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Cement Based Anode in the Electrochemical Realkalisation of Carbonated Concrete

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A nondestructive technique to revert concrete carbonation is the electrochemical realkalisation (ER) that consists in applying a direct current between the steel bars and an external auxiliary electrode placed on concrete surface with the aim of restoring the alkalinity loss. In the present work is studied the use of a conductive mortar as anode in the electrochemical realkalisation of concrete. Carbonated reinforced concrete specimens with a water/cement ratio of 0.65 were evaluated. ER was applied by 5 day period with a current density of 2 A/m^2 and using as anode a layer of modified mortar (add 25% graphite powder by cement weight) saturated with a 1M Na_2CO_3 solution. The pH in the concrete was determined by phenolphthalein test and potentiometric titration. After the ER treatment the pH value of the carbonated concrete samples had increased from 8 to 12.

Keywords: Electrochemical, Carbonation, Concrete, Conductive mortar, Graphite powder, Realkalisation.

1. INTRODUCTION

In reinforced concrete structures (RCS) generally the corrosion attack is avoided by the high alkalinity of the pore solution. A passive layer on the steel surface provides a suitable corrosion resistance [1]. In urban and industrial environments without presence of chloride sources (marine and deicing salts) the carbonation of concrete is the principal cause of corrosion in RCS. The carbonation process is the chemical reaction in the pore water of dissolved hydration products, mainly calcium hydroxide, with carbon dioxide (CO_2) from the atmosphere reducing the pH of the pore solution from 12.5 to less than 9 [2]. The carbonation process in general is described by reactions (1) and (2).

$$CO_2 + H_2O \rightarrow H_2CO_3 \tag{1}$$

$$H_2CO_3 + Ca(OH)_2 \rightarrow CaCO_3 + 2H_2O \tag{2}$$

When the pH of pore solution is reduced below 11.5 the passive layer on steel surface is broken and the steel corrodes [3]. The steel corrosion mechanism induced by carbonation in reinforced concrete has been studied widely in last years and still is an area of active scientific research [4-8].

The electrochemical rehabilitation methods for reinforced concrete (cathodic protection, CP, electrochemical chloride extraction, ECE, and electrochemical realkalisation, ER) have proved to be an effective option for corrosion control eliminating the causes of corrosion reactions. The Electrochemical Realkalisation (ER) is a nondestructive technique to revert the concrete carbonation and consists in applying a direct current between steel bars and an external auxiliary electrode placed temporarily on concrete surface. The ER stops reinforcement corrosion induced by carbonation increasing the pH around the steel and recovering the passive condition [9]. With the aim of restoring this alkalinity loss that brings the carbonation, a direct current density (1–2 A/m^2) [10] is applied between the steel bars (cathode) and the external auxiliary electrode (anode) of steel or activated titanium immersed in a sodium carbonate solution. The realkalisation of carbonated concrete can be obtained generally in a short period of time (3 to 14 days) [11]. On steel surface the pH is restored by the generation of OH^- which is introduced into the concrete. The initial reaction that takes place on steel surface is the oxygen reduction (3), however the oxygen dissolved in the pore solution is consumed in the first hours of applied the cathodic polarization and reaction (4) takes place.

$$O_2 + 2H_2O + 4e^- \rightarrow 4OH^- \tag{3}$$

$$4H_2O + 4e^- \rightarrow 4OH^- + 2H_2 \tag{4}$$

In the anode the electrochemical reaction depends of material electrode, if material is steel the anodic reaction is the oxidation of iron (5)

$$Fe \rightarrow Fe^{2+} + 2e^- \tag{5}$$

And when the anodic material is electrochemically inert (MMO activated titanium or carbon) the oxidation occurs according to (6) in alkaline electrolytes and (7) in carbonated media. The oxygen evolution is present in both cases [12].

$$4OH^- \rightarrow 2O_2 + 4H^+ + 4e^- \;; \qquad \text{pH} > 10 \qquad (6)$$
$$2H_2O \rightarrow O_2 + 4H^+ + 4e^- \;; \qquad \text{pH} < 10 \qquad (7)$$

Polder et. al.[13] report that in the anode surface the hydroxide consumption is equivalent to acid production, which may dissolve the alkaline components of concrete. The sodium carbonate solution avoids the acidification and destruction of concrete paste stabilizing the pH between 10.5 and 11[14].

Nowadays the development of new anodic systems in electrochemical methods to stop the corrosion on RCS is an area of great interest between corrosion engineers and scientist. Some examples of anodic systems developed include activated titanium mesh, thermal sprayed zinc, conductive polymers, and conductive organic paints. All these systems have disadvantages like high installation cost, high contact resistivity, different thermal expansion coefficient compared to concrete and poor wear resistance [15].

An additional type of anode system includes the use of conductive mortar or cement paste overlay containing carbonaceous materials as secondary electrode. Generally, the electrical conduction in cement paste is through the motion of ions in the pore solution [16,17]. The dry concrete is not a good electricity conductor, the electrical resistivity of dry concrete is in order of 10^4 (dry outdoors) to 10^8 Ohm-cm (oven dry) [18]. In fact it can be considered as a semiconductor material. The difference in electrical resistivity most to water contained in the pore structure of concrete and dissolved hydration products; hence the cement paste is an ionic conductor. An improvement of its electrical properties can be obtained by additions of conductive carbonaceous materials (carbon fibers, coke breeze, and graphite powder) [19], which are electronic conductors. In studies developed on mortars and cement pastes added with carbon fibers and graphite powder Chung D.D.L.[19] reports electrical conductivity values of $1.41\text{x}10^{-1}$ S/m adding to mixture 1% vol. of carbon fiber with 10μm of diameter and $4\text{x}10^{-3}$ S/m (9.18% vol.) for coke powder with particle size <75 μm. Some applications of conductive mortar or cement paste include the electrical grounding of building structures, lightning protection, deicing of bridge deck, roadway monitoring, electromagnetic shielding, strain sensor, conductive floor panels and corrosion control in reinforced concrete structures (CP and ECE).

The use of conductive mortar overlay as anode in electrochemical techniques to corrosion control in reinforced concrete presents certain advantages over the materials that are commonly applied for this techniques. Within the advantages are: lower price in comparison with activated titanium mesh or thermal sprayed zinc, the conductive mortar overlay can be in permanent form on the structure's surface and reused for later electrochemical treatments in case of necessity, another advantage is to obtain an uniform current distribution since the conductive mortar overlay can be applied over its large surface area of the structure with a small covering thickness of conductive mortar overlay.

The idea of a conductive cement based material layer as electrode in electrochemical techniques to attenuate or avoid the corrosion in reinforced concrete structures is not new, in the United States at the beginning of the 70's Stratfull [20] used a conductive layer made with asphalt concrete and coke breeze as anode in impressed current cathodic protection system applied on a bridge deck. Since this work it has been applied and studied the impressed current cathodic protection in concrete structures with conductive mortar anodes, some cases can be found in the literature [21-25]. On the other hand, Perez et al.[26] recently applied with success a cementitious conductive paste layer mixed with graphite powder as auxiliary electrode in the electrochemical chloride extraction (ECE) demonstrating that the use of this material is feasible and the efficiencies obtained are similar to those obtained with the use of MMO activated titanium anode. Based on the prior, this work aims to investigate the use of a conductive mortar layer mixed with coke powder, as anode in ER to increase the pH around the reinforced steel in carbonated concrete.

2. EXPERIMENTAL PROCEDURE

2.1 Specimen Preparation

Prismatic specimens (15 x 30 x 7cm) of reinforced concrete were fabricated. The details of the mixture are presented in Table 1. As reinforcement it was used a corrugated steel rod with 3/8" diameter and partially coated with a non-corrosive paint leaving an exposed area of 259.26cm^2 (Figure 1). The concrete cover thickness is 1.5cm. After its fabrication and before performing the studies the specimens were cured by 28 days at 25°C and RH of 100%.

Table 1. Conductive Mortar and Concrete Mixture

Mixture	Cement CPC-30R*, kg/m^3	H_2O	Ratio w/c	Coarse aggregate kg/m^3	Fine aggregate kg/m^3	Carbonaceous material **
Concrete	300	195	0.65	1080	740	---
Conductive mortar***	1	0.39	---	---	1	25

* According to NMX-C-414-ONNCCE [27]
**Percent of cement (%), Carbonaceous material: coke powder with particle size < 1mm.
*** The mortar mixture appears in cement proportion.

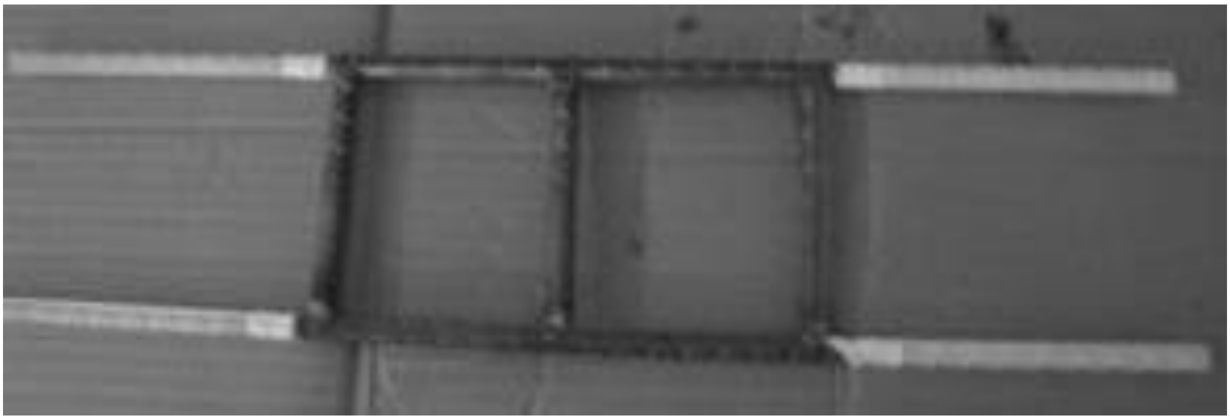

Figure 1. Steel bars coated with non-corrosive paint

2.2 Accelerated Carbonation

The average carbon dioxide content in the atmosphere is 0.03% by volume or air and in urban or industrial zones around 0.1%. The carbonation is a slow process, and therefore to obtain short term results it is necessary to accelerate the process. For this reason, the concrete specimens were introduced to a carbonation chamber. Inside the chamber the concrete specimens were exposed to a controlled environment, CO_2 concentration of 25% by volume of air, at 27± 2 °C and RH of 80 ± 9%, by a 3 week period to guarantee the carbonation front reaches the reinforcement steel surface. Besides, concrete cubic blocks (10 x10 x 10cm) with the same mixture were elaborated and exposed in the carbonation chamber in order to verify periodically the depth of carbonation front by phenolphthalein test.

2.3 Electrochemical realkalisation

After the accelerated carbonation process a conductive mortar layer with 1cm thickness was applied on one face of the prismatic concrete specimen (the characteristics of conductive mortar mixture are shown in table 1), the electric conductivity of conductive mortar mix was 0.631 mS/cm (± 8%), measured by four probe method in prismatic samples (7.5 x 7.5 x 2.5 cm). As primary anodes two graphite rods (diameter 0.25 inch) were used, Figure 2. After a day of applied the conductive mortar layer on prismatic specimen it was applied a 1M Na_2CO_3 solution until soaking the surface of the conductive layer. A DC power supply (Steren PRL-25, 15V/30A) was connected to the steel and primary graphite anode, the prismatic concrete specimens were connected in parallel to DC power supply. The current density applied was 2A /m^2 (52mA current intensity), and the initial voltage difference applied between anode-cathode of 8.8V, to reach the level of current required. The ER treatment was 5 days and the values of voltage difference and current intensity were monitored daily. To compare the performance of conductive mortar anode, the same electrochemical treatment was carried out on prismatic specimens using as anode a steel mesh immersed in pulp paper soaked with 1M Na_2CO_3 solution. The criteria to determine if the ER has been effective in both cases were obtained from the NACE SP0107-07 [28].

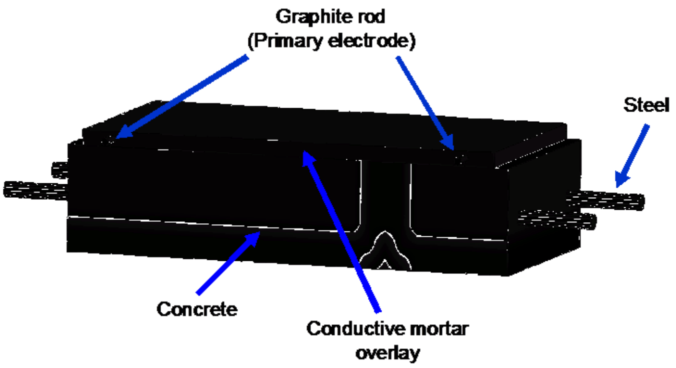

Figure 2. Conductive mortar anode on prismatic concrete specimen.

2.4 Realkalisation width and pH measurement

The measurement of carbonation depth front after the accelerated treatment in the carbonation chamber, and after the ER, was done spraying a 1% phenolphthalein solution on the surface of a freshly broken concrete. Additionally pH measurements were obtained using a pH meter Hanna Instruments HI 253, drilling powder samples from carbonated, non-carbonated and realkalised concrete zones. The powder samples were dissolved in distilled water with a solid-liquid ratio of 1:10. The initial and end pH of conductive mortar during the ER was obtained with pH-meter, drilling powder samples of conductive mortar. In the case of steel mesh anode with pulp paper the pH was measured placing the pH-meter electrode in the pulp paper. In figure 3 are showed the zones of taking samples to pH measurement.

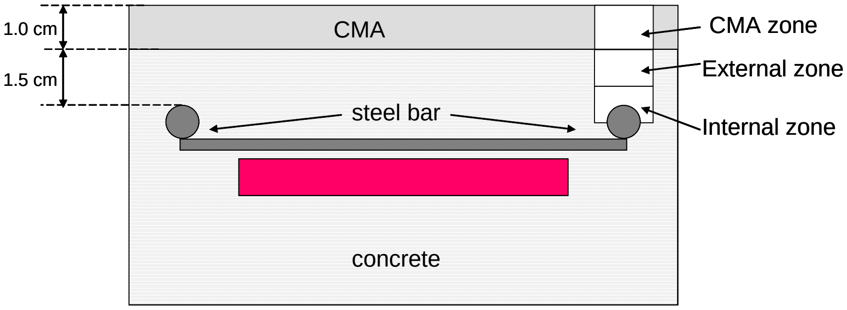

Figure 3. Zones from where were obtained the samples to pH measurements.

2.5 Corrosion rate measurement

The corrosion state of steel in prismatic concrete specimen it was determined by corrosion potential (E_{corr}) and current density (i_{corr}) before and after the electrochemical realkalisation. The i_{corr} was obtained by linear polarization resistance (LPR), the used parameters of LPR were: potential scan ± 20mV from rest potential, and 10mV/min scan rate. A conventional three electrode cell was used, where the working electrode was the steel rebar, a stainless steel plate as counter electrode and a copper /copper sulfate (SCE) as reference electrode. The measurements were carried out using an ACM Instruments model Gill AC 100 kHz potentiostat/galvanostat.

3. RESULTS AND DISCUSSION

3.1 Accelerated carbonation

The carbonation front depth of concrete cubic blocks reach 3±0.3cm after 3 weeks in the carbonation chamber, The average pH values measured with pH-meter on carbonated and non-carbonated zone were 12±0.5 on non-carbonated zone and 8±0.3 on carbonated from cubic blocks.

These can be considered as criterion to assure that the carbonation has reached the reinforcement in the prismatic beams (concrete cover thickness is 1.5cm) exposed in carbonation chamber.

3.2 Total density charge

In figure 4 are shown the current density and the applied voltage during the 5 days of ER with conductive mortar anode, the observed variations must be due to the humidity loss in the mortar layer, thus it was necessary to fit the exit voltage to maintain the current around 52mA. From this graphic of current density was calculated the total charge density. The total density charge calculated during the treatment was of 235.26A h /m^2.

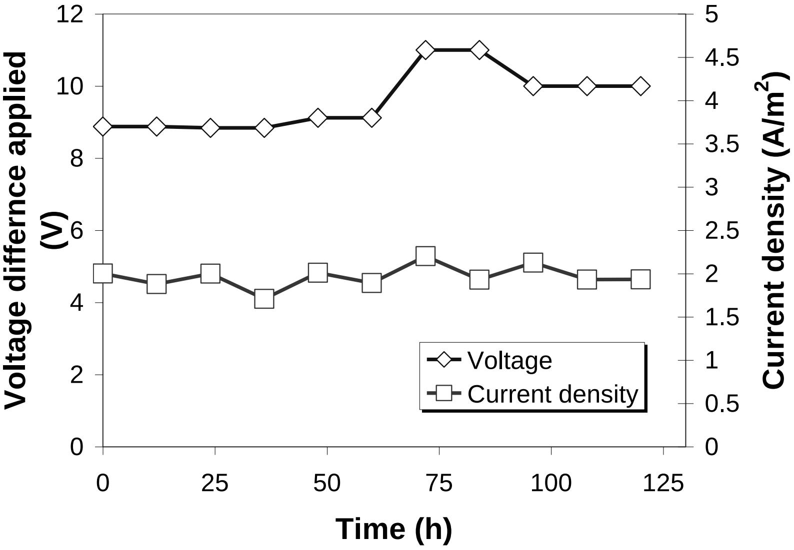

Figure 4. Voltage difference and current density applied during the ER treatment to prismatic concrete specimen.

3.3 Realkalisation width

The phenolphthalein test was carried out to evaluate the carbonation depth after the ER treatment. The maximum depth of carbonation front after applied the ER on prismatic specimen face, where conductive mortar layer was applied is 0.8cm, figure 5, and in the zone around the steel bar the pink coloration of the phenolphthalein reached 1.07cm. On the right steel rod the pink coloration has extended 1.5cm, including the totality of the concrete cover, this is attributed to the combined effect of OH^- generation on the reinforcement steel surface and the penetration of the Na_2CO_3 solution from the conductive mortar layer. By other hand, in the figure 5, it is possible to be appreciated that the ingress of alkaline electrolyte in the concrete did not appear of uniform way, is observed a colorless zone in superior surface of concrete, between the steel bars.

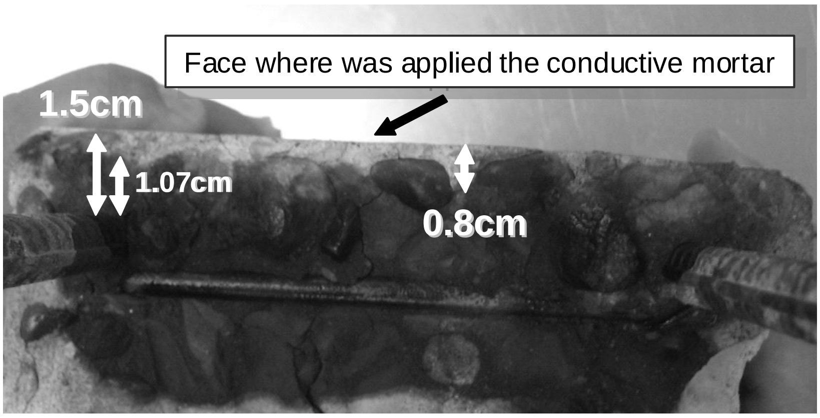

Figure 5.Carbonation front after ER with conductive mortar anode.

The NACE SP0107-2007 suggests two criteria to determine if the ER treatment has been effective, the first establishes that the charge density, by unit of steel area, during the treatment must be at least of 200A h /m^2. And the second criterion dictates that the effectiveness of the ER treatment is demonstrated by the pH test, using a solution of phenolphthalein, indicating that the realkalisation degree, by the pink coloration around the reinforcement, has a minimum of 10mm. The results reported, in figures 4 and 5, indicate that these two criteria were satisfied since the charge density, in the present study, reach a value of 235A h /m^2 and the minimum extension of the pink coloration around the steel surface is 1.07cm.

3.4 pH value

The pH measured on prismatic concrete specimens before and after the ER, using conductive mortar anode (CMA) or steel mesh anode (SMA) are presented in figures 6 and 7. In external concrete surface (external zone, figure 3) the realkalisation of concrete is generated by the penetration of alkaline solution of Na_2CO_3 from exterior through capillary absorption and diffusion. In the case of CMA the restoration of pH was from 8.7 (carbonated) to 11.7 (realkalised), figure 6. And in the ER with SMA immersed in pulp paper soaked with Na_2CO_3 solution, the pH increase before and after (8.3 to 11.8 respectively) the results of electrochemical treatment is similar to obtained using the CMA to the same period of time. In the internal zone the increase of pH is due to generation of hydroxyl ions according to reactions (3) y (4), and figure 7 shows the pH of concrete around the steel rebar (internal zone, figure 3), with the use of CMA it can observe that the pH increase from 8.13 (previous to ER) to 12.06 after the 5 days of ER, and with the SMA the pH values increased from 7.9 to 11.83. As can be observed the application of CMA or SMA in ER of carbonated concrete no presents a great difference in the results of pH after 5 days of treatment.

In table 2 are show the initial and end pH of CMA and anolyte around SMA, and in both cases is observed a decrease of pH during the 5 days of treatment. The reduction in pH is generated by the anodic reaction (6) with generation of O_2 and OH^- consumption. In the case of SMA the O_2 generation was confirmed with the formation of bubbles on surface of pulp paper in SMA. And in CMA the bubbles appear in the junction between concrete and CMA at the beginning of the treatment, first two days when the conductive mortar was totally soaked with Na_2CO_3 solution, after the second day the CMA it was not soaked and bubbles were not observed. The pH values of anolyte with the SMA obtained are in agreement with the reported by F. Gonzalez, et al. [29] who used a MMO Ti Mesh anode immersed in an alkaline electrolyte (1M Na_2CO_3) for the realkalisation of concrete specimens made with cement CPC30R reaching a pH value of 10.5 after 13 days of treatment.

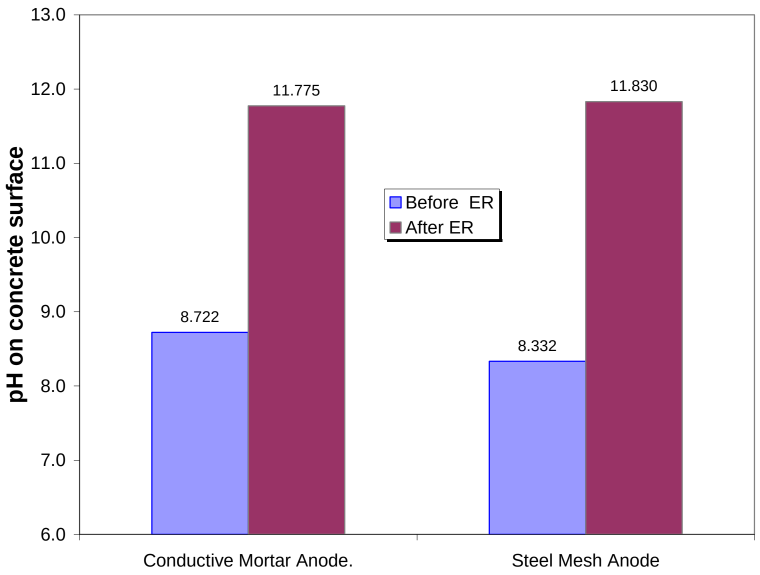

Figure 6. pH values of concrete on prismatic specimen surface (external zone) before and after Electrochemical Realkalisation.

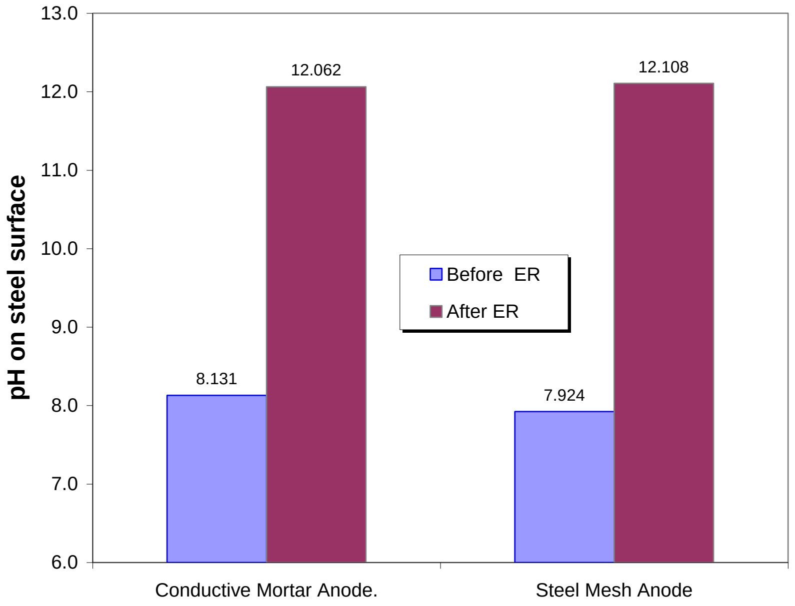

Figure 7. pH values of concrete on steel surface (internal zone) before and after Electrochemical Realkalisation.

Table 2. Initial and end pH of CMA and SMA anolyte during the ER.

Anode	Initial	End
Conductive mortar	12.385	11.024
Steel mesh	12.421	10.825

3.5 Corrosion current density and potential

The Figure 8 compares corrosion potential (E_{corr}) versus corrosion current density (i_{corr}) of steel immersed in prismatic concrete specimens from before carbonation until five days after the ER.

Previous to carbonation the electrochemical parameters of prismatic specimens displayed potentials between -100 to -150 mV versus Cu/$CuSO_4$ (vs. SCE), according to ASTM C876 [30] suggests that the corrosion probability is 10%, and the i_{corr} measured before carbonation with LPR method presented values of 0.38 to 0.54 uA/cm^2 which locates the systems in a region from low corrosion to moderate corrosion, the interpretation of i_{corr} it was considering the criteria proposed by Broomfield [31] and represented in figure 8 by vertical dashed lines.

After the carbonation process the values of E_{corr} were found between -120 to -150 mV (vs. CSE) and i_{corr} of 1.3 to 1.58 uA/cm^2, which locates the system in high corrosion condition, in this case the E_{corr} values are very positive and contradictory, Arup and Klinghoffer [32] suggest that the misleading results of corrosion potential in carbonated concrete are consequence of the difference in concentration of hydroxyl, hydrogen and metal ions that precipitate out on carbonation front leading to a "junction potential".

During the 5 days of ER the potential reach values of -2500 mV (vs. CSE) with a current density applied of 2A/m^2 for both cases (CMA and SMA). After 5 days of finished the electrochemical treatment the readings of corrosion potential for specimens with SMA were between -566.4 and -505.33 mV (vs. CSE) and an i_{corr} of 0.38 to 0.46 uA/cm^2, it locates the system in a zone of low corrosion. For concrete treated with CMA after the ER the potentials readings showed values of -583 to -479 mV (vs. CSE) with a current density from 0.69 to 0.98 uA/cm^2, high corrosion condition. In this last case the specimens have not been depolarized totally, however it can be observed a reduction in i_{corr} as consequence of electrochemical treatment of realkalisation, changing the condition from high corrosion to low corrosion for the concrete treated with SMA and for CMA the corrosion condition change from high corrosion to moderate.

Considering the values of i_{corr} obtained from LPR, in both cases, show that the realkalisation could not repassivated the steel at least with the duration of treatment applied; but a reduction in the corrosion rate it was obtained. The difference in i_{corr} reduction between the concrete treated with CMA and SMA is attributed to the weak ingress of alkaline solution of Na_2CO_3, as can be observed in figure 5, the penetration of solution occur of preferential form in the edge of specimens for the concrete treated with CMA.

On base to previous, it is demonstrated that the use of an auxiliary electrode of conductive cementitious material with addition of 25% of coke breeze is able to increase the pH of carbonated concrete by an electrochemical way.

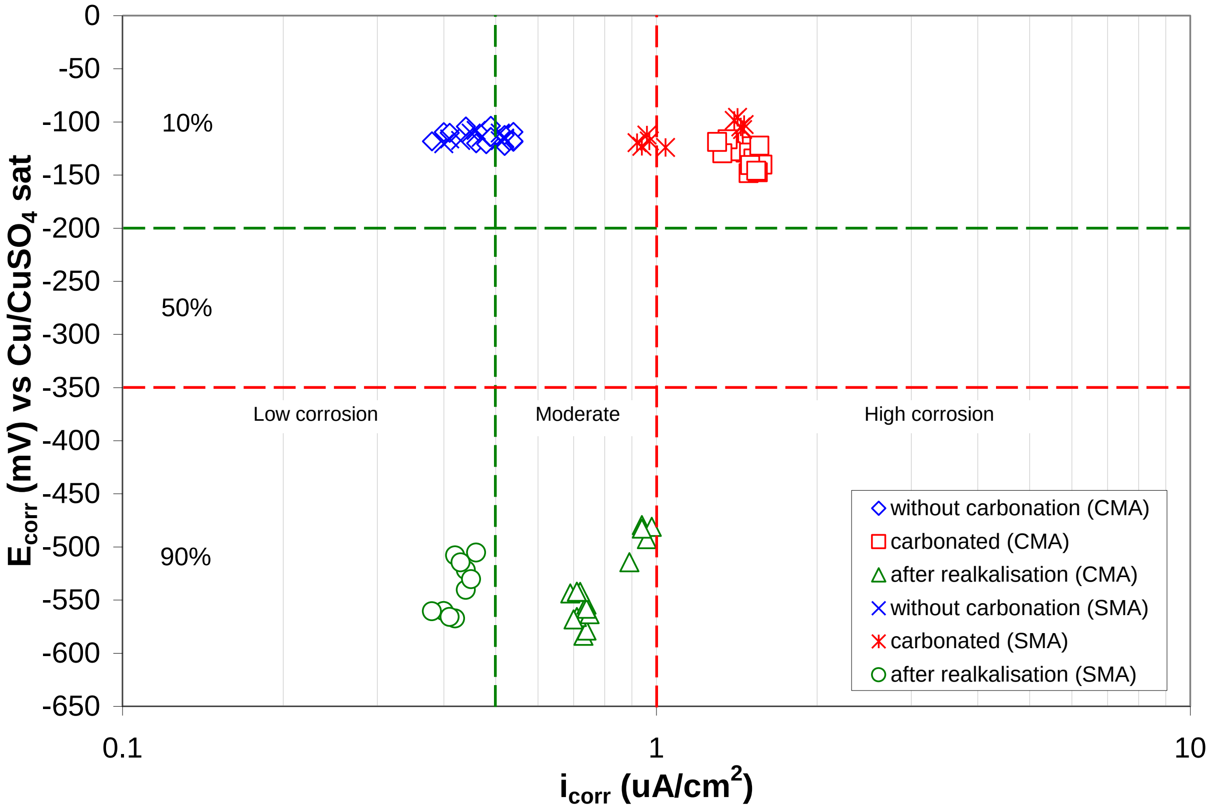

Figure 8. Corrosion potential (E_{corr}) versus corrosion current density (i_{corr}) of prismatic concrete specimens, conductive mortar anode (CMA) and steel mesh anode (SMA)

4. CONCLUSIONS

The results of the present study at laboratory show that:

The application of ER using as anode a conductive mortar, applying a current density of 2 A/m^2, during 5 days, satisfies the NACE SP0107-2007 criteria to determine if the ER treatment has been effective. In the present study, the charge density reach a value of 235A h/m^2 and an extension of the realkalisation width by phenolphthalein test around the steel of 1.07cm.

Comparing the pH increase measured in concrete around the steel no present a great difference with using as auxiliary electrode CMA or SMA; in both cases the average pH obtained after the ER is 12.

The determination of corrosion condition by LPR shows a reduction in the corrosion current density after the ER with the CMA from high corrosion to moderate, but not is enough to reach the passive condition of steel, at least with duration of test.

And finally, the use of a conductive mortar anode added with 25 % of graphite powder increase the pH of carbonated concrete from 8 to 12, around the steel bars, and it makes its use possible in the electrochemical realkalisation of concrete.

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