Durability of RC structures made with chloride-contaminated raw materials

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Isfahani

Synopsis: Within the SeaCon project "Sustainable concrete using seawater, salt-contaminated aggregates, and non-corrosive reinforcement", financed by the Infravation program, an experimental study is being carried out, aimed at demonstrating the safe utilization of chloride-contaminated raw materials for the production of a sustainable concrete, when combined with non-corrosive reinforcement to construct durable and economical concrete infrastructures. Experimental tests are on going to assess the corrosion behavior of austenitic (S30403 and S24100) and duplex stainless steels (S31803 and S32304) reinforcing bars, and for comparison of carbon steel, embedded in concretes made with chloride-contaminated raw materials and subjected to different environmental conditions. This paper focuses on the results of tests carried out on reinforced concrete slabs exposed to ponding with a 3.5% NaCl solution for approximately one year, in order to simulate the effect of the further penetration of chlorides. Results showed that this condition led, in few days, to the initiation of corrosion on the carbon steel bars embedded in concretes made with chloride-contaminated raw materials. Neither the initial contamination nor the further penetration of chlorides led to the onset of corrosion on any of the stainless steel bars embedded in concrete made with chloride-contaminated materials.

Keywords: carbon steel, chloride contaminated raw-materials, chloride-induced corrosion; durability; seawater, stainless steel, sustainability

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INTRODUCTION

The concrete production requires a huge consumption of natural resources, such as fresh water to mix and cure concrete and virgin rocks for aggregates and Portland cement production, leading to a consistent environmental impact [1]. Hence, there is the need of looking for alternative solutions to minimize this impact.

Several solutions have been proposed to increase the sustainability of the construction industry, such as the use of supplement cementitious materials (SCM) as partial replacement of Portland cement or the use of recycled aggregates as a portion of total aggregates used in concrete [2-6]. These strategies allow to reduce the consumption of virgin rocks, to recycle waste materials that otherwise should be disposed of and to reduce the emission of CO_2 in the environment due to the lower consumption of Portland cement. However, they do not limit the consumption of fresh water, which is of particular concern since in many parts of the world fresh water is a depleting resource. As a matter of fact, the World Meteorological Organization has reported water scarcity and water stress conditions in countries by 2025, due to the increase in world population and the consequent increase of fresh water consumption for human and industrial needs.

Seawater, being available in several parts of the world, could be a valid alternative to reduce the consumption of fresh water in concrete technology; however, presently, its use is prohibited in many countries for reinforced concrete structures. As a matter of fact, the use of seawater may affect some properties, such as the setting time, the workability, the kinetics of hydration of portland cement and, as a result, the early strength and the long-term development of strength of concrete [7]. The main aspect which prohibits the use of seawater as mixing water is its high content of chloride salts which can promote corrosion of conventional reinforcement made of carbon steel. In fact, it is well known that chlorides above a certain threshold may locally destroy the passivity film present on carbon steel rebars embedded in concrete, thus pitting corrosion occurs. For such reason, design standards worldwide aim at preventing the use of chloride-contaminated materials and at limiting the further penetration of chloride during the service life of the structure. The risk of inducing corrosion on the rebar could be limited if seawater, as well as of other chloride-contaminated raw materials, were employed in combination with non-corrosive reinforcement.

Within the framework of ERA-NET Plus Infravation 2014, an infrastructure innovation program on "Advanced systems, materials and techniques for next generation infrastructure" (http://www.infravation.net/), the SEACON project - Sustainable concrete using seawater, salt-contaminated aggregates, and non-corrosive reinforcement (http://seacon.um-sml.com) is being carried out. This project, that involves ten academic and industrial partners and collaborators, both from Italy and US, is aimed at demonstrating the safe utilization of seawater and salt-contaminated aggregates (natural or recycled) for a sustainable concrete production when combined with non-corrosive reinforcement, e.g. stainless steel and GFRP bars, to construct durable and economical concrete infrastructures. Experimental tests are on going to assess the corrosion behavior of austenitic and duplex stainless steels reinforcing bars, and for comparison of carbon steel, embedded in

concretes made with chloride-contaminated raw materials (including seawater) and exposed to different environmental conditions. This paper focuses on the results of tests carried out on reinforced concrete specimens exposed to ponding with a 3.5% NaCl solution for approximately one year.

EXPERIMENTAL PROCEDURE

Concrete slabs made with chloride contaminated raw-materials and reinforced with different types of stainless steel bars and, for comparison, of carbon steel bars were cast, in order to study the effect of the further penetration of chlorides on the corrosion behavior of reinforcement. The slabs were subjected for approximately one year to ponding with a 3.5% NaCl solution and the corrosion behavior of the bars was monitored.

Materials and mixes

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A limestone Portland cement (reference cement), designated according to European standard EN 197 as CEM-II/A-LL 42.5R, and a cement contaminated with chloride ions produced by Buzzi (SeaCon cement), with a composition similar to that of CEM-II/A LL 42.5R, but with the ground limestone replaced by a process dust (containing higher amount of Cl⁻ compared to cement) were used. Fly-ash was added to the cements. De-ionized water as well as natural seawater collected from the Mediterranean sea were employed. As natural aggregate, two siliceous sands (0-2 mm [0-0.08 in.] and 0-4 mm [0-0.16 in.]) and one gravel (4-12.5 mm [0.16-0.49 in.]) were employed, whilst as recycled concrete aggregate (RCA), old chloride-contaminated concrete specimens stored from previous researches (made with Portland cement, *w/c* ratio of 0.5, crushed limestone aggregate and chloride contamination ranging from 1% to 5% by mass of cement) were crushed and sieved to obtain a fraction passing between 4 mm [0.16 in.] and 12.5 mm [0.49 in.].

The raw materials were used to cast five mixes. Mix *Ref* is the reference mix with virgin ingredients (without chloride); mixes *Cem*, *Sea* and *RCA* are mixes contaminated by chlorides respectively from the cement (SeaCon cement), the seawater (used as mixing water) and the RCA (used to partially replace the coarse aggregate). In Mix *Cem-FA*, the SeaCon cement was used and a higher amount of fly ash, which can improve the durability of the concrete by reducing the mobility of the chloride ions, was added. Concrete with water/cement ratio of 0.52 was obtained by mixing 335 kg/m³ [21 pcf] of cement (reference or SeaCon cement), 175 l/m³ [11 pcf] of water (de-ionized water or Sea water), 30 kg/m³ [1.9 pcf] of fly ash (80 kg/m³ [5 pcf] were used for Cem-FA). 1824 kg/m³ [114 pcf] of silicious aggregate were used for mixes *Ref*, *Cem* and *Sea*; 1763 kg/m³ [110 pcf] of silicious aggregate were employed for mix *Cem-FA* and, finally, 1437 kg/m³ [90 pcf] of silicious aggregate and 365 kg/m³ [23 pcf] of RCA aggregate were utilized for mix *RCA*. An acrylic-based superplasticiser was added in order to achieve a class of consistence S4. **Table 1** shows the mix proportions of the concretes, the results of the slump test and the expected chlorides. The expected chlorides ranged from 0.02% mass of cement in the Mix *Ref* to 1.1% vs mass of cement in the *Sea* concrete.

As reinforcement, besides 16 mm [0.63 in.] carbon steel rebars, 16 mm [0.63 in.] stainless steel rebars of grades 304L, 22-05, 23-04 and 20 mm [0.79 in.] XM-28 rebars were considered; these grades were considered on the basis of the results obtained in previous experimental works as well as literature research [8-10]. **Tables 2** and **3** show the mechanical properties and the chemical composition of the steel bars. The stainless steel bars were subjected to commercial sand blasting and pickling, in order to remove the oxide scale produced during hot forming, whilst carbon steel bars were subjected to sand blasting. Before testing all the reinforcement were degreased with acetone.

Specimens

Compressive strength of the different concrete mixes was tested on two plain specimens $100 \times 100 \times 100$ mm³ $[3.9 \times 3.9 \times 3.9 \text{ in.}^3]$. Reinforced $350 \times 120 \times 80$ mm³ $[13.8 \times 4.7 \times 3.1 \text{ in.}^3]$ prism slabs, with a hollow on the upper surface with dimension of $310 \times 80 \times 30$ mm³ $[12.2 \times 3.1 \times 1.2 \text{ in.}^3]$, were cast for ponding tests in order to study the effect of the further penetration of chlorides (Fig. 1). Specimens were reinforced with one bar of each type of steel, with a concrete cover thickness of 10 mm [0.39 in.]. Before casting, the two ends of each bar were masked with a styrene-butadiene-modified cement mortar and coated with a self-amalgamating tape; a length of the bar of 80 mm [3.15 in.] was exposed to the concrete. The slabs were equipped with titanium wires (as internal reference electrode) along the bars and a titanium mesh (as counter-electrode) at the bottom for electrochemical measurements. To account for the variability of the results, the ponding tests were carried out on two replicate slabs made with each concrete mix.

Items of investigation

- After casting, the specimens were covered with a plastic sheet and stored in laboratory at 20°C [68°F]. After 24 hours, they were demoulded and cured at 20°C [68°F] and 95% relative humidity. Compressive strength of concrete was tested after 1, 7 and 28 days of moist curing according to EN 12390-3.
- Reinforced slabs were cured for 7 days, afterwards they were subjected to ponding with a 3.5% NaCl solution for approximately one year in air-conditioned laboratory ($T = 20^{\circ}C$ [68°F]). The ponding solution was monthly

- 1 replaced. During the exposure period, the corrosion behavior of steel embedded in the reinforced slabs was
- 2 monitored by means of electrochemical measurement of half-cell potential of steel (E_{corr}) versus a saturated
- 3 calomel electrode (SCE), placed on the specimen surface in the central part of each bar, and corrosion current
- density (i_{corr}), measured through linear polarization resistance measurements (R_p) as: $i_{corr} = B/(R_p)$, where B was
- 5 assumed equal to 26 mV.

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- 6 After approximately one year of exposure, a sample was cored in the lateral non-reinforced part of the slab and
- 7 the chloride profile was measured by analyzing the total chloride content (acid soluble) of concrete slices at
- 8 depth intervals of 10 mm [0.39 in.].

EXPERIMENTAL RESULTS AND DISCUSSION

Compressive strength of concrete

Fig. 2 reports the average values of compressive strength measured at different curing time on all mixes. Compressive strength increased in time, as a consequence of hydration processes, especially in the first 7 days of curing; higher values in terms of compressive strength were observed for the mixture with an higher fly ash content (*Cem-FA*). A target of 40 MPa [5.8 ksi] was obtained in terms of compressive strength after 7 days of moist curing. Concretes made with chloride-contaminated raw materials did not have any negative influence on the compressive strength with respect to the reference concrete, after the different curing times.

Corrosion of embedded reinforcement

The monitoring of the corrosion potential ($E_{\rm corr}$) and the corrosion current density ($i_{\rm corr}$) of the bars was carried out after the 7 days of moist curing and the subsequent exposure to ponding with a chloride solution. Figs. 3 and 4 show, as examples, the trend in time of the corrosion potential and the corrosion current density of carbon steel bars and stainless steel of grade 304L embedded in the five concrete mixes. In the Figures the color of the symbol indicates the type of concrete, whilst the filling color the replicate specimen. The time was referred to the beginning of exposure for all specimens, to compare measurements taken in the same day and to account possible variations of the corrosion parameters due to variation of exposure conditions. Before the exposure to ponding, the corrosion potential of both carbon steel and 304L stainless steel bars, embedded in the different concretes, was between -250 mV and -150 mV vs SCE, suggesting that the bars were in passive conditions, in spite of the presence of chlorides in the raw materials used for casting. Only the carbon steel bars embedded in the *Sea* concrete showed corrosion potential lower than -350 mV vs SCE and corrosion current density around 4-5 mA/m².

In the first days after the beginning of the ponding, a slight increase of the corrosion potential and a decrease of the corrosion current density occurred on all the bars in all the mixes, except the carbon steel bars in *Sea* concrete in which the potential remained around -300 mV vs SCE.

In time, due to ponding with the NaCl solution, chloride penetration took place through the concrete cover. In

In time, due to ponding with the NaCl solution, chloride penetration took place through the concrete cover. In Fig. 3, it can be observed that after only 34 days of ponding the potential of the carbon steel bar in the Ref concrete dropped to about -300 mV vs SCE and the corrosion current density increased up to about 2.5 mA/m² (grey triangular symbol in Fig 3b). The observed sharp decrease in the corrosion potential and the increase in corrosion current density indicated that corrosion initiated on this bar. After about 76 days from the beginning of the ponding, corrosion initiated also on the carbon steel bar embedded in the replicate Ref concrete. In time, corrosion occurred also on the carbon steel bars embedded in the two Cem, Cem-FA and RCA replicate slabs (on one replicate slab made with Cem-FA corrosion occurred after approximately one year of exposure, indicating the higher resistance to chloride penetration of this concrete). After about one year of exposure, the carbon steel bars embedded in the concretes Ref, Cem, Cem-FA and RCA had a corrosion potential in the range of -500/-400 mV vs SCE and a corrosion current density around 10 mA/m² (potential values around -300 mV vs SCE were measured on one of the Cem-FA replicate slab). Even the carbon steel bars embedded in the Sea concrete experienced a drop, after about 50 days from the beginning of ponding, in the corrosion potential and an increase in the corrosion current density. From the initial values around -300 mV vs SCE for the corrosion potentials and of 2-3 mA/m² for the corrosion current density, the potentials decreased to values lower than -400 mV vs SCE and the corrosion current densities increase to values higher than 10 mA/m². It might indicate that, although corrosion initiated before the exposure to ponding, the further penetration of chloride contributed to

As far as the 304L stainless steel bars are concerned, in Fig. 4 it can be observed that both the corrosion potential and the corrosion current density, except in the initial exposure period, remained constant during the exposure period in all the mixes. Fewer fluctuations were observed and among the replicate slabs no significant differences in corrosion potential and corrosion current density were detected. The corrosion potential was around -100 mV vs SCE after about one year of exposure, whilst the corrosion current density was lower than 1 mA/m². In spite of the further penetration of chloride in time due to the ponding with NaCl solution and the expected increase of the amount of the chlorides near the bars, corrosion did not occur on the 304L stainless steel rebars, indicating that in the nearby of the bars the critical chloride content was not yet reached.

Fig. 5 summarizes the average values and the interval of variability of corrosion potential and corrosion current density measured on all the specimens during approximately one year of exposure to ponding with a 35g/l sodium chloride solution. Only for carbon steel bars, which is the only type of steel which experienced the onset of corrosion, the average values in terms of corrosion potential and corrosion current density calculated in steady conditions before and after corrosion initiation has been distinguished in the histogram. By considering that in the first days of exposure a slight increase of the potential and a slight decrease of the corrosion current density were observed in all types of concrete (i.e before reaching steady values), in the calculation of average, minimum and maximum values, such measurements were not considered. The average values showed in the Figure were calculated from the average values obtained between the replicate slabs, since no significant differences in corrosion potential and corrosion current density were observed between them.

As previously observed, corrosion occurred on all the carbon steel bars and the initiation time varied from about 30 to 120 days, except on one bar embedded in the *Cem-FA* concrete, where corrosion occurred after about 1 year. Before the occurrence of corrosion, the corrosion potential and the corrosion current density were respectively around -100 mV vs SCE and 1 mA/m², regardless the type of concrete (carbon steel bars in *Sea* concrete were an exception). After the occurrence of corrosion for carbon steel bars, potential values were lower than -400 mV vs SCE and the corrosion current densities were higher than 10 mA/m². None of the stainless steel bars showed initiation of corrosion during the exposure to ponding. No significant variation can be observed among the different grades of stainless steel embedded in the different mixes. The corrosion current densities of the duplex stainless steels, i.e. 22-05 and 23-04, were higher than those of the austenitic stainless steel, i.e. 304L and XM-28. However, the high values of current density did not indicate the initiation of corrosion as confirmed by the corrosion potential values (the values measured from the beginning of exposure in concretes made with chloride-contaminated raw materials were higher than -200 mV vs SCE, as well as that on the bars embedded in the *Ref* concrete), and might be due to the surface conditions of the tested bars.

Chloride profiles

After approximately one year of exposure to the ponding, a core was taken from all the slabs and the chloride content at interval depths of 10 mm was measured. Fig. 6 shows the chlorides profiles evaluated on all the slabs: comparable results between the two replicate slabs, i.e. between the two replicate mixes, were obtained. In the Figure the vertical dotted lines represent the bars depth (except for XM-28, that have an higher diameter). For Sea concretes the amount of chlorides was almost constant through all the depth of the slabs and, excluding the first layer, it was between 1.2 and 1.8% vs mass of cement. For this type of concrete, the presence of an initial chloride content around 1% vs mass of cement, and hence of a limited concentration difference between the solution and the concrete, led to a poor driving force for chloride diffusion. For the other mixes the chlorides content decreased increasing the depth and, at each depth, the highest values were measured on the RCA mixes, whilst the lowest on the Cem-FA concretes, confirming that an higher amount of fly ash led to a lower chloride penetration. From Fig. 6 a rough estimation of the chloride content at the bars depth, after 1 year of exposure, can be determined. Considering that the bars are at a depth of 10-26 mm [0.39-1 in.], the value measured at the interval depth of 10-20 mm [0.39-0.79 in.], can be considered representative of the average chloride content. It can be observed that the average chloride content was in the range 1.24-2.9% vs mass of cement for all the different mixes (the lowest value was measured on the Cem-FA concrete, whilst the highest value was measured on the RCA concrete). Certainly, highest chloride contents were present in the upper part of the bars (it can be estimated that these values were lower than 1.8-3.5% vs mass of cement, which are the chloride contents measured in the first layers). For the stainless steel bars considered in this work, since corrosion did not occurred yet on any of the bar, these values can be considered lower than the critical chloride threshold for pitting corrosion initiation. Hence, on the basis of the results obtained on the reinforced slabs, where bars were embedded at a depth of 10 mm, exposed to ponding with a chloride solution for one year, the stainless steel bars used in this work, even the less alloyed, seemed to be suitable in combination with concrete made with chloride contaminated raw materials and exposed to the further chloride penetration. Conversely, as expected, carbon steel bars are not suitable to be used in chloride contaminated environments, even when embedded in chloridefree concrete made with a limestone Portland cement and a water/cement ratio of 0.52.

FURTHER RESEARCH

Since the duration of the tests, i.e. 1 year, was significantly lower than the required service life of a real reinforced concrete structure, it will be useful to prolong these ponding tests until the expected chloride concentration at the end of the service life of a real RC structure will be reached at the depth of the bars. An estimation of the chloride content which can be reached at the depth of the bar in a real reinforced concrete structure could be made by means of performance-based approaches for the service life design (e.g. the "Model Code for Service Life Design", published by the International Federation for Structural Concrete (*fib*) [11]). Furthermore, since the occurrence of corrosion is affected by the exposure conditions (e.g. temperature) it would be also interesting to evaluate the corrosion resistance of the different types of stainless steel in different

environmental conditions, in order to detect the suitable combinations of stainless steel and type of concrete that can be employed in the different scenarios.

3 CONCLUSIONS

- Tests aimed at studying the role of chloride-contaminated raw materials on the concrete compressive strength showed that, in the case of the mixes studied in this work, chloride contamination did not significantly affect the strength at the different curing ages.
- Corrosion tests on reinforced concrete slabs exposed to ponding with a 3.5% NaCl solution showed that the further penetration of chloride ions led to the initiation of corrosion on the carbon steel bars embedded in the
- 9 different mixes suggesting that the initial chloride content, due to the use of chloride contaminated raw
- materials, was only slightly lower to the chloride content which promotes the initiation of corrosion in the
- studied exposure conditions. The further penetration of chloride did not lead to the onset of corrosion on any of the stainless steel bars embedded in concrete made with chloride contaminated materials and the chloride
- 13 content detected after 1 year of exposure at the bar depths can be considered as a minimum value for the critical
- 1.5 content detected after 1 year of exposure at the bar depths can be considered as a minimum value for the critical
- chloride threshold. These tests are still ongoing to reach, at the bar depths, the chloride content expected during
- the design service life of a real structure.

16 ACKNOWLEDGMENTS

- 17 The project was made possible with the financial support received from the Infravation Program under Grant
- 18 Agreement No. 31109806.005-SEACON. The opinions in this paper are those of the authors and not necessarily
- 19 those of the sponsors or collaborators.

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TABLES AND FIGURES

Table 1–Composition of the five concrete mixes

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	Ref	Cem	Sea	RCA	Cem-FA
reference cement, kg/m³ (pcf)	335 (21)	-	335 (21)	335 (21)	-
seacon cement, kg/m³ (pcf)	-	335 (21)	-	-	335 (21)
fresh water, kg/m ³ (pcf)	175 (11)	175 (11)	-	175 (11)	175 (11)
sea water, kg/m ³ (pcf)	-	-	175 (11)	-	-
fly ash, kg/m ³ (pcf)	30(2)	30(2)	30 (2)	30 (2)	80 (5)
0-2 mm sand, kg/m ³ (pcf)	490 (31)	490 (31)	490 (31)	484 (31)	473 (29)
0-4 mm sand, kg/m ³ (pcf)	596 (37)	596 (37)	596 (37)	588 (37)	576 (36)
gravel, kg/m³ (pcf)	739 (46)	739 (46)	739 (46)	365 (23)	714 (45)
RCA, kg/m ³ (pcf)	-	-	-	365 (23)	-
<i>w/c</i> ratio	0.52	0.52	0.52	0.52	0.52
w/b ratio	0.48	0.48	0.48	0.48	0.42
slump, mm (in.)	200 (8)	185 (7)	185 (7)	200 (8)	210 (8)
expected chlorides (% mass vs cem)	0.02	0.23	1.1	0.78	0.23

Table 2—Mechanical properties (U.S. = ultimate strength; $Y.S._{0.2\%} =$ yield strength at 0.2% of deformation; $A_{gt} =$ total elongation at maximum force) and main alloy elements of the stainless steel bars

Steel	Designa	Mechanical properties			Main alloy elements (%)							
name	EN 10088-1	UNS	U.S., MPa (ksi)	Y.S. _{0.2%} MPa (ksi)	$A_{ m gt} \ (\%)$	С	Cr	Ni	Мо	Mn	N	
304L	1.4307	S30403	760 (110)	543 (79)	24	0.021	18.51	8.61	0.15	1.31	0.146	
22-05	1.4462	S31803	838 (122)	607 (88)	24	0.021	22.56	5.26	3.332	1.48	0.158	
23-04	1.4362	S32304	750 (109)	524 (76)	18	0.019	23.37	4.27	0.30	1.23	0.095	
XM-28	-	S24100	923 (134)	672 (97)	-	0.11	17.41	2.23	0.18	12.02	0.3	

Table 3–Mechanical properties (U.S. = ultimate strength; $Y.S._{0.2\%} =$ yield strength at 0.2% of deformation; $A_{gt} =$ total elongation at maximum force) and main alloy elements of the carbon steel bars

Steel	Designation	Mechai	Main alloy elements (%)												
name	EN 10027-1	U.S., MPa (ksi)	Y.S. _{0.2%} MPa (ksi)	$A_{ m gt} \ (\%)$	C	Mn	Си	Si	Ni	Cr	S	Мо	P	V	N
С	B450	590 (86)	540 (78)	10.9	0.145	0.87	0.55	0.14	0.16	0.10	0.042	0.04	0.017	0.001	0.009

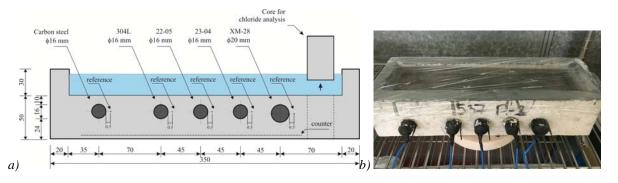


Fig. 1–Geometry (a) and photo (b) of reinforced slab for ponding tests (dimensions in mm [1 mm = 0.039 in.])

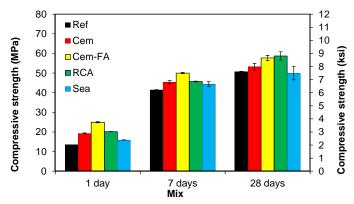


Fig. 2—Compressive strength (average value of two replicate specimens) and interval of variability of concretes after different curing time in moist conditions

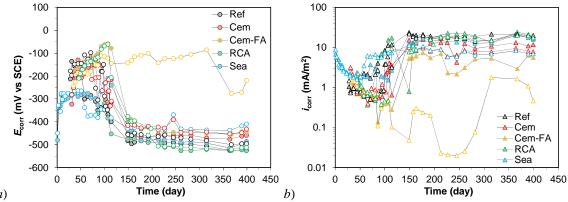


Fig. 3–Evolution in time of the corrosion potential (a) and corrosion current density (b) of carbon steel bars embedded in the different concretes and exposed to ponding with a 3.5% NaCl solution (filled symbols indicate the replicate specimens)

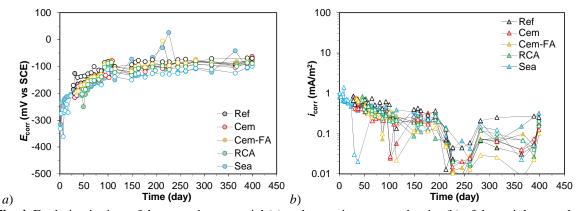


Fig. 4—Evolution in time of the corrosion potential (a) and corrosion current density (b) of the stainless steel of grade 304L embedded in the different concretes and exposed to ponding with a 3.5% NaCl solution (filled symbols indicate the replicate specimens)

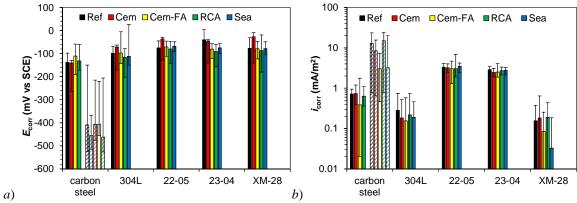


Fig. 5–Average values and interval of variability of corrosion potential (a) and of corrosion current density (b) of the different types of bars embedded in the different concretes before (full fill) and after (texture fill) corrosion initiation.

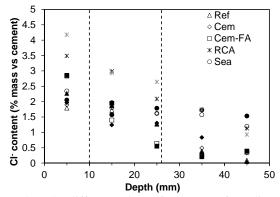


Fig. 6–Chloride profiles measured on the different mixes after 1 year of ponding exposure (dotted lines indicate the bars depth, except for XM-28) [1 mm = 0.039 in.]