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Electro-chemical chloride extraction: Influence of C_3A of the cement on treatment efficiency

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Abstract

The aim of this study was to clarify whether the C_3A content of cement had a significant effect on electrochemical chloride extraction (ECE) treatment efficiency. It is known that a higher C_3A content in a cement gives it superior chloride complexing ability resulting in the formation of an “insoluble” calcium chloro aluminate compound.

ECE was applied using cylindrical specimens made from concrete containing two levels of C_3A (4.3% and 9.05%). Specimens were 5 cm in diameter and 10 cm in height. Steel was placed in the axial direction with an embedded length of 7 cm. These specimens were immersed in an NaCl solution and dried in a stream of air at 40 °C for 10 months. The corrosion was monitored by half-cell potential and polarization resistance measurements. After steel depassivation, ECE was applied for 20, 30, 40 and 50 days using a constant current density of 1 A/m^2 of steel. At the end of the treatment, the specimens were maintained at 20 °C and 70% RH in order to observe the evolution of the steel (electrochemical measurements).

The results show that, after 30 days of treatment, the chloride content remained constant in the specimen. This was probably due to OH^- ion formation on the steel. The OH^- ions “contribute” to the current transport, decreasing the ECE efficiency. Concerning the C_3A content, ECE efficiency was slightly affected by C_3A because only a part of the bound chloride ions was released. From the point of view of corrosion, half-cell potential showed a shift in the positive direction, indicating little corrosion activity at 20 °C and 70% RH. However, polarization resistance measurements showed that 2 months post treatment corrosion rates were significant, although the corrosion rate decreased from 6 $\mu A/cm^2$ to 2.5 $\mu A/cm^2$.

Keywords: Corrosion; Electrochemical chloride extraction; C_3A

1. Introduction

Chloride ions are considered to be the major cause of premature corrosion of steel reinforcement. They are common in nature and small amounts are usually unintentionally included in the mix ingredients of concrete. Dissolved chloride ions may also penetrate unprotected hardened concrete in structures exposed to marine environments or to deicing salts.

Chloride ions can be eliminated by a process known as electrochemical chloride extraction (ECE). This process has been used to decontaminate structures such as bridge decks [1]. The removal is accomplished electrochemically by using a noble anode and an electrolyte suitable to the structure surface, and passing direct current between this anode and the reinforcing steel which acts as the cathode. Since anions or negatively charged ions migrate toward the anode, it is possible to make chloride ions migrate away from the reinforcing steel and out of the concrete structure. A total charge between 3000 and 1000 A h/m^2 is applied during the treatment [2–6]. At the same time, positive ions (Na^+, K^+ and Ca^{2+}) migrate away from the anode towards the reinforcements.

This electrochemical technique produces hydroxyl ions at the cathode or the steel, while water is decomposed at the anode. As shown in the following reactions, hydrogen gas

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can also be produced at the cathode (depending on the potential):

$$H_2O + 1/2O_2 + 2e^- \rightarrow 2OH^-$$

$$2H_2O + 2e^- \rightarrow 2OH^- + H_2$$

And chlorine release and/or acidification of the electrolyte can occur at the anode:

$$2OH^- \rightarrow H_2O + 1/2O_2 + 2e^-$$

or

$$H_2O^- \rightarrow 2H^+ + 1/2O_2 + 2e^-$$

$$2Cl^- \rightarrow Cl_2 + 2e^-$$

On the other hand, it is well known from the literature that the chloride ions can be present in concrete in two forms [7]:

- free or weakly bound chloride ions;
- chloride combined in compounds such as calcium chloro aluminate.

It is the free and weakly bound part of the chloride which promotes corrosion [7] even if bound chloride may participate in corrosion initiation when the establishment of pH gradients is required to sustain passive film breakdown [8]. Nevertheless, the chemical combining of chloride ions is frequently considered as a beneficial effect in that it reduces the rate of chloride penetration in concrete [9].

It is also well documented that a higher C_3A content gives cement greater chloride complexing ability resulting in the formation of an “insoluble” calcium chloro aluminate compound.

According to Metha [10], chemical binding of penetrating chlorides cannot be expected unless the C_3A content is higher than about 8%.

Recent experimental observations have shown that a C_3A content as high as 8.6% does not effectively reduce chloride penetration when concrete is immersed in seawater [11] and other studies have found more corrosion-induced distress associated with high C_3A content [12].

It has also been reported that the majority of chloride binding takes place during the first 28 days of curing [13].

Whatever the explanation, the C_3A content may change the electrochemical treatment efficiency.

In addition, it is believed that the components with bound chlorides and free chlorides in the pore solution are connected by a chemical equilibrium [14,15]. This implies that if the free chlorides are removed, bound chlorides will dissolve until the new equilibrium is reached. This phenomenon could limit the treatment efficiency because bound chlorides would be dissolved and available for their deleterious role of promoting the corrosion of reinforcing steel. It can also adjust the forecasts of the models if it is not taken into account correctly [16–18].

The aim of this work was to observe ECE efficiency and determine whether the C_3A content was a factor influencing this efficiency.

ECE efficiency was evaluated by the reduction in the chloride content and the subsequent electrochemical behavior of the system, i.e. the true effect of ECE on steel corrosion rate.

2. Experimental procedures

2.1. Preparation of tests slabs

Two series of concrete were prepared, containing cements with C_3A contents of 9.05% and 4.3%. The chemical composition of the cements is listed in Table 1. Other constituents were identical in both concretes and both series were cast with the same cement/sand/aggregates/water ratio. Details of the concrete mix are given in Table 2.

Cylindrical specimens of 5 cm diameter and 10 cm height were cast and a steel rod of 10 mm diameter was placed in the axial direction with an embedded length of 7 cm (Fig. 1).

After unmolding, specimens were cured for 28 days at 20 °C and 50–60% RH. After curing, specimens were submitted to immersion–drying cycles. They were immersed in a solution containing NaCl (35 g/l) and dried in a stream of air at 40 °C.

One set of tests was made prior to the electrochemical treatment. The tests included chloride analyses (free and total) and electrochemical measurements (half-cell potential and polarization resistance).

After 10 months of immersion–drying cycles, tests revealed a high chloride content and a high steel corrosion activity. Then, ECE was applied.

2.2. Application of ECE and measurements

Before treatment, specimens were immersed in the $Ca(OH)_2$ (electrolyte of extraction) for 12 h in order to “saturate” the specimen, reduce the initial electrical resistance and “facilitate” the ECE process.

ECE was performed using a constant current density of 1 A/m^2 of steel surface with $Ca(OH)_2$ as the electrolyte and titanium mesh as the external anode. The experimental set up for ECE is shown in Fig. 2.

The treatment periods were 10, 20, 30, 40 and 50 days. Chloride analyses and electrochemical measurements were

Table 1
Chemical composition of cements used (cement 1=CEM I 52.5 R; cement 2=CEM I 52.5 R PMES)

	CaO	SO_3	SiO_2	Al_2O_3	MgO	Fe_2O_3	Na_2O	K_2O	C_3S	C_2S	C_4AF	C_3A
Cement 1	62.4	2.96	20.1	4.9		2.34	0.21	0.74	56.3	15.3	7.11	9.05
Cement 2	67.2	2.10	22.7	2.9	0.71	2.00	0.29	0.10	70.7	12.6	6.8	4.30

Table 2
Concrete mix used

Component	Formulation (kg/m^3)
Sand (0/4 R)	810
Aggregates (4/10 R)	1000
Cement	300
Water	210
Ratio W/C	0.7
Compressive strength	40 MPa

conducted for each period. One specimen was used for each period. For the chloride analyses, cores were taken from the specimen, each core was dry-cut into 5-mm-thick slices and total chloride (acid soluble chlorides) and free chloride (water soluble chlorides) were determined by potentiometric titration with $AgNO_3$ according to AFREM recommendations [19].

Half-cell potential and polarization resistance were measured by a Calomel electrode and a Voltalab potentiostat respectively. A three-electrode method was used to measure the polarization resistance. The steel was cathodically and anodically polarized 20 mV from the half-cell potential using a sweep rate of 12 mV/min. This sweep is recommended by Gonzalez et al. [20].

Electrochemical measurements were conducted before extraction and at varying times after interruption of the extraction current.

3. Results

3.1. Variation of chloride profile in concrete made using cement 1 with 9.05% C_3A

Figs. 3 and 4 show free and total chloride variation during the treatment versus time and depth of samples for the concrete specimens made using cement 1 with 9.05% C_3A. Chloride content is given as a percentage by weight of cement (% wc).

As can be seen in Fig. 3, free chloride was removed significantly in the first 30 days of treatment, during which the content went from 2.2 to 0.47% wc (about 78% removed) in the 5 mm nearest to the steel. In the 30 to 40 day and 40 to 50 day periods, chloride ion variation was smaller. The final free chloride ion content was 0.22% wc (about 90% removed). This free chloride content is below the limit fixed by European standards, which accept 0.4% wc [21], and below the threshold indicated by numerous authors [22].

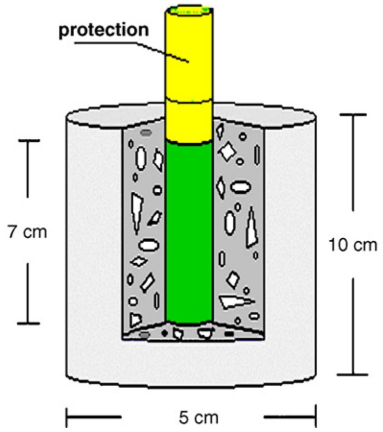

Fig. 1. Concrete specimen for the electrochemical tests.

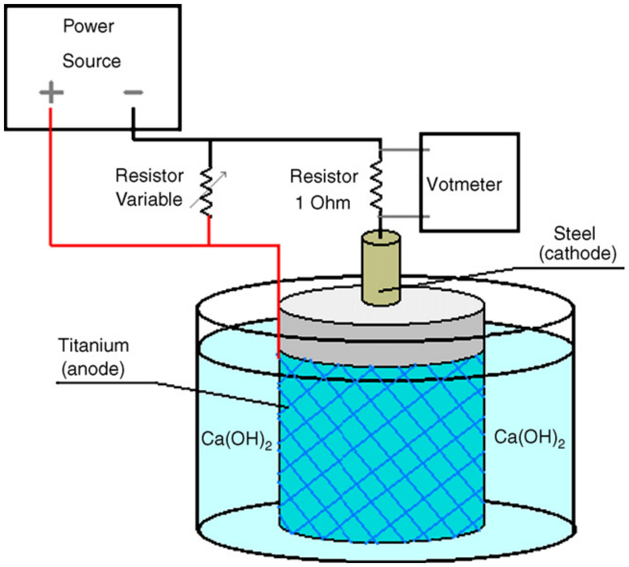

Fig. 2. Experimental ECE set-up.

Concerning total chloride ion variation (Fig. 4), as happened with free chloride ions, the chloride content had decreased markedly after 30 days of treatment. The amount of total chloride decreased from 4.4 to 1.3% wc close to the steel (about 70% removed).

In the subsequent periods up to 40 and 50 days, the variation was smaller. The final total chloride ion content near the steel was 0.91% wc, i.e. about 79% of total chloride removed.

3.2. Variation of chloride profile in concrete made using cement 2 with 4.3% C_3A

Figs. 5 and 6 show free and total chloride variation with time and depth of cover concrete for the concrete specimens made using cement 2 with 4.3% C_3A. Chloride content is given as a percentage by weight of cement (% wc).

The results show that, after 30 days of treatment, the proportion of free chloride ions had decreased from 3.4 to 0.66% wc (about 80% removed) near the steel. However, after this period, the chloride ions were removed more slowly. The final chloride content near the steel was 0.21% wc (about 94% removed).

For the total chloride variation (Fig. 6), the same evolution was observed as for free chloride ions. After 30 days of treatment, the chloride content had decreased markedly from 4.8 to 1.25% wc (about 73% removed) near the steel. No significant changes were observed at 40 or 50 days. The final chloride content near the steel was 0.70% wc, that is to say about 85% had been removed.

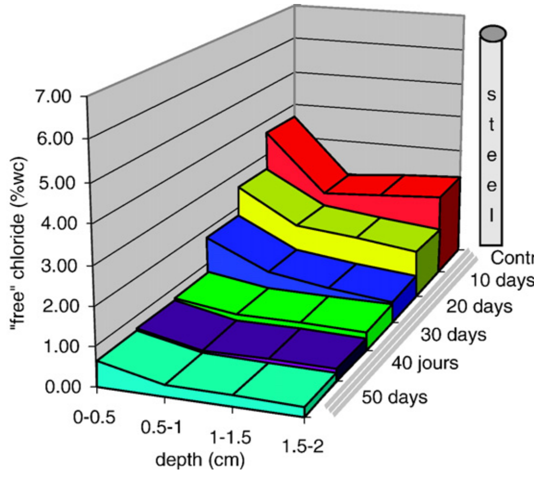

Fig. 3. Free chloride content variation (cement 1 with 9.05% C_3A).

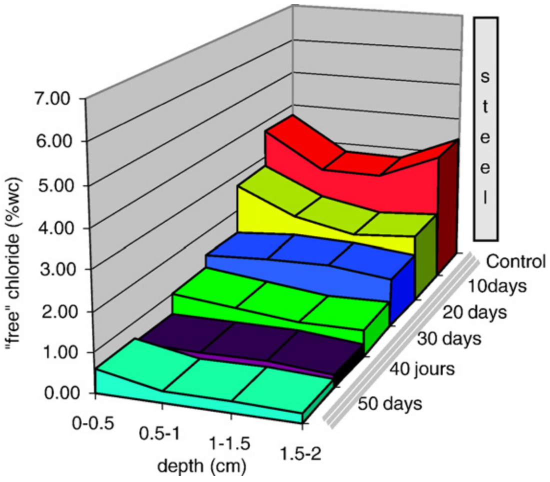

Fig. 5. “Free” chloride content variation (cement 2 with 4.3% C_3A).

The amount of free chloride removed close to the steel from specimens with 4.3% C_3A was 4% higher than from specimens with 9.05% C_3A. Concerning total chloride ions, the chloride removed from specimens with 4.3% C_3A was 6% more than from specimens with 9.05% C_3A. That is to say that the influence of C_3A content is small.

The free chloride remaining in the specimens was 0.22% wc for 9.05% C_3A and 0.21% wc for 4.3% C_3A. This suggests that there is a limit to ECE.

The total chloride remaining in the specimens was 0.91% wc for 9.05% C_3A and 0.70% wc for 4.3% C_3A. This confirmed that the “chemically” bound chlorides in specimens made with 9.05% C_3A were not released by ECE.

3.3. Variation (by mass) of chloride profile for the two cements

If the data are re-calculated or expressed in kg/m^3 of concrete and the volume of concrete is introduced for each depth, a chloride mass balance can be obtained. These results are re-plotted in Figs. 7–9. Now, the variation over time in the amounts of free, total and bound chloride (determined by comparing acid-soluble and water-soluble chloride from the same cores) is plotted. That is to say, the concrete volume found by chloride ions during migration (concentric circles) has been eliminated.

Fig. 7 shows the variation of the free chloride content in the specimen during treatment. As expected, free chloride content was higher in specimens with 4.3% C_3A before treatment, i.e. free chloride content was limited by the C_3A content as mentioned in the literature [7].

It can be observed that the migration rate of free chloride ions was not constant during treatment and that this migration rate was not equal for the two concrete types. For example, for specimens with 9.05% C_3A, the migration rate during first 20 days was about 42 mg/day. This migration rate supposes total extraction in 34 days. However, the migration rate decreased from 42 to 30 mg/day between the 20th and 30th day, from 30 to 8 mg/day between the 30th and 40th day, and from 8 to 2 mg/day between the 40th and 50th day.

For the total chloride content shown in Fig. 8, the migration rate was slightly higher than the migration rate observed

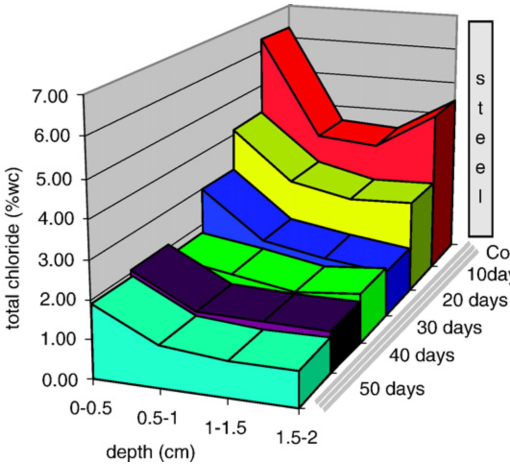

Fig. 4. Total chloride content variation (cement 1 with 9.05% C_3A).

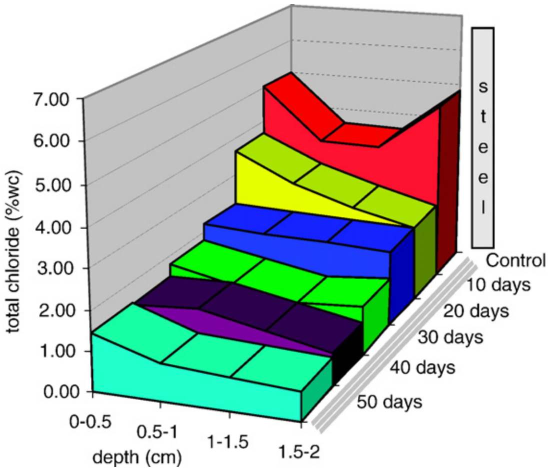

Fig. 6. Total chloride content variation (cement 2 with 4.3% C_3A).

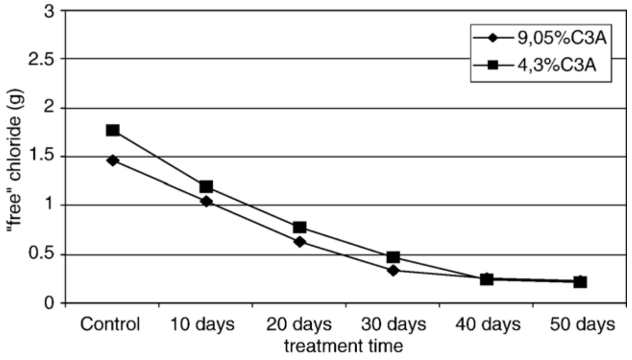

Fig. 7. Variation of "free" chloride content in specimens.

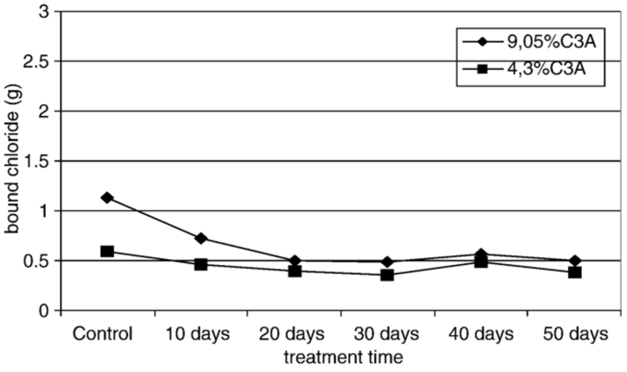

Fig. 9. Variation of bound chloride content in specimens.

for free chloride, but this migration rate also decreased during the treatment. Thus, migration rates decreased from 83 to 63 mg/day between the 10th and 20th days, from 63 to 31 mg/day between the 20th and 30th days. Between the 30th and 40th days, migration rate was constant. Finally, in the last period of treatment, a migration rate of 9 mg/day was observed.

According to the results obtained in the present study, no firm conclusion can be drawn as to the variation of the quantity of "bound" chloride ions. This is deduced from the fact that the migration rate of "free" chloride ions and total chloride ions is not equal. The variation in the values for "bound" chloride ions is depicted in Fig. 9. Initially, during the first 10 days of treatment, for specimens made with 9.05% C_3A, the amount of "bound" chloride ion decreases very steeply. After 20 days, these ions remain stabilized at a much lower value than those measured before treatment.

The variation of bound chlorides with treatment time and depth of cover concrete is shown in Figs. 10 and 11. It can be seen that:

- the bound chloride content before treatment was higher in specimens made with 9.03% C_3A than in those made with 4.3% C_3A as mentioned in the literature [7];
- the physically bound chloride ion content was more modified when the ions were close to the electrodes (depth 0–0.5 and 1.5–2 cm) as mentioned by Tritthart [23];
- the physically bound chloride content was initially greatly modified but stabilized after the 20th day;
- the final chemically bound chloride content remained above 0.5% wc.

3.4. Electrochemical behavior

Polarization resistance and half-cell potential were used to observe the evolution of the steel/concrete interface. Electrochemical measurements are greatly influenced by humidity and temperature. Therefore, all electrochemical studies were made under the same conditions. Before tests, the specimens were immersed in water for 12 h in order to stabilize the half-cell potential and to avoid overload in the potentiostat. In this case, corrosion rate is not controlled by oxygen diffusion to the steel surface. This fact was verified by electrochemical impedance spectrum (EIS) tests [24].

Tables 3 and 4 present the values measured before treatment and those recorded at several times after treatment.

Half-cell potential and corrosion rate values were very high before treatment, which is typical of concrete containing chlorides.

After the treatment periods, the steel had a very high half-cell potential. Such potentials are attributable to the very high polarization of the steel during treatment. This implies that, under these conditions, care has to be taken with the

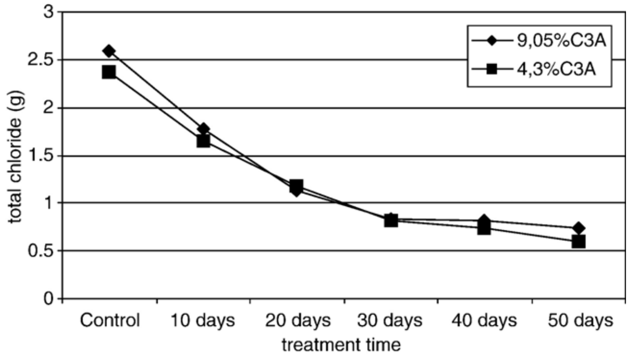

Fig. 8. Variation of total chloride content in specimens.

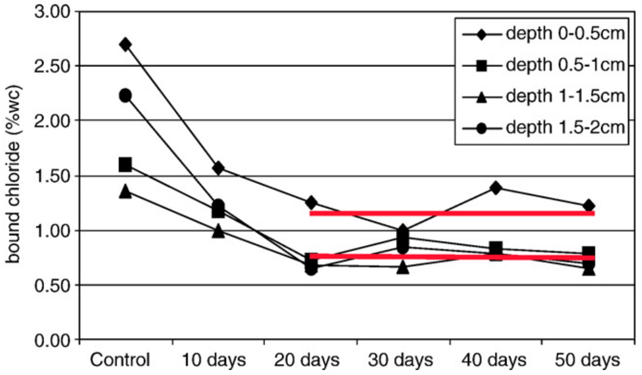

Fig. 10. Variation of bound chloride from specimens with 9.03% C_3A.

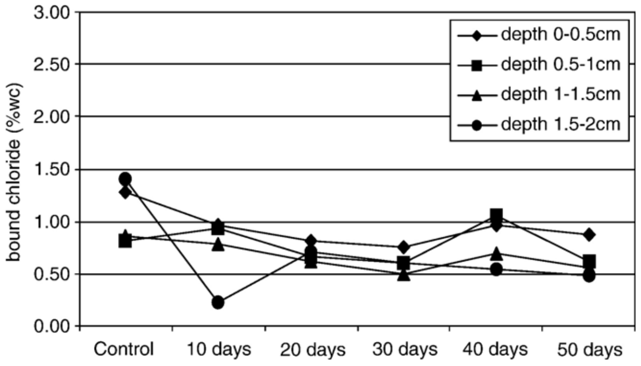

Fig. 11. Variation of bound chloride from specimens with 4.3% C_3A.

interpretation of half-cell potential values. The criteria given by ASTM C876-80 [25] do not apply here.

After treatment, specimens were maintained under laboratory conditions (20 °C and 60% RH). Depolarization was observed and the half-cell potentials measured were those shown in Table 3. These results show that, for 10 days of treatment, half-cell potential changed from −365 to −335 mV vs. calomel (SCE). For 30 days of treatment, half-cell potential changed from −616 to −153 mV and for 50 days from −577 to −172 mV. These results indicate that the steel was passivated according to the criteria of ASTM 89 950.

Table 4 shows the values measured for corrosion rate (polarization resistance) before the treatment, at several periods during treatment and 2 months after completion of the treatment.

Before treatment, the corrosion rates were very high. They were between 5.28 and 7.62 μA/cm^2, indicating significant active corrosion. These values are of the same order as those observed by other authors under field and laboratory exposure [26,27]. After completion of the treatment, although the steel was polarized, corrosion rates decreased only slightly or remained constant. Two months after completion of the treatment, the corrosion rate was much lower than the values measured before treatment. Corrosion rate values were reduced from 6.48 to 2.65 μA/cm^2, from 7.62 to 2.19 μA/cm^2 and from 6.59 to 2.89 μA/cm^2 at 10, 30 and 50 days, respectively.

Table 3
Half-cell potential E before, during and after treatment (E in mV vs. SCE)

Specimen and treatment time	Before treatment	After 10 days treatment	After 30 days treatment	After 50 days treatment	2 months after the end of treatment
1 (10 days)	−635	−1122			−335 [a]
6 (10 days)	−658	−1120			
9 (10 days)	−647	−1127			
2 (30 days)	−640		−1075		
5 (30 days)	−616		−1080		−153 [a]
8 (30 days)	−622		−1070		
3 (50 days)	−619			−1069	
4 (50 days)	−571			−1065	
7 (50 days)	−577			−1025	−172 [b]

[a] Specimens maintained in air (20 °C 60% HR) for 2 months after treatment.
[b] Specimens maintained in air (20 °C 60% HR) for 1 month after treatment.

Table 4
Polarization resistance tests before, during and after treatment (I_{corr} in μA/cm^2)

Specimen and treatment time	Before treatment	After 10 days treatment	After 30 days treatment	After 50 days treatment	2 months after the end of treatment
1 (10 days)	6.48	5.49			2.65 [a]
6 (10 days)	5.28	4.93			
9 (10 days)	5.52	4.64			
2 (30 days)	6.61		5.84		
5 (30 days)	7.62		4.97		2.19 [a]
8 (30 days)	5.97		4.44		
3 (50 days)	6.26			4.85	
4 (50 days)	5.93			4.36	
7 (50 days)	6.59			4.90	2.89 [b]

[a] Specimens maintained in air (20 °C 60% HR) for 1 month after treatment.
[b] Specimens maintained in air (20 °C 60% HR) for 2 months after treatment.

In general the results indicate that corrosion rates decreased by 50%. However, they still remained higher than the limit of 0.05–0.1 μA/cm^2 proposed for passive conditions [28], although half-cell potentials showed a shift in the positive direction, indicating little active corrosion. Similar results have been observed by other researchers [29,30].

4. Discussion

4.1. Ion migration

The results presented show that the "free" or total chloride migration rate is not constant throughout the treatment. This can be attributed to two factors: a decrease in chloride concentration and an increase in hydroxyl concentration. These explanations were advanced by Andrade et al. [30], who presented a simulation of the process and suggested that the effects were better represented by considering the chloride transference number.

Elsener et al. [31] also suggest that the greater the quantity of chloride removed, the lower the transfer number and treatment efficiency.

Tritthart [23] observed that the chloride content decreased at the various depths. He mentioned that the differences were due to the direction of migration of the chloride ions. Thus the chloride content decreased most slowly near the steel and most rapidly near the anode. He observed that the chloride content initially fell steeply, particularly in the region next to the cathode, but subsequently changed little. He proposed that, at the start of treatment, when the chloride concentration in the specimen was high, the chloride concentration fell relatively rapidly. Later, when the chloride concentration in the specimen became smaller, the proportion of total current, which was carried by chloride ions, was reduced. As a consequence, the rate of decrease of the total chloride content became slower.

According to the results obtained in the present work, it seems clear that there is "competition" between OH^- and Cl^- in carrying the current. Considering that the process is applied with a constant current, OH^- formation seems to be the main reason for the decrease of chloride migration rate. This could explain the large decrease in chloride ion after 30 days of treatment. In addition, we recall from electrochemistry that the mobility of OH^- ions is greater than the mobility of chloride ions. In other words, the proportion of the total current carried by chloride is reduced and that carried by OH^- is increased.

Elsener et al. [31] and Tritthart [23] did not mention OH^- formation and its influence on treatment efficiency. It is Andrade et al. who speak of OH^- formation and the competition between OH^- and Cl^-. It is therefore reasonable to assume that not only chloride ion diminution but also OH^- release may be responsible for the decrease in chloride ion migration rate.

An interesting point is the variation of bound chloride ion. It was seen that these ions were also bound after 28 days of curing and their proportions were modified during the treatment.

Enevoldsen et al. [32] have suggested that the “chemically bound” chlorides can be released into the paste pore solution by ECE. However, the fact that an amount of bound chloride (0.5% wc) is found even after treatment indicates that the chemically bound chloride is not totally released. Only a part of the bound chloride (which may considered as “adsorbed”) is brought into solution under the effect of the direct current.

Elsener et al. [31] mention that there is a chemical equilibrium between bound chlorides and free chlorides. Thus, after removal of the “free” chlorides and a sufficient waiting time, bound chlorides are dissolved until the new equilibrium is reached. This hypothesis could explain the corrosion rate observed 2 months after treatment.

4.2. Electrochemical tests

Half-cell potential and polarization resistance measurements indicate a reduction in the corrosion rate. However, although “free” chloride content was reduced to 0.2% wc (threshold 0.4% wc), the corrosion rate remained high even 2 months after treatment. Two possible explanations for this behavior may be:

- the chloride content or “threshold” for a repassivation of the steel by ECE is lower than 0.2% wc;
- the free chloride ions are modified due to a chemical equilibrium between bound chloride (0.73% wc) and “free” chloride (0.2% wc).Unfortunately, the free chloride ion analysis was not performed 2 months after treatment.

Another interesting observation is that the comparison of chloride analyses before and after treatment and the half-cell potential measurements cannot be used alone to evaluate ECE efficiency. From electrochemical considerations, when a half-cell potential changes from −150 mV to more positive values, only a little active corrosion is to be expected [25]. However, from the results it can be seen that, although the half-cell potential clearly shows a shift to more positive values, the corrosion rate still remains higher than the passivation limit. This fact has also been observed by other authors [29,30,33].

It is interesting to note that other works found corrosion rates higher than before treatment [29,30,33,34]. This was not the case in this study.

5. Conclusions

The following conclusions can be drawn from the present study:

- A large proportion of the chloride ions can be removed from concrete by ECE.
- The treatment is limited by transference number decrease of the chloride ions and OH^- formation on the steel which preferentially carries the current towards the outside.
- The corrosion rate decreased 50% as a result of the chloride ion decrease and OH^- generation on the steel.
- The treatment efficiency seems to be unaffected or only slightly affected by C_3A content because the “physically” bound chloride can be released during treatment.
- Neither the half-cell potential, nor the chloride ion evolution can be used alone to determine the treatment efficiency. A polarization resistance technique is needed to verify that the steel is really repassivated after treatment.

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