

MONITORING OF ELECTROCHEMICAL CHLORIDE EXTRACTION (ECE) FROM REINFORCED CONCRETE ELEMENTS

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Abstract

The main objective of this study is to monitor the evolution of ionic conductivity within the recovering layer of reinforced concrete structures by electrochemical chloride extraction (ECE) during treatment operations. The ECE process consists in the application of a direct electrical field between the reinforcement grid, inside the concrete, and an external metallic mesh to promote the migration of chloride anions away from the concrete. Since chloride anions are the main single ions responsible for the reinforced concrete structure corrosion and degradation, ECE is an obvious advantage in terms of concrete structure renovations that are subjected to maritime conditions and in the early stages of concrete contamination, this is a degradation process that is common in coastal areas.

Laboratory tests were made using concrete prisms contaminated with chloride ions in contact with a saturated calcium hydroxide solution. The extraction process was followed by measuring the ionic conductivity using the two pins technique. Two stainless steel rods embedded in the concrete prism were used as pins.

Keywords and phrases: electrochemical chloride extraction (ECE), process monitoring, concrete conductivity, concrete corrosion.

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Variations of ionic conductivity with the depth in concrete covering layer are discussed and it is shown that this procedure can be used as a monitoring technique based on the ECE process.

1. Introduction

Concrete structure degradation is a problem with enormous economical and environmental impacts. One of the main causes of degradation is the steel corrosion reinforcement. As known, the steel within the concrete is a chemical environment that is favourable to its passivation. However, this corrosion resistance reduces drastically when high concentration of chlorides or carbon dioxide is present in the environment, namely, in coastal, industrial, urban areas, as steel will suffer active corrosion and the concrete steel structure will degrade progressively allowing the onset of an ever increasing quantity of aggressive agents [1].

Different techniques have been used to rehabilitate structures subjected to corrosion by chlorination and carbonation. The solutions that should be adopted are influenced by the state of degradation and contamination of the structure and can be divided into three main groups: preventive, curative, and reconstructive techniques. The main purpose of preventive techniques, normally applied to new or recent structures with low levels of degradation, is to reduce the aggressor's levels of contamination. Within this group are included: the application of an external coating that functions as a barrier in order to reduce the diffusion of external aggressive agents, corrosion inhibitors, steel galvanization, and cathodic protection. Curative techniques are applied to intermediate degradation states and generically include electrochemical re-alkalinization, cathodic protection, electrochemical chloride extraction, and also localized reconstruction techniques. And finally, reconstructive techniques which apply to terminal states of structure degradation when the existing structure must be partially or totally replaced [2].

The contamination of offshore structures by chloride ions is a question of great technological and economical importance. In 1973, the Kansas Department of Transportation, being aware of this issue, developed the concept of chloride ion extraction in concrete by electrochemical migration. There have been several studies that show it is possible to remove chlorides from concrete by using electrochemical means. There is even a set of rules for practical application for businesses of this specialty [3-5]. Briefly stated, one of the great advantages of applying the ECE technology is the relative low cost of implementation compared to a traditional repair involving demolition and reconstruction of concrete elements contaminated with aggressive agents.

ECE, also known as desalinization, consists in the application of an electric field between the reinforcement, inside the concrete and an external electrode formed by a mesh of stainless steel or titanium (both inert in this environment and operational conditions) immersed in an electrolyte reservoir. Figure 1 shows schematically the ECE process.

Electrolytes that have been used include sodium borate (Na_3BO_3) and lithium borate (Li_3BO_3) as buffer solutions, or calcium hydroxide ($\text{Ca}(\text{OH})_2$) and sodium hydroxide (NaOH). In this process, the chloride ions tend to migrate away from the concrete due to the electric field formed between the reinforcement (cathode) and the outer mesh (anode). Of course, due to charge compensation requirements, cations also migrate in the regions closer to the reinforcement and hydroxide ions are produced within the concrete as a result of cathodic reactions. A typical treatment uses currents between 1 and 5A/m^2 at a maximum voltage of 50V, during 10 to 50 days in order to remove 20 to 50% of chlorides (by cement mass) present in the structure [2].

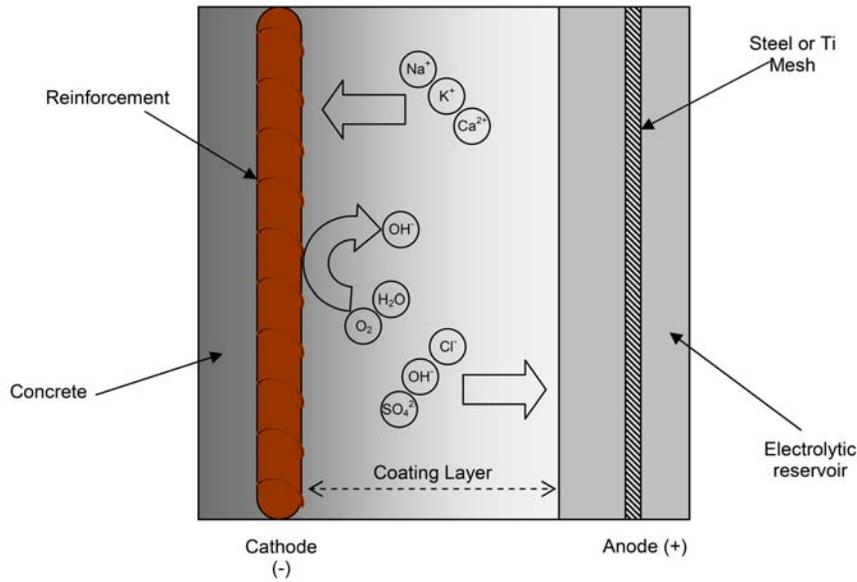
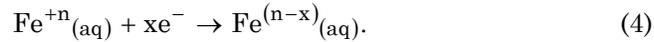


Figure 1. Schematic representation of an ECE process.

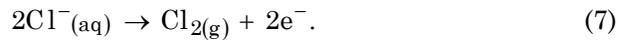
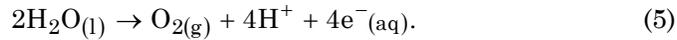
The application of the ECE process to a reinforced concrete structure is conditioned by some requirements [5-17]. Firstly, the structure should be contaminated with chloride ions and the reinforcement under attack by these same ions. The existence of corrosion derived from chloride ions should be checked by using the set of inspection techniques. In order to ascertain chloride ion contamination level in the structure, concrete chemical analysis at different depths and at different zones, must be performed before the ECE in order to be able to program the whole treatment process, including time and current required relevant parameters in defining the process [9]. Another important requirement for successful implementation of ECE is the existence of the reinforcement's electrical continuity in order to be able to distribute power to all areas that will be addressed. Indeed, the efficiency of the extraction process depends on the uniform current distribution that is obtained in the structure [10].

Since the process involves the application of DC (direct current) to promote ion migration, net oxidation, and reduction processes occur at the electrodes. Thus, at the cathode, the same is expected to occur following semi-reduction reactions depending on the environment near the rebars:



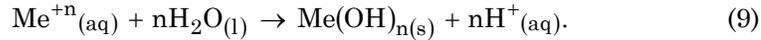
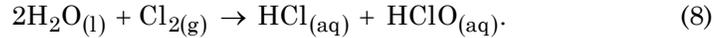
Reaction (1) tends to occur very slowly because the oxygen concentration near the cathode (steel reinforcement) is very small and tends to decrease during the process. Reaction (3) will only occur in a very localized manner on the surface of the reinforcement. Reaction (4) is always associated with an iron oxide. Thus, with application of very high currents in the ECE process, the main product formed from the cathode is hydrogen. The formation of hydrogen near the reinforcement can cause a weakening of the rebar-concrete bond [6] and hydrogen embrittlement problems, which are of particular concern in the presence of pre-stressed and post-stressed reinforced concrete structures [7, 8]. Pre-and post-stressed steel microstructures possess voids, particularly grain boundaries and defects, with the capacity to hold atomic hydrogen produced during the reactions (2) and (3), making steel weak and brittle, causing premature rupture.

The following reactions may occur at the anode mesh:



The lower the pH level of the electrolyte, the greater the formation of chlorine is favoured (7). At high pH levels, water oxidation occurs preferentially (5).

Moreover, the following chemical reactions may additionally occur:



Relative to the ionic motion during the process, and the extraction itself, it appears that the ions move towards the opposite pole under the influence of the applied electrical field in the concrete pores structure. This ionic motion occurs according to the laws of electrolyte mass transport, based on three components, migration, diffusion, and convection, which can be described by the Nernst-Planck equation [19-24].

It is generally accepted that the ECE technique has a high potential for treatment of structures contaminated with chloride ions. There are still some aspects under study in order to improve the understanding of the process itself, the consequences for the structure and to increase the process efficiency. Thus, the first objective of this study is to contribute to the understanding of this technique from a practical perspective.

The second objective is to evaluate the contribution of monitoring techniques already applied to the degradation of concrete structures, such as ionic conductivity, to gather relevant information on the progress of ECE that may allow real time control of the treatment process.

ECE involves movement of different ion species, DC concrete conductivity (resistivity) measurements during the desalination process that is directly proportional to ion concentrations present at different covering layer depths, may be used to understand the process itself and to give practical information about the proceedings being undertaken in laboratory and work sites.

There are some studies that use embedded sensors in the structure, namely, chloride ion sensitive electrodes [28], to obtain information throughout the process and those which use the measurement of concrete resistivity as a technique for monitoring the process [10-25].

2. Experimental

(a) ECE testing

Galvanostatic ECE laboratory tests were conducted on prismatic samples of reinforced concrete, whose covering layer was contaminated with 5% chloride ions by mass of cement, for 6 weeks. Table 1 presents the parameters used in three typical tests.

Table 1. ECE testing parameters

| Parameter | Test I | Test II | Test III |
|-----------------------------|-----------|---------|----------|
| Concrete type | A | B | B |
| Anode type | 316L mesh | Ti mesh | Ti mesh |
| Current (A/m ²) | 1 | 1 | 2 |

Dimensions of the concrete specimens used were $50 \times 30 \times 10\text{cm}^3$, with reinforced squared mesh of $15 \times 15\text{cm}$ of 8mm steel located at 50mm of probe surface. Two types of concrete were used: an intermediate and common quality concrete used to construct common structures, hereafter named Type A concrete, and a second superior quality concrete used in chemically more aggressive environments (EQ1) C32/40, hereafter named Type B concrete. The concrete compositions of each type are given, respectively, in Tables 2 and 3. The concrete was prepared in the laboratory by using a small cement mixer.

Table 2. Composition of intermediate quality concrete (Type A)

| Composition | (kg/m ³) |
|-------------|----------------------|
| Sand | 624 |
| Gravel 1 | 484 |
| Gravel 2 | 727 |
| Cement (c) | 367 |
| Water (a) | 180 |
| w/c Ratio | 0.51 |

Table 3. Composition of superior quality concrete (Type B)

| Composition | (kg/m ³) |
|----------------|----------------------|
| Gravel (10/20) | 580 |
| Gravel (5/10) | 590 |
| Sand (0/4) | 515 |
| Sand (0/2) | 200 |
| Cement | 280 |
| Additives | 100 |
| Water | 170 |
| Adjuvant | 3.42 |

Samples were prepared by using a uniform concrete covering layer (5cm), contaminated with calcium chloride (Merck, pro analysis) to obtain a content of 3 to 4% chloride by mass of cement, corresponding to about 0.4% of the total mass. After preparation, the specimens were cured for periods of 28 days, after which ECE experiments were done. Two types of anode were used: stainless steel AISI 316L and activated titanium coated with a mixture metal oxides (Ti-MMO), supplied by *Corrpro* (see Figure 2), which met the standards recommended by NACE. The mesh was applied to the entire outer test surface of the concrete specimen.

The process was conducted by immersion test specimens in aqueous solutions saturated with calcium hydroxide and applying constant currents of 1 or 2A/m², using stainless steel and titanium coated with

Ti-MMO anodes. The galvanostatic process was implemented by using a continuous power supply of Kioto model PS230230 and two multimeters to monitor the voltage and current, both Kioto, model KT-1992HZ.

Saturated with calcium hydroxide ($\text{Ca}(\text{OH})_2$) used as electrolyte solution were prepared from Merck *pro analysis* calcium hydroxide powder. The high pH level of these solutions allows only a low release of chlorine gas. In fact, the gas evolution with a characteristic chlorine odour was observed.

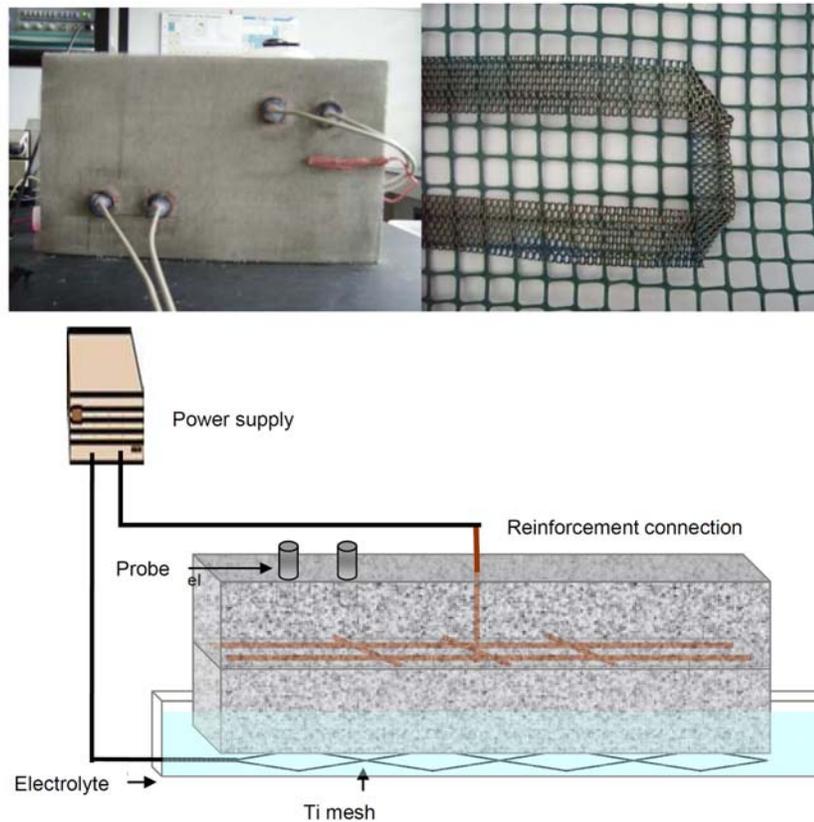


Figure 2. Test specimen and Ti mesh.

To monitor ECE progress, concrete samples were collected during the test period, by drilling of the specimen samples, and analyzing the powder for total chloride content at depths of 1.5cm and 3cm, according to

the specification LNEC 231-1981E (Cement-Determination of halides), which applies the Volhard method to 2g concrete samples, after drying in an oven at 100°C for 1 hour, and subject to an initial attack with nitric acid. Subsequently, an excess of standard silver nitrate solution is added and the excess silver ion is back titrated with standard potassium thiocyanate in the presence of iron III as an indicator.

The potential of the steel reinforcement was measured throughout the testing period with the aid of an external reference Ag/AgCl electrode (Hanna).

(b) Measurement of concrete ionic conductivity

The ionic conductivity of the covering layer concrete was measured at two depths by using the two pins technique. In this technique, the concrete resistivity is determined through the solution resistance, R , by dividing the value of the AC voltage applied between two similar electrodes by the respective alternating current intensity response. To reach the conductivity value, ρ , the “cell parameter” must be known. The cell constant is influenced by the distance between the electrodes, L , and their areas, A , and depends also on the applied electric field. The relation between resistivity, electrolyte resistance, and geometric parameter of the cell is expressed through the following Equation (10):

$$R = \rho(L/A). \quad (10)$$

The probe system used to determine the conductivity is shown schematically in Figure 3. The system consists of two stainless steel 316L ring probes with 5mm thickness placed at two different levels of measurement separated 10mm from each other by a high-density polyethylene (HDPE) ring. Electrical connections of probe to the feeding (signal generator) and reading (multimeter) equipments are protected inside of epoxy resin block.

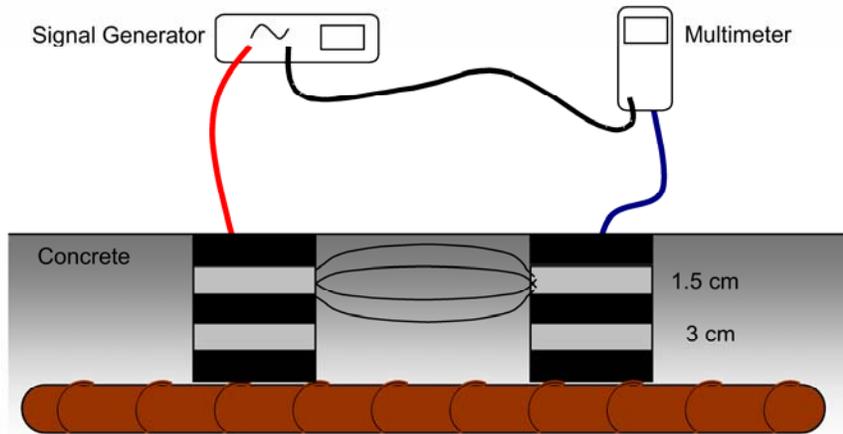


Figure 3. Schematic representation of the system for the measurement of concrete ionic conductivity.

The two system probes were placed in the concrete covering layer with a separation of 5cm by boring using a drill with a plug end-type TE-C-GB 30/45-11/8 with a diameter close to that of the probes. A cement grout was used to ensure a perfect seal of the probe holes.

The measurement was performed by applying a sinusoidal wave current, with a frequency of 108Hz using a Tektronix signal generator model TDS 1002, between the SS rings at the same depth and measuring the AC potential difference between the same rings.

The cell constant of the probe system was determined by using an aqueous calcium hydroxide solution, prepared from pro-analysis powder (Riedel-de Häen), with concentration values of 0.001, 0.01, 0.1 mol·dm⁻³ and measuring conductivity with the use of a WTW conductimeter, model LF 330/SET. A probe system was placed 5cm apart from each other in a closed 31 × 23 × 16.5cm³ container and similar tests were carried out to those performed with concrete samples. A cell constant of 0.3cm⁻¹ for the sensors installed at the depth of 1.5cm and of 0.25cm⁻¹ for the sensors installed at the depth of 3cm were taken in this work (cf. Figure 3).

3. Results and Discussion

(a) ECE process

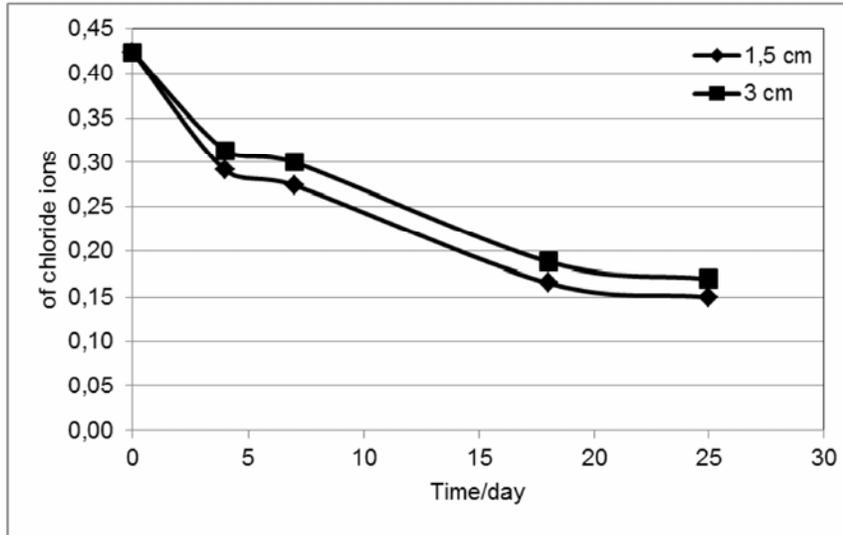
Figure 4 shows the variation of chloride ion total concentration for each of the three tests (cf. Table 1) and for each of the two depths: 1.5cm and 3cm, during the test period.

Results show, first of all, a significant influence of concrete type on the ECE speed. Variation of chloride ion concentrations with time in experiments I and II, show chloride extraction is faster in the less compact and more porous concrete Type A, where a reduction to half of the initial content of chloride was achieved in about 10 days as opposed to 20 days needed to achieve the same reduction with the higher quality concrete Type B. Higher concrete porosity decreases tortuosity facilitating the mass transfer processes.

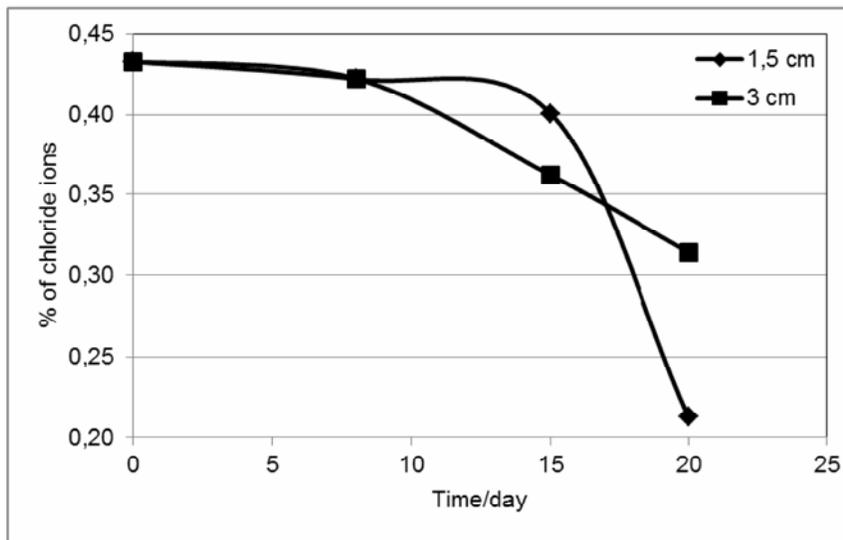
As expected, the extraction rate increases along with the current applied (cf. Figures 4(b) and 4(c)). These results show that the extraction process is about three times faster when the process current is doubled, using the same type of concrete.

Another relevant aspect observed in the results is the fact that there is a tendency to reach an extraction limit (cf. Figures 4 and 6) that lays somewhere around 0.15% of chloride ions. These observations are consistent with the fact that during the ECE process free ions, i.e., dissolved ions in the electrolyte pore solutions, are those that are extracted while chloride ions that are chemically bound to the cement and aggregates remain within the concrete [26, 27].

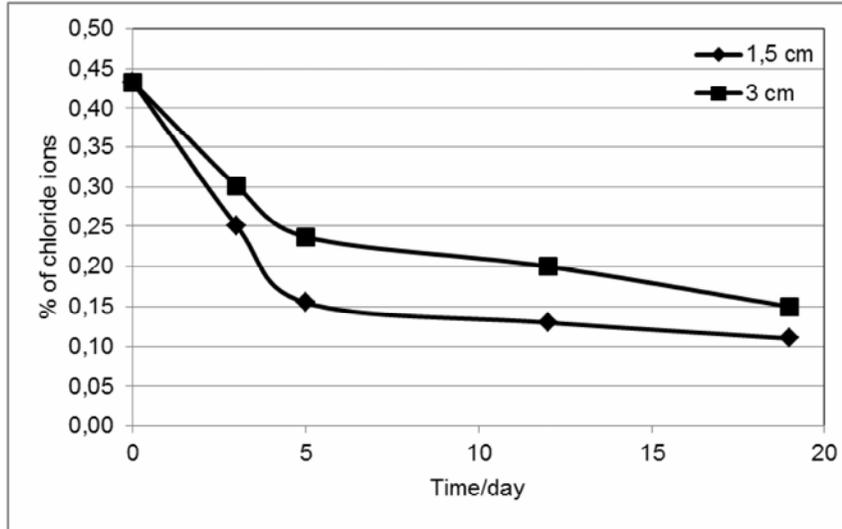
Admitting that there is a chemical equilibrium between the chloride ion solution within the pores and chemically bounded ions, extending the ECE process would be only efficient, if there is space for the system to evolve back to a balanced state.



(a)



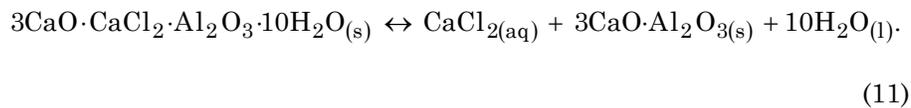
(b)



(C)

Figure 4. Chloride ion concentration variation: (a) Test I (Type A concrete, $1A/m^2$ current, stainless steel 316L mesh anode); (b) Test II (Type B concrete, $1A/m^2$ current, Ti mesh anode); and (c) Test III (Type B concrete, $2A/m^2$ current, Ti mesh anode).

In fact, the chloride ions within the concrete, may be “free”, i.e., dissolved in the pores electrolyte solution, or “connected”, i.e., chemically bonded to the cement or aggregates materials. The ions that are extracted during ECE process are these free ions, dissolved in the electrolyte pore solutions that can move. However, a balance between “free” ions and “connected” ions has been proposed by several authors and 331 eventually involves the following reaction [26, 27]:



Since chloride ion extraction is a faster process than reaction (11), there is always a part of the chloride ions that are not removed because

they are chemically bounded. When ECE process is interrupted the balance tends to be restored and “free” chloride ions are generated in aqueous solution, and therefore, it can still be harmful to the structure. In this sense, it was proposed, application of several ECE cycles with a relatively long stoppage time in order to restore the balance between chloride ions in the solution and bounded ions to increase the chloride extraction percentage in the structure [28].

The analysis also allows another relevant observation: The chloride ion concentration tends to decrease at a higher speed and stay at lower values at depths closer to the surface. Despite beginning with a uniform chloride ion concentration across the concrete covering layer, as the ECE process unfolds, concentration profiles start to build up within the same layer maintaining higher concentration levels near the reinforcement. This fact is justified considering that near the reinforcement hydroxyl ions are formed through anodic reactions (1) and/or (2), and have a mobility 2.5 times higher than the chloride ions, which causes the current to be progressively carried in a higher fraction by this ion at the expense of chloride ion retardation. On the other hand, in more remote levels of the reinforcement, closer to the concrete’s outer surface, the number of chloride ions transported tends to decrease gradually thus promoting greater ion extraction.

This observation could be a drawback to the ECE process, because near the rebars there will always be conditions for a build-up of high chloride ion concentrations making it impossible to actually reduce the reinforcement corrosion (pitting corrosion). Thus, it would be more practical to apply the process to structures, where the chlorination threshold has still not been reached at the reinforcement bars, as suggested by some authors [18] or to apply very high currents at the early stages of the an ECE to ensure an effective removal of chloride ions from the reinforcement neighbourhood. However, the latter solution may cause problems of hydrogen release, weakening of the steel and concrete/ reinforcement bond [8].

Thus, the problem raised by several authors about continuation of corrosion after applying the ECE process [15-18], has eventually, as a first explanation, the formation of a very alkaline environment near the reinforcement causing steel alkaline depassivation, but can also be caused by the build-up of high chloride ion concentration near the rebars due to an incapacity to extract these ions through the ECE at low currents, continuing the active corrosion. After stopping the ECE process, the chloride ion concentration has a tendency to homogenize by diffusion processes. Despite the relative slowness of this process, an ECE can be effective, as evidenced by the significant number of work sites, where the ECE process has been applied with clear positive results in medium and long term periods [4].

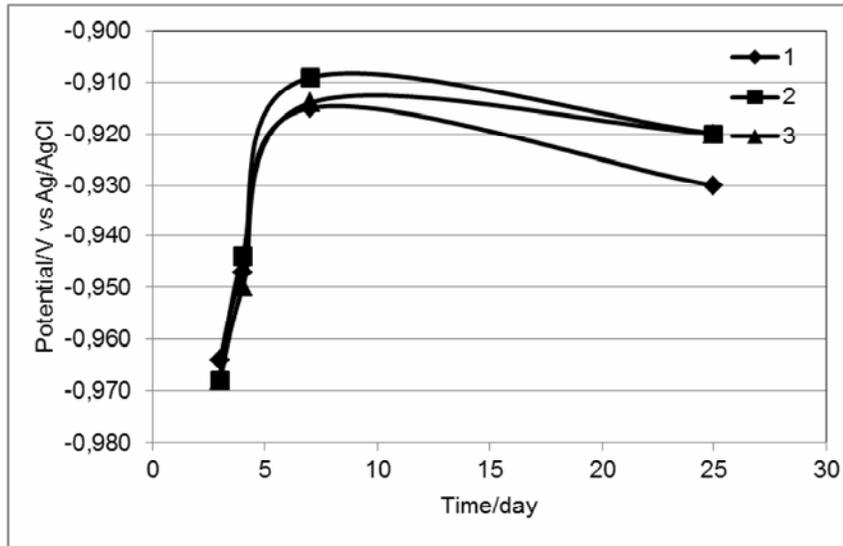
Evidence that repassivation does not happen, and therefore corrosion does proceed, even after ECE application, is illustrated in Table 4 by the values of the potential of steel reinforcement read after stopping ECE current injection and its total depolarization of the reinforcement.

Table 4. Reinforcement steel potential after stopping the ECE application

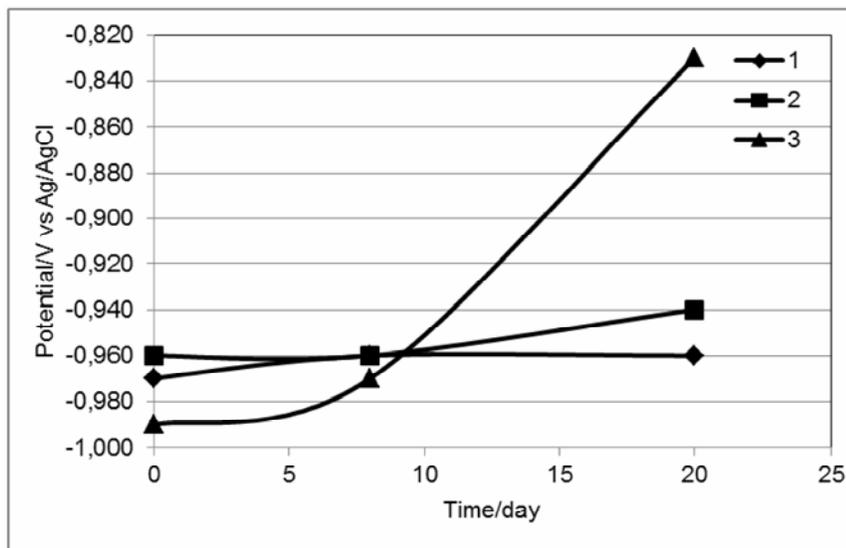
| Test | Potential (mV vs Ag/AgCl) |
|------|---------------------------|
| I | - 300 |
| II | - 367 |
| III | - 420 |

Figure 5 shows values of the potential of steel reinforcement *versus* the Ag/AgCl reference electrode, measured at the same three surface points, during the ECE application, and taken from the surface not immersed. Since hydrogen release reaction (1) only occurs for electrochemical potential lower than -1000mV vs. Ag/AgCl (about -1100mV vs. Cu/CuSO_4), measured potential values lay always in potential regions, where there is no hydrogen release. Therefore, we may consider that the main cathodic reaction was that described by Equation (2).

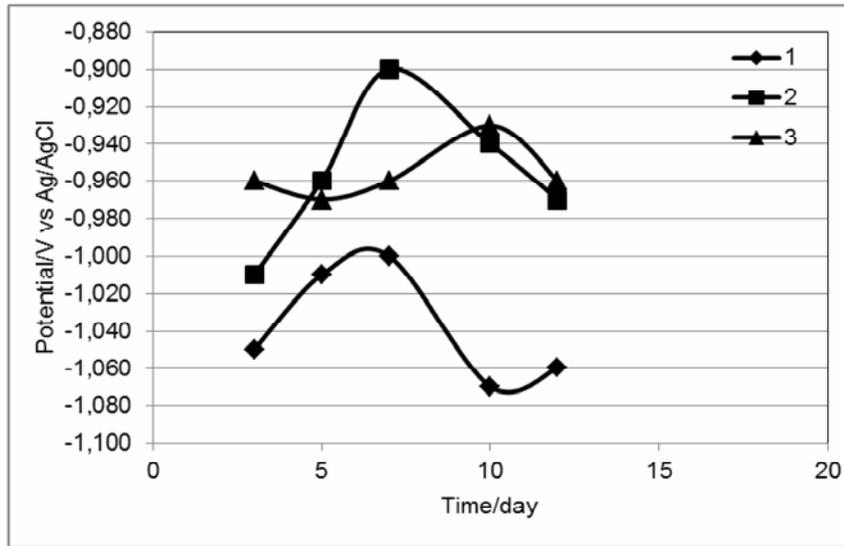
One last observation worth mention is that, there was no significant differences in the results for ECE performance by using 316L SS or titanium mesh, except that Ti mesh showed higher durability than the steel 316L mesh. In fact, a clear corrosion process on the 316L SS surface was observed.



(a)



(b)

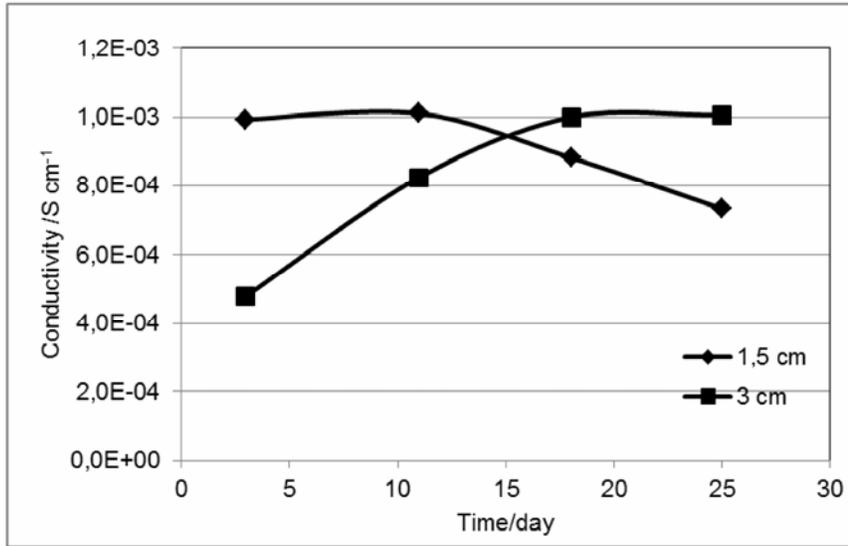


(c)

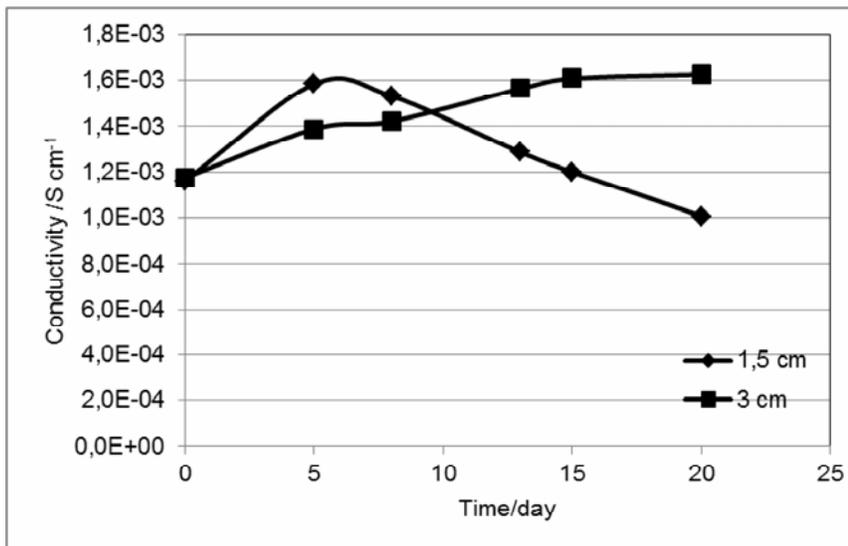
Figure 5. Variation of the steel reinforcement potential at three surface points during testing period: (a) Test I (Type A concrete, $1\text{A}/\text{m}^2$ current, stainless steel 316L mesh anode); (b) Test II (Type B concrete, $1\text{A}/\text{m}^2$ current, Ti mesh anode); and (c) Test III (Type B concrete, $2\text{A}/\text{m}^2$ current, Ti mesh anode).

(b) Monitoring of concrete ionic conductivity

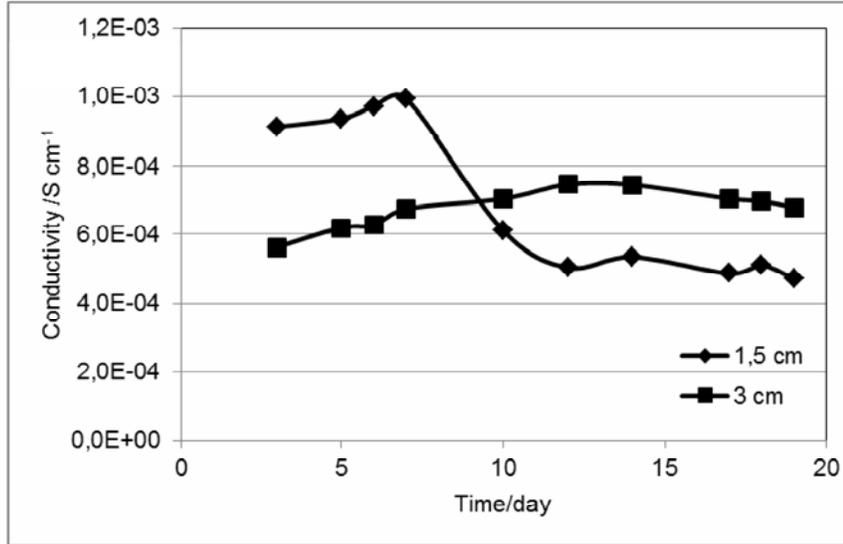
Figure 6 shows the variation of the concrete ionic conductivity during the test period for each of the three specimens (cf. Table 1) and for the two depths of 1.5cm and 3cm.



(a)



(b)



(c)

Figure 6. Variation of the concrete conductivity at two depths during testing period: (a) Test I (Type A concrete, $1\text{A}/\text{m}^2$ current, stainless steel 316L mesh anode); (b) Test II (Type B concrete, $1\text{A}/\text{m}^2$ current, Ti mesh anode); and (c) Test III (Type B concrete, $2\text{A}/\text{m}^2$ current, Ti mesh anode).

The first observation that can be made is the relatively high concrete conductivity (low resistivity) presented by all the probes, which can be justified by the presence of very high chloride ion contamination levels.

The results show differences of the ionic conductivity, depending on depth: the conductivity tends to increase during the ECE process at inner layers of the concrete covering layer and to decrease at the outer layers. These observations are consistent with the fact that at layers near the reinforcement hydroxyl ions, with high ionic mobility (λ_i) (cf. Table 5), are formed due to the cathodic reactions, increasing the ionic concentrations and conductivity, during the process. As opposed to the outer layers, where there is an overall decrease in the conductivity

during the process due to the successive removal of chloride ions and slower input of ions from outside, since this is a diffusion process, compared with migration in an electric field. Moreover, in order to maintain the electro neutrality, positive ions must also leave the concrete. Thus, conductivity also tends to decrease. All experiments show, however, that at layers closer to the surface (1.5cm), there is an initial trend for increase in ionic conductivity resulting from an initial increment of chloride ions coming from inner layers.

Table 5. Ion mobility some ions with interests for the ECE process under analysis [19]

| Ion | $\lambda_i, \text{S}\cdot\text{m}^2/\text{mol}$ |
|-----------------|---|
| H ⁺ | 0.03496 |
| Li ⁺ | 0.00387 |
| Na ⁺ | 0.00501 |
| K ⁺ | 0.00735 |
| Ca ⁺ | 0.01190 |
| OH ⁻ | 0.01991 |
| Cl ⁻ | 0.00764 |

The justification given earlier becomes even more evident by comparing ionic conductivity results with chloride ion concentration variation during the testing period. This comparison shows clearly that ionic conductivity variations follow closely the ECE process speed variations. For instance, results for probe at 1.5cm, show that the period of overall increase in concrete conductivity coincide precisely with that of higher chloride ion extraction speed and efficiency. After this initial increasing period, conductivity tends to decrease, probably due to lack of chloride ions and hydroxyl ions, which start to migrate outwards, but not yet in considerable concentrations.

Results on the conductivity (resistivity) variation through the concrete covering layer during ECE presented in this work are coherent with those observed by other authors [9, 25, 26]: large variations in the early stages of the ECE followed by a plateau or a decrease, depending on several factors, including the depth at which conductivity is measured. In the early stages of the process, considering the high concentration of chloride ions and its mobility (cf. Table 5), a significant part of the current is carried precisely by these ions, but as chloride ions are extracted and constant current is maintained (galvanostatic process), other ions become more important as charge carriers, including hydroxyl ions that are produced at the cathode. Thus, the chloride ion extraction efficiency, defined as the ratio of current carried by chloride ions to the total current will decrease as the chloride ion concentration decreases.

Figure 7 shows a schematic representation of the proposed model. To simplify it, we assume three regions in the covering layer. Zone I, near the concrete surface, where the chloride ion concentration decreases rapidly to a relatively constant value, and where the current transmission is ensured essentially by the chloride ions. The conductivity in this area presents a constant decrease.

Zone II, an intermediate zone, where at an early stage and during the bulk of the ECE process, an increase of concrete ionic conductivity is observed. It works as a path zone for chloride ions, which maintains a relatively constant concentration of chloride ions, but where simultaneously the concentration of other ionic species, including hydroxyl ions, increases. Similarly to zone I, after an initial increase conductivity tends to decrease after most of the chloride ions have been extracted, which implies a significant decrease in ECE speed.

Zone III, near the reinforcement bars, where the concrete ionic conductivity grows at the expense of hydroxyl ions formed by cathodic reactions. Since these ions possess a high mobility, they are the main responsible for current transmission.

This model may provide a possibility to determine the duration and to monitor the progress of ECE process through the continuous measurement of concrete ionic conductivity at the intermediate zone (zone II) of the covering layer, where there is increasing/decreasing change of this parameter. In fact, when the ionic conductivity in this intermediate zone begins its decline, it is inefficient to continue ECE process for much longer.

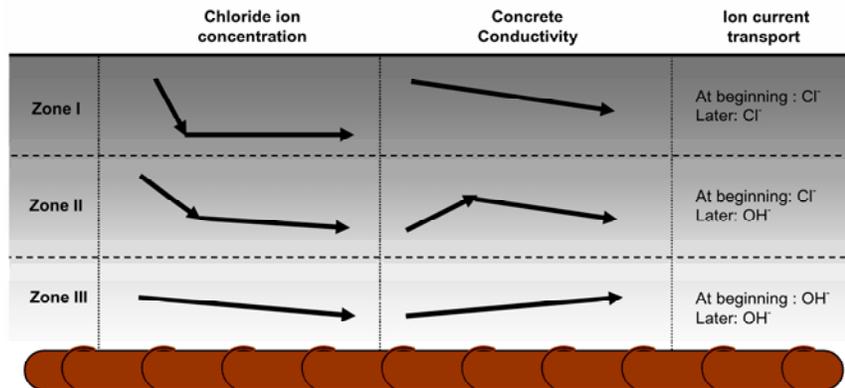


Figure 7. Schematic representation of the concrete ionic conductivity variations, in different regions of concrete covering layer during the ECE process.

4. Conclusion

The following conclusions can be made:

- ECE is a good process for rehabilitation of structures that are not yet in very advanced stages of chloride contamination and do not show high corrosion rates of reinforcements, having clear advantages in terms of cost and ease of application compared to other rehabilitation processes.
- It is likely that the absence of steel repassivation is not due to relatively high concentration of chloride ions remaining near the reinforcement, but results from the high concentrations of hydroxyl ions formed there by the cathodic reactions, which tend to be the main current carriers.

- The ECE process tends to be more effective at high current inputs.
- Monitoring the concrete ionic conductivity during the ECE process, with the help of probes embedded in the concrete at different covering depths, and in particular at the intermediate layer, can be a useful way to follow the process in a simple and economic manner.

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