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# Study of an electrochemical chloride extraction treatment on a both carbonated and chlorinated reinforced concrete.

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## Abstract

Civil engineering structures and historical buildings can suffer from corrosion of the embedded reinforcing steel once the concrete cover is totally carbonated and/or when chloride ions reach the steel/concrete interface. On field, these two types of contamination can be encountered separately or combined requiring implementation of proper repair methods.

In this study, in a first step, corrosion of both chlorinated and carbonated reinforced concrete specimens were followed during casting and accelerated contaminations. In a second step, electrochemical chloride extraction was performed as a repair treatment (cathodic polarization, 1 A/m<sup>2</sup> of steel surface during 8 weeks). The efficiency of the treatment and its impact on the cement matrix and at the concrete-steel interface were studied during the treatment, after rebar depolarization and in the long term (several months) in order to evaluate its durability. In order to achieve these aims, three analysis means were used: electrochemical characterization, scanning electron microscopy and Raman microspectroscopy.

In this paper, results obtained on specimens cast with CEM III cement (often encountered on reinforced concrete historical monuments) with chlorides addition into the mix water and further accelerated carbonation are presented. Study of rebar's electrochemical characteristics during the artificial aging highlighted an increase of corrosion rate from negligible value of 0.1  $\mu\text{A}/\text{cm}^2$  after fabrication to about 10  $\mu\text{A}/\text{cm}^2$  after carbonation. SEM observations confirmed this phenomenon with the identification of a corrosion layer on most of the concrete/steel interface after carbonation. The ECE efficiency was demonstrated by a chloride extraction of about 97% at the rebar level which yields a decrease of chloride ions concentration below the theoretical threshold value of 0.4 % by mass of cement after two weeks. Simultaneously a realkalisation ring was observed on concrete around the reinforcement bar which reached almost 1 cm after 56 days. After depolarization, results showed that a duration of 28 days of ECE treatment allowed the stabilization of the corrosion state of the rebar. Raman microspectroscopy allowed to study in situ, thanks to specific cells, the corrosion products' reduction during the treatment.

Keywords: reinforced micro-concrete; chloride; carbonation; cathodic treatment

## **Introduction**

Complete cover carbonation and chloride ions penetration until the steel/concrete interface are the two main causes of reinforced concrete degradation induced by corrosion. These two types of contamination can be encountered separately or combined on civil engineering structures as well as on historical buildings requiring implementation of proper repair methods.

This study aims to characterize the corrosion processes then the electrochemical chloride extraction (ECE) treatment in a both carbonated and chlorinated reinforced concrete. For this purpose, four series of specimens based on two cements and two aging processes were casted. In this paper the study's complete protocol is presented. First results obtained on one series of specimens during the aging then during the treatment are presented and discussed in a second time. SEM-EDS analyses and Raman microspectroscopy were used to characterize the corrosion product layer at the steel/concrete interface. SEM-EDS gave information about corrosion products distribution, morphology and elements of composition while Raman microspectrometry allowed the identification of crystallized corrosion phases formed. Non-destructive-testing (NDT) electrochemical characterization was used to monitor the evolution of the state of corrosion of the rebar (active/passive).

## **Methodology**

In this section the specimens' characteristics and the experimental program established to follow the evolution of corrosion state during the aging and during the electrochemical treatment are presented.

### **Specimens formulation and design**

Design and composition of specimens used for this study must allow and facilitate chemical reactions within the concrete cover. To achieve this, cylindrical specimens of reinforced concrete of 10 cm in height and 4 cm in diameter (Fig 1) were casted using the composition already used in Tong's thesis [1] that allows on a reduced scale to stay close to the granular distribution of a concrete (Tab 1).

*Table 1: "Micro-concrete" composition*

<b>siliceous Palvadeau aggregates</b>					<b>Cement</b>	<b>Water</b>	<b>W/C ratio</b>
<b>(kg/m<sup>3</sup>)</b>					<b>(kg/m<sup>3</sup>)</b>	<b>(kg/m<sup>3</sup>)</b>	
0/0.315	0.315/1	0.5/1	1/4	2/4	387	251.55	0.65
159	295	192	686	229			

Two types of cement were selected:

- CEM I (52.5 N CP2; Holcim, Lumbres) which is a usual Portland cement which will allow results comparisons. The specimens were named type 1.
- CEMIII/A (52.5 L PM ES CP1 NF; Lafarge, Le Havre), which is a cement frequently encountered on concrete historical monuments. Furthermore, the use of this cement which contains blast furnace slag is promoted for the construction of new structures because of its lower impact on environmental issue (less CO<sub>2</sub> produced). Therefore, the results will help improve knowledge about the behavior of reinforced structure using this cement. The specimens were named type 3.

Rebar was a smooth round steel type S 235JR of 5 mm in diameter with an active surface of 10 cm<sup>2</sup>.

The open porosity measured after curing (28 days for CEM I cement and 90 days for CEM III cement) was about 18 % ± 1 %.

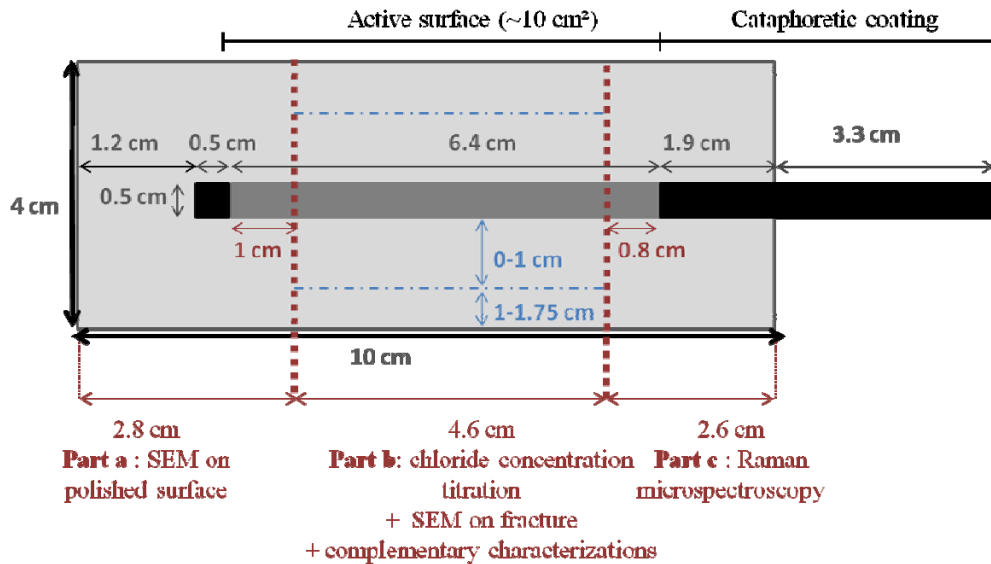


Figure 1 : Specimen design and sampling for the analytical characterizations

### Experimental set-up

A specific set-up (Fig 2) was designed in order to study, in a first step, the corrosion of a both carbonated and chlorinated reinforced concrete and then, in a second step, the efficiency and impact of the electrochemical repair (during the treatment and after its end).

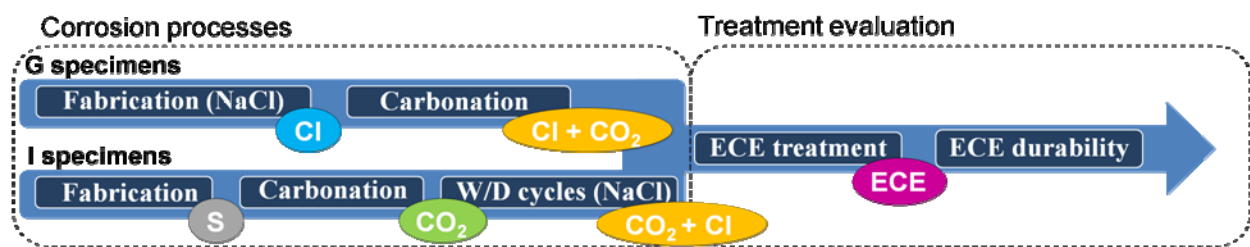


Figure 2 : Experimental set-up

Two different aging procedures were studied:

- Chloride addition to the mix (5 % of cement mass of NaCl) (Cl) and then carbonation until complete carbonation of the concrete cover (50 % CO<sub>2</sub>; 70 % HR) (Cl+CO<sub>2</sub>). The specimens were named type G.
- Carbonation until reaching the concrete/steel interface (50 % CO<sub>2</sub>; 70 % HR) (CO<sub>2</sub>) and then chloride penetration by wetting/drying cycles (five days in a solution of 35 g/L NaCl and two days in the laboratory at room temperature) (CO<sub>2</sub>+Cl). The specimens were named type I.

Evolutions of carbonation and chloride contamination by wetting/drying cycles were followed thanks to colour indicators, respectively phenolphthalein (0.5 % in ethanol) and silver nitrate solutions (0.1 N).

After the aging step, the electrochemical chloride extraction treatment was then conducted on 37 specimens of each series (G1, I1, G3 and I3). The treatment principle is presented on Fig 3. As indicated in the NF EN 14038 part 2 standard [2], a current density of 1A/m<sup>2</sup> of steel was applied between the rebar and a titanium/platinum grid (counter-electrode) during 8 weeks. The electrolyte used was a disodium tetraborate solution (25g/L of Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub> 10H<sub>2</sub>O) to promote ionic exchanges and facilitate chloride ions entrapment. The treatment monitoring was performed controlling and/or following the room temperature, the applied current, the voltage of each specimen and the voltage of the whole specimens on a one hour frequency base.

Three control specimens were taken from the treatment after 3, 7, 14, 28 and 56 days, 56 days corresponding to the end of the treatment. These times were selected as several authors observed that the extraction of chloride ions is more efficient during the treatment's beginning [3-6]. Electrolyte was renewed at each term (except 3 days) to avoid a loss of treatment efficiency due to the increase of chloride ions concentration into the electrolyte. Regarding the chloride extraction evolution, concrete's free and total chloride concentrations were measured on two of the control specimens by AgNO<sub>3</sub> potentiometric titration (on the first centimetre around the rebar and on the 0.75 cm of external cover). Moreover the electrolyte chloride titration was also performed. Phenolphthalein test was performed on the surface of part c (Fig 1) of the two same control specimens. The third control specimen was kept to perform an electrochemical characterization after depolarisation.

Simultaneously to the treatment, two specimens (which were not submitted to the current) were used to observe lixiviation phenomenon. They were immersed in the tetraborate based solution and Cl<sup>-</sup> titration was performed after 14, 28 and 56 days of treatment.

After treatment, the electrochemical characteristics of the reinforced specimens were checked every month to verify rebars are depolarized (storage at 60 %HR and 20°C). Cycles of relative humidity are currently running and electrochemical characterizations are performed to ensure the durability of the treatment.

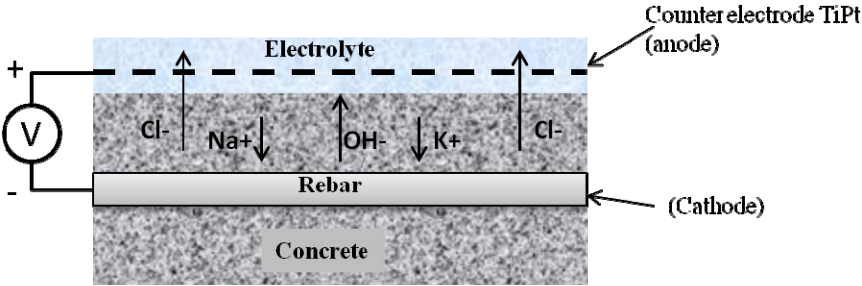


Figure 3 : Simplified principle of electrochemical chloride extraction

Analysis means

To study the corrosion process of the rebar, three main analyses means were used on different parts of the cylinder (Fig 1):

- A Jobin-Yvon Horiba (LABRAM) Raman spectrometer was used. Spectra were obtained with a helium-neon red laser (0.1 mW for these measurements). For these study acquisitions a x50 objective was used to focus laser light and collect spectra for duration of 600 seconds. Specific designed cells were prepared from the regular one

after the aging step to follow the concrete/steel interface “in-situ” during the corrosion initiation and propagation and then during the treatment by Raman microspectroscopy. After cutting as presented Figure 1, a thin glass slide was pasted on the polished surface to isolate the steel/concrete interface from a direct contact with the external environment. After preparation, these samples were immersed in a 25g/L Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub> 10H<sub>2</sub>O solution. Regular observations at a magnification of 50 were made until the detection of pitting corrosion. Raman spectra were then acquired in different areas of interest. The same areas were followed during the ECE treatment and after treatment.

- A JEOL 5600LV scanning electron microscope (SEM) equipped with a micro-analyzer X in dispersal of energy Oxford Link Pentaflet was used to observe corrosion at the concrete/steel interface. Two types of samples were examined: polished cross sections to evaluate corrosion layers evolution and fractured samples to observe both corrosion crystallised phases’ morphology and eventual modifications of the cement matrix.
- An electrochemical characterization was performed with a Bio-Logic VMP2Z potentiostat (5 channels) at each step of the process. The half-cell potential ( $E_{\text{corr}}$ ), the linear polarization resistance (LPR) and the electrical concrete resistance ( $R_e$ ) were measured and the corrosion rate  $i_{\text{Corr}}$  ( $\mu\text{A}/\text{cm}^2$ ) was calculated according to the formula (1).

$$i_{\text{Corr}} = \frac{B}{R_p S} \quad (1)$$

With:  $B = 26$  mV,  $R_p$  (ohm) the linear polarization resistance and  $S$  ( $\text{cm}^2$ ) the active steel surface ( $10 \text{ cm}^2$  in this study)

### Corrosion processes

In this section results obtained on G3 specimens casted with a CEM III/A cement, aged first by NaCl addition to the mix and then by carbonation are presented.

#### Electrochemical characterization

Fig 4 present the half-cell potential (a), the resistance (b) and the corrosion rate (c) of specimens casted with the CEM III/A cement (S), G3 specimens casted with chloride addition (Cl) and G3 specimens after complete cover carbonation (Cl+CO<sub>2</sub>).

From Fig 4-a, half-cell potentials measured for the three studied contamination states are similar with values in the range of -500 mV to -600 mV. The fact that the half-cell potential is significantly electronegative even without contamination (S) can be explained by the use of the CEM III/A cement [7-10]. From Fig 4-b, the resistances of non-contaminated specimens (S) and chlorinated specimens (Cl) are of the same order with average values and standard deviations respectively of 746 ohm and 35 ohm for specimens “S” and 971 ohm and 31 ohm for specimens “Cl”. In the case of both chlorinated and carbonated specimens (Cl+CO<sub>2</sub>) the resistance decreased by a 10 factor with average values and standard deviations respectively of 80 ohm and 12 ohm. This could be explained by a better ionic conductivity due to chlorides freeing during carbonation. Finally, the corrosion rate after curing (Fig 4-c) was below  $0.1 \mu\text{A}/\text{cm}^2$  for both I3-S and G3-Cl specimens which indicates a negligible level of corrosion according to the Rilem [11]. For both chlorinated and carbonated specimens a drastic increase of corrosion rate ( $8 \pm 1 \mu\text{A}/\text{cm}^2$ ) demonstrated a high level of corrosion [11]. This phenomenon is in good agreement with the hypothesis of chlorides freeing during carbonation.

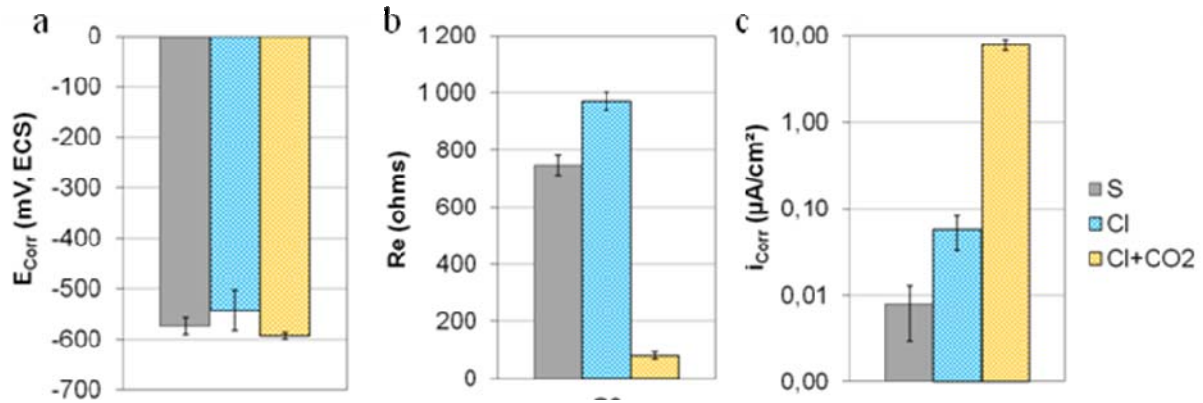


Figure 4. (a) Half-cell potential, (b) resistance and (c) current density of non-contaminated specimens (S) and G3 specimens after curing (Cl) and after carbonation (Cl+CO<sub>2</sub>)

#### Steel / concrete interface characterization by scanning electron microscopy

After casting, the steel/concrete interface of both non-contaminated (S) and chlorinated (Cl) specimens was characterized by an absence of corrosion (Fig 5-a) as well as the rebar prints observations (Fig 5-b).

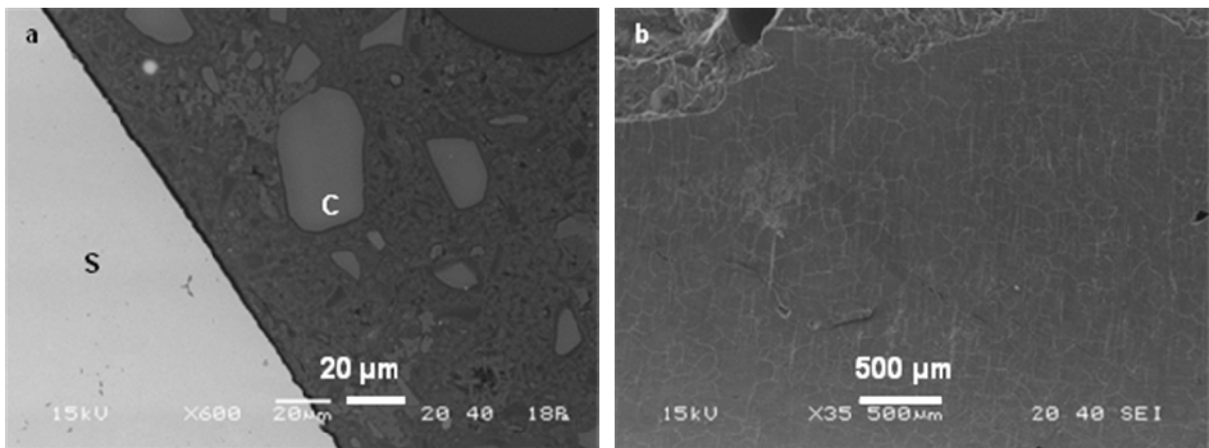


Figure 5 : (a) BEIC-SEM image (x600) of the steel/concrete interface on cross section of G3 specimen, after casting (Cl) without evidence of corrosion ; (b) SEI-SEM image (x35) of the rebar print of G3 specimen, after casting (Cl) without evidence of corrosion

Fig 6-b shows a representative area of the interface of a G3 specimen after carbonation (Cl+CO<sub>2</sub>) with a corrosion layer of 5 to 15  $\mu\text{m}$ . The observation on fracture after carbonation (Fig 6-b) showed numerous areas of corrosion in the rebar print. The crystallisation morphology of corrosion products mainly containing Fe, O and Cl is presented on Fig 7.

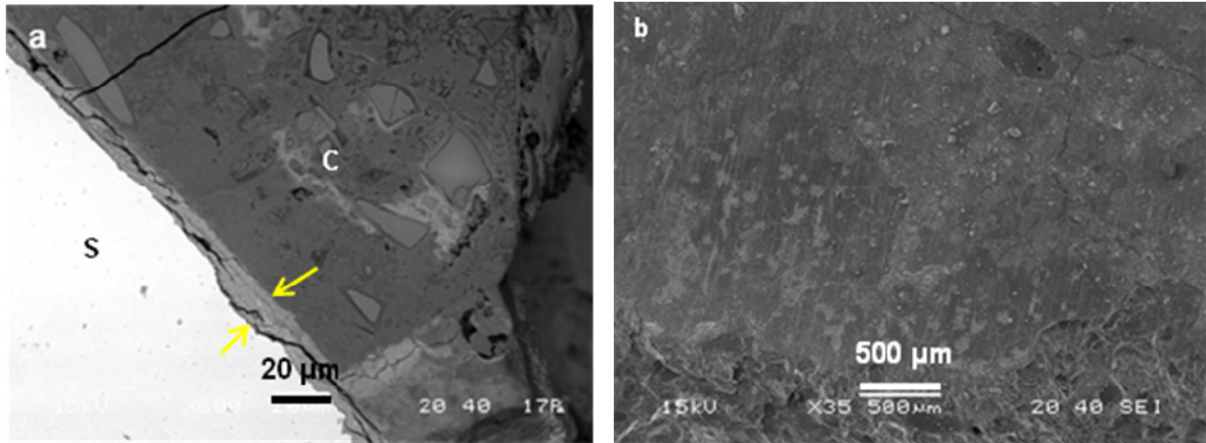


Figure 6 : (a) BEIC-SEM image (x600) of the steel/concrete interface on cross section of G3 specimen, after carbonation (Cl+CO<sub>2</sub>) with evidence of a corrosion layer; (b) SEI-SEM image (x35) of the rebar print of G3 specimen, after carbonation (Cl+CO<sub>2</sub>) with evidence of corrosion products cristallisation

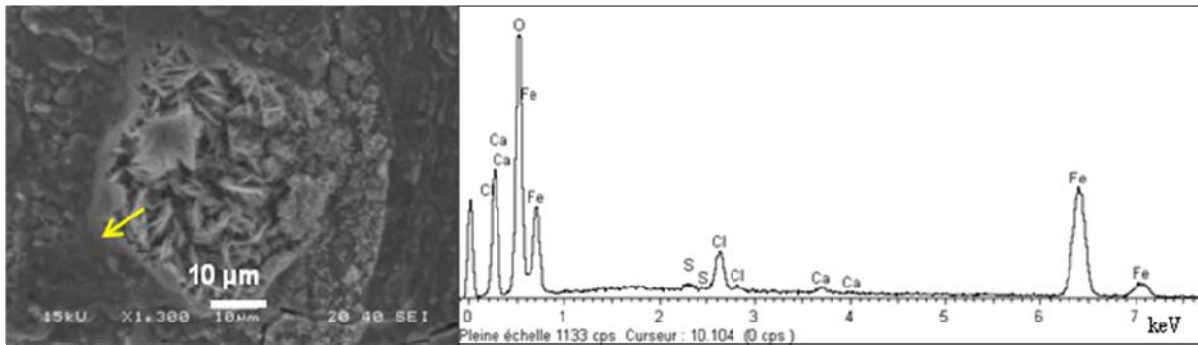


Figure 7 : SEI-SEM image (x 1300) of corrosion products cristallisation on the rebar print of G3 specimen, after carbonation (Cl+CO<sub>2</sub>) and EDS associated spectrum

These observations are in good agreement with electrochemical characterization results. Indeed corrosion products were observed only after carbonation of the G3 specimen, chlorinated during the casting, which corresponds to the drastic increase of the corrosion current.

#### Corrosion products characterization by Raman Spectroscopy

Corrosion in-situ following were made on a specimen G3 both chlorinated and carbonated. Chlorinated green rust (Fig 4) was the predominant phase found (main Raman bands at 434 cm<sup>-1</sup> and 506 cm<sup>-1</sup> and a chloride presence characteristic band around 320 cm<sup>-1</sup>) [12] which evidenced the presence of chloride into the corrosion processes. Moreover, green rust oxidation into FeIII oxyhydroxyde did not occur which proved the good isolation of the interface from a direct exposure to oxygen from the air.



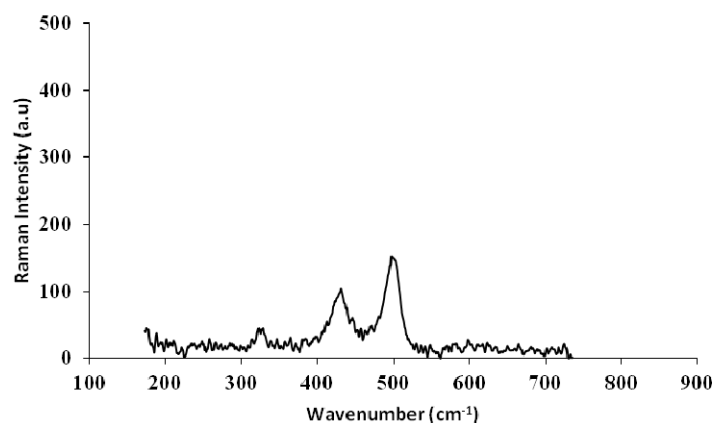


Figure 8 : Chlorinated green rust Raman spectrum

### **Electrochemical cathodic treatment on G3 specimens**

ECE treatment implies several phenomena particularly corrosion products transformations, chloride ions extraction, OH<sup>-</sup> formation and ionic migration. This section aims to study these different effects.

#### **Chloride concentration and pH evolution**

Fig 9 shows the evolution of chloride concentration during the ECE treatment as well as the pH evolution at the rebar vicinity. The chloride threshold for preventing corrosion on new structures from the EN206 standard [9] has also been added.

Regarding the chloride extraction, the evolution of chloride ions extraction was in good agreement with the increasing chloride ions concentration titrated into the electrolyte. After 3, 7, 14 and 28 days the chloride ions extraction was respectively of about 58, 70, 86 and 93 % in the external cover and 40, 81, 94 and 97 % in the first centimetre around the rebar. The extraction was faster during the seven first days of treatment which is in accordance with literature results [3-6]. Results show that the free chloride concentration became less than the EN206 chloride threshold of 0.4 % mass of cement after 7-14 days of treatment.

The extraction of chloride ions decreases with time as the chloride concentration gets slower and therefore the resistance increases.

Regarding non polarised specimens, chlorides extraction efficiency was similar to the one obtained under cathodic polarization from 28 days of immersion.

Fig 9 highlights that there was initially no difference between total and free chloride ion concentrations which means that after carbonation, as electrochemical characterization results suggested, most chlorides were freed. This can also explain the particularly good extraction results obtained during this study even without polarization.

Regarding the pH evolution (Fig 9), observed pH increase was probably due to the hydroxyl ions formation around the rebar and maybe the cations migration towards the rebar. Based on results obtained and the NF EN 14038 part 1 standard [13], concerning required thickness of concrete with basic pH around the rebar, the minimum treatment duration required is either 56 days to obtain a 10 mm realkalised concrete zone around the rebar or around 28 days considering the experimental rebar diameter (5 mm).

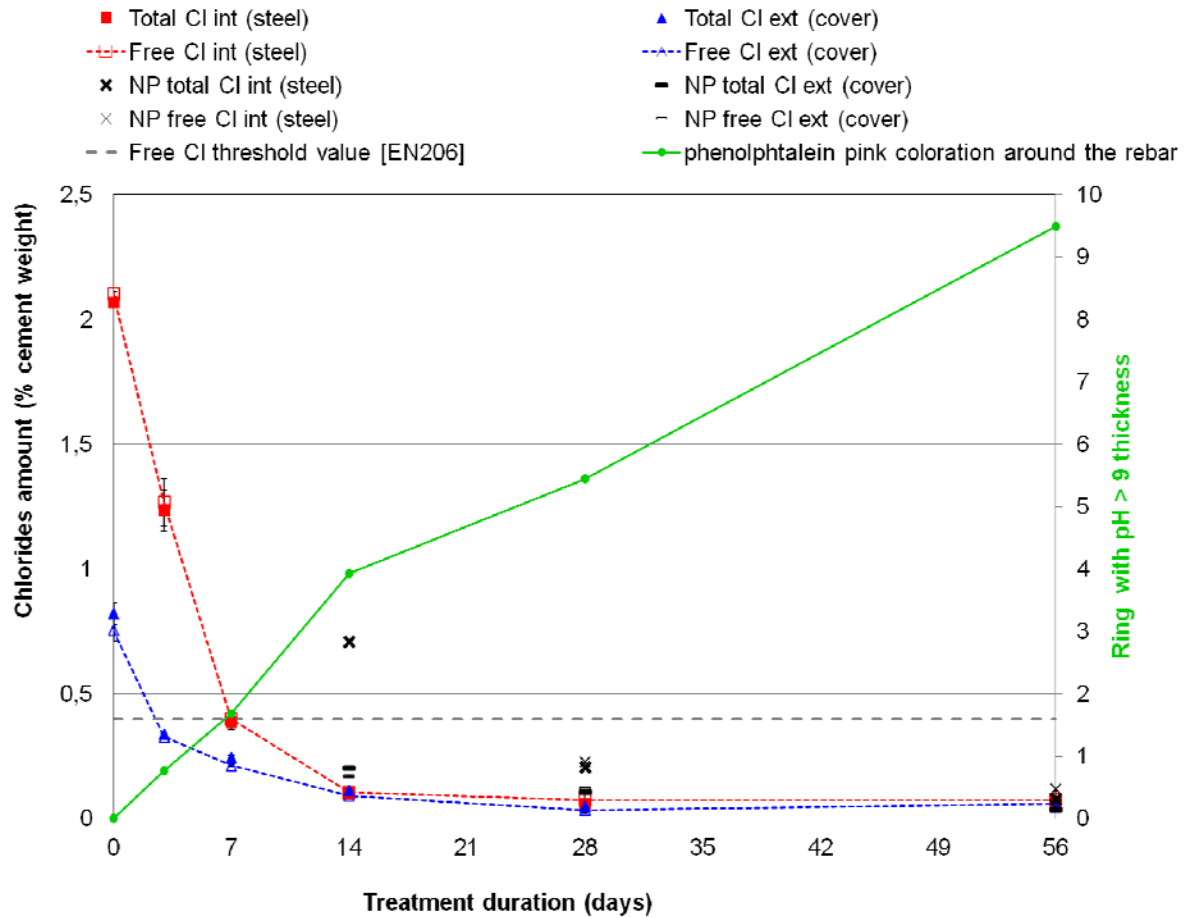


Figure 9: Evolution of chloride concentration and ring with pH>9 thickness during ECE treatment

### Electrochemical characterization

Fig 10 compares previous electrochemical characterization results to those obtained after the electrochemical cathodic treatment (56 days) after 10 weeks of depolarization. These results show a stabilisation of the rebar corrosion state to a negligible corrosion level after treatment with a half-cell potential of  $-161 \pm 19$  mV and a corrosion current of  $0.03 \pm 0.01$   $\mu\text{A}/\text{cm}^2$ . This last is however higher than the corrosion current of a non-contaminated specimen (S). Regarding non polarised specimens, results obtained show that, even with a similar amount of extracted chloride than for polarised specimens, corrosion current is still high. This underline the fact that both chloride extraction and pH increase are needed to stop or at least decrease the corrosion state of the rebar.

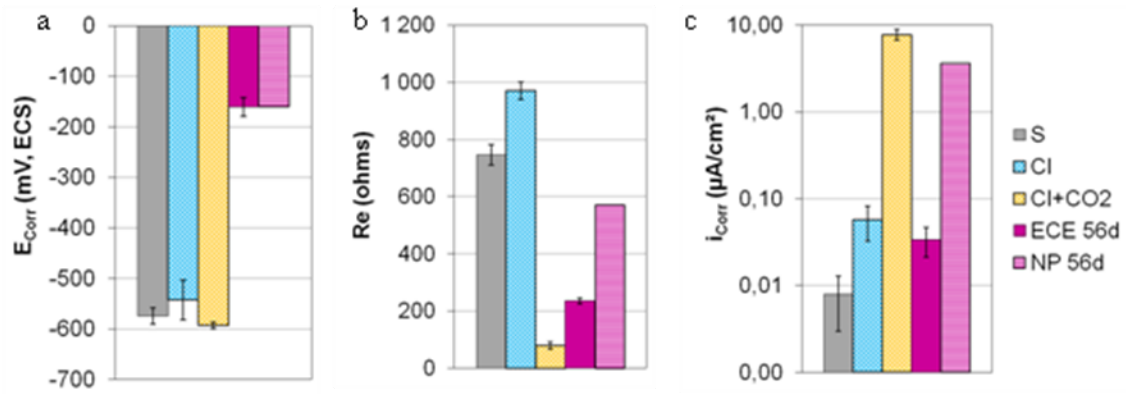


Figure 10. (a) Half-cell potential, (b) resistance and (c) current density of non-contaminated specimens (S) and G3 specimens after curing (Cl), after carbonation (Cl+CO<sub>2</sub>), after electrochemical treatment (ECE 56 days) and after 56 days of immersion (NP 56d)

### Corrosion products characterization by Raman Spectroscopy

During ECE treatment, regular Raman acquisitions (successive the first 5 hours and then every day) were performed to follow the phases transformations induced. After two days of polarization, the main phase detected was magnetite (main Raman band at 670 cm<sup>-1</sup>) (Fig 11). This phase remained stable during 20 days of polarization after which the treatment was ended. No remaining chlorinated green rust was identified at the end of the treatment. Magnetite is a more stable corrosion product which explained the stabilization of the rebar corrosion state obtained by electrochemical characterization. A study of the treatment durability should however be performed.

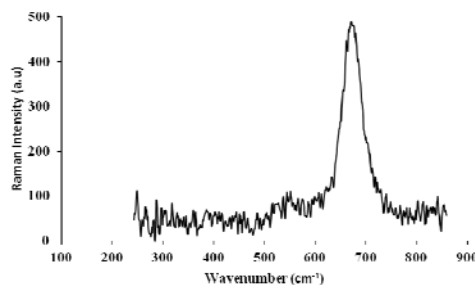


Figure 11 : Magnetite Raman spectrum

### Conclusion

In this experimental work, the set-up design adopted to study, first the corrosion process on both chlorinated and carbonated reinforced concrete specimens, and second, the electrochemical cathodic treatment were presented.

Results obtained on G3 specimens allowed establishing the following conclusions:

- Non-contaminated specimens as well as chloride contaminated G3 specimens showed a negligible corrosion after curing as indicated by corrosion rates less than 0.1  $\mu\text{A}/\text{cm}^2$ .
- After the carbonation process, as expected, corrosion increased rather drastically as evidenced by corrosion rates in the range of 10  $\mu\text{A}/\text{cm}^2$ . SEM results confirmed this behaviour with the observation of corrosion products on most of the interface (5-15

- Efficiency of the electrochemical treatment based on chloride extraction as well as pH increase of the concrete around the  $\mu\text{m}$  corrosion layer on 80-90% of the concrete/steel interface).reinforcement was demonstrated. Two weeks of treatment were needed to obtain a  $\text{Cl}^-$  concentration below the threshold value of 0,4 % mass of cement while 56 days of treatment lead to one centimeter of concrete with  $\text{pH}>9$ . After the end of the treatment the corrosion current was below  $0.1 \mu\text{A}/\text{cm}^2$  corresponding to a negligible level of corrosion which demonstrated that the corrosion was halted.
- Comparison with results obtained on immersed and non polarised specimens showed that both chloride extraction and pH increase are needed to stabilise the corrosion level of the rebar. Indeed, without polarization, the pH did not increase and although a significant amount of chlorides were lixiviated, the corrosion current remained in the high corrosion level.

The following of treatment durability and Raman spectroscopy results will complete the experimental work on G3 specimens. Comparison with G1 specimens [14] and with the two other types of specimens will give a larger view on this subject.

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