



# Chloride Electroremediation in reinforced structures: preliminary electrochemical tests to detect the steel repassivation during the treatment



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## ARTICLE INFO

### Article history:

Received 14 January 2015

Received in revised form 1 June 2015

Accepted 1 June 2015

Available online 3 June 2015

### Keywords:

Electroremediation

Electrochemical Chloride Extraction

Nondestructive techniques

## ABSTRACT

Application of electroremediation techniques in concrete is being increasingly used not only to remove different harmful pollutants from the cement matrix, but also for the reduction of different substances that provoke undesirable reactions in the material, as could be chlorides inducing corrosion or efflorescence in construction materials. Electrochemical chloride extraction (ECE) in concrete has aroused great interest in the understanding of the mechanisms involved in this type of techniques and has been massively used in laboratory experiments. However, there is an important need to develop new techniques to detect the exact moment at which the rebar is repassivated, or in other words, the moment at which the treatment has been finished. The usual methods to control the electroremediation efficiency involve extraction and chemical analysis (chloride ions) of cores from the structure. Electrochemical techniques applied to the corrosion rate quantification,  $I_{\text{corr}}$ , by the Polarization Resistance technique,  $R_p$ , are the suggested nondestructive indicators of the efficiency of the technique at the end of the treatment, but it is not possible to use these parameters as passivity indicators during the treatment. In this work, the ECE treatment has been monitored through different electrochemical techniques: accumulated electric charge density ( $\sigma$ ), instant off potentials ( $E_{\text{off}}$ ), polarized and depolarized potentials ( $E_{\text{pol}}$ ,  $E_{\text{dep}}$ ), and Polarization Resistance measurements ( $R_p$ ) registered during the treatment (current off and current on), electrolyte resistivity measurements ( $R_x$ ), and electrochemical impedance spectroscopy (EIS), comparing the on-site results with the final results obtained after destructively analyzing the results of the ECE treatment. From these preliminary results, a resistivity index  $R_x/R_{\text{initial}}$  in which initial resistivity ( $R_{\text{initial}}$ ) and resistivity values taken along the treatment ( $R_x$ ) are considered to evaluate the treatment progression. Apart from this, the polarized steel potentials seem to postulate as feasible convenient indicators of the repassivation of the steels.

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## 1. INTRODUCTION

The possibility to remove chloride ions from concrete by an electroremediation process was first studied in the USA in the 1970s [1]. Then, Electrochemical Chloride Extraction, ECE (also called chloride removal or desalination), was patented by the Norwegian company Noteby in 1986 [2], using a water-retaining substance, such as paper fiber pulp or retarded shotcrete, wetted

with calcium hydroxide or tap water. Other developed variations were also patented [3–5]. Considerably lower voltages, preferably less than 30 V, are used in ECE. Not until the beginning of the 1990s, research reports were published on this method [6–25]. So, the electroremediation applied to chloride extraction using impressed current has been used for over 30 years on a relatively large scale with good success [17,25–27], even though some side effects have been detected [28,29]. These side effects were related with the accumulation of alkali ions around the steel, locally increasing the pH, that can provoke that potentially reactive aggregates may become reactive (alkali silica reactions, ASR) as a result of increased hydroxyl ion content arising from electrochemical treatment. Some authors also generated controversy about the efficiency of the treatment and its ability to repassivate the embedded steel [30,31].

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Because the original concrete surface is left unchanged after the treatment, ECE may be particularly suited for structures with special architectural values such as monuments.

For ECE, a direct current is applied between the reinforcement (cathode) and an anode that is placed temporarily on the outer surface of the concrete. The anode is preferably an activated titanium wire mesh, but recently some attempts have been tested using conductive mortars with carbon fibers with good results [32,33].

Titanium wire mesh anodes have several properties that are important to ensure the treatment such as very low power consumption per unit of product, low maintenance cost, high strength, flexibility to operate cells at very high current density, and resistance to adverse environments and thus no corrosion.

The anode is surrounded by an electrolyte, which is an aqueous solution in its basic form. Chloride ions migrate from the reinforcement to the anode. Due to a relatively high current density of 1–2 A/m<sup>2</sup>, relatively large amounts of chloride can be removed from the concrete within a relatively short time, usually 6–10 weeks. After that, the anode, the electrolyte, and the incorporated chloride ions are removed from the structure.

ECE is an “all-over” method for protecting reinforcement in concrete. It does not harm reinforcement that is not corroding. So, in general a complete surface will be treated. Only in special cases, the treatment will be limited to specifically corroding areas. The design of chloride extraction should be based on the amount and distribution of reinforcing steel and on the chloride profile present [34–36]. Using this information, the anode system, the process parameters, and the testing procedure for final acceptance are chosen.

ECE is normally based on impressed current, but some trials using sacrificial anodes have been proposed [37]. No power source is needed, but the treatment time is longer than that with impressed current.

Therefore, the main objective with this treatment is not only to lower the corrosion rate by shifting the polarized potential, but also to use the secondary effect of the current to repel ions of the same sign [6,8,13,21]. It is implicitly assumed that this repulsion will provoke the lowering of the corrosion rate by the simple disappearance of the chlorides. Other complementary electrochemical treatments can also be applied, injecting inhibitor from the anolyte to the rebar [38].

After the first studies that appeared in the 1990s in which the reduction in the chlorides content was the most relevant result [6,9,25], other authors proposed that apart from the chloride reduction, another important effect was the repassivation of the steel [15,39,40]. In fact, simultaneously to the rejection of chlorides, a build-up of hydroxide ions results not only due to the cathodic reduction of oxygen (as in the case of cathodic protection) but also by electrolysis of water:  $2\text{H}_2\text{O} + \text{e}^- \rightarrow 2\text{OH}^- + \text{H}_2$ , with the evolution of hydrogen gas.

Different authors have tried to model the treatment and the whole process [8,10,18,19,21,39,41–44], but it has not been still possible to find accurate model. Literature data from research and applications indicate that about 50% of the total chloride content can be removed [17,45]. It is thought that this percentage represents the free chlorides present in the pore solution.

The first version of the European standard related with the correct application of ECE treatment has been published recently [46]. The design current density is typically in the range of 0.5–2 A/m<sup>2</sup> of concrete surface area. An upper limit has to be specified to avoid deterioration of the concrete.

During the treatment, a form of monitoring of its progress is necessary. The criteria concern the content of chloride in contact with reinforcing steel. That means drilling cores before and after the treatment; so, it is a destructive method. The total (acid-

soluble) chloride content should preferably be below 0.4% by mass of cement at the end of the treatment [47–49]. On structures with nonhomogeneous chloride distribution, a direct comparison of the total chloride content before and after the treatment is difficult. For electrolyte confined in tanks, monitoring the chloride in the electrolyte is another method [12,15]. The problem is that it is not easy to use this sort of tank to place the anode for its application on site in real reinforcement structures, and the quantification of the removed chloride does not provide reliable information on the passivation of steel.

So, another manner to verify the condition after the treatment is to measure the corrosion potential ( $E_{\text{corr}}$ ) or the corrosion rate ( $I_{\text{corr}}$ ). For the corrosion rate determination, the linear polarization resistance method (LPR, also called  $R_p$ ) needs to be applied. The corrosion potential should recover values similar to those typical of lack of corrosion (more anodic than –350 mV vs. Saturated calomel electrode, SCE) [50], and the corrosion rate measured on site should be lower than 0.1  $\mu\text{A}/\text{cm}^2$  [51]. However, these values are not always achieved some days after the treatment is finished [39]. It must be mentioned that about this application of the polarization resistance after the treatment to determine if the passivation of the steel occurs, some contradictory results have been found [7,24]. Important investigations about short term and long term efficiency of ECE have also been published [52], concluding by the  $R_p$  determination that corrosion rate increases some time after the treatment [53].

There are no methods to measure the advancement of the treatment during its application more than the recording of the charge passed and therefore the only possibility of controlling the progression of the treatments is drilling cores to measure the decrease of the chloride content near the reinforcements. In this way, the main conclusion of the COST (European Cooperation in Science and Technology) Materials Action 521 “Corrosion of steel in reinforced concrete structures,” supported by the European commission, summarizes the state of the art and gives valuable information on preventive measures, monitoring, and maintenance for reinforced concrete structures. About electrochemical chloride removal, its main conclusion was that the current practice and its domain of applicability is known, but ECE can be improved by lowering the costs and finding new criteria for effectiveness using nondestructive methods.

In a previous paper [54], the monitoring of the total electrical charge was considered to standardize the minimum value needed for enough decrease of chlorides, also considering the electrolyte electrical resistance. In this paper, preliminary results on new methods for monitoring the efficiency of the ECE treatments are presented. Conclusions are based not only on different parameters registered during the current disconnections (as instant off potentials or 24 h potential decay, as are analyzed in cathodic protection) but also on the results obtained from nondestructive electrical and electrochemical techniques such as electrochemical impedance spectroscopy measured in polarized conditions.

## 2. EXPERIMENTAL

### 2.1 Specimen fabrication

Mortar specimens (size 7 × 7 × 7 cm) were fabricated and cured in a 100% relative humidity, RH, chamber for 28 days. Two different types of specimens were fabricated by adding NaCl or CaCl<sub>2</sub> (2.5% and 5% Cl<sup>-</sup> by weight of cement, respectively, which imply approximately 0.54–1.08% by weight of sample) in the mixing water to study the effect in the cement matrix pore solution during the ECE.

The mortar dosage was:

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