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Limitations and advantages of electrochemical chloride removal in corroded reinforced concrete structures

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Abstract

Existing experience on electrochemical methods for the rehabilitation of corroded reinforced concrete structures (RCS) shows that chlorides can be removed from these structures and that carbonated concrete can be realkalised. However, the current state of know-how has not advanced sufficiently to dissipate serious doubts about the efficiency of electrochemical chloride removal (ECR) and electrochemical realkalisation (ERA). A fundamental controversy remains to be resolved, which can be summarised in the following question: *Once the causes of corrosion have been removed, does the corrosion of corroded RCS cease?*

The present work attempts to find an experimental response to this question, determining the corrosion rates of corroded rebars embedded in cement mortar slabs with chloride additions, before and after the application of ECR, and of clean and prerusted steel electrodes embedded in small chloride-free mortar specimens. According to the results obtained, if ECR is applied preventively it is an efficient procedure for delaying the start of corrosion. However, if applied too late it does not assure the repassivation of corroded RCS and is therefore useless.

Keywords: Steel; Reinforced concrete; Electrochemical chloride removal; Electrochemical rehabilitation methods; Repassivation

1. Introduction

Current know-how on reinforced concrete structure (RCS) durability indicates:

- The great importance of RCS corrosion in scientific, economic and social terms [1,2].
- The practically unlimited durability of passive RCS [3,4].
- The dramatic reduction in RCS durability when the passive state of the rebars is lost [4–6].
- The causes of loss of the passive state [1–3,7,8].
- The enormous cost of repairing RCS damaged by rebar corrosion [1–3,6].
- The interest, in view of the preceding points, aroused by RCS electrochemical rehabilitation methods: ECR and ERA [9–22].
- The certainty that ECR and ERA are capable of removing, in just a few weeks, the causes of RCS corrosion [13–22].

But the state-of-the-art has not found a fully satisfactory response to the fundamental question: *Once the causes of corrosion have been removed, does the corrosion of corroded RCS cease?*

The present work seeks to find an experimental response to this question by determining the corrosion rates of corroded rebars embedded in large cement mortar slabs with chloride additions before and after the application of ECR. Corrosion rates are also determined for clean and prerusted steel electrodes embedded in small chloride-free mortar specimens with the aim of simulating an ideal situation not achievable in real conditions, in which 100% chloride removal from the structure is achieved.

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Fig. 1. Small cement mortar specimens, without chloride additions, in which clean and prerusted steel electrodes were tested.

2. Experimental materials and methods

2.1. Materials

The study has assessed the behaviour of steel electrodes, in clean condition and with different prerusting grades, embedded in small cement mortar specimens of 8 × 5.5 × 2 cm, as shown in Fig. 1, manufactured with a cement/sand/water ratio of 1/3/0.5. The specimens were always used without chloride ion additions with the aim of guaranteeing the passivation of the clean steel electrodes and, if possible, to achieve the repassivation of the corroded electrodes.

In order to check the effect of the steel prerusting grade on the possibility of passivation, the following types of electrodes were used:

- Clean steel, recently pickled in a 50% hydrochloric acid solution inhibited with 4 g/l of hexamethylen tetramine, thoroughly rinsed in running water and distilled water, and immediately dried with compressed air.

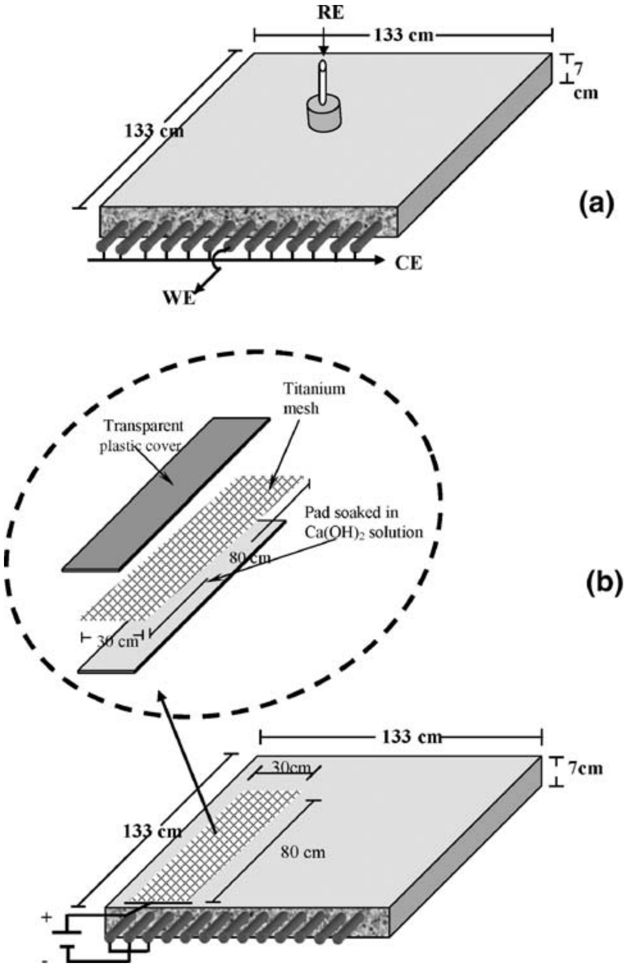
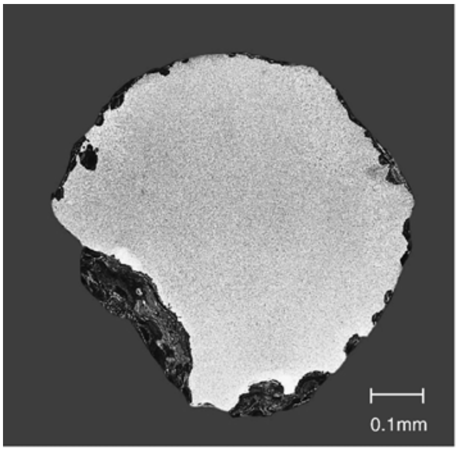
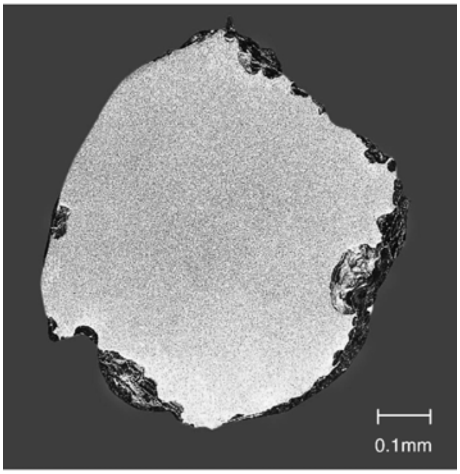


Fig. 2. Schematic representation of the mortar slabs used, showing (top) the situation of the electrodes during R_p measurements and (bottom) the area where chloride removal was performed.



(a) With ECR



(b) Without ECR

Fig. 3. Cross sections of the rebars in the slab with chloride additions: (a) in slab area with ECR, and (b) in slab area without ECR.

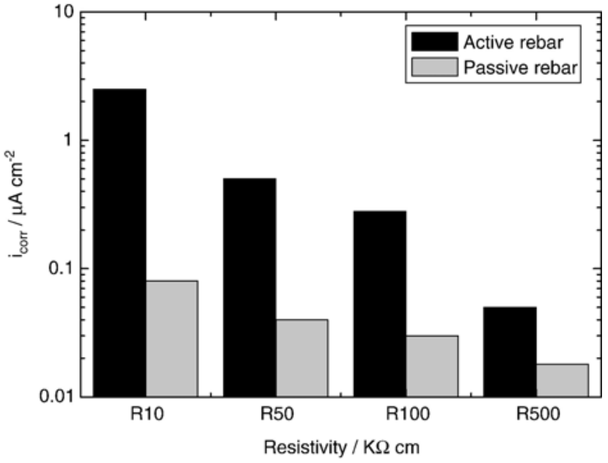


Fig. 4. Influence of concrete resistivity on the corrosion rate of rebars embedded in slabs with chloride additions (active) and without (passive).

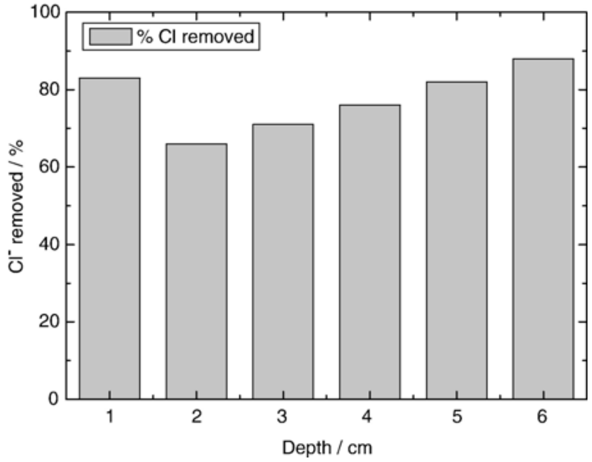


Fig. 6. Percentage of chlorides removed from the slab after applying the procedure on both sides.

- Steel electrodes with different prerusting grades achieved by contaminating their surface with 500 mg of Cl$^-$/m^2 and exposing them for different times, between 1 and 28 days, in a humidity cabinet before trying to repassivate them in the cement mortar.

2.2. Electrochemical chloride removal

For the ECR tests, two cement mortar slabs of 133 × 133 × 7 cm, manufactured more than a decade ago with a cement/sand/water ratio of 1/3/0.5, were used, as schematically represented in Fig. 2. The slabs were usually conserved in a dry laboratory environment, with relative humidity levels of 30–40%, except when they were wetted with frequent additions of water for the performance of previous studies [23–26]. One slab was manufactured without additives, to represent passive reinforcements, and the other with the addition of 3% $CaCl_2$, to simulate the behaviour of active reinforcements. The rebars in the latter were strongly corroded (Fig. 3). Thirteen rebars of 0.8 cm in diameter were embedded in each slab at intervals of 9 cm, crossing the slab from one side to the other. The area where the rebars emerged into the atmosphere was insulated with tape in order to prevent differential aeration phenomena.

Measurements were made of the E_{corr} and the corrosion rate of rebars in both slab types. For the performance of potential measurements a saturated calomel reference electrode (RE) was placed on the surface of the slabs, being situated in a hole in the centre of a stainless steel cylinder in order to assure its stability (Fig. 2). One of the rebars acted as the working electrode (WE) and the two adjacent rebars as counter electrode (CE), in such a way as to achieve a uniform distribution of the electrical signals across the entire surface of the WE. In the area where chlorides had been removed, a CE of 40 cm in length was placed on the surface of the slab for the performance of R_p measurements.

ECR was begun after several weeks of progressive wetting of the slabs, once their behaviour had become stabilised so that the E_{corr} and R_p values no longer suffered significant alterations. ECR was carried out for 6 weeks at a current density of 5 A/m^2

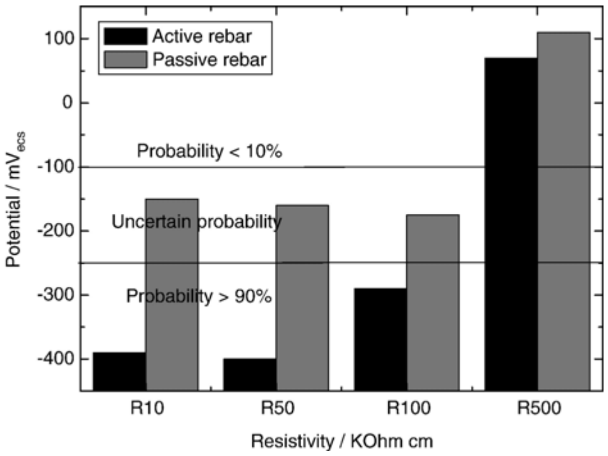


Fig. 5. Dependence of the potentials of active and passive rebars on the concrete's degree of wetness (resistivity).

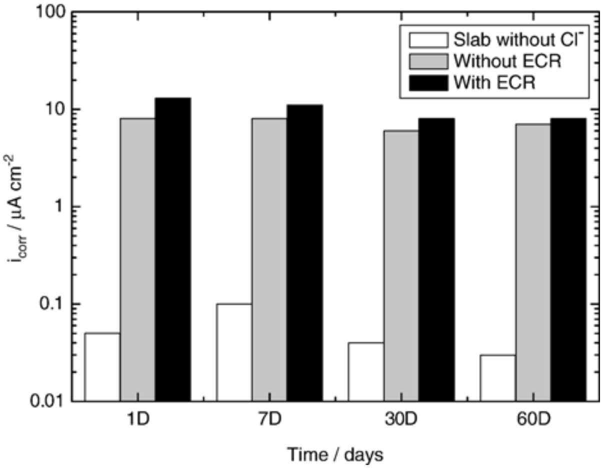


Fig. 7. Comparison of i_{corr} values determined at different times in areas of the slab with chlorides where ECR was performed on both sides and in areas where the procedure was not applied. Results referring to a slab without chloride additions are also included.

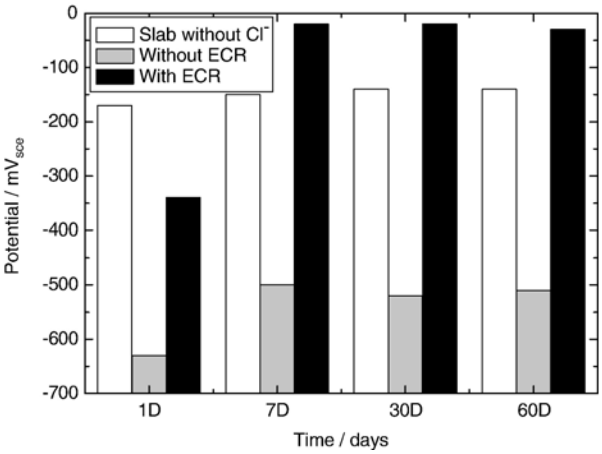


Fig. 8. Comparison of E_{corr} values determined at different times in areas of the slab with chlorides where ECR was performed on both sides and in areas where the procedure was not applied. Results referring to a slab without chloride additions are also included.

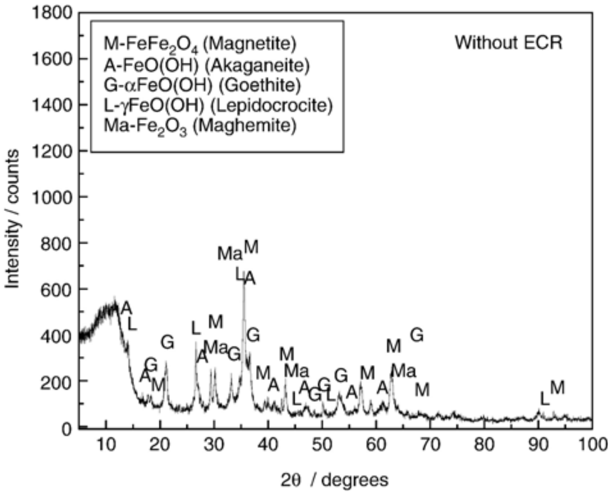


Fig. 10. X-ray diffractogram of a sample of corrosion products on rebars situated in slab areas where ECR was not performed.

in relation with the rebar surface; first on one side of the slab and then on the other. After each removal the evolution of the electrochemical parameters was determined for a time of approximately 2 months. The removal process was performed on a slab area of approximately 30 × 80 cm, short-circuiting 3 rebars to act as the cathode and using a titanium mesh as an external anode, placing between this and the slab surface a pad soaked in $Ca(OH)_2$ saturated solution in order to facilitate the passage of the current. The pad was wetted twice a week and was covered with a transparent impermeable plastic in order to impede the evaporation of the electrolyte. Fig. 2b illustrates the experimental set-up.

2.3. Methods

The evolution in time of the behaviour of the corroded and clean (passive) electrodes was monitored by means of polarisation resistance (R_p) measurements, both on the small specimens (Fig. 1) and on the large mortar slabs (Fig. 2), imposing a potential step of 10 mV and measuring the current after 15 s. This waiting time has been sufficient to achieve an approximate and reliable R_p value (not exact), considering that in a few seconds, or tenths of a second, a quasi stationary response was achieved in potentiostatic measurements on passive or active rebars, respectively [27]. The R_p measurements were taken with a 1286 Solartron potentiostat with positive feedback that automatically compensates for the IR-drop between the WE and RE electrodes. Polarisation curves were also determined, though less frequently, at a potential scanning rate of 1 mV/s. Both procedures were applied before and after ECR and provide a quantitative indication of the corrosion rate, i.e. the efficiency of the electrochemical rehabilitation methods. In addition, periodic controls of the corrosion potential (E_{corr}) were made in an attempt to deduce, in this case qualitatively, whether or not the corroded electrodes have been repassivated.

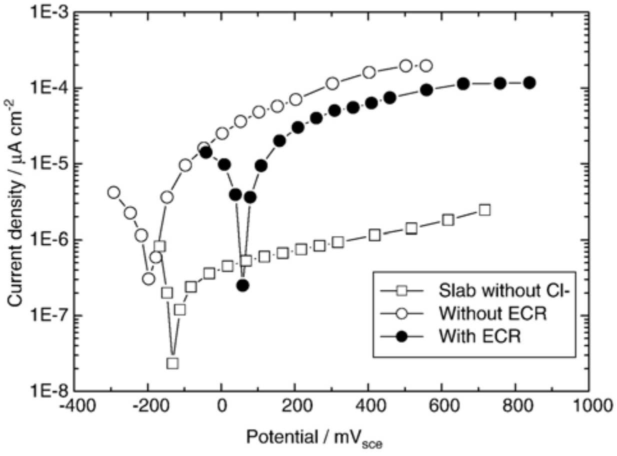


Fig. 9. Polarisation curves obtained in slab areas without ECR and with ECR on both sides. For comparative purposes the polarisation curve for a rebar in the slab without chloride additions is included.

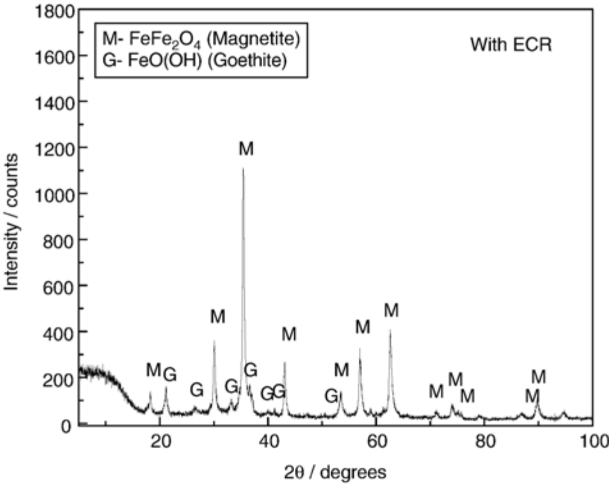


Fig. 11. X-ray diffractogram of a sample of corrosion products on rebars situated in slab areas where ECR was performed on both sides.

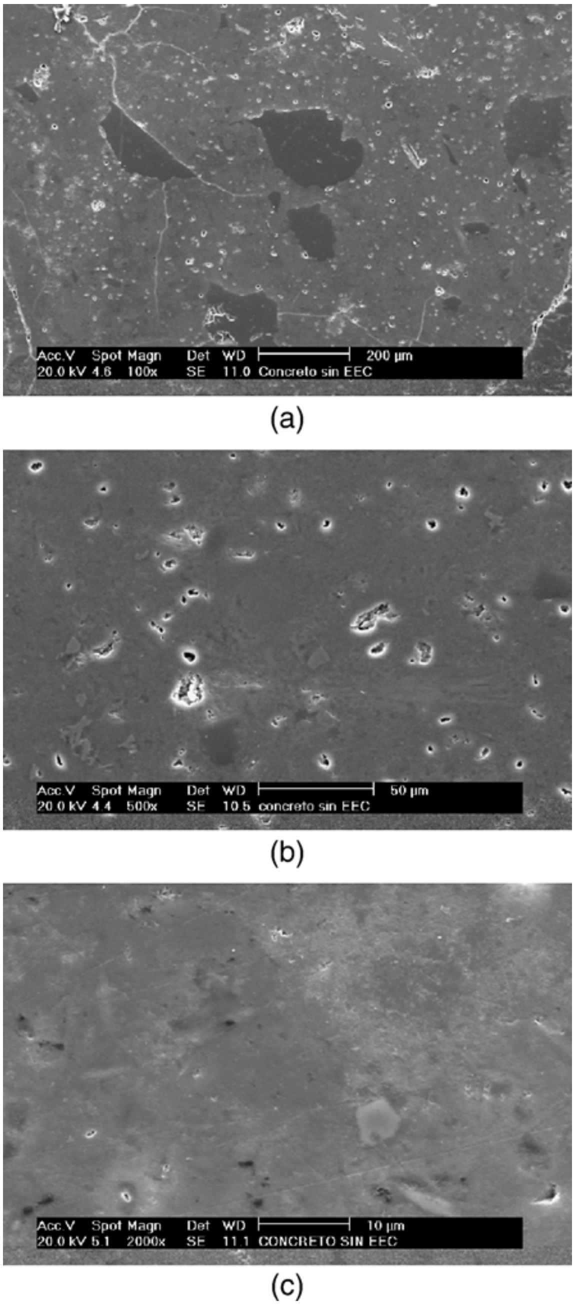


Fig. 12. Micrographs obtained at 100 (a), 500 (b) and 2000 magnifications (c) of concrete in the slab with chloride additions before the application of ECR.

Before and after the removal process, mortar samples were taken (cylinder of 3.2 cm in diameter) from both sides of the slabs in order to determine chloride profiles as a function of depth.

X-ray diffraction was used to analyse corrosion products from the rebar surfaces in areas of the slab where ECR had been performed and in areas where chlorides had not been removed, in order to check for possible modifications derived from the application of the high cathodic potentials used. On the other hand, samples taken from both parts of the slab were observed by SEM in order to identify possible morphological changes caused to the mortar by the passage of the electric current.

3. Experimental results

The tests began with very dry slabs, after years of storage in the indoor laboratory atmosphere, which were progressively wetted before the performance of ECR. Figs. 4 and 5 present data that indicates the great influence of the degree of mortar wetness (resistivity) on the corrosion rate and on the corrosion potential of the rebars, above all when they are in the active state in the slab with chloride additions. The resistivity ρ of the mortar or concrete can be estimated by measuring the ohmic resistance R between a small disc electrode placed on the mortar

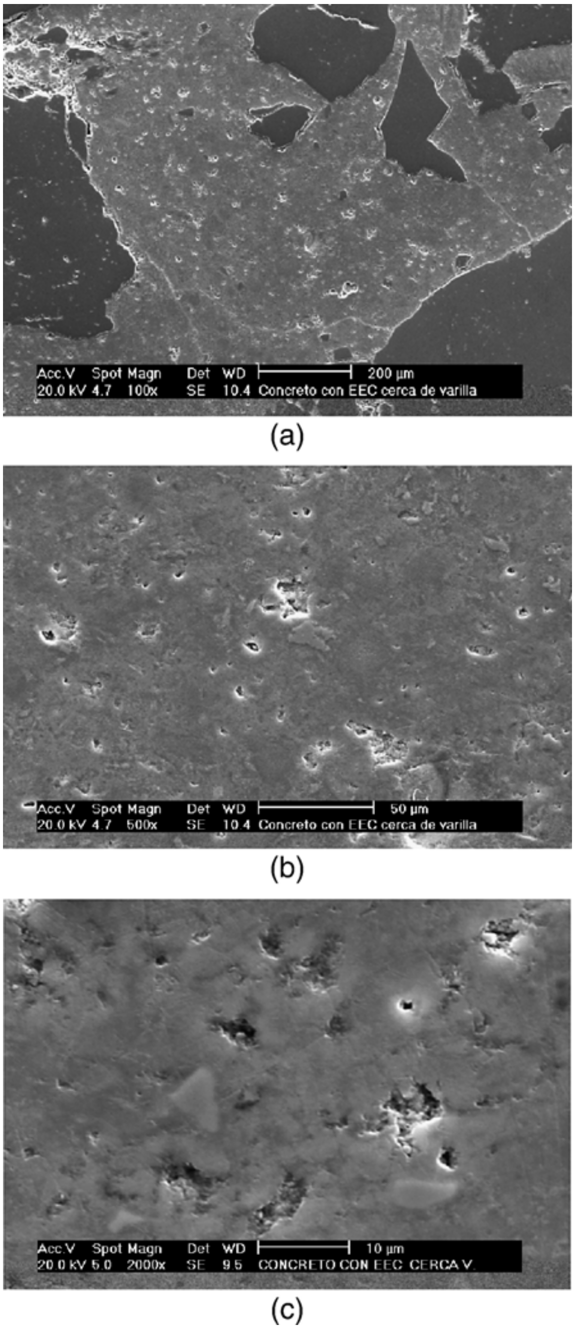


Fig. 13. Micrographs obtained at 100 (a), 500 (b) and 2000 magnifications (c) of concrete in the slab with chloride additions after the application of ECR on both sides.

slab and the lattice of metallic rebars embedded in it, and using the formula:

$$\rho = 2\mathrm{RD}$$

where D is the disc diameter [28]. The behaviour of the active rebars tends to progressively approximate that of the passive rebars as the resistivity of the mortar or concrete grows.

Fig. 6 displays the percentages of chlorides that are removed at different depths in the slab, after applying the procedure on both of its sides. After 6 weeks of removal the remaining chloride contents are below the maximum thresholds allowed by most standards for new structures, 0.4% of the cement weight [29].

The evolution of i_{corr} and E_{corr} in the slab with chlorides is represented in Figs. 7 and 8, respectively, comparing the values obtained in areas with and without chloride removal and relating these with the values corresponding to passive rebars in the slab without chlorides. It is noted that while ECR does not significantly change the rebar corrosion rates, it is however translated into a surprising ennobling of the E_{corr} values.

Fig. 9 compares the polarisation curves of rebars situated in areas of the slab with chlorides where the removal process was applied and in areas not subjected to treatment. For comparative purposes the polarisation curve of one of the passive rebars in the chloride-free slab is included.

The X-ray diffractograms in Figs. 10 and 11 correspond to the corrosion products of rebars situated respectively outside and inside the slab areas where ECR was applied. Peaks are identified in the untreated area which fit the spectra for magnetite ($FeFe_2O_4$), akagenite ($FeO(OH)$), goethite (α-$FeO(OH)$), lepidocrocite (γ-$FeO(OH)$) and maghemite (Fe_2O_3). In contrast, in the general spectrum of the area with ECR, the application of very high negative potentials, up to 40 V, has given rise to changes in the corrosion products, and magnetite peaks are the only significant ones in the spectrum. Nevertheless, the presence of a very small proportion of goethite is also revealed, being attributable to the peaks observed in positions 2 theta: 22.22, 36.649 and 33.24.

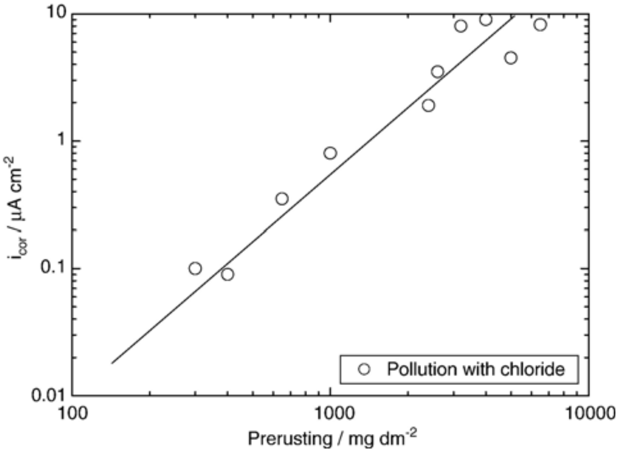


Fig. 14. Relationship between i_{corr} values after 30 days of testing in steel electrodes with different prerusting grades embedded in chloride-free cement mortar specimens with a cement/sand/water ratio of 1/3/0.5.

The micrographs in Fig. 12, at 100, 500 and 2000 magnifications, show aspects of the mortar structure in the slab area where ECR was not applied, and Fig. 13, at the same magnifications, shows morphological details of the mortar affected by the passage of the removal current.

Fig. 14 establishes the relationship that exists between the steel prerusting grade and the i_{corr} values measured one month after embedding the electrodes in the small chloride-free mortar specimens shown in Fig. 1. Despite the dispersion of results, a clear proportionality is seen between the measured i_{corr} values and the prerusting grade of the electrodes.

4. Discussion

The simplest and commonest way of checking the success of ECR consists of determining the depth-concentration profile before and after application of the rehabilitation treatment. It is common to accompany this data with E_{corr} measurements, relating the success of the treatment with significant shifts in this variable towards less negative values, but in general without specifying the conditions in which the measurements were made. This approach may give rise to serious errors in the appreciation of the active or passive state of the rebars, since depending on the resistivity of the concrete, i.e. the degree of wetness of its pore network, E_{corr} values can vary by several hundreds of mV, in the case of both active and passive rebars (Fig. 5).

However, these qualitative checks should not satisfy the researcher, because if they are necessary conditions for the rebars to return to the passive state, they may be insufficient. The fact that chlorides are removed is beyond doubt [13–20], as is proven for instance by the results in Fig. 6; and the ennobling of the potentials is, at times, also beyond doubt (Fig. 8). But curiously, the specialised literature often fails to specify the conditions in which potential measurements are performed, and the results demonstrate that the same structure can be catalogued as passive, doubtful, or active (Fig. 5) simply by imposing a suitable degree of wetness to achieve “appropriate” E_{corr} values. Consequently, potential measurements alone are insufficient and must always be accompanied by some type of quantitative determination of rebar corrosion kinetics, either through the estimation of i_{corr} from polarisation resistance measurements (Figs. 4, 7 and 14); from the obtainment of polarisation curves (Fig. 9); or from the direct measurement of the corrosion process time constant by the application of short duration galvanostatic pulses [22,23], etc.

Electrochemical rehabilitation methods (ERA and ECR) cannot be considered an end in themselves but rather a means of repassivating rebars: the only guarantee of RCS durability. The best way of assessing the efficiency of electrochemical rehabilitation methods, i.e. the perfection of the hypothetical repassivation of the rebars, is to determine the quantitative parameters mentioned in the preceding paragraph. i_{corr} values differ enormously, by around two orders of magnitude, between the active and passive states for high degrees of mortar or concrete wetness, but tend to be more similar and confusable in very dry structures with resistivities of >100 kΩcm (Fig. 4). On the other hand, the plot of the polarisation curves, with very different responses for the active and passive states (Fig. 9), also allows a reliable estimation of the efficiency of the rehabilitation treatment.

The tests performed on the slab with chloride additions reproduce the usual conditions of application of the ECR procedure on real structures, in terms of both the current density used and the duration of the removal process, with the advantage that removal is carried out on both sides of the slab (of the rebars), something that is not possible in most real structures. It is managed to remove 68–88% of the initial chlorides (Fig. 6), reducing the residual content below the traditionally accepted threshold of 0.4% of the cement weight [29]. Nevertheless, i_{corr} values in the slab areas where the method was applied do not differ significantly from those determined in areas not subjected to removal, maintaining a stable value of $\approx 10\ \mu A/cm^2$, characteristic of the active state (Fig. 7). Consequently, the answer to the fundamental question: *Once the causes of corrosion have been removed, does the corrosion of corroded RCS cease?* must necessarily be negative.

Attention is drawn to the great ennobling of E_{corr} values registered as a consequence of ECR (Fig. 8) for which no completely convincing explanation is available. The E_{corr} value is dependent upon many factors, of which the degree of mortar wetness is one of the most important (Fig. 5). This change in E_{corr} is also very probably partly due to the modifications produced in the surface oxyhydroxides by the passage of high cathodic currents during the removal process (Figs. 10 and 11), and above all to the appreciable increase in mortar porosity that takes place in the rehabilitated zones (Figs. 12 and 13). These changes in the E_{corr} are maintained in time (Fig. 8). According to our experience, the ease of oxygen renewal at the metal/corrosive medium interface has a great influence on the E_{corr}, which is much more negative in the absence of oxygen than in well aerated media. Therefore it is explainable that greater porosity facilitates the provision of oxygen and thus the transition to more positive E_{corr} values. In relation with the effect of the ECR on concrete porosity, Siegwart et al. [19] have shown that the pore size and distribution is altered by ECR, with the appearance of a greater number of pores of a smaller size after treatment, while Castellote et al. [21] mention an increase in total porosity caused by the application of an electrical field, as well as changes in the size distribution due to the increase in the range of smaller capillary pores, which they attribute to the dissolution and removal of portlandite and ettringite.

However, although this ennobling is frequently used as a demonstration of the efficiency of electrochemical rehabilitation methods, according to the accumulated experience it does not imply the recovery of the passive state (Figs. 7 and 9).

A situation as favourable for repassivation as that achieved by surrounding the previously corroded electrodes with a new chloride-free cement mortar would never be possible with ECR. In fact, ECR never achieves complete removal (Fig. 6). Nonetheless, the i_{corr} of strongly corroded electrodes can reach and maintain values between 1 and 10 $\mu A/cm^2$, typical of the active state, months after being embedded in the mortar specimens (Fig. 14), demonstrating that the intended repassivation has not taken place. Only in the case of incipient corrosion is it possible to return to corrosion rates typical of the passive state: $\leq 0.1\ \mu A/cm^2$.

This does not mean that ECR is dismissible as a rehabilitation procedure, since it is capable of achieving considerable reductions in the chloride content of contaminated structures (Fig. 6). Therefore it is an excellent preventive method if implemented before the chlorides reach the rebar level in sufficient quantities to trigger the transition of the rebars from passive to active state. Thus the corrosion initiation time can be extended by successive interventions, if necessary, until the service lifetime of the structure envisaged in the design phase is reached without the need for costly repairs. However, strictly speaking, ECR cannot be considered a rehabilitation method, because according to the results of this research it is incapable of restoring deteriorated structures to their original state, i.e. the passive state (Figs. 7, 9 and 14).

The results also prove that a close relationship exists between the prerusting grade and the corrosion rates after rehabilitation treatment. This behaviour would be expectable when the attack kinetics is under cathodic control, resulting in a reduction rate of the oxidising agent in the medium which is proportional to the surface development of corrosion products; something that is characteristic of the active state, since in the passive state the kinetic control is anodic, being exercised by protective films of just a few nm [30].

5. Conclusions

- ECR is capable of removing the cause of corrosion but cannot halt the damage if the process of deterioration is advanced.
- ECR may be considered an appropriate preventive method but, strictly speaking, not a rehabilitation method.
- Chlorides are not necessary to continue corrosion that has already begun.
- ECR cannot be considered an end in itself, but a means to achieve the repassivation of rebars in RCS damaged by corrosion.
- ECR is not capable of repassivating highly corroded steel surfaces.
- The E_{corr} depends on many factors and a significant ennobling of this variable after the application of ECR does not in itself demonstrate the efficiency of the procedure.
- The high current densities required in the practical application of ECR give rise to the reduction of some ferric compounds in the corrosion products to magnetite, increase the porosity of the mortar, and very probably, as a consequence, weaken the bonds between the steel rebars and the concrete.

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