentration is much lower in the cathode side than in the anode side, which corresponds to the direction of electromigration. The profile shows, that the desalination was not finished as the concentration in the anode side still exceeded 0.1 wt%, but compared to the initial concentrations which was >0.4 wt% the concentration had decreased significantly here as well. The overall transport number for Cl^- in the experiment was 0.38 which is even better than in the lab experiments 1 and 2 (where it was about 0.3), and the removal could have progressed in case of prolonged duration, as the transport number in the last period was 0.25.

The on-site experiment gave very good results. The desalination has been efficient in the outer 10 cm of the wall (at least) and all together 131 g Cl, was removed into the anode poultice at a relatively high transport number.

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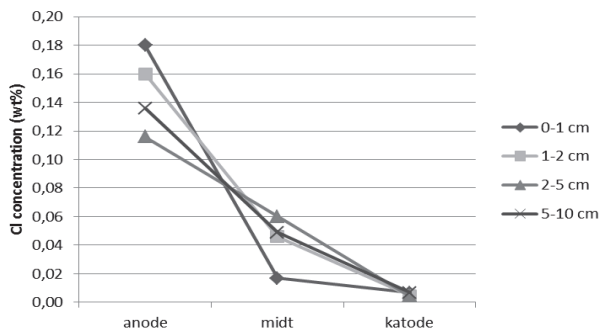
Figure 13:   On-site experiment. Cl^- concentration profiles from anode to cathode in four different depths

4   Conclusions

This investigation was focused on some major questions in relation to full scale desalination of monuments and buildings. The experiments were made with sandstones from a historic warehouse. The Cl⁻ removal rate was very similar whether the electrodes were placed on the same side or opposite sides of the stone to be desalinated. Diffusion into the poultice is important in the first day of electrodesalination only. A part of a stone which is not placed directly between the electrodes was desalinated, but with a slower rate than the part between the electrodes. The electric field is strongest in the part of the stone with highest concentration of ions. The experiments in the present investigation were made with stone samples which were contaminated with salts from environmental exposure and thus the salt concentrations varied significantly between samples and in each sample. The experiments could be repeated with stones prepared so the salts were initially homogeneously distributed in order to verify these findings. An on-site experiment with electrodes at the same side of the wall gave a very good result, and even better than the laboratory experiment with electrodes at same side. The transport number was 0.38 and 0.32 in pilot and laboratory scale, respectively. The desalination had progressed and at least into the depth of 10 cm, from where the deepest samples were taken.

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