

Corrosion of steel rebar embedded in natural pozzolan based mortars exposed to chlorides

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ABSTRACT

Corrosion of steel is considered the most important durability problem of reinforced concrete. The application of supplementary cementitious materials has been proposed in order to mitigate this durability problem, reduce the production costs and control the emission of greenhouse gases. Mexico is rich in volcanic areas from which natural pozzolanic materials can be obtained. This paper examines the use of such natural pozzolans as a partial substitute of normal portland cement in reinforced mortar specimens. Compositions with substitution levels of 0%, 10% and 20% by mass of normal Portland cement of natural pozzolanas were investigated. The specimens were exposed to penetration of chlorides. Compressive strength, corrosion potential, polarization resistance, electrical resistivity, and chloride content of the mortars were determined in order to characterize the physical, mechanical, electrical, and electrochemical behavior of the mortar as well as the embedded steel. It was found that the use of pozzolan has resulted in a significant increase in mortar resistivity and corrosion initiation time for the same cover depth, and as a result, decreases the rate of corrosion of rebars once corrosion was initiated.

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1. Introduction

Concrete and reinforced concrete are the most widely used construction materials. The durability of reinforced concrete structures is assured by both the physical and chemical protection of the embedded steel against corrosion. Related works [1–5] have shown that the hydration of cement results in a high pH (~13) of the pore solution, which sustains electrochemical stability conditions for the embedded rebars. Concrete functions also as a physical barrier, delaying the ingress of aggressive agents that may initiate corrosion of steel. However, concrete is a porous material and therefore steel protection resulting from the penetration of aggressive substances is not may not remain perfect over time. This protection depends mainly on the quality of the concrete cover (as specified by the mix design and quality of its constituents) and also of the care exercised during its casting and curing.

Corrosion of steel rebars in reinforced concrete structures is often caused by exposure to chloride exposure originating mainly from the application of deicing salts or from marine environment [6]. Chlorides penetrate into concrete through water by diffusion and absorption. The repeated and prolonged exposure may eventually result in a high concentration of chlorides at the concrete - rebar interface. Some of the chlorides are chemically bound by the hydration products of cement, adsorbed by the gel pores (adhered

physically) or remain in ionic (free) form. Such free chlorides are available for the aggressive reaction on the surface of rebar. However, the distribution of ions among these three phases is not permanent, because the free chloride ion equilibrium is maintained in the pore solution. Then, only the chlorides over this equilibrium can be bound [7].

In the USA, the damage caused by the corrosion process results in tremendous economic losses amounted around \$276 billion dollars per year [8]. This ever growing need to improve the durability of reinforced concrete and also reduce the production costs, has lead to the use of diverse formulations of concrete, including the use of supplementary cementitious materials. Because of their morphology, chemical composition, mineralogy and particle size, some materials possess pozzolanic activity [9]. In example, the ability to react with calcium hydroxide in presence of water producing a cementing material [9,10]. These materials can thus be used as an additive or as a partial substitution of portland cement.

The use of pozzolanic materials, such as fly ash, has become a common practice in recent years. This material is a pulverized coal combustion by-product of the power plants and is the most extensively used by-product material in the United States [11]. Several studies have focused on the role of fly ash on chloride-induced corrosion [12–15]. It has been observed that the electrical resistivity of the cementing matrix increased [12–14], and consequentially led to a decrease in the chloride diffusion coefficients of concrete. Particularly, the use of fly ash has been shown to increase the chloride binding capacity of hydrates thus reducing the level of

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Table 1

Chemical composition of cements and pozzolan additive

%	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	SO ₃	Na ₂ O	K ₂ O	TiO ₂	P ₂ O ₅	Mn ₂ O ₃	LOI
NPC	18.88	4.35	1.86	64.46	1.24	3.35	0.31	0.60	0.20	0.11	0.04	2.6
CPC	17.6	4.70	1.77	64.74	1.23	3.26	0.37	0.80	0.19	0.13	0.02	4.6
NP	59.48	17.03	4.70	4.70	1.85	0.00	1.64	1.11	0.67	0.03	0.09	7.8

chlorides in the pore solution [16]. Nevertheless, it has been demonstrated that increasing the fly ash content leads to decreasing the threshold chloride level (Ccrit) [17].

Fly ash is less expensive than cement clinker and its application does reduce the cost of construction. However, it is also a waste material that requires an additional effort of recollection, treatment and characterization. In addition to the use of fly ash, natural pozzolan (NP), silica fume and slag have been evaluated as candidates for the reduction of steel corrosion [18]. Natural pozzolans (NPs) have been regularly used for temperature control in massive concrete structures and also as a supplementary cementitious material. Such NPs are commonly used to increase the resistance of concrete to sulphate attack and they are also recognized to contribute to the control of the alkali-aggregate reaction [11].

There are few works related to the influence of pozzolans on the corrosion kinetics in reinforced concrete. Koulombi et al. [19] examined the efficiency of Greek NPs in mitigating reinforced concrete corrosion. In this investigation NP from Santorin was used to partially replace the portland cement. Mortar specimens with a rebar of 5 mm diameter were tested. After curing, the specimens were immersed in an aggressive 3.5% per mass NaCl solution for 5 months. According to this investigation, the mass loss of the rebars was reduced almost by 40% for the specimens with this NP. However, in another work [20], it was found that the pozzolanic activity does not always improve the concrete protective properties because the latter depends on the chemical composition of the pozzolan used.

México has important volcanic areas in which natural pozzolanic materials with mineralogical characteristics similar to artificial pozzolans (such as fly ash, blast furnace slag and silica fume) are available.

In the cement industry, there is an ongoing research on sustainable development; in that context partial substitution of cement with materials possessing pozzolanic characteristics is sought [21,22]. It is also that pozzolanic materials improve some properties of both fresh (increase the setting time, cohesion and workability, reduce segregation and bleeding, etc.) [9,10] and hardened concrete (reduce permeability, porosity, increase the long-term strength) [12–24].

The objective of this work is to examine the effect of Mexican natural pozzolan on corrosion of steel rebars initiated by accelerated chloride ingress. Laboratory tests were conducted on mortar specimens with different proportions of NP. Corrosion potential, polarization resistance and mortar resistivity were measured in order to evaluate the corrosion process. In addition, the compressive strength and the chloride content of mortars were examined in order to characterize the mechanical behavior and mortar resistance to chloride penetration.

2. Experimental program

2.1. Materials

Commercial grade portland cement (CPC, CPC30R according to NMX-C-414-ONNCE normative [25]), a normal portland cement (NPC, type I according to ASTM C 150 [26]) and a NP (from the Puebla State of Mexico) were used in the mortar mixtures. The NP

composed of andesite and was used in proportion of 0%, 10% and 20% (substitution by mass) of NPC. The pozzolan had a pyroclastic texture with crypto-crystalline matrix with acidic glassy phases containing fragments of feldspar and iron hydroxide [27]. According to NMX-C-414-ONNCE, the CPC is composed of clinker, gypsum and mineral additives. The chemical composition of the materials used in this research program is presented in Table 1. Three mortar formulations (denoted as NPC, NPC10P, NPC20P) were investigated as shown in Table 2. For all mortars, a W/Cm ratio of 0.65 was used (Cm: total of material used, i.e. cement + pozzolan).

2.2. Preparation of specimens

Cylindrical mortar specimens of 50 mm diameter and 130 mm height were constructed with a single rebar (10 mm diameter and 140 mm length) at the center. Industrial grade A60 steel according to ASTM A370 [28] (designated G42 according to NMX-C-407-ONNCE [29]) was used as embedded steel in all specimens. In order to confine the exposed area of the steel, the rebar was partially coated with a non-corrosive paint, as illustrated in Fig. 1, leaving an exposed area of 15 cm². Sixteen specimens (per composition) were cast and then stored in a curing room at 20 °C and RH of 95% for 24 h. Following this period, the specimens were demoulded and cured at 20 ± 1 °C and RH of 100% for 28 days (for all compositions). After the curing period, specimens were exposed to the aggressive solution and the exposure time began into account.

Additionally, four mortar cylinders (100 mm diameter and 200 mm height) were fabricated per composition for the compressive strength tests exclusively. Mortar specimens were cast, compacted and cured according to the ASTM C 192/C192 [30]. These specimens were maintained in a curing room at 20 °C and RH of 95% for 24 h, after that, they were cured at 20 ± 1 °C

Table 2

Proportions of materials used in the research program

Mortar	W/Cm	Cement, kg/m ³	Sand/Cm	NP, %
CPC	0.65	401	4:1	0
NPC	0.65	401	4:1	0
NPC10P	0.65	361	4:1	10
NPC20P	0.65	321	4:1	20

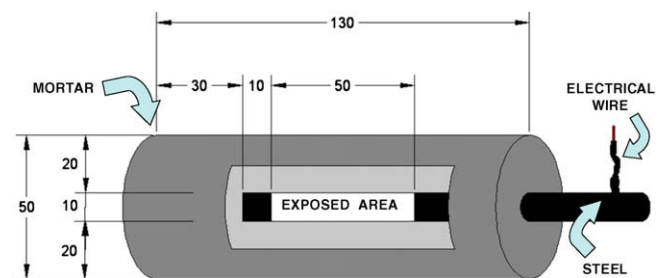


Fig. 1. Schematic representation of mortar specimen (all dimensions are in mm).

and RH of 100% for 28 days for all compositions and 56 days for NPC, NPC10P and NPC20P compositions. CPC compositions were not cured until 56 days. The compressive strength results obtained, according to ASTM C 39 [31], at the age of 28 and 56 days as presented in Table 3.

2.3. Exposure conditions

In normal conditions, the evolution of chloride ingress is slow and has an evolution rate measured in years. In order to accelerate this process, the specimens were exposed to chlorides using the cycles consisting of 3-day partial immersion in a NaCl solution having a concentration of 35 g/L followed by 4-days drying period at 40 °C in a forced ventilation oven i.e. wetting and drying (WD) cycles. Under such conditions, the corrosion is induced only by the chlorides, since water of the cement matrix prevents the carbonation of the specimen [32].

2.4. Corrosion measurements

Corrosion potential (E_{corr}) and linear polarization resistance (R_p) were obtained using a potentiostat/galvanostat VoltaLab PGZ-301 connected to a personal computer. Also, electrochemical response of the steel–mortar interface was recorded in a frequency interval of 1–100 kHz by means of the Electrochemical Impedance Spectroscopy technique. The polarization applied to system was 10 mV peak-to-peak starting from the free corrosion potential, E_{corr} , (this test was applied just before the R_p technique). The results were treated using the software package Zview 2.0 in order to obtain the mortar resistivity. The R_p technique was applied using

a scan rate of 12 mV/min (for steel in a passive state the scan rate was reduced to 6 mV/min) and a potential shift of 20 mV in both anodic and cathodic direction. The R_p results were translated to current density (i_{corr}) using the Stern and Geary [33] relationship ($i_{\text{corr}} = B/R_p$) with B defined as the anodic–cathodic slope. For steel in a passive state, i.e. $E_{\text{corr}} > -250$ mV vs. saturated calomel electrode (SCE), a value of $B = 52$ mV was used, whereas for steel in an active state, i.e. $E_{\text{corr}} < -250$ mV vs. SCE, $B = 26$ mV was adopted [34]. Measurement configurations comprising three electrodes were used, with the rebar acting as a working electrode, a SCE as reference and a 304 L stainless steel as auxiliary electrode. No compensation for the IR drop effect was considered, since the potentiostat did not allow for determination of this parameter. In addition, the electrochemical tests were performed at a time interval of 7 days, at the end of the immersion period (end of the third day) and the measurement conditions assumed a low mortar resistivity. Four specimens of each composition were analyzed.

2.5. Visual examination of steel surface

Some specimens were subject to the tensile splitting test in order to examine the state of corrosion of the rebar. At the same time, different mortar samples were collected from the mortar–steel interface, in order to determine the total chloride (i.e. acid soluble) concentration.

3. Experimental results

3.1. Compressive strength

At an age of both 28 and 56 days, the test results show that mortars containing NP material have a slightly reduced compressive strength (10% in average) when compared with NPC and CPC. This effect can be attributed to a somewhat slower chemical reaction of the natural pozzolan used. Favorable temperatures and continuous curing for a prolonged period of time may be needed by pozzolans in order to develop completely the pozzolanic reaction [11]. It can be expected that the compressive strength of mortars with pozzolan will be higher than that of the NPC and CPC at higher ages, because of the effect of pozzolanic reaction results in the densification of the cement matrix.

Table 3
Compressive strength of mortar investigated

Composition	Compressive strength, MPa	
	28 days	56 days
CPC	36.0	—
NPC	41.6	42.0
NPC10P	34.5	37.0
NPC20P	39.0	37.5

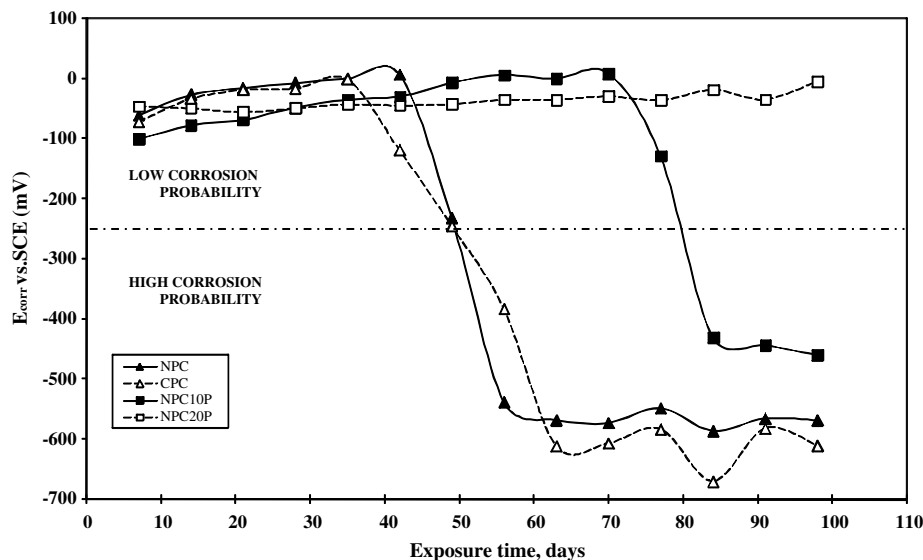


Fig. 2. Corrosion potential evolution for different mortar specimens.

3.2. Corrosion potential

The evolution of the corrosion potential, E_{corr} , of specimens vs. time (during to the WD cycles) is demonstrated in Fig. 2. The dotted horizontal line presents the border between regions of high and low probability of corrosion, as proposed by Andrade and Alonso [35]. Each E_{corr} measurements were performed at a time cycle of 7 days.

During the first 35 days the values near to -100 mV vs. SCE were obtained. During this period, all four compositions were in the low corrosion probability zone and showed similar behavior. However, after 40 days, the E_{corr} of NPC and CPC specimens gradually become more negative eventually resulting into corrosion potential values near to -600 mV. During the same period, the NPC10P and NPC20P specimens remained at a nearly constant potential value. After 80 days of WD exposure, the NPC10P specimens demonstrated a shift of E_{corr} to more negative values suggesting a higher corrosion probability. In contrast, during the complete test-

ing period, the NPC20P mortars had values near to -100 mV and, therefore, remained in a condition of low corrosion probability.

3.3. Corrosion rate

Fig. 3 shows the corrosion rate for different mortar specimens. Four different zones have been proposed [35] for the corrosion rate as a function of the i_{corr} . These zones are bordered by the dotted horizontal lines.

During the initial period of the exposure, all four compositions demonstrated similar behavior, with i_{corr} values below $0.1 \mu\text{A}/\text{cm}^2$. After 40 days, the i_{corr} of CPC and, slightly later, of NPC specimens was increased gradually, entering the high corrosion rate zone with values over $1.0 \mu\text{A}/\text{cm}^2$ at 60 days, and even reaching $3.0 \mu\text{A}/\text{cm}^2$ at 90 days. In contrast, for the NPC10P and NPC20P specimens, the i_{corr} values ranged between 0.05 and $0.07 \mu\text{A}/\text{cm}^2$, maintaining these specimens in a low corrosion rate state. However, after 77 days, a notable activation of the NPC10P specimens

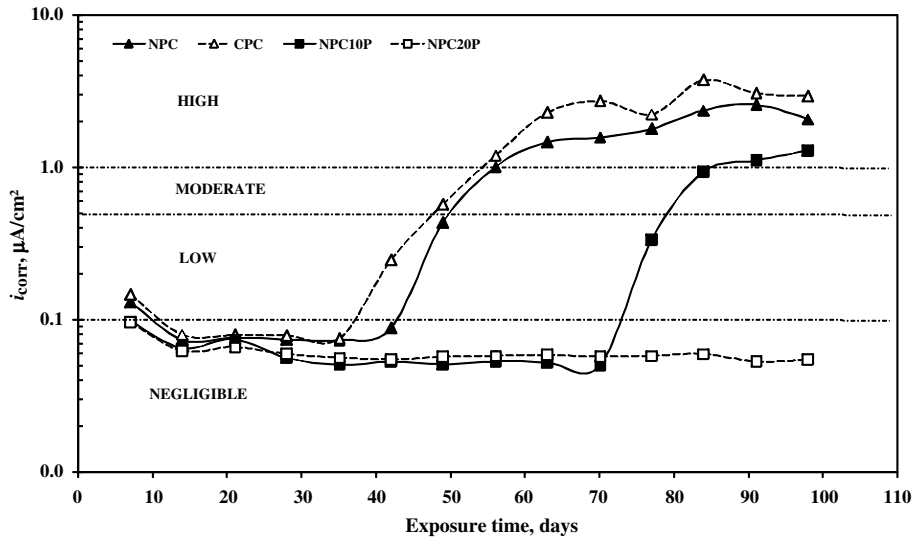


Fig. 3. Corrosion rate of mortar specimens.

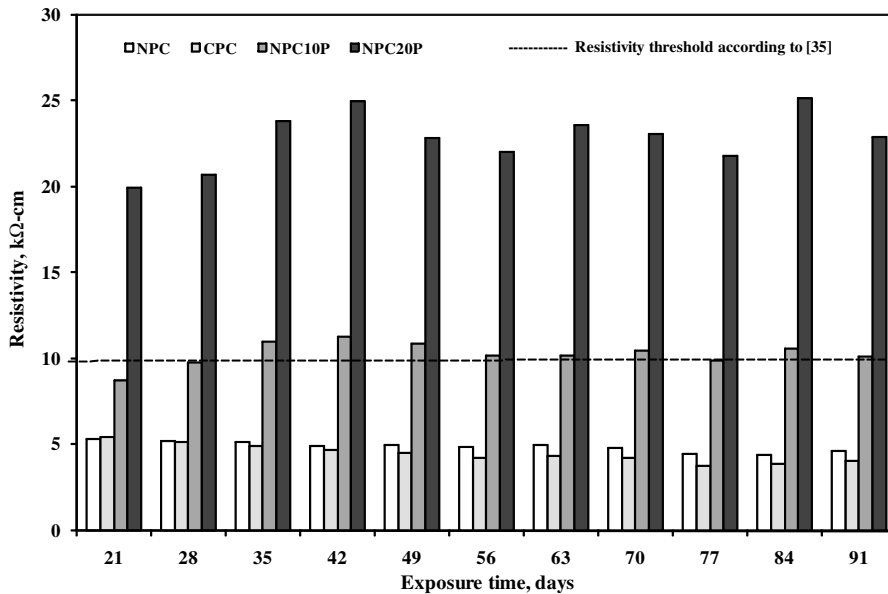


Fig. 4. Resistivity evolution of investigated mortars.

occurs with gradual increase of the i_{corr} values (up to $1.0 \mu\text{A}/\text{cm}^2$). During the whole testing period, the NPC20P specimens did not show any increase in the corrosion rate value.

3.4. Resistivity of mortars

It is known that the electric resistivity of cementitious materials, ρ , is directly influenced by the porosity and the ionic concentration of the pore solution. The evolution of resistivity of mortars is presented in Fig. 4 as obtained indirectly by EIS test. The resistivity of NPC20P specimens is almost twice than that of NPC10P and four times than those of NPC and CPC. The porosity decrease due to the use of pozzolan, results in higher resistivity which delays the penetration of chlorides into the mortar. Therefore, there is a significant change in the resistivity obtained when using NP in mortar. The scatter observed on the resistivity values for one same composition do not show any clear trend about the decrease of resistivity due to chloride content. This behavior can be attributed to changes (at the time of electrochemical test) in temperature and moisture content, parameters that are known to produce a pronounced effect on resistivity values.

3.5. Chloride content

The total chloride concentration near the mortar–steel interface after 77 days of exposure to WD cycles vs. mass of cementitious material (% by mass Cm.) is shown in Fig. 5. The maximum chloride concentration allowed in the design of new reinforced concrete structures exposed to aggressive or coastal environment is marked by a dotted line [36,37]. It can be observed, that all specimens have a chloride concentration that is higher than permitted. Nevertheless, only the NPC and CPC specimens showed chloride concentrations at the mortar–steel interface close to 1.3%. In contrast, the chloride concentration for the NPC10P and NPC20P specimens was 0.8% and 0.5%, respectively.

3.6. State of rebar surface

The state at surface of the embedded steel bar for different mortar compositions after 77 days of WD cycles is demonstrated

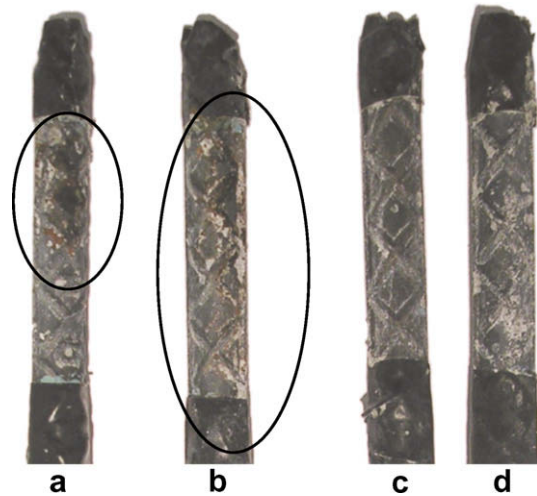


Fig. 6. Surface state of the steel bars after 77 days of exposure for: (a) NPC, (b) CPC, (c) NPC10P and (d) NPC20P.

in Fig. 6. Because of the active corrosion process in the NPC and CPC specimens zones of notable degradation are detected. The specimens containing natural pozzolan (NPC10P and NPC20P) did not show any degraded areas visible by the naked eye. Based on these results allow to conclude that the specimens with natural pozzolan provided better protection to the embedded steel bar than those constructed with commercial portland cements.

4. Discussion

For the exposure conditions corresponding to WD cycles (in which the pH of exposure solution is kept constant during the chloride attack), corrosion potential and corrosion rate undergo three different stages. The first stage is observed during the first days of exposure with values of $E_{\text{corr}} > -250 \text{ mV}$ which represents a passive state of the embedded rebar. This state was also confirmed by the i_{corr} values, which were lower than $0.1 \mu\text{A}/\text{cm}^2$.

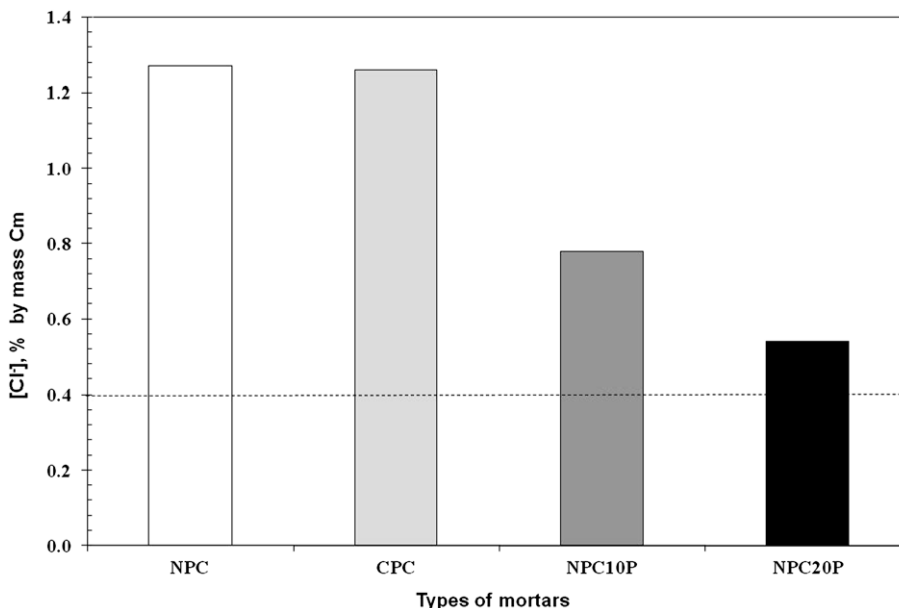


Fig. 5. Total chloride concentration in mortar–steel interface at 77 days of exposure.

The second stage (between 50 and 60 days, for the NPC and CPC and between 75 and 85 days, for NPC10P) is characterized by a gradual transition from the passive state towards an active corrosion state. During this period E_{corr} evolved from -250 to -500 mV. This stage is also characterized by a significant increase in corrosion rate i_{corr} .

During the third stage, E_{corr} is lower than -550 mV vs. SCE, for the NPC and CPC specimens and not more than -400 mV for the NPC10P. This corresponds to an active corrosion state according to ASTM C876 [38]. The i_{corr} values show that these specimens are in a high corrosion rate zone with values above $1.0 \mu\text{A}/\text{cm}^2$.

The use of pozzolanic materials reduces the porosity of the mortar matrix [12–14], which is directly related to electrical resistivity. The tests performed showed an increase in mortar resistivity for all compositions with NP. The best result was obtained when 20% NP was used. The results obtained are compatible with the exponential law [39,40] proposed for the influence of fly ash on the resistivity due to the decrease in porosity. It is known that low porosity influences the ingress of aggressive agents (chlorides). Although, results of chloride diffusion coefficient were not obtained in this work, the results of the chloride content are in accordance to those reported by Salta [41], who substituted 50% of NPC by fly ash, and found a reduction of the chloride diffusion coefficient by 50% vs. NPC.

Figs. 2 and 3 demonstrate the beneficial effect of NP on the initiation of corrosion rate of reinforcing steel. Specimens with 10% and 20% of NP remained passive during 70 and 100 days, however, the specimens based on commercial grade portland cement (NPC and CPC) became active after approximately 50 days of WD cycles. This behavior can be attributed to the reduction of porosity owing to the pozzolan material used, as it has also been observed by other researchers [7,9,10].

Fig. 7 presents the evolution of corrosion rate as a function of resistivity, at different periods. The dotted lines represent the limits for the corrosion rate (horizontal lines) and resistivity (vertical lines) [35]. It is observed that NPC and CPC specimens show resistivities below $10 \text{ k}\Omega \text{ cm}$ and i_{corr} values increasing gradually from 0.1 to $4.0 \mu\text{A}/\text{cm}^2$. These results agree with the data reported by Andrade and Alonso [35]. On the other hand, the NPC10P showed resistivity values near the threshold. In this case,

the corrosion rate may increase and other parameters in addition to resistivity (chloride content, oxygen, humidity, etc.) may control the kinetics of the corrosion process. In a similar way, NPC20P specimens showed high resistivity and, therefore, low corrosion rates in comparison to NPC and CPC. In the case of NPC20P, the kinetics of the corrosion process is controlled by the low porosity (evidenced by the high mortar resistivity of these specimens) that influenced in the low chloride content of the mortar. As can be observed in Fig. 5, between 40% and 50% of diminution of chloride content (measured within 10-mm depth around steel rebar) was obtained for mortars with 10% and 20% of NP at 77 days of exposure time.

5. Conclusions

The use of Mexican natural pozzolan as a partial 10–20% substitution of portland cement has a beneficial effect on the corrosion behavior of steel due to following factors:

- it results into a significantly higher mortar resistivity;
- a 20% replacement of cement by pozzolan leads to a significant increase of the induction time and reduces the corrosion rate by one order of magnitude, as verified by the visual inspection of surface state of the embedded steel rebar;
- the exposure to WD cycles in 35 g/L NaCl solution provides an improved protection for the reinforcement steel as compared to normal and composite portland cement.

Therefore, the use of natural pozzolans influences both the initiation and propagation time of the progress in steel corrosion. The addition of finely ground natural pozzolan and its subsequent reaction with cement hydrates provides an effective pore filling effect. It can be expected that the high resistivity will be the most important parameter at later ages of hardening.

The reduction of compressive strength observed for the specimens with 20% of natural pozzolan is considered acceptable taking into account the reduction of the chloride ingress and improved protective properties. The compressive strength of such concrete can be improved if superplasticizer is used for the preparation of mortars.

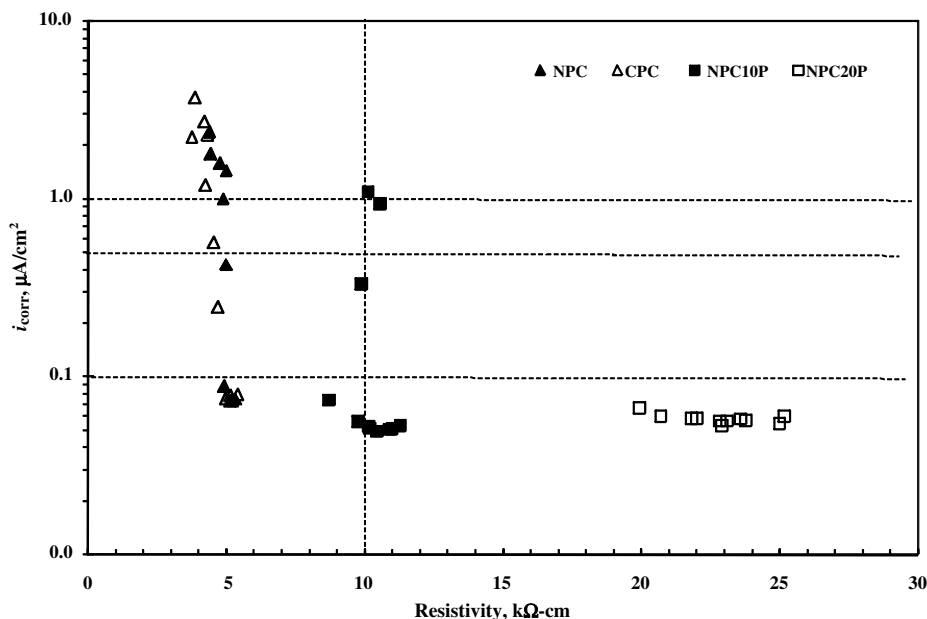


Fig. 7. Relation between ρ and i_{corr} for fabricated specimens.

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