

Effect of Surface Coatings in the Corrosion of Reinforced Concrete in Acid Environments

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This study involves the protective effect of organic coatings used on reinforced concrete in an acid environment similar to those existing in certain metallurgic processes, in which the concrete degradation and the steel corrosion cause significant costs to the metal production. A 0.50 w/c concrete was prepared and then characterized through physical and mechanical tests such as electrical resistance, capillary absorption, total absorption, total porosity, chloride permeability, and tensile and compressive strength. The uncoated, acrylic, and epoxy coated concrete cylinders were exposed to an artificial acidic solution for 589 days. The corrosion of the steel reinforcement was evaluated by means of corrosion potential and polarization resistance. The morphology of the corrosive attack was observed using scanning electron microscopy (SEM) and the composition of the corrosion products was determined using an X-ray diffractometer (XRD). The deterioration of the concrete and its level of contamination were evaluated by measuring the concentration profiles of chloride, acidity and sulfate. The results show that the reinforcing steel reached an active state in the uncoated concrete and in the acrylic coated concrete, whereas it remained in a passive state in the epoxy coated concrete. Under the conditions established in this study and based on chemical analyses and on the electrochemical tests, it was determined that the coating which performed better as a barrier in an acidic medium was the epoxy coating.

Keywords: corrosion, concrete, reinforcing steel, sulfuric acid, coatings.

1. INTRODUCTION

An important aspect of the organic coatings in concrete is that they may increase the service life of concrete structural elements [1]. However, the possibility to use coatings over the steel is possible in

reinforced structures only they are not exposed to earthquakes, because of the problems in chemical adherence [2-4].

Certain organic coatings, combined with the concrete cover, protect the reinforced concrete's steel by creating a physical barrier against the aggressive media. Moreover, the aqueous environment found inside the concrete acts as an electrolyte solution, defined mainly by OH⁻ ions, producing a highly alkaline environment. When in contact with the normal content of O₂ in the air (through the exposed structure), this creates a strongly adhesive, compact and invisible layer, protecting the steel bars from corrosion. Such phenomenon will occur as long as a high quality concrete is used and its physical-chemical characteristics do not vary because of the action of external environment conditions [4-10].

In an aggressive environment the service life of a reinforced concrete structure is reduced, showing premature deterioration due to reactions with aggressive substances. In general, the durability of concretes is related to the type of cement, their mechanical properties and resistance to local climate, chemical attacks and abrasion [11-13].

In relation with chemical attacks to reinforced concrete, studies carried out by different groups show that the most common cause of activity of reinforcing steel is the chloride ions diffusion, reaching the steel-concrete interface [2,14-17]. Dehwah and Obserholster reported that the diffusion of chloride ions is generally 10 to 100 times faster than that of sulfates. [18-19].

Nevertheless the presence of sulfate ions in concrete structures can become hazardous, due to the effect of a combination with the calcium aluminates hydrates of the Portland cement clinker. This reaction can cause a volume increase up to 250%, specially in humid environments, by producing ettringite ($3\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot 3\text{CaSO}_4\cdot 32\text{H}_2\text{O}$) or thaumasite ($\text{Ca}_6\text{Si}_2(\text{CO}_3)_2(\text{SO}_4)_2(\text{OH})_{12}\cdot 24\text{H}_2\text{O}$), an expansive salt. Internal tensional stresses derived from this expansion process can not be absorbed by the material, forming a series of cracks and delaminations on the concrete cover [20-23].

The presence of sulfate ions and low pH represent the most aggressive conditions for concrete structures. Studies on the performance of concrete in acid solutions (H₂SO₄, HNO₃, HCl) from industrial waters have shown a large and fast concrete deterioration, due to the transport of soluble calcium salts away from the cement matrix and, thus, changing the concrete pore solution pH. In this case, the corrosive action impact will depend on the quantity and concentration of acid acting on the concrete, which might cause complete disintegration of the material under these conditions [20-24].

Zhou et al. [25] indicated that there is no formation of expansive salts (ettringite and/or thaumasite) in the degradation of concrete, when under the low pH environment of sulfuric acid. This, compared to the salts found in concrete without acid attack. Instead, the observed degradation is caused by the neutralization reaction between the acid and the alkaline substances in the concrete.

Similarly, Fattuhi et al. [26] studied the effects of sulfuric acid on concrete cylinders with different quantities of cement; this showed that the lower the quantity of cement, the lesser the effect of the acid in the concrete. Fattuhi et al. [27] also analyzed the effect of applying different protective surface coatings to concrete, concluding that the best protection was obtained from a coating prepared from a mixture of styrene and butadiene. However, the resulting protection is only effective for a limited period (50 days).

Zenonas realized a modeling of corrosion protection for reinforced concrete structures with surface coatings [17] and Aguiar [1] conclude that epoxy coating has the best performance against acid media.

Currently, most copper mining companies show considerable damages in concrete slabs, caused by the overflow of acidic solutions from their chemical processes. Accordingly, the protection of concrete using organic surface coatings appears as an alternative to diminish this degradation problem.

In order to accomplish a solution to this problem, the research carried out in this paper studies the physico-chemical properties of reinforced concrete cylinders, made of pozzolanic Portland cement covered with acrylic and epoxy coatings. This, by analyzing its performance against the action of sulfuric acid in similar concentration levels to those found in industrial processes.

2. EXPERIMENTAL PROCEDURE

2.1. Materials

The studies were carried out using 10 x 20 cm concrete cylinders with and without reinforcement. The reinforced concrete cylinders were fabricated using a corrugated steel bar, (ASTM A36/A36M-08) where the steel composition is Fe with C: 0.17-0.27 max., P 0.04 max., Si 0.30 max., S 0.05 max., Cu 0.2 max.; with an elastic limit of 280 MPa and a tensile strength of 440 MPa. The steel bar had 1 cm in diameter, 16 cm long with an exposed area of 33.93 cm², located 2.5 cm from the edge of the cylinder.

The concrete (nominal 25 MPa compressive strength) was prepared with pozzolanic Portland cement (composition shown in Table 1) and a 0.50 w/c ratio (content shown in Table 2), in accordance to ASTM C31/C31M-08 [28] standard. The concrete involved in this research used a 0.50 w/c ratio, similar to that used in copper refining processes in Chile, instead of the regular 0.70 w/c ratio, due to local conditions related to earthquake stresses and requirements of Chilean Standard (NCh 170 Of. 85) [29].

The cylinders were cured in a humidity chamber (H.R.90-100% and T°17-23 °C) for 28 days, in accordance to ASTM C 172-08 [30] and ASTM C143/143M-08 [31] standards.

Table 1. Cement composition (%).

Compound	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	Na ₂ O	K ₂ O	SO ₃
Content, %	21.5	4.6	3.3	62.0	2.7	0.2	0.4	2.2
Compound	P ₂ O ₅	TiO ₂	P.I.	Free Cal	C ₃ S	C _s S	C ₄ AF	C ₃ A
Content, %	0.09	0.30	2.8	0.50	66.0	16.0	11.1	6.6

Table 2. Content by m³ of concrete.

	Content of 1 m ³ of concrete			
Mixture	Portland Pozzolanic Cement H25 / Kg	Water / L	Gravel / Kg	Sand / Kg
w/c: 0.50	308	154	1039	959

Before painting the concrete cylinders, they were cleaned to eliminate any surface contamination, keeping the roughness level needed and increasing the net surface area to ensure an adequate adhesion between coating and concrete. The paints used were an epoxy resin (bisphenol A + epichlorhydrine) with a polyamine aliphatic hardener and an acrylic resin (Table 3). The thickness of the paint coating was $50 \pm 0.3 \mu\text{m}$. Three similar samples were used for each test condition.

Table 3. Characteristics of the coatings.

Characteristics	Epoxy resin	Acrylic resin
Resin	Bisphenol A + epichlorhydrine	Mono component
Hardener	Polyamine aliphatic	----
Density, (Kg/dm ³)	1.5	0.9
Solid content, (%)	100	23
Adherence to concrete	Very good	Good
Chemical attack resistance	Maxim	Medium
Consumption by layer, (g/m ²)	750	200
Colour	Green	Uncolored

2.2. Methods

Concrete compressive strength (ASTM C39), indirect tensile strength (ASTM C496), electrical resistivity, total water absorption, % total void content (ASTM C642), capillary absorption (Fagerlund method) and chloride permeability (ASTM C1202) were evaluated.

All cylinders were immersed in a solution containing 180 g/L H₂SO₄, 70 ppm Cl⁻ and pH 1.5 because this composition is used in copper metallurgic processes. Exposure included 5-day wet and 2-day dry cycles throughout the duration of the study.

Steel reinforcement electrochemical measurements were evaluated using open-circuit corrosion potential (OCP) and linear polarization resistance (LPR), with an anodic scanning direction of ± 10 mV respect to the OCP at a scan rate 0.1 mVs⁻¹. This was carried out using a Potentiostat-Galvanostat Gsec 2.0, with an external copper/copper sulfate reference electrode (Cu/CuSO₄ saturated), and a

copper sheet as the counter electrode. The electrochemical system used was reported by the authors in a previous works [22].

The morphology of the corrosive attack was observed using the JEOL JSM-5410 scanning electron microscope, coupled to an EDX 9100 energy dispersive spectrometer, and the identification of the corrosion products obtained on the steel was performed using the X'PERT PRO of PANalytical diffractometer analysis using a CuK α radiation and a pyrolytic graphite monochromator. The equipment power was 40mA and 40kV in grazing beam, using an angle of incidence of 1 degree and a nickel filter. The rebar with the corrosion products was taken in an oxygen free chamber to avoid formation of other oxides present at the steel/concrete interface before opening the sample to the environment. The analysis of steel corrosion products was made at the middle of the rebar.

The samples were processed at the end of the exposure period to determine the pH profile, the free chlorides and sulfates concentrations, as a function of the concrete depth. The pH quantitative determination was done by an aqueous suspension, from pulverized concrete, obtained from cut slices of unreinforced cylinder in accordance to ASTM 1218/C1218M-99 [32] and a Corning Acholar 425 pH meter. The determination of free chlorides was carried out on pulverized concrete using the Volhard volumetric method, according to the UNE standard 112-010-94. The determination of sulfates was done via gravimetry according to the Spanish standards for clay: UNE 370-75 and UNE 7131. The concrete cylinders were evaluated after 56 and 589 days (1.6 years).

3. RESULTS AND DISCUSSION

3.1. Physico-mechanical characterization of the concrete specimens

The physico-mechanical characterization of the concrete is shown in Table 4 and it represents the values established for a 0.50 w/c ratio, which allows a diffusion of the aggressive elements. The values for the mechanical and physico-chemical properties of the designed concrete are those normally corresponding to a H25 concrete (25 MPa of compressive strength).

Table 4. Physical–Mechanical Properties of the Concrete.

Property	Mixture
Compressive strength, (MPa)	22.4
Tensile strength, (MPa)	1.7
Total Absorption, (%)	3.4
Total Porosity, (%)	13.8
Effective Porosity, (%)	13.1
Capillary Absorption, (Kg/m ² s ^{1/2})	2.7 x 10 ⁻²
Resistance to water penetration, m (s/m ²)	1.1 x 10 ⁸
Capillary Sorption, S (m / s ^{1/2})	2.1 x 10 ⁻⁴
Chloride Permeability, (Coul)	6910

3.2. Surface appearance of the coated and uncoated concrete

The initial surface appearance of the cylinders and the appearance after a 56 day testing time in an industrial acidic medium are shown in Figure 1. The uncoated concrete showed a clear deterioration, with weight loss and holes due to superficial material loss, but the deterioration degree of the acrylic coated concrete was lower and there was no apparent change in the epoxy coated concrete. However, after a 589 day exposure to the medium, the acrylic coating showed localized deterioration with surface delaminations and 33% of debonded coating. A lower deterioration was observed in the epoxy coated concrete with 2% of debonded coating. The uncoated specimens showed the highest degradation with intense concrete loss after 589 days.

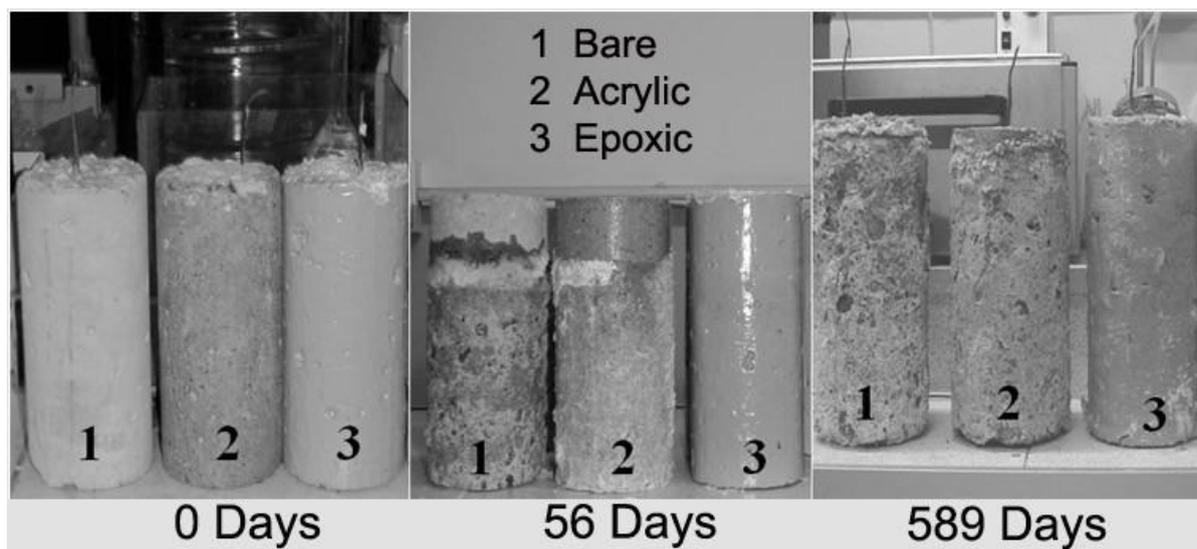


Figure 1. Surface appearance of concrete specimens at different exposure periods.

3.3. Chemical characterization of exposed concrete samples

Once the exposure period ended, slice sections of the specimens were pulverized and then chemically analyzed. The pH profile, chloride, and sulfate content were analyzed, in order to find a relationship between the level of attack on the steel and the formation of the corrosion products.

3.3.1. pH profile

Figure 2 shows the pH variation (acidity) as a function of the concrete depth for the different samples. After a 56-day exposure, all samples were a basic pH close to 12 in the medium similar to the initial concrete condition, because the Chilean Pozzolanic Portland cement used, has low quantity of Na_2O and K_2O compare with OPC, moreover, variability of composition is permitted. In this study, the initial condition of the concrete was pH 12.

After 589-day exposure, uncoated concrete cylinders and acrylic coated cylinders had pH values next to 6. On the other hand, in the epoxy coated concrete, the pH around the steel reinforcement remained basic (pH 10).

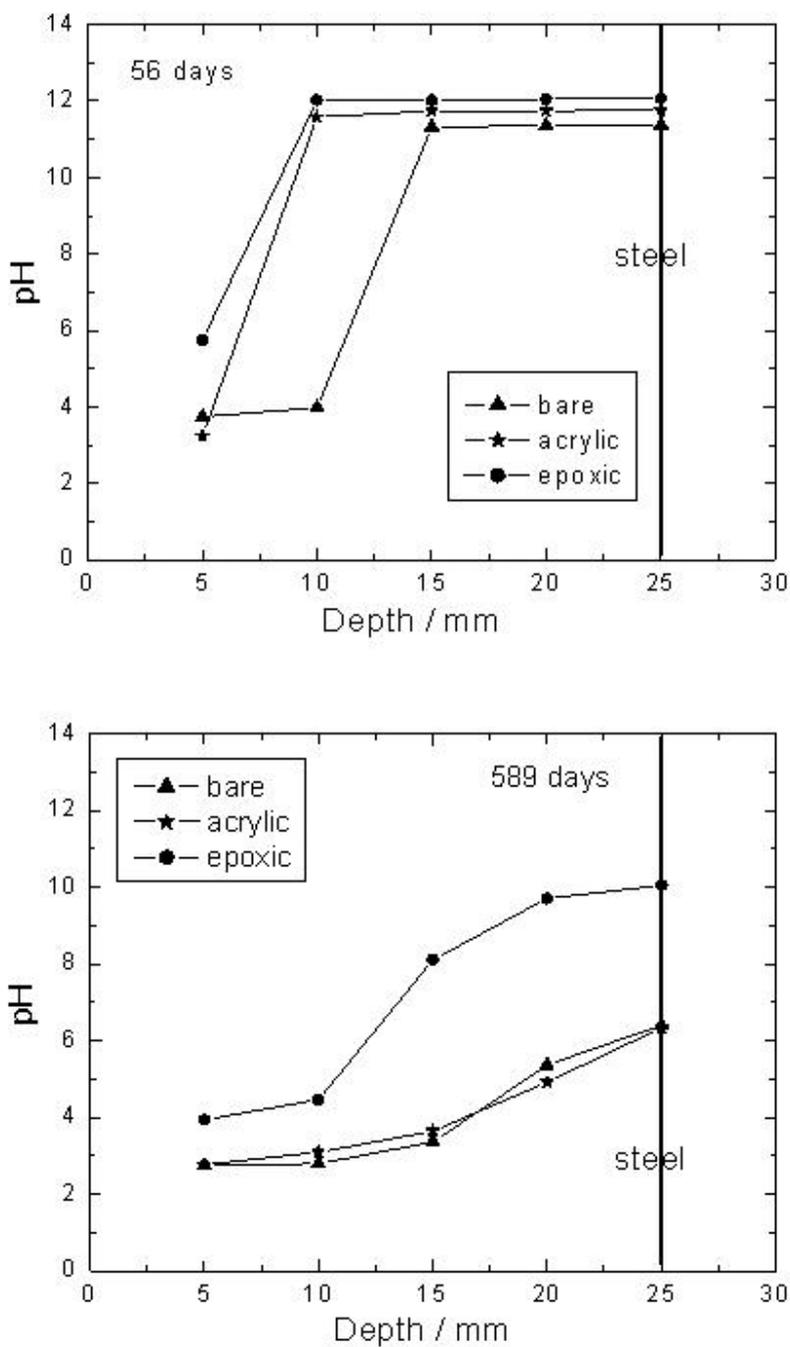


Figure 2. pH as a function of concrete depth.

The acrylic coating does not show protective characteristics, as its performance is quite similar to that observed in the uncoated concrete in relation to the penetration of hydrogen ions to the steel surface.

The development of superficial crystallized salts was observed in the uncoated cylinders, which corresponds to $\text{CaSO}_4 \times 2\text{H}_2\text{O}$, analyzed qualitatively with BaCl_2 solution, coincident with the results of Zhou et al. [22], because they found that calcite or calcium carbonate (CaCO_3) formed on the concrete surface is replaced by $\text{CaSO}_4 \times 2\text{H}_2\text{O}$ in presence of sulfuric acid, in accordance with equation 1.



Moreover, the presence of sulfuric acid in the medium causes degradation in the concrete surface due to neutralization reactions (eq. 2) observed in the hydroxides formed from the reaction of the cement with water, thus causing the formation of calcium sulfate dehydrate or gypsum.



3.3.2. Profile of sulfate content

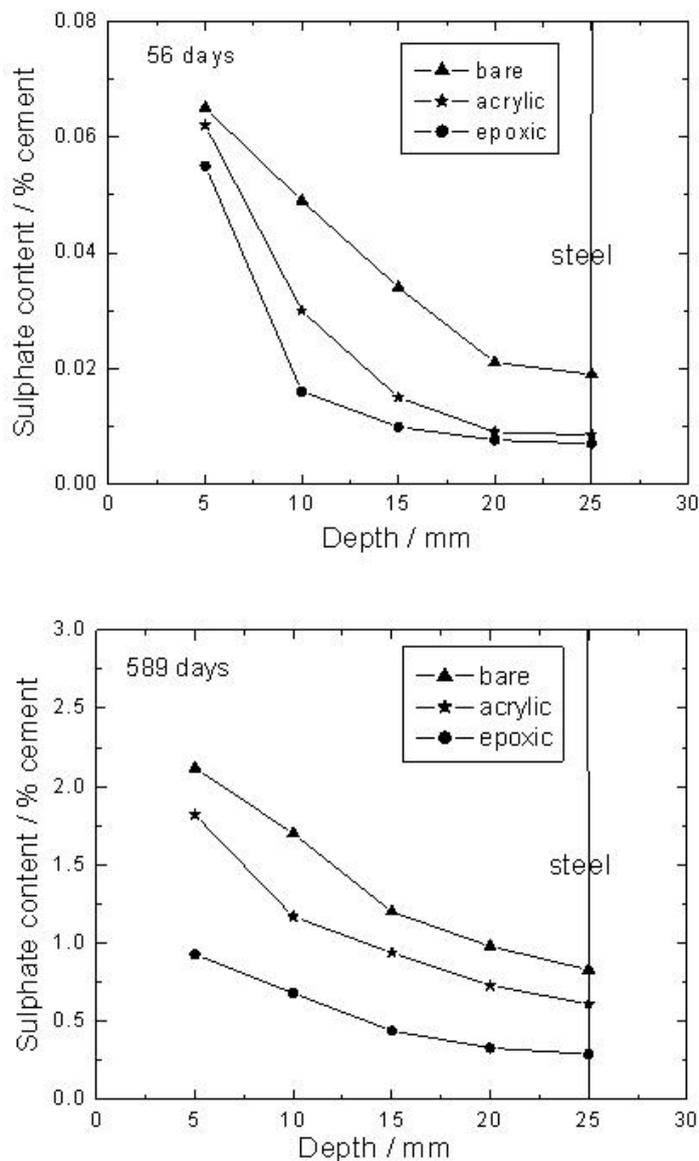


Figure 3. Variation in the content of sulfates as a function of concrete depth.

Figure 3 shows the variation in the sulfate (SO₄²⁻) content by weight of cement as a function of the concrete depth for the different cylinders. The results show that the concrete was slightly contaminated with the sulfate ion after a 56-day exposure, with an external value of 0.07 % and a 0.01 % value at the steel/concrete interface depth.

On the other hand, quantities of 2.5 % (kg SO₄²⁻/kg cement) after a 589-day exposure is shown in Figure 3, corresponding to 7.5 kg SO₄²⁻/ m³ concrete. According to this, the uncoated concrete under laboratory conditions is more exposed to be attacked by this type of ion, as is shown in figure 4A.

Additionally, the action of the sulfates in the concrete does not only depend on the concentration of sulfate ions but also on the positive ion of the sulfate salt. Therefore, having detected the presence of gypsum, the calcium sulfate may act on the hydrated calcium aluminate to form a hydrated calcium sulfoaluminate complex (ettringite), as in the following reaction:



In general, the formation of ettringite takes place in a strongly expansive way, due to the significant volume increase involved in its formation and as it causes crystallization due to the incorporation of 25 water molecules (Figure 4B).

Moreover, Table 5 shows the diffusion coefficients for the sulfate ion calculated from the data presented in Figure 3. The values obtained decrease as exposure time and also in relation to the protective coating.

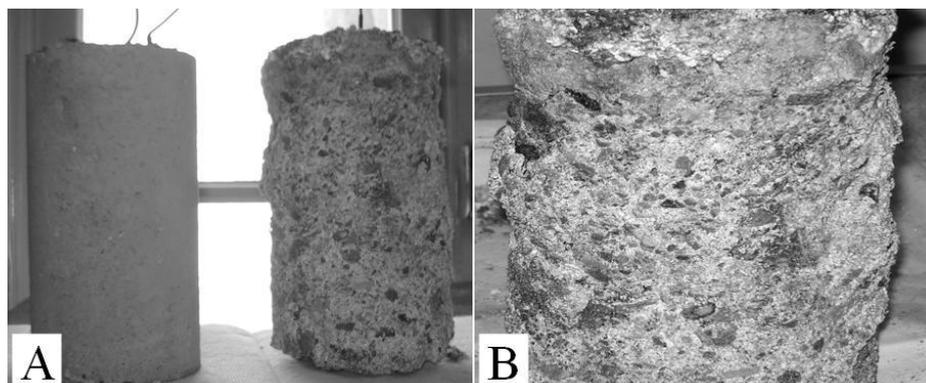


Figure 4. Surface appearance of concrete samples after 589 days. (A) exposed and unexposed sample (B) enlargement of exposed sample

Table 5. Diffusion coefficient of sulfate ion in the samples.

Sample	Diffusion coefficient of sulfate ion D (m ² s ⁻¹)	
	56 days	589 days
Bare	37.3 x 10 ⁻¹²	5.6 x 10 ⁻¹²
Acrylic	12.5 x 10 ⁻¹²	4.4 x 10 ⁻¹²
Epoxic	6.4 x 10 ⁻¹²	3.7 x 10 ⁻¹²

3.3.3. Profile of free chloride content

In Figure 5 the penetration profile of free chloride ions through the concrete is shown. There is a general agreement among researchers concerning that the threshold limit for chloride concentration changing the steel passivity is approximately 0.4% (4000 ppm) by weight of cement [4-7]. As it can be observed in figure 5, the chloride content close to the reinforcing steel in the uncoated concrete is low (0.004%), thus proving the passive state of the steel after 56 days. The analysis also shows minimal chloride content levels for the painted cylinders.

On the other hand, the chloride content near the reinforcement reaches 0.03 % for the uncoated concrete, and around 0.01% for the coated concrete after 589 days of exposure (Fig. 5). These results are in accordance with the low chloride content in the testing solution; therefore, chlorides would not be responsible for the corrosion of the reinforcement.

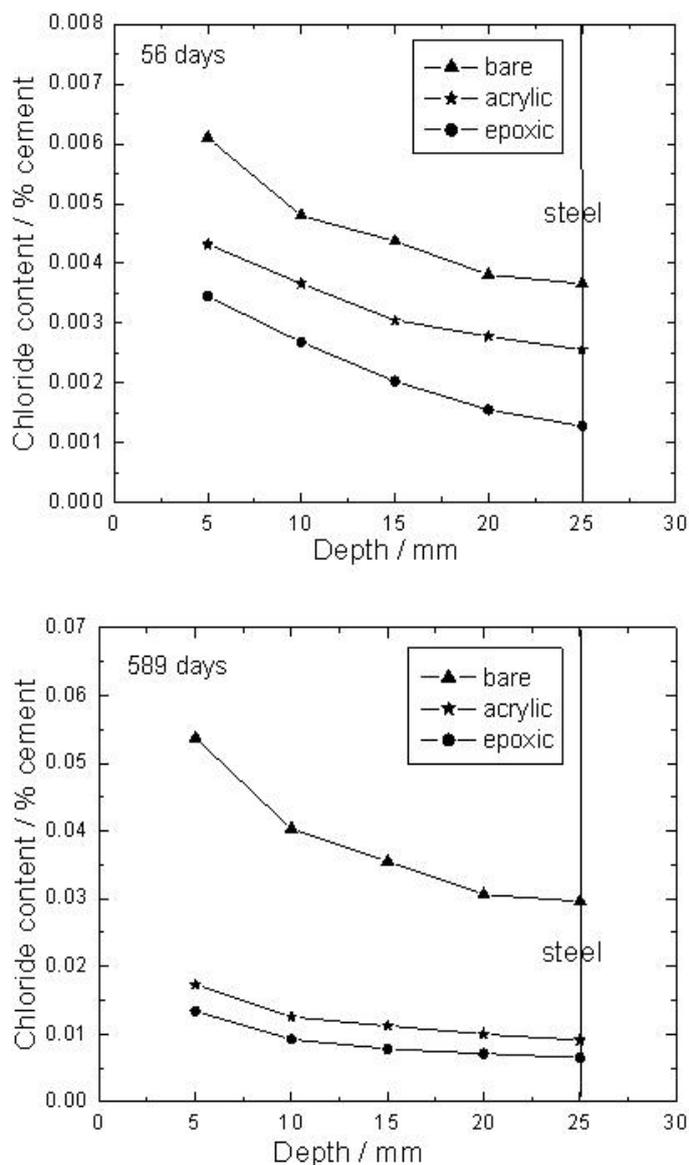


Figure 5. Variation in the content of free chlorides as a function of concrete depth.

Additionally, Table 6 shows the diffusion coefficients for chloride ion calculated from the information presented in Figure 5. The diffusion coefficient values obtained change with the exposure time and also with the protective coatings. Also, comparing these values with the diffusion coefficients obtained for the sulfate ion, these are higher due to the smaller size of chloride ion.

Table 6. Diffusion coefficient of chloride ion in the samples.

Sample	Diffusion coefficient of chloride ion $D \text{ (m}^2\text{s}^{-1}\text{)}$	
	56 days	589 days
Bare	16.1×10^{-11}	11.5×10^{-12}
Acrylic	15.1×10^{-11}	10.9×10^{-12}
Epoxic	5.6×10^{-11}	8.5×10^{-12}

3.4. Corrosion potential and corrosion current densities of the steel

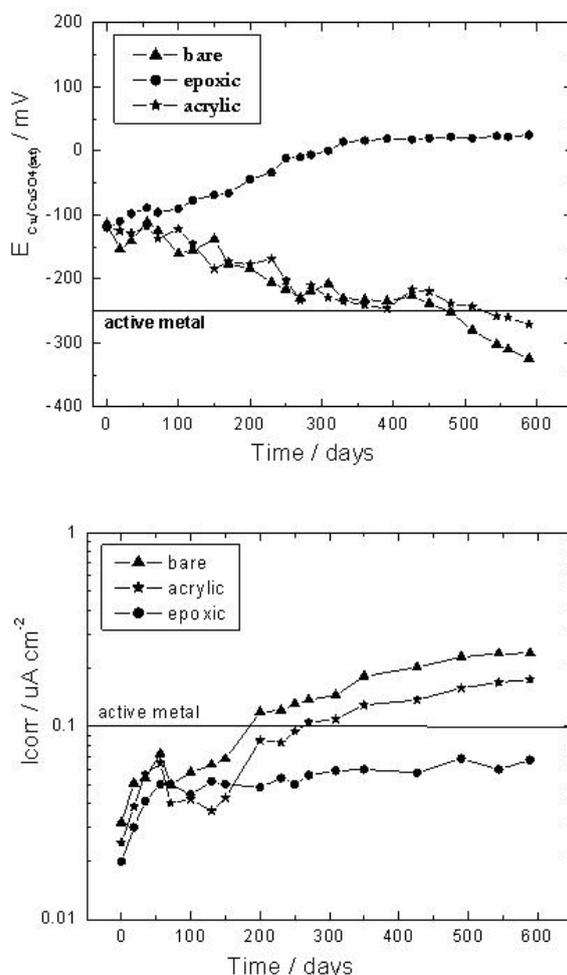


Figure 6. Electrochemical measurements of the steel in concrete. a) Corrosion potential b) Corrosion current.

Figure 6 shows a correlation between the evolution of the corrosion potential and the activation of the current density of the steel bars for the different testing conditions. The moment in which the steel reinforcement is activated is clearly indicated ($E_{\text{corr}} < -250$ mV vs. Cu/CuSO₄ and $i_{\text{corr}} > 0.1 \mu\text{Acm}^{-2}$) [14, 20-22].

The passive state of the bars was corroborated at the initial conditions. Rebar corrosion potential in all specimens is similar (-117 mVsce) and the current density slightly similar (0.02-0.03 μAcm^{-2}).

In the development of the study and after 589 days, bar corrosion potential in the specimens with epoxy paint showed a passive state, with positive values (24 mVsce), contrasting with the specimens coated with acrylic and uncovered: the potential values of these bars decrease presenting an active state for a similar exposure time (-268 and -323 mVsce, respectively). These data are consistent with the current density values for bars in concrete with acrylic paint and without paint, increasing and reaching after 589 days values between 0.17 and 0.24 μAcm^{-2} , differing for the specimens with epoxy paint after 589 days, with values of 0.06 μAcm^{-2} .

The variation in the electrochemical values is related to the barrier effect of coating against the access of water and ions to the reinforcing bar. After 589 days, in the specimen with epoxy paint, the free chloride ion content in the bar/concrete interface is 5 times lower as well as the sulphate content is three times lower than the other specimens. The order of admission of ions and water of the coatings was: epoxy < acrylic < bare.

Moreover, the pH values of concrete change according to the type of coat: with acrylic and uncovered: pH 6 due to more ions and water permeability than with epoxy: pH 10, which supports respectively the active and passive state of the reinforcing bar, according to Pourbaix diagram.

Therefore, the results show that both, the steel bar in uncoated concrete and in acrylic coated concrete were in active state after 589 days of exposure to the medium, whereas the steel bar in the epoxy coated concrete remained in a passive state.

Figure 7 show the cross sections at the middle of the steel from the uncoated concrete sample (Figure 7A) [22] and the steel from the epoxy coated concrete sample (Figure 7B). A general attack caused by corrosion can be observed in both samples, with an average thickness of corrosion product of 4.7 μm and corrosion current around 0.24 μAcm^{-2} for the steel in the uncoated concrete sample, and 1.9 μm for the epoxy coated concrete, with corrosion current around 0.07 μAcm^{-2} . The cracks in the corrosion product formed on the steel of the uncoated concrete (Figure 7A) probably produced an increase in the corrosion current.

The chemical composition of the epoxy coatings interferes with the intrusion of water and ions in the concrete. The epoxy paints have an evaporative process in a chemical reaction between the curing agent and the hardener. Thus, creating intertwined chains during the hardening process of this type of thermostable polymer and making it more impermeable to the intrusion of water and ions in the concrete.

However, the acrylic paint is produced by evaporation of the solvent (evaporative drying, where there are no chemical processes involved) and the result of the process generally is a linear or cross-linking type of polymer with certain degree of permeability.

The epoxy coating may be a barrier that is not associated with the electrochemical performance of the rebar, but in the diffusivity performance of the concrete.

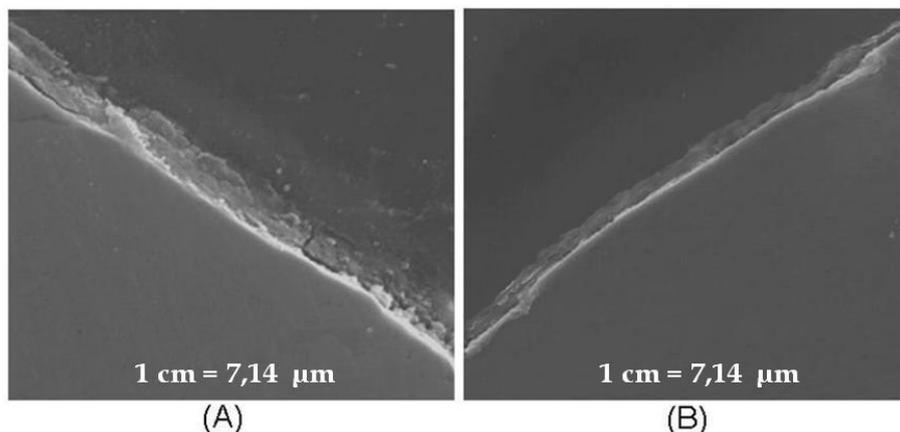


Figure 7. SEM on a cross-section of steel. (A) uncoated concrete (B) epoxy coated concrete

3.5. Composition of the corrosion products

The results obtained from X-ray diffraction for the composition of the corrosion products formed on the steel in different samples showed the presence of lepidocrocite, goethite and magnetite. The XRD pattern for the corrosion products in the steel epoxy coated concrete is shown in Figure 8.

Furthermore, the EDAX results obtained from the corrosion products show an element composition of 8.81% O, 2.82% Al, 1.04% S, 18.33% Ca, 69.10% Fe for the steel embedded in the uncoated sample and 13.29% O, 0.28% Si, 9.22% Ca, 0.21% S, 77.15% Fe for the steel of the epoxy coated sample, respectively. These data corroborate the barrier effect of the epoxy coating, as the sulfur content in the corrosion product (originated from the H₂SO₄ solution) is lower than in the corrosion product obtained from the steel without epoxy barrier on the concrete. The oxygen and iron presence confirm the presence of different species of iron oxide.

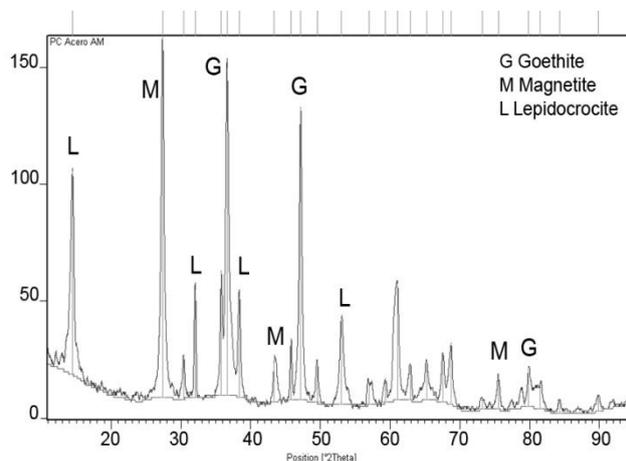


Figure 8. XRD pattern of corrosion products in epoxy coated concrete.

4. CONCLUSIONS

The results of the concrete sample exposition to an industrial chemical acid solution for 589 days lead to the following conclusions:

A correspondence among pH, chloride and sulfates on the steel surface, as well as between the corrosion potential and the corrosion current density of the steel with the coating type on the concrete was observed. The lower pH, the higher chloride and sulfate content cause diminishing of corrosion potential and increasing of corrosion current density.

The corrosion products observed in the different samples were lepidocrocite, goethite and magnetite and the thickness of this layer is 2.5 times higher for the steel in the uncoated concrete.

Based on the chemical analysis and electrochemical variables and the conditions of this study, it was stated that the best coating against acidic media is the epoxy paint.

Because the acidic environment of the mining process requires solutions to give the durability of reinforced concrete structures, epoxy paint on concrete can be a possibility of corrosion protection of structural steel and the disintegration of the concrete due to their high tightness in the acidic medium.

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