



## Metallic corrosion of steels embedded in calcium aluminate cement mortars

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### Abstract

In the present paper, the corrosion levels of reinforcing steels embedded in Calcium Aluminate Cement (CAC) mortars have been studied. Experiments were designed to investigate the influence of the following factors in steel corrosion: cover thickness, type of steel (carbon steel (CS) and stainless steel (SS)), temperature at mixing and curing, influence of chloride concentration, nitrite ion as corrosion inhibitor and carbonation of mortar. The reinforcing steel bars do not become more corroded because of the carbonation of the CAC mortar cover; in some cases, this carbonation seems to have an inhibitor effect in the steel corrosion. The higher the process of carbonation in CAC, the higher the intensity of the X-ray reflections of Friedel's salt and the smaller the values of corrosion rates. © 2001 Elsevier Science Ltd. All rights reserved.

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### 1. Introduction

Along several decades, from the 1950s through the 1970s, High Alumina Cement (recently denominated Calcium Aluminate Cement, CAC) was widely used in Spain for reinforced and prestressed floor beams fabrication, mainly in areas with high demand of building construction like Catalonia, Valencia, Balearic and Canary Islands.

Last decade, an important number of failures and defects was detected, many of which were related to the use of CAC, showing the need to investigate the changes in properties and behaviour of this type of cement. Meanwhile, the use of CAC is highly restricted in general construction and totally banned in prestressed concrete by Official Rules in Spain and other countries.

The fact that under specific ambient conditions, CAC concrete strength decreases dramatically some years after mixing and casting has already been known long time ago. The explanation of this loss of strength is the process called “conversion” [1–4]. Conversion of CAC concrete takes

place in buildings in normal conditions of use, and is a result of a change in the composition of the hydrated cement from a metastable to a stable form. As a result, many properties change and, consequently, the behaviour of the reinforcing steel is affected.

Durability of both concrete and steel govern the remaining service life of the structure. At present, there are very few references in the specialised bibliography concerning the corrosion levels that reinforcing steels embedded in CAC mortars and concretes present [5–7]. This study tries to characterize the durability of the structures built with CAC concrete.

The following objectives were established prior to starting the experiments:

1. To obtain new data and deeper understanding of the steel corrosion process in CAC mortars.
2. To investigate the influence of the following factors in steel corrosion:
  - Cover thickness.
  - Type of steel: carbon steel (CS) and stainless steel (SS).
  - Temperature at mixing and curing.
  - Influence of chloride concentration.

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- Nitrite ion as corrosion inhibitor.
- Carbonation of mortar.

For these tasks, three sets of experiments were designed and carried out as described below.

## 2. Experimental programme

### 2.1. Materials and specimen preparation

CAC/R Electroland cement was used. Mortars were prepared according to Spanish Standard [8] requirements. The composition of CAC employed is given in Table 1. Silica sand was always employed as aggregate, and distilled water was always used. The mortar combined 1 part (mass) of cement and 3 parts of sand. The w/c ratios used were 0.4 and 0.7.

Analytical-grade NaCl was used in the mix water in order to achieve total chloride ion concentrations of 0.5%, 1%, 1.5% and 2%, as a corrosive agent. Also, analytical-grade  $\text{NaNO}_2$  was used in order to achieve total nitrite ion of 4%, as an inhibitor. All proportions are in relation to cement weight.

CS and SS bars were used for the experiment. The exposed areas used were 9 and 15 cm<sup>2</sup>, respectively. Bars were previously cleaned in HCl/H<sub>2</sub>O solutions and degreased in acetone. Adhesive tape was used for limiting the exposed area.

### 2.2. Influence of cover thickness in steel corrosion

Experiment 1 was designed for the following objectives:

- To check the influence of cover thickness in CAC mortars.
- To test the corrosion behaviour of SS.
- To test the influence of the temperature at mixing and curing in the steel corrosion levels.

Four cubic cells, 10 cm side, were prepared for this experiment as shown in Fig. 1. Two cells were mixed and cast at 5°C, and the other two at 60°C. Two w/c ratios, 0.4 and 0.7, were used for individual cells in either pair. No chloride ions were added at fabrication.

Positioning of steel bars was arranged as shown in Fig. 1. Cover thickness are 1, 2, 3 and 4 cm for CS bars, and 1, 2 and 3 cm for SS bars. Note the inverted V arrangement to allow equal cover conditions for either steel type. Individual bars have been identified within each cell by a capital letter

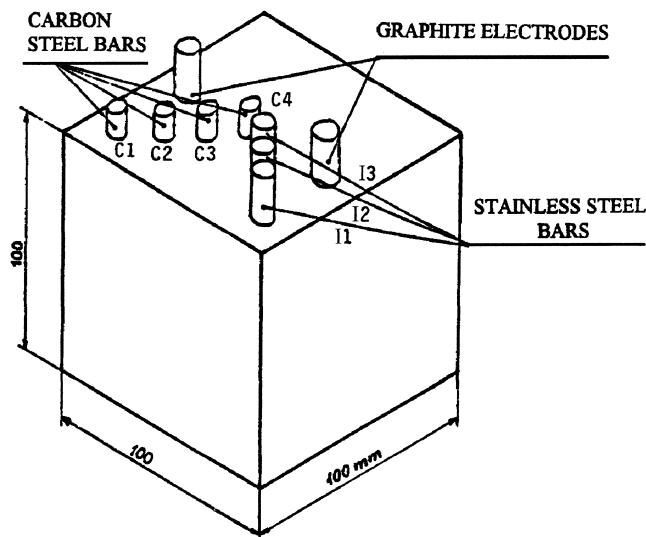


Fig. 1. Shape and dimensions of the cells tested in Experiment 1.

(C for CS, I for SS) plus a digit meaning the cover thickness in centimeters.

After demoulding, the cells were partially submerged in a seawater solution. Test measurements were taken and recorded daily for the first week. Then for the following 8 weeks, the reading interval was set to 3 days and changed to once a week for the last 4 months of the experiment. Finally, the cells were demolished to perform direct weighing of the electrodes.

### 2.3. Influence of chloride ion

Experiment 2 was designed for the following objectives:

1. To test the influence of chloride ion concentration in steel (CS) corrosion embedded in CAC mortars.
2. To determine the critical concentration above which the steel corrosion is not acceptable (concentration threshold).

The test specimens were 2 × 5.5 × 8 cm and contained, embedded, two identical bars. A graphite bar was also embedded as counterelectrode. The mortar specimens were prepared in the laboratory, at 5°C and 60°C, respectively, and 90% RH.

Five mortar specimens were prepared at 20°C with a w/c ratio of 0.7. Chloride ions were added in five different concentrations, 0%, 0.5%, 1%, 1.5% and 2% by cement weight. After demoulding, all the samples were partially submerged in distilled water at ambient temperature ( $\approx 20^\circ\text{C}$ ) for the rest of the experiment time.

### 2.4. Influence of carbonation process

Measurements of the rate of corrosion of steel rebars have been carried out in carbonated and uncarbonated

Table 1  
Chemical analysis of CAC (wt.%)

CaO	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	FeO	TiO <sub>2</sub>
38–40	2–3.5	40–42	8–12	2.5–5.5	1.5–2.0

mortar specimens without additives and also with NaCl as a corrosive agent, and  $\text{NaNO}_2$  as a corrosion inhibitor (Experiment 3).

The purpose of this third experiment is to study the steel (CS) behaviour when it is embedded in carbonated mortars made with CAC.

This experiment has two different parts:

- (A) Study of carbonated CAC mortars without additions.
- (B) Study of carbonated CAC mortars with additions.

Four samples, of identical shape as in Experiment 2, were prepared for this study, combining two w/c ratios (0.4 and 0.7) and two mixing temperatures ( $5^\circ\text{C}$  and  $60^\circ\text{C}$ ). After the curing period, the samples were hastily carbonated in a  $\text{CO}_2$  atmosphere. In the process of carbonation, the test specimens were maintained after the curing period in a chamber holding a  $\text{CO}_2$  atmosphere at a RH of  $\cong 47\%$  [9]. After the carbonation period, samples were stored in HR = 100%,  $20^\circ\text{C}$  ambient. Corrosion rates,  $I_{\text{corr}}$ , and corrosion potentials,  $E_{\text{corr}}$ , were recorded at different intervals along a time period of 6 months.

Chloride ions as an active corrosion agent and nitrite ions as a corrosion inhibitor have been tested as additions to the mortar mix, both separately and jointly, in proportions 2% and 4%, respectively, by cement's weight.

### 2.5. X-ray diffraction spectra

The XRD experiments were carried out in a Scheifer powder diffractometer with a graphite monochromator and an NaI (TI) scintillation detector using  $\text{Cu K}\alpha$  radiation. The aperture slit of  $3^\circ$  and the detector slit of  $0.15^\circ$  were used. After being crushed in an agate mortar to  $40\ \mu\text{m}$ , the samples were preserved in acetone at  $5^\circ\text{C}$ . The XRD spectra were taken at a speed of  $1^\circ\ \text{min}^{-1}$ , and the intensities were calculated from the maxima of diffraction after discounting the background.

### 2.6. Measurement techniques

The electrochemical technique used to measure the instantaneous corrosion rates was the polarization resistance technique, through the well-known Stern–Geary formula [10]:  $I_{\text{corr}} = B/R_p$ .  $I_{\text{corr}}$  was calculated assuming values of  $B = 26\ \text{mV}$  for corroding steel or  $52\ \text{mV}$  for passive steel.  $R_p$  and corrosion potential ( $E_{\text{corr}}$ ) were periodically measured during the time of the experiment, and the weight loss was measured, for each electrode, at the end of the test. All potentials are referred to saturated calomel electrode (SCE). The electrochemical loss estimated from the integration of the  $I_{\text{corr}}$ –time curves has been compared with the corresponding gravimetric losses. The good agreement obtained between the two weight loss values validates the  $B$  values employed. Conductivity was measured always at the end of the test, using a GECOR6 instrument from GEOCISA. This

technique is based on the IR drop from a pulse between the sensor counterelectrode and the rebar network.

## 3. Results and discussion

Fig. 2 (Experiment 1) shows the evolution of corrosion current density,  $I_{\text{corr}}$ , and corrosion potential,  $E_{\text{corr}}$ , with time corresponding to each of the steel types in contact with the studied CAC mortars (in specimens with w/c = 0.4 and at  $60^\circ\text{C}$  curing temperature). The zone between 0.1 and  $0.2\ \mu\text{A}/\text{cm}^2$ , corresponds to the limits of an active corrosion in terms of decrease of service life and of tolerable corrosion level [11].

After the first 3 weeks, the corrosion trends for CS and SS are quite similar, although with different values, the remaining SS in the passive zone, while CS reaches important corrosion levels.

Comparison of corrosion levels of C1 and C2 against C3 and C4, with clearly lower values for the latter, confirm that cover thickness is a determining parameter for CS corrosion. This conclusion is coherent with the fact that corrosion levels not only depend on  $\text{Cl}^-$  concentration but also on other depth-depending factors such as  $\text{O}_2$  concentration, humidity, etc. However, this conclusion is not applicable to SS rebars ( $I_1, I_2, I_3$ ).

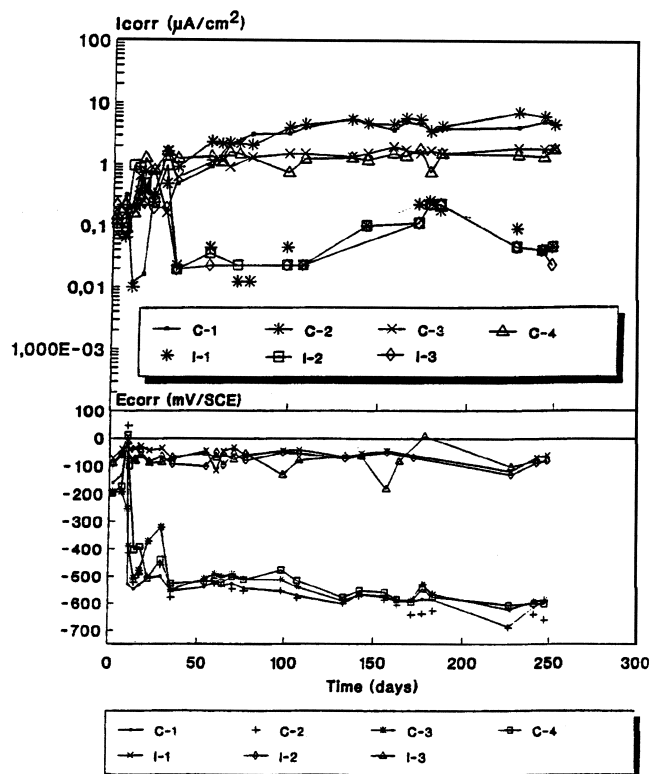


Fig. 2. Evolution of  $I_{\text{corr}}$  and  $E_{\text{corr}}$  with time. Cell number 3: w/c = 0.4, temperature =  $60^\circ\text{C}$ . C: carbon steel; I: stainless steel. Numbers mean cover depth in cm.

As can be expected, a thicker cover may produce a delay in the transition period from passive to active states, but with different steady corrosion levels.

Of course, in specimen manufactured with a w/c ratio of 0.7, the levels of corrosion are higher for every cover thickness due to the high porosity. However, the conclusions are similar for both w/c ratios.

The trends in the  $E_{\text{corr}}$  measurements (Fig. 2) confirm the interpretation of the  $I_{\text{corr}}$  data. Within the three initial weeks of the experiment,  $E_{\text{corr}}$  values are similar for all the seven bars, but, afterwards, corrosion becomes much more active for CS ( $\cong -600$  mV) than for SS, which remains in passive conditions throughout the experiment ( $\cong -100$  mV).

Fig. 3 illustrates the influence of temperature (referred also to CS specimens with w/c=0.4 and 3 cm cover thickness) (Experiment 1). Corrosion rates at 5°C are much lower than those at 60°C. This fact is attributed to the conversion process that produces an important increase in the mortar porosity, and a consequent loss of the cover capability to protect steel against corrosion.

### 3.1. Influence of chloride ion concentration

Chlorides may be introduced during manufacture or service. During service, chlorides from external sources may penetrate zones beyond the cover. A question of practical importance, which has to be considered when chloride-bearing concreting materials are specified or when investigations of the durability of reinforcement concrete structures are made, concerns the threshold level of chloride ion, which is liable to stimulate the steel corrosion in concrete. Thus, for concrete made from OPC, it is generally accepted that a low risk is associated with chloride contents (by weight of the cement) of <0.4%, an intermediate risk for chloride contents of 0.4–1.0% and high risk for levels >1.0% [12].

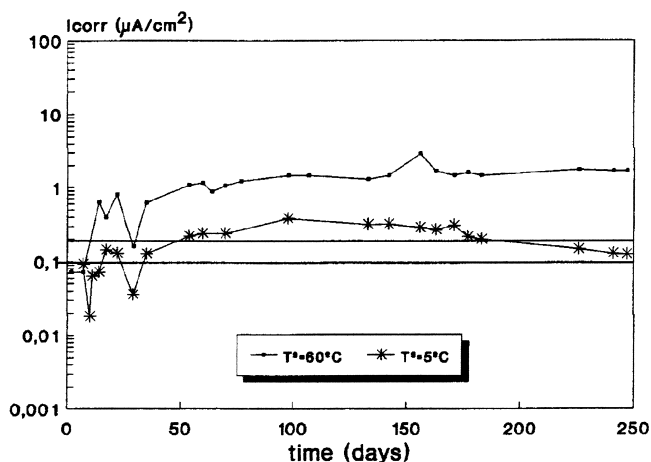


Fig. 3. Influence of the temperature in the steel corrosion levels (CS, specimen with w/c=0.4 and with a cover thickness of 3 cm in cubic cells partially submerged in sea water).

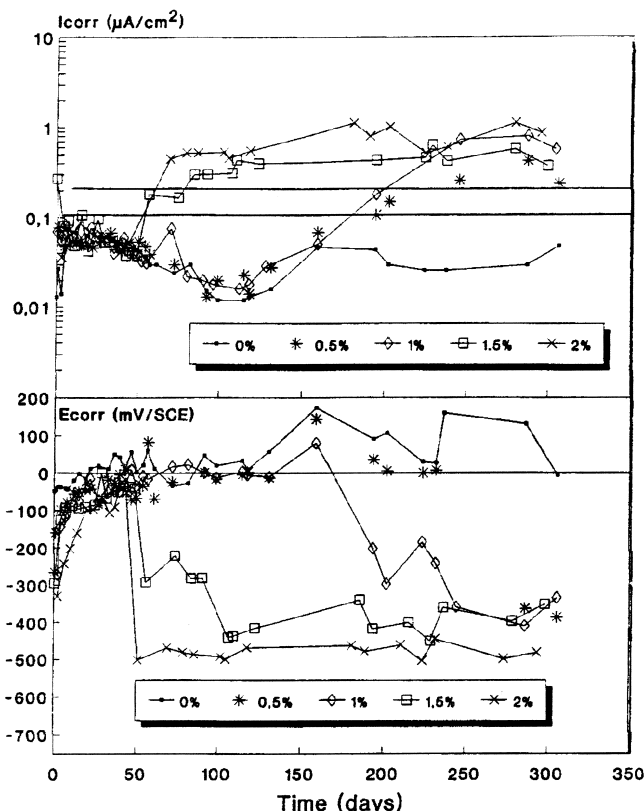


Fig. 4. Evolution of  $I_{\text{corr}}$  and  $E_{\text{corr}}$  with time for CS bars in CAC mortars part. submerged in distilled water (w/c=0.7, temperature=20°C).

However, there is not much information regarding the behaviour of reinforcement embedded in mortar made of CAC [5–7]. Measurements of the rate of corrosion of steel rebars have been carried out in specimens without and with NaCl as a corrosive agent.

Fig. 4 (Experiment 2) shows the evolution of the  $I_{\text{corr}}$  and  $E_{\text{corr}}$  with time, corresponding to different concentrations of chloride ions.

At the beginning, all the samples provided the same  $E_{\text{corr}}$  and  $I_{\text{corr}}$  values, but after 50 days, those cells with chloride ion concentrations (1.5% and 2%) presented a sudden increase in  $I_{\text{corr}}$  values, meaning important corrosion levels. The rest of the samples still remained passive ( $I_{\text{corr}} < 0.1 \mu\text{A}/\text{cm}^2$ ) after 160 days of age. This sudden increase marks the end of the initiation stage and, subsequently, the beginning of the propagation stage of corrosion. Samples without chloride ions remained passive throughout the experiment.

Plotted  $E_{\text{corr}}$  values vs. time are also presented in Fig. 4, showing similar conclusions as mentioned above. It can be clearly seen that, after 50 days, the  $E_{\text{corr}}$  values of the samples with higher chloride ion concentrations decreased significantly ( $-500$  mV), meaning active corrosion conditions of steel. Lower concentration samples do not reach this state until the age of 220 days.  $E_{\text{corr}}$  remained fairly constant for chloride-free cells (passive conditions). These results are similar from those described by other authors [5].

### 3.2. Resistivity results

Fig. 5 shows the evolution of resistivity with time by specimens of CAC mortar and OPC mortar prepared with and without  $\text{Cl}^-$ . An increase in the resistivity value is observed due to the progress of the hydration reaction. The resistivity values are notoriously higher than those provided by the OPC mortar. These results are coherent bearing in mind that mortars of CAC and OPC manufactured with the same w/c ratio presented different porosities, being greater than those of OPC, as was described in a previous paper [13]. Additionally, the hydration process in CAC, with the formation and interlacing of microcrystals and the agglutinative “gel of alumina”, consumes water faster than the development of the gel of calcium silicate hydrates and calcium hydroxide in OPC mortars.

### 3.3. Study of carbonated HAC mortars without additions

The carbonation of the concrete cover is one of the two main factors that may produce corrosion of reinforcement steel bars (along with the presence of chloride ions in the mortar). However, this fact is not clearly demonstrated when mortars are made with CAC. Carbonated CAC structures have been found with their reinforcing steel bars completely clean and without oxide traces; on the other hand, some carbonated CAC structures have been found with their steel bars completely corroded [14].

In Fig. 6 (Experiment 3), the results for bars embedded in carbonated CAC mortar samples without additions are presented. In the beginning of the process,  $I_{\text{corr}}$  values are very low; however, as can be seen in the figure, there is an important increase of corrosion values 50 days after mixing, which marks the end of the carbonation process. Despite this increase, the  $I_{\text{corr}}$  values never exceed  $0.2 \mu\text{A}/\text{cm}^2$  remaining in the area marked as the transition zone ( $0.1 \mu\text{A}/\text{cm}^2 < I_{\text{corr}} < 0.2 \mu\text{A}/\text{cm}^2$ ), as can be seen.

It can be drawn up from the results that, in a carbonated mortar of CAC with different calcium aluminate hydrates

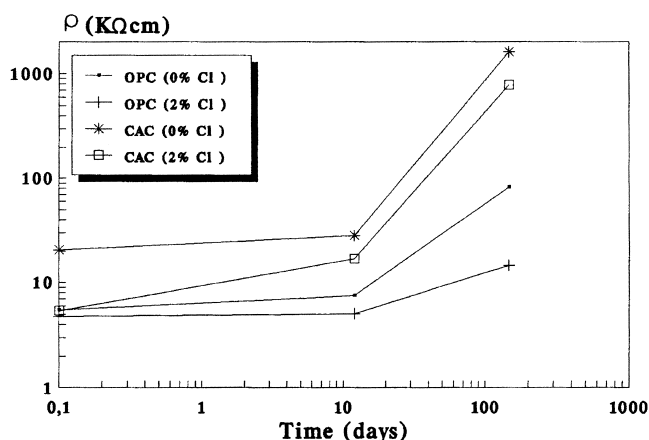


Fig. 5. Variation of resistivity presented by the specimens with time.

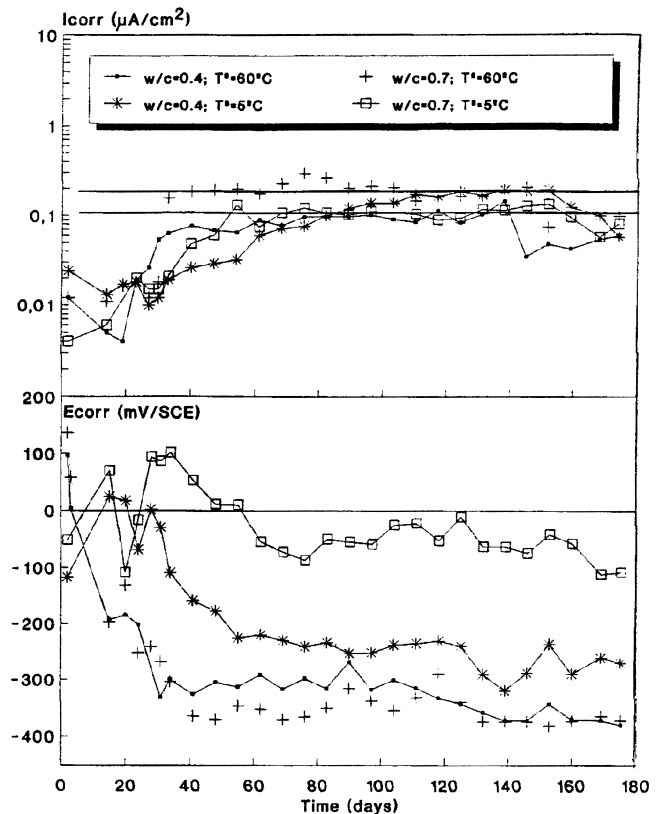


Fig. 6. Evolution of  $I_{\text{corr}}$  and  $E_{\text{corr}}$  with time. Carbonated samples without additions.

initially present, the observed  $E_{\text{corr}}$  variation range (+150 to  $-400$  mV, Fig. 6) indicates that not only is the generalised corrosion possible, but also passivity can take place. That confirms that potential values alone cannot be considered in CAC mortars as the absolute indicator of passive state (Standard ASTM C876).

All the specimens show a very similar behaviour, although the higher the w/c ratio is, the higher the corrosion rate becomes.

These results show that the reinforcing steel bars remained passive throughout the experiment, despite the fact that mortar cover was completely carbonated. When the bars are embedded in OPC mortars the corrosion values are higher than these ones, reaching important corrosion rates [15].

### 3.4. Study of carbonated CAC mortars with additions

Two samples were prepared, at  $20^\circ\text{C}$ , adding both nitrite and chloride ions to each one. A third sample was made with nitrite ions only and a fourth one with chloride ions only. One sample of the former pair and the other two were then hastily carbonated.

As can be seen in Fig. 7 (Experiment 3), the addition of nitrite ions has an important effect, acting as a strong inhibitor of corrosion ( $I_{\text{corr}} < 0.2 \mu\text{A}/\text{cm}^2$ ). Only in the case of nitrite+chloride ions in a carbonated mortar the  $I_{\text{corr}}$  values exceed the limited zone, reaching  $1 \mu\text{A}/\text{cm}^2$ . How-

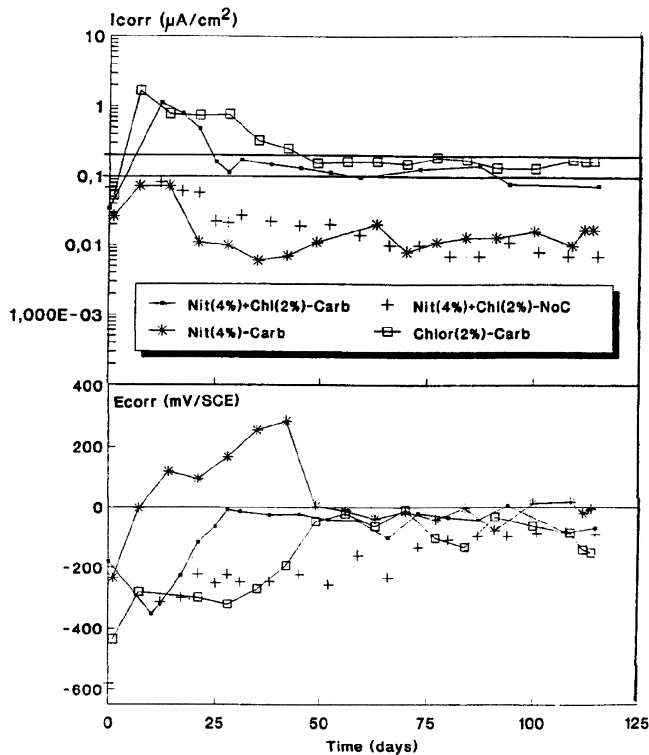


Fig. 7. Evolution of  $I_{\text{corr}}$  and  $E_{\text{corr}}$  with time. Carbonated samples with additions,  $w/c=0.7$  and temperature =  $20^\circ\text{C}$ .

ever, these high values only appear in the beginning of the evolution; after the chloride ions attack, the inhibitor agent goes into action, and places the reinforcing steel bars within the passive zone ( $0.1 \mu\text{A}/\text{cm}^2 < I_{\text{corr}} < 0.2 \mu\text{A}/\text{cm}^2$ ).

If results of samples with nitrite and chloride ions are compared, it can be observed that the steel bars embedded in

the carbonated mortar are more corroded than the bars of the noncarbonated mortar. In the first case, the values reach  $1 \mu\text{A}/\text{cm}^2$ , but there is a sudden decrease, remaining then the steel in the passive zone. In any case, the steel bars reach important corrosion levels.

It is observed that the worst case is the carbonated mortar sample with 2% by cement's weight of chloride ions (without nitrite ions). The trend of this specimen is very similar to the one of the carbonated mortar with nitrite and chloride ions, but the  $I_{\text{corr}}$  values are higher, reaching even  $2 \mu\text{A}/\text{cm}^2$ .

Despite this fact, the corrosion levels do not seem to be as important as they are when using OPC. Garces et al. [13] confirm the general knowledge that the attack, in OPC mortars, is important when carbonated mortar is involved and/or it contains depassivating ions.

Fig. 7 shows that 50 days after the mixing, the  $I_{\text{corr}}$  values decrease until they become  $< 0.1 \mu\text{A}/\text{cm}^2$ . At the same time, the  $E_{\text{corr}}$  values show an increase, reaching values next to  $-100 \text{ mV}$ . In bars embedded in OPC mortars, corrosion implies the development of a continuous and generalized corrosion process that can even increase in presence of depassivating ions such as  $\text{Cl}^-$ . Conversely, in CAC mortars, the carbonation process implies the inhibition of the corrosion process of bars embedded in mortars with a 2% content of chloride ions, as shown in Figs. 6 and 7. This unexpected behaviour is a breakdown of the ideas explaining the carbonation corrosion mechanisms in OPC mortars. The drop in porosity values and the maintenance of pH values within the alkalinity range are factors that may explain such behaviour. Finally, the influence of the formation of Friedel's salt ( $\text{Ca}_4\text{Al}_2\text{O}_6\text{Cl}_2 \cdot 10\text{H}_2\text{O}$ ) in the above-mentioned experiments is also a fact to be considered in the steel corrosion behaviour.

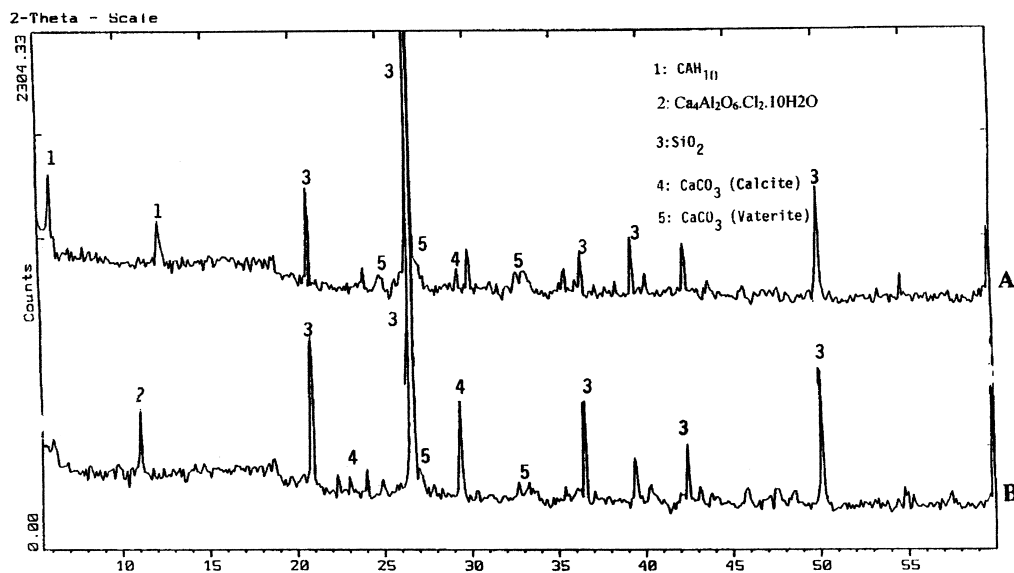


Fig. 8. XRD spectra of the CAC mortars ( $w/c=0.7$ ; at  $20^\circ\text{C}$ ; 120 days: age of curing) ((a)  $\text{Cl}^-$ : 2%; (b)  $\text{Cl}^-$ : 2% +  $\text{CO}_2$ ).

### 3.5. X-ray diffraction analysis

Phases present have been studied from XRD spectra corresponding to the mortar specimens at the end of the experiments (Fig. 8). Independently of conversion of hexagonal phases to the more stable cubic phase,  $\text{Cl}^-$  added as sodium chloride to the mix water combines with Ca and Al as Friedel's salt,  $\text{Ca}_4\text{Al}_2\text{O}_6\text{Cl}_2 \cdot 10\text{H}_2\text{O}$ . Thus, the normal hydroxyaluminates characteristic of CAC are partly replaced by the chloroaluminate. There is a lack of information about the levels of chloride likely to remain uncombined for long periods in the solution phase associated with concretes made from various cements in different conditions of natural exposure. The source of the chloride ions may be significant, and it has been suggested that chlorides, which penetrate concrete from the external environment, tend to remain uncomplexed to a greater degree than those present at the time of manufacture [12]. It is also unclear that the extent to which carbonation, especially in CAC concrete, may cause decomposition of calcium chloroaluminate hydrates.

Fig. 8 shows the XRD spectra of two different specimens of CAC mortars at the end of the test ( $w/c = 0.7$ ; at  $20^\circ\text{C}$ ; 120 days: age of curing) ((a)  $\text{Cl}^-$ : 2%; (b)  $\text{Cl}^-$ : 2% +  $\text{CO}_2$ ). In diffractogram (a), the predominant phase is the hexagonal  $\text{CAH}_{10}$ , while  $\text{CaCO}_3$  is detected in small quantities as calcite and vaterite and Friedel's salt do not appear. However, in diffractogram (b), the predominant phases are  $\text{CaCO}_3$  (as calcite and vaterite) and Friedel's salt. Therefore, mortar carbonation does not apparently imply the Friedel's salt decomposition in CAC mortars. At variance with this results, Page and Vennesland [16] state that Friedel's salt decomposition may be produced if the pH of the pore solution decreases, as is the case if silica fume is added to the cement pastes made of OPC. On the other hand, as Figs. 7 and 8 show, a clear relationship can be established between the carbonation process — Friedel's salt formation and the level of corrosion intensity values: The higher the process of carbonation, the higher the intensity of the X-ray reflections of this compound and the smaller the values of corrosion rates.

## 4. Conclusions

(1) The coating thickness is a determining parameter in the corrosion levels of the corrugated CS. On the other hand, it is not a determining factor when the SS is used, as the steel bars remain passive whatever the coating thickness is. This fact outlines the possible convenience of using this type of steel in concrete structures, at least when the steel bars must have a thin cover.

(2) In CAC mortars, contents of chloride ions higher than 1% by cement's weight produce important corrosion levels, situating the steel in the corrosion active zone.

(3) The reinforcing steel bars do not become more corroded because of the carbonation of the CAC mortar

cover; in some cases, this carbonation seems to have an inhibitor effect in the steel corrosion.

(4) The nitrite ions are strong inhibitors of corrosion of reinforcing steel embedded in CAC mortars.

(5) The higher the process of carbonation in CAC mortars, the higher the intensity of the X-ray reflections of Friedel's salt and the smaller the values of corrosion rates.

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