

Experimental study on the effectiveness of Electrochemical Chloride Extraction (ECE) for reinforced concrete.

Justine De Brabandere^{1*}, Manon Wybo², Bjorn Van Belleghem¹, Emile Godefroidt¹ and Tim Soetens¹

¹SANACON bv, Ghent University spin-off, Nijverheidsweg 1/A, 9820 Merelbeke, Belgium

²Magnel-Vandepitte Laboratory for Structural Engineering and Building Materials, Ghent University, Technologiepark-Zwijnaarde 60, 9052 Ghent, Belgium

Abstract. Chloride-induced corrosion is a widespread damage phenomenon in reinforced concrete structures that leads to a substantial reduction in their service life. Fortunately, different repair techniques are available, including electrochemical techniques, such as cathodic protection (CP) and electrochemical chloride extraction (ECE). While CP has proven to be an effective method to stop reinforcement corrosion, ECE, where an electrical field is used to force chloride ions out of concrete, is less seen in practice. In this research, the influence of different parameters on the effectiveness of ECE is investigated. The results of this research show that for concrete samples containing around 0.75 m% Cl⁻-cement mixed-in chlorides the removal rate ranged from 50-60% in the concrete cover to 20-30% around the reinforcement and behind it, irrespective of different influencing factors. However, when a higher initial chloride concentration, namely 2.68 m% Cl⁻-cement was present within the concrete, the removal rate was 1.4 times higher. Besides, when an ingressed chloride profile was created with approximately 0.75 m% Cl⁻-cement at the level of reinforcement, the removal rate was between 1.7 and 2.8 times higher. Lastly, it must be noted that an examination of the corrosion potential indicated that all samples had a low probability of reinforcement corrosion after ECE-treatment, showing that ECE successfully removed enough chlorides to reduce the risk for active corrosion.

1 Introduction

Chloride-induced corrosion is one of the main causes reducing the intended service life of reinforced concrete structures [1]. Due to the propagation of corrosion, expansive corrosion products are formed, leading to cracking and even spalling of the concrete. Already cracked concrete serves as a catalyst of the corrosion process since the harmful substances can now penetrate more easily through the cracks. Besides these damage phenomena that give a visual warning, a reduction of the cross-section of the reinforcement bars can also occur during the corrosion process, leading to a lower load-bearing capacity of the reinforced concrete [2]. The chlorides, which are responsible for the initiation of the corrosion process, can be present in the concrete due to mixing-in during the production process or due to chloride ingress from the environment over time [3].

Fortunately, there are different repair interventions for chloride-induced corrosion, namely electrochemical techniques, such as cathodic protection (CP) and electrochemical chloride extraction (ECE). While, CP has already proven to be a long-term effective method in halting chloride-induced corrosion, ECE is still in an experimental phase and has not been widely implemented in practice.

ECE is a repair technique that forces chloride ions out of reinforced concrete by applying an electrical field.

The negative side of a power source is connected with the reinforcement, and the positive side is connected with an anode that is placed on the concrete surface. Due to the created electrical field, the negatively charged chloride ions migrate towards a positively charged applied anode and accumulate in the electrolyte around the anode. Additionally, hydroxyl ions are generated at the reinforcement, which contributes to the restoration of the passivity [4, 5]. The typical duration of treatment of this technique ranges from four to eight weeks, and the applied current density is around 1 to 5 A/m² steel [6, 7, 8].

Although ECE has been around since the 1990s, it has not been widely applied in practice, resulting in a lack of detailed information in the literature regarding its effectiveness [8]. Therefore, the aim of this research is to investigate the influence of different factors on the effectiveness of this technique and to identify when ECE might be an effective repair option.

2 Materials and methods

2.1 Concrete samples

Eight reinforced concrete samples of 300 × 300 × 70 mm were made for this research. The reinforcement net consisted of BE 500 S steel bars with a diameter of 10 mm and a length of 250 mm.

* Corresponding author: justine.debrabandere@sanacon.be

These eight samples each have one different parameter to clearly observe the influence of this factor on the effectiveness of the ECE treatment. The reference sample S1, which is shown in Figure 1, has the following parameters:

- Type of cement: CEM I 52.5 N;
- Concrete cover: 25 mm;
- Steel reinforcement density: 0.9 m² steel/m² concrete;
- Chloride concentration: 1 m% Cl⁻~cement ;
- Source of chlorides: mixed-in (by adding NaCl in the mixing water);
- Applied voltage: 48 V;
- ECE treatment schedule: intermittent (alternating seven days on - seven days off).

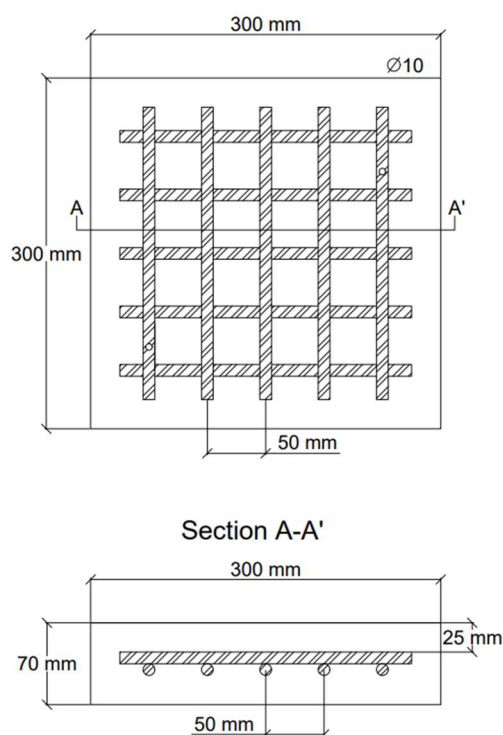


Fig. 1. Sample 1 (S1): the reference sample.

Samples 2 to 6 show a variation in a parameter related to the intrinsic properties of the concrete samples, while in samples 7 and 8 the variable parameter is related to the ECE setup.

Table 1 gives an overview of the varied influencing parameters in each sample.

Due to the variation of three chloride contents and the use of two different types of cement, a total of four different concrete mixes were made, which are listed below:

- CEM I 0 m% Cl⁻~cement;
- CEM I 1 m% Cl⁻~cement;
- CEM I 4 m% Cl⁻~cement;
- CEM III/A 1 m% Cl⁻~cement.

Table 1. Influencing parameter of each sample

Sample	Influencing parameter	
S2	Type of cement	CEM III/A 42.5 N
S3	Concrete cover	15 mm
S4	Steel reinforcement density	0.5 m ² steel/m ² concrete
S5	Chloride concentration	4 m% Cl ⁻ ~cement
S6	Source of chlorides	Ingressed chlorides*
S7	Applied voltage	24 V
S8	ECE treatment schedule	Continuously on

*An ingress chloride profile is simulated by performing a custom adjusted rapid chloride migration test (RCMT), which will be explained in paragraph 2.2.

All four mixes had a W/C-factor of 0.5 and a cement content of 320 kg/m³. The aggregate composition consisted of sand 0/4, gravel 2/8, and gravel 8/16. Additionally, to enhance the workability of the concrete mixes, a superplasticizer, namely MasterGlenium 51 con. 35% NL, was used into all concrete mixes at a dosage of 2 ml per kilogram of cement. For mixes 2, 3, and 4, the chlorides were added by dissolving NaCl in the mixing water.

Following casting, the samples were placed in a humid room to cure, maintaining a relative humidity of at least 95% and a temperature of 20 °C. The next day, the samples were demoulded and stored in this humid room, where they remained for an additional 27 days. After a total curing period of 28 days, the samples were stored indoors for 84 days, before being moved outside for the ECE treatment.

2.2 Rapid chloride migration test (RCMT)

Sample 6, where no NaCl was added in the mixing water, was used to simulate a chloride ingress scenario. Since allowing the chloride ingress process to occur naturally was not feasible within the framework of this research, a rapid chloride migration test (RCMT) was conducted instead. At the time of the test, the sample was approximately two months old. The RCMT setup was based on the NT Build 492, though adjusted to accommodate the prismatic sample used in this research.

Firstly, the sample was prepared by submersing it in a container filled with a saturated Ca(OH)₂ solution, where it was kept for five days. Secondly, the experimental setup was created as illustrated in Figure 2, where the sample was placed in a container, with a perforated stainless steel plate positioned both beneath and on top of the concrete sample. Then, to facilitate chloride penetration from the top, the reservoir formed on top of the concrete sample was filled with the catholyte solution. This catholyte solution was 10% NaCl by mass, dissolved in tap water (100 g NaCl in 900 g water). The container in which the sample was placed was then filled with the anolyte solution. This

was a solution of NaOH in tap water (approximately 12 g NaOH in 1 litre water).

Finally, the power source was turned on which created an electric field through the sample, allowing negatively charged chlorides ions to penetrate the concrete from the top surface.

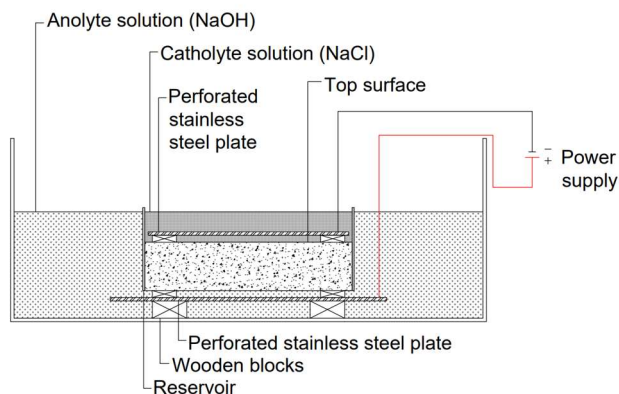


Fig. 2. The test setup for the RCMT.

The total duration of the RCMT was 40 hours on a voltage of 20 V to ensure sufficient penetration of chlorides into the concrete.

2.3 Electrochemical chloride extraction (ECE)

Before starting the ECE treatment, the test setup had to be prepared. Firstly, a reservoir was made at the top surface of the concrete samples to hold the electrolyte during the ECE treatment. Secondly, a mixed metal-oxide coated titanium (TiMMO) mesh with measurements of 250 mm × 250 mm was placed inside the reservoir on top of the concrete. This mesh can conduct a nominal current of 30 mA/m². Afterwards, the retainer on top of the sample was filled with the electrolyte, namely tap water, and was wrapped in a plastic film to avoid evaporation. Lastly, the TiMMO mesh anode was connected with the positive side of a power source and the reinforcement was connected with the negative side of the power source. The power source was set on a voltage controlled mode with a voltage of 48 V. The final test setup is shown in Figure 3.

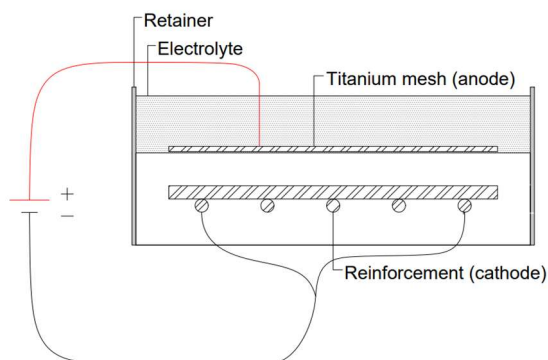


Fig. 3. The test setup for the ECE treatment.

The ECE treatment was applied for a total of seven weeks. For all samples, except sample 8, these seven weeks consisted of alternating between seven days

where the power source was on and seven days where the power source was off. This intermittent ECE schedule was based on different research, described in articles [5, 9, 10], which showed a higher efficiency of the treatment when an intermittent ECE treatment was applied. For sample 8, the power source was continuously on. During the on-periods, the current of all samples was measured.

2.4 Intermediate measurements

Several measurements were performed at different timepoints for the RCMT and ECE treatment. However, in this article, only the initial and the final measurements will be discussed in paragraph 3. For a full discussion of all the intermediate results, the author refers to [11].

2.4.1 Corrosion potential measurements

The corrosion potential of the steel reinforcement gives an indication of the corrosion risk. The results are interpreted based on the guideline in the ASTM C876-91 standard (1990) [12].

This steel potential was measured, before the ECE-treatment and then each time at the beginning and at the end of each off-period. The measurements were done by connecting a high-impedance voltmeter between the steel reinforcement and a Cu/CuSO₄ reference electrode (CSE). The reference electrode was placed on the top surface of the sample and measurements were conducted within a fixed grid of 50 mm × 50 mm, positioned above the reinforcement bars.

2.4.2 Total chloride concentration

To evaluate the effectiveness of the RCMT in forcing chlorides to ingress in sample 6 and the effectiveness of the ECE in extracting chlorides out of the samples, the total chloride concentrations were determined. This total chloride concentrations consist of free, physically bound and chemically bound chlorides [13].

Before and after the RCMT and before, during each off period and after the ECE treatment, drilling dust was taken from the concrete samples in increments of 10 mm with a drill bit of 20 mm. Then, the chloride concentrations of the obtained drilling dust of the different depths from each concrete sample was measured by means of direct potentiometry with calibration solutions, also known as the ‘Rapid Chloride Test’ (RCT). Through this approach, chloride profiles in the samples could be established.

2.4.3 Free chloride concentration

Out of the total chlorides present in the concrete, it are only the free chlorides that can be extracted during the ECE process. Therefore, the free chloride concentration is the maximum amount of chlorides that can be extracted. So, based on this free chloride concentration, the maximum removal rate can be determined [5, 13].

For sample 1 and sample 6 (after RCMT), the initial free chloride concentration was determined with a test

method also known as a ‘Rapid Chloride Test Water-soluble’ and is described in ASTM C1218/C1218M-20. This test was conducted on the same drilling dust as the test for the total chloride concentration. It must be noted that during this test, a part of the physically bound chlorides is detected. However, this is not a problem since both originally free chlorides as well as dissolved physically bounded chlorides can be extracted during the ECE treatment [5, 13].

3 Results and discussion

It is noteworthy that in all graphs of this paragraph, the chloride concentration and removal rate for each tested 10 mm depth interval are given at the average depth of the interval.

3.1 Rapid chloride migration test (RCMT)

3.1.1 Chloride concentrations

The results of the total chloride concentration measurements before and after the RCMT for sample 6 are given in Figure 4. As expected a distinct chloride profile was observable after the RCMT, where the highest concentration was present at the surface, namely a total chloride concentration of 1.46 m% Cl⁻ ~cement, and decreased progressively. At the level of reinforcement the total chloride concentration was 0.74 m% Cl⁻ ~cement. Hereby, the RCMT was successful in achieving a chloride ingress profile over the depth.

Additionally, for sample 6, the free chloride concentration after the RCMT was also tested and shown in Figure 4. This resulted in similar or even higher chloride concentrations than the total measured concentrations. This higher free chloride concentration suggests a small measurement error in the test. However, based on these results, it can be assumed that almost all chlorides in sample 6 are present as free chlorides. This can probably be explained by the fact that the chlorides were forced to ingress by an RCMT, which led to a higher free chloride concentration than in a mixed-in or a naturally ingressed scenario.

3.1.2 Corrosion potential measurements

Before the RCMT, sample 6 had a concentration of around 0.05 m% Cl⁻ ~cement of total chlorides, which resulted in quite positive corrosion potential measurements, namely an average of -73 (± 5.5) mV vs. CSE. As chlorides were ingressed in the concrete sample during the RCMT, a higher chance of active rebar corrosion could be expected after the RCMT. This resulted in more negative corrosion potential values, namely an average of -330 (± 22.9) mV vs CSE.

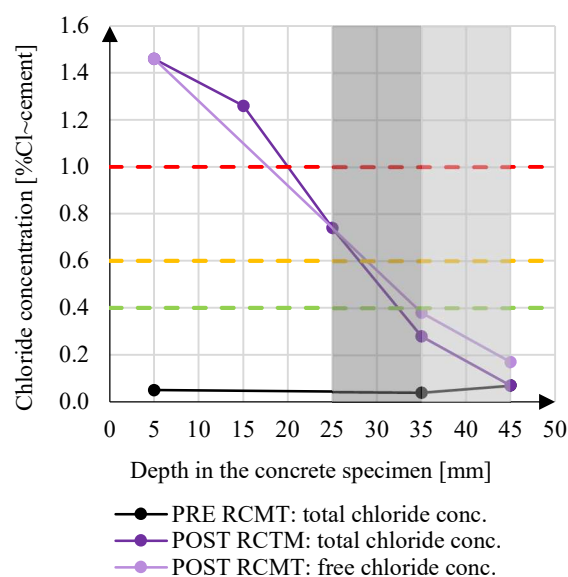


Fig. 4. The initial total chloride concentration (PRE RCMT), the final total and free chloride concentration (POST RCMT) for sample 6 at the different tested depths (with an indication of the reinforcement (first layer and second layer)).

3.2 Electrochemical chloride extraction (ECE)

3.2.1 Chloride concentrations

The averages of the initial total chloride concentrations at the different tested depths, namely 0-10 mm, 30-40 mm, and 40-50 mm, of all samples except sample 6 are given in Table 2. The average initial total chloride concentrations for all samples that were theoretically made with 1 m% Cl⁻ ~cement is between 0.68 and 0.81 m% Cl⁻ ~cement. For sample 5, with theoretically 4 m% Cl⁻ ~cement, this initial total chloride concentration is 2.68 ± 0.16 m% Cl⁻ ~cement. The initial total chloride concentration of sample 6 for the ECE is given in Figure 4, namely the POST RCMT curve. The total chloride concentration of sample 6 at the level of reinforcement, namely at 20-30 mm depth, is similar to the other samples.

Table 2. The average initial total chloride concentration of all samples.

Sample	Average initial total chloride concentration [m% Cl ⁻ ~cement]
S1	0.81 ± 0.06
S2	0.79 ± 0.06
S3	0.77 ± 0.09
S4	0.77 ± 0.03
S5	2.68 ± 0.16
S7	0.68 ± 0.05
S8	0.67 ± 0.02

The final remaining total chloride concentrations of all samples, after the full ECE treatment of seven weeks are given in Figure 5. The reinforcement net is indicated in grey. However, for sample 5, the placement of this net differs from the other samples (and thus from the indication on the graph in Figure 5), ranging from 15 mm to 25 mm.

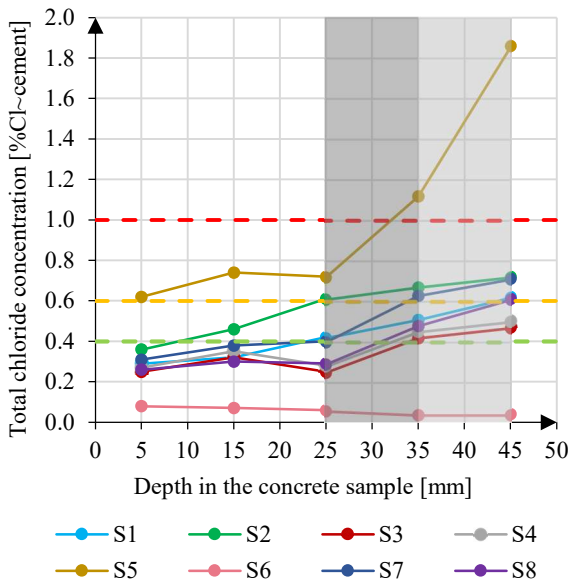


Fig. 5. The final remaining total chloride concentration of all samples at the different tested depths (with an indication of the reinforcement (first layer and second layer)).

Firstly, it is observable that samples 1 to 4, 7 and 8 namely the samples with an average initial total chloride concentration of 0.75 m% Cl⁻ ~cement, ended with similar remaining total chloride concentrations. The other samples, namely samples 5 and 6, deviate notably from this group. Sample 6, characterized by an ingressed initial chloride profile, demonstrated remarkably lower remaining total chloride concentrations. Conversely, sample 5, with higher initial chloride levels, exhibited significantly higher remaining total chloride concentrations.

Secondly, it can be observed that generally the remaining total chloride concentration for the samples, except sample 6, increased with increasing depth.

For the samples with an initial total chloride concentration of 0.75 m% Cl⁻ ~cement the average of the remaining total chloride concentrations was taken and plotted in Figure 6. Based on these initial and final total chloride concentrations, the corresponding average removal rate can be determined. This is an indicator for the amount of chlorides extracted relative to the initial total chloride concentration and is visualized in Fig. 6.

In Figure 7, a noticeable decrease in the removal rate with greater depth can be observed. For all samples, except sample 6, this can be explained by the increase in remaining total chloride concentration with greater depth. However, for sample 6, this can be explained by the initial decrease in total chloride concentration with greater depth after the RCMT treatment.

Additionally, this decrease in removal rate is larger at the 30-40 mm and 40-50 mm depths, namely between the two reinforcement bars and partially behind the reinforcement. This higher removal rate in the concrete cover was expected as the electrical field is concentrated there, resulting in more chlorides to extract in this region. However, it must be noted that there is still a chloride removal after the reinforcement. The removal rate at the 40-50 mm depth is still 16% for the samples 1 to 4, 7, and 8, 31% for sample 5, and 43% for sample 6.

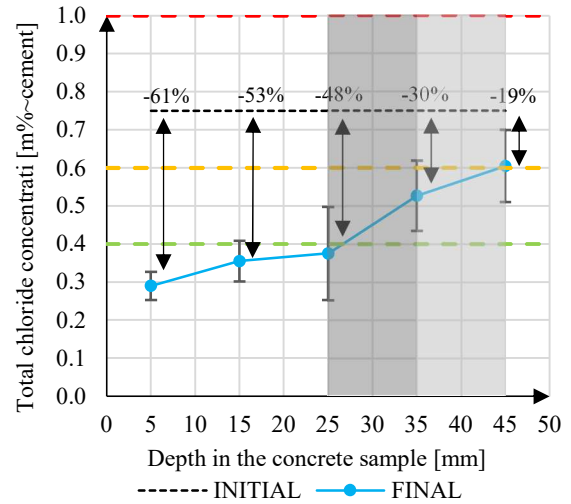


Fig. 6. The average of the initial and of the final total chloride concentrations of samples 1 to 4, 7, and 8 with corresponding removal rates (with an indication of the reinforcement (first layer and second layer)).

To be able to compare the removal rates alongside the remaining total chloride concentrations of sample 5 and 6 with the other samples, Figure 7 is provided.

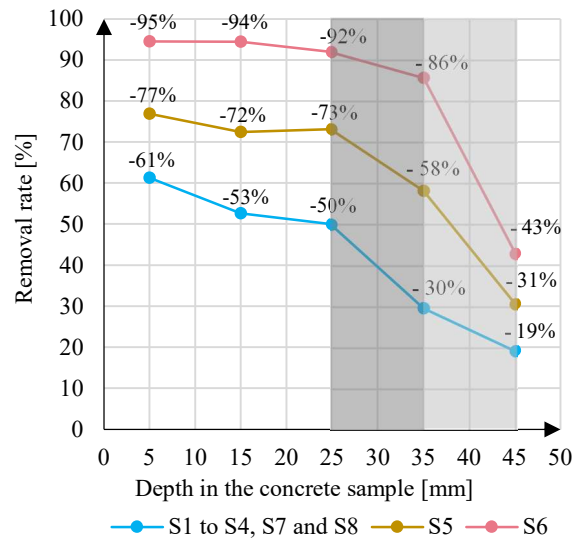


Fig. 7. The removal rates of samples 1 to 4, 7, and 8 and the removal rates of sample 5 and sample 6 (with an indication of the reinforcement (first layer and second layer)).

Samples 1 to 4, 7, and 8 (0.75 m% Cl⁻ ~cement initial total chloride concentration)

In Figure 5 it can be seen that samples 1, 3, 4, and 8 showed almost no difference in remaining total chloride concentrations. Sample 2 and sample 7 showed a slightly higher remaining total chloride concentration compared to sample 1, especially sample 2. The small deviation in the remaining total chloride concentration can be attributed to the fact that these two samples had a lower current, namely between 0.10 A and 0.30 A instead of between 0.25 A and 0.7 A (for S1). This lower current is due to a lower applied voltage (S7) or the usage of CEM III/A instead of CEM I (S8), which results in a higher resistance. However, the comparable outcomes of samples 1, 2 and 7 imply that the higher

current used in sample 1 did not significantly increase the removed chloride concentration. This may be due to the generally high currents across all samples. Literature findings indicate that beyond a certain threshold, increasing the current density only leads to a small increase in extraction efficiency. In article [14], it was found that a part of the electrical energy (current) was transformed into thermal energy, by comparing the environmental temperature and the internal temperature of samples with different current densities [14, 15].

Figure 6 shows that the average removal rate for these samples with an initial total chloride concentration of 0.75 m% Cl⁻ ~cement in the concrete cover is around 50-60%, leading to a remaining total chloride concentration for these first 3 depths below 0.4 m% Cl⁻ ~cement. The removal rate for the last two depths is goes from 30% to 20%.

Additionally, for sample 1, the initial free chloride concentration was tested, which resulted in an average free chloride concentration of 0.60 ± 0.12 m% Cl⁻ ~cement. Based on these results and the initial total chloride concentration, the chemically bound chloride concentrations is determined to be around 0.21 m% Cl⁻ ~cement. By comparing this initial free and bound chloride concentration with the final remaining total chloride concentration, it can be seen that at the surface this 0.29 m% Cl⁻ ~cement remaining total chloride concentration closely mirrors this bound chloride concentration. This indicates that almost no chlorides can be extracted anymore and thus the maximum removal rate was approximately obtained. However, for the last two depths this maximum removal rate was far from reached.

Sample 5 (2.7 m% Cl⁻ ~cement initial total chloride concentration)

The removal rates for sample 5, as shown in Figure 7, are around 1.4 times higher than the removal rates of the samples with an initial total chloride concentration of 0.75 m% Cl⁻ ~cement. For the first three depths, namely the concrete cover, this is around 75%, while for the last two depths the removal rate is 58% and 31% respectively. However, the remaining total chloride concentrations still stayed quite high. For the first three depths, the remaining total chloride concentration was between 0.62 m% Cl⁻ ~cement and 0.74 m% Cl⁻ ~cement, which is 3 times higher than these concentrations for the samples with an initial total chloride concentration of 0.75 m% Cl⁻ ~cement. For the last two depths, the remaining total chloride concentrations are 4 times higher than the samples with an initial total chloride concentration of 0.75 m% Cl⁻ ~cement, namely 1.12 m% Cl⁻ ~cement and 1.86 m% Cl⁻ ~cement respectively. Additionally, they are even above 1 m% Cl⁻ ~cement. So, despite its high removal rates due to its higher initial chloride concentration, sample 5 still ended the ECE treatment with significantly high remaining total chloride concentrations.

Sample 6 (chlorides ingressed by RCMT)

As shown in Figure 7, sample 6 has a removal rate above 90% for the first three depths, which is around 1.7 times higher than the removal rates of the samples with

an initial total chloride concentration of 0.75 m% Cl⁻ ~cement. For the last two depths, the removal rate was 86% and 43% respectively, which is 2.8 and 2.3 times higher than the removal rates of the samples with an initial total chloride concentration of 0.75 m% Cl⁻ ~cement. The final remaining total chloride concentration was quite constant for all the depths and was around 0.06 m% Cl⁻ ~cement, which is equal to the initial total chloride concentration (pre ECE treatment). For sample 6, the maximal removal rate was thus reached. Additionally, as mentioned before in paragraph 3.1.1, the initial free chloride concentration for sample 6 was tested, which showed that almost all chlorides in sample 6 were free chlorides. Based on these results, it can be concluded that ECE is efficient in removing the free chlorides out of the concrete.

3.2.2 Corrosion potential measurements

The results of the averages of the corrosion potential measurements of the steel reinforcement with the Cu/CuSO₄ reference electrode before and after the ECE treatment are given in Figure 8. Initially the potentials of all samples, except sample 5 and sample 6, are similar and around -250 mV vs CSE. Sample 5 and sample 6 have a significantly more negative potential. For sample 5, this can be explained by the higher initial chloride concentration. For sample 6, the initial chloride concentration at the level of the reinforcement is similar. However, this total chloride concentration consisted of more free chlorides compared to the other samples, which resulted in a more negative potential and thus a higher risk for active reinforcement corrosion.

The results after the ECE treatment, measured after seven days of off-period, show that the corrosion potential of the steel became more positive for all the samples, and was above the limit of -200 mV. This means that, according to ASTM C876, the chance of having no corrosion is above 90%.

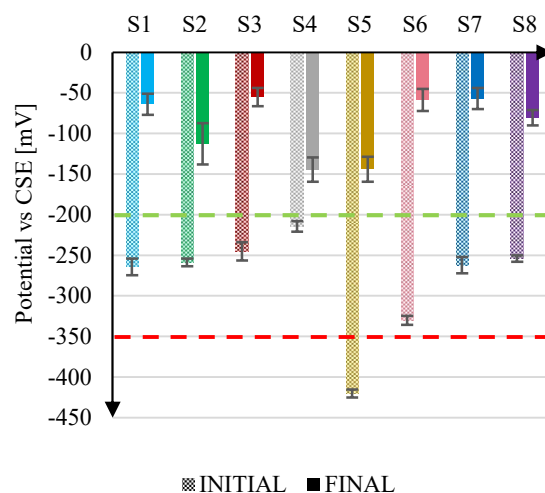


Fig. 8. Evolution of the average corrosion potentials vs CSE of the steel reinforcement of all samples.

4 Conclusion

Certain conclusions can be drawn from examining the influence of different parameters on the effectiveness of an ECE treatment.

- (1) Irrespective of the variable parameters, it is found that the removal rate decreases with an increasing depth into the concrete sample. The removal rates of the different samples were always notably higher within the concrete cover than deeper in the concrete. Therefore, it can be concluded that ECE is particularly effective in removing chlorides in the concrete cover. However, in all samples chlorides were still extracted between the two reinforcement layers and even beyond the reinforcement.
- (2) It can be seen that when performing an ECE treatment on a concrete sample containing around 0.75 m% Cl⁻-cement mixed-in chlorides, factors such as reducing the steel density, reducing the concrete cover, or using a continuous ECE treatment schedule instead of an intermittent one (alternating seven days on - seven days off) have little to no effect on the achieved removal rate. This research revealed that after a treatment of seven weeks, chloride removal rates for samples with CEM I and 0.75 m% Cl⁻-cement ranged from 50-60% in the concrete cover to 20-30% around the reinforcement and behind it.
- (3) It was observed that when a profoundly high current was applied, an increase in current did not lead to a significantly higher removal rate. Two parameters, namely using CEM III/A instead of CEM I and reducing the voltage from 48 V to 24 V, led to relatively lower currents. However, similar removal rates were observed, which may be due to the generally high currents across all samples. Literature findings indicate that beyond a certain threshold, increasing the current density only leads to a small increase in extraction efficiency, since a part of the electrical energy (current) is transformed into thermal energy [14, 15].
- (4) Two factors showed a big influence on the removal rate. A higher initial chloride concentration of 2.68 m% Cl⁻-cement in the concrete resulted in higher removal rates by a factor 1.4 compared to the samples with 0.75 m% Cl⁻-cement. In the concrete cover this removal rate is around 75%, while between the reinforcement layers and behind the reinforcement it is 58% and 31% respectively. However, despite the enhanced removal, the remaining total chloride concentrations are still above 1 m% Cl⁻-cement after the full ECE treatment. This indicates that the performed treatment was insufficient for an initial chloride concentration of 2.68 m% Cl⁻-cement.

Furthermore, using a rapid chloride migration test (RCMT) to introduce chlorides into a concrete sample notably affected the effectiveness of the ECE treatment. Since an RCMT resulted in a greater concentration of free chloride compared to a mixed-in scenario, the removal rates significantly

increased. In this study, the removal rates ranged from 90-95% in the concrete cover to 45-85% between the reinforcement layers and behind the reinforcement, which is between 1.7 and 2.8 times higher compared to the samples with 0.75 m% Cl⁻-cement. Therefore, it can be concluded that ECE is effective in extracting free chlorides from the concrete. However, in practical applications involving chloride ingress in real structures, such high removal rates are not expected, since a bigger part of these chlorides is bound.

- (5) The corrosion potential measurements of the steel reinforcement with the Cu/CuSO₄ reference electrode showed a global increase after the ECE treatment, which resulted in more positive values than -200 mV vs CSE. According to ASTM C876, this indicates a low risk for active reinforcement corrosion. Thus, it can be concluded that irrespective of the remaining total chloride concentrations and the removal rates obtained in this research, ECE treatment can achieve a low likelihood of steel corrosion.

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