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Corrosion Behavior of Steel Embedded in Ternary Concrete Mixtures

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The main reason for the premature failure of reinforced concrete structures is corrosion of the reinforcements. The use of new mortars based on ternary mixtures, an alternative to ordinary Portland cement (OPC), requires extensive research in order to check its passivating properties for reinforcements and the instability or permanence of the passive state achieved. Pozzolans and slag extend the market for concrete by improving specific properties of concrete products, allowing them to be constructed with other materials or placed in environments that would have precluded the use of Portland cement alone. In properly formulated concrete mixtures, pozzolans and slag have been shown to enhance long-term strength, decrease permeability, increase durability, and reduce thermal cracking of bulk concrete. Steel reinforcements have been exposed for 13 months in mortars immersed in a 3.5% NaCl solution. The effect of mortar composition using ternary mixtures of fly-ash (FA), micro-silica (MS), and granulated blast furnace slag (GBFS) was tested. OPC was also tested as a reference. Electrochemical characterization was performed, measuring corrosion potential, linear polarization resistance, and electrochemical noise resistance. The best passivating properties were shown by the mixture of 10% FA and 10% GBFS.

Keywords: Steel corrosion, reinforced concrete, ternary mixtures, fly-ash, micro-silica, granulated blast furnace slag, Portland cement

1. INTRODUCTION

Reinforced concrete (RC) combines the good compressive strength properties of concrete and the excellent mechanical strength properties of steel. Thus, RC materials can be used by designers, architects and civil engineers to meet high mechanical strength, fire resistance, durability, shape adaptability and low cost requirements [1-3]. Fortunately, if concrete is correctly executed its high pH guarantees the passive state of the rebar. In the passive state, corrosion is insignificant and if this state is maintained reinforced concrete structures (RCS) durability can be practically unlimited.

Pozzolans include a wide range of predominately glassy materials like fly-ash (FA); micro-silica (MS) or silica fume (SF), waste material from the silicon and ferrosilicon metal industry; and natural pozzolans, and geologic deposits of clay. These materials may be calcined prior to use in order to increase their activity. Pozzolans are not new to the construction materials industry, and in fact have been used for construction purposes for thousands of years [5].

FA is the most commonly used supplementary cementitious material (SCM). In 2001 the United States produced about 68 million tons of FA [4]. FA is the inorganic residue collected from flue gases exiting the boilers in pulverized coal generating stations. FA particles are collected in electrostatic precipitators or bag houses and then transferred to a storage silo or sluice pond. FA has a spherical morphology and exhibits a rather wide range of bulk chemical compositions, which has resulted in the creation of two classes of FA in ASTM C 618-03 Standard [6], based on the oxide mixture content (SiO_2+Al_2O_3+Fe_2O_3). Class F FA has an oxide mixture content of 70% or more, while class C FA has an oxide mixture content of between 50% and 70%. Class F FAs are typically pozzolanic, although some authors have noted that they may occasionally exhibit some self-cementitious properties [7]. Class C FA can also exhibit self-cementitious properties [6]. The beneficial properties normally associated with FA are increasing resistance to sulfate attack [8], and reducing expansion caused by alkali-silica reactions (ASRs) (aggregate-alkali reaction, formation of thaumasite, etc.) [9]. Alkaline cements also show better durability behavior than OPC because systems based on activated FA are less prone to expansion due to ASRs than Portland cement systems [10]. Furthermore, these materials adhere extraordinarily well to reinforcing steel, feature high-volume stability, high fire resistance, and low thermal conductivity, and would foreseeable afford excellent durability in aggressive environments [11].

Granulated blast furnace slag (GBFS) has been used for many years as a SCM material in Portland cement concrete, either as a mineral admixture or as a component of blended cement [12].

A ternary mixture is simply a mixture of three components. In the case of a ternary mixture of cementitious materials, the components could for instance be OPC, FA, and GBFS. Likewise, the combination could be a blended cement (already a binary mixture) and slag. The concept behind ternary mixtures is not new. In fact, Abdun-Nur refers to a ternary mixture that was being commercially produced over 40 years ago [13]. Both the American Concrete Institute (ACI) slag document [14], and the silica fume (SF) document [14], give some brief documentation of concrete containing ternary cementitious mixtures. However, ternary mixtures are becoming more prevalent because they can enhance performance and reduce costs. The reduction in cost is associated with the fact that most SCMs are by-products. Moreover, the use of these materials also decreases the amount of Portland cement that must be manufactured, thus making the cement industry more sustainable. One of the issues related to the development of ternary mixtures is the number of concrete mixes that need to be formulated and tested to ensure the performance of the mixture. When FA, SF, GBFS, or natural pozzolans are used in combination with Portland or blended cement, the proportioned concrete mixture should be tested to demonstrate that it meets the required concrete properties [16-18].

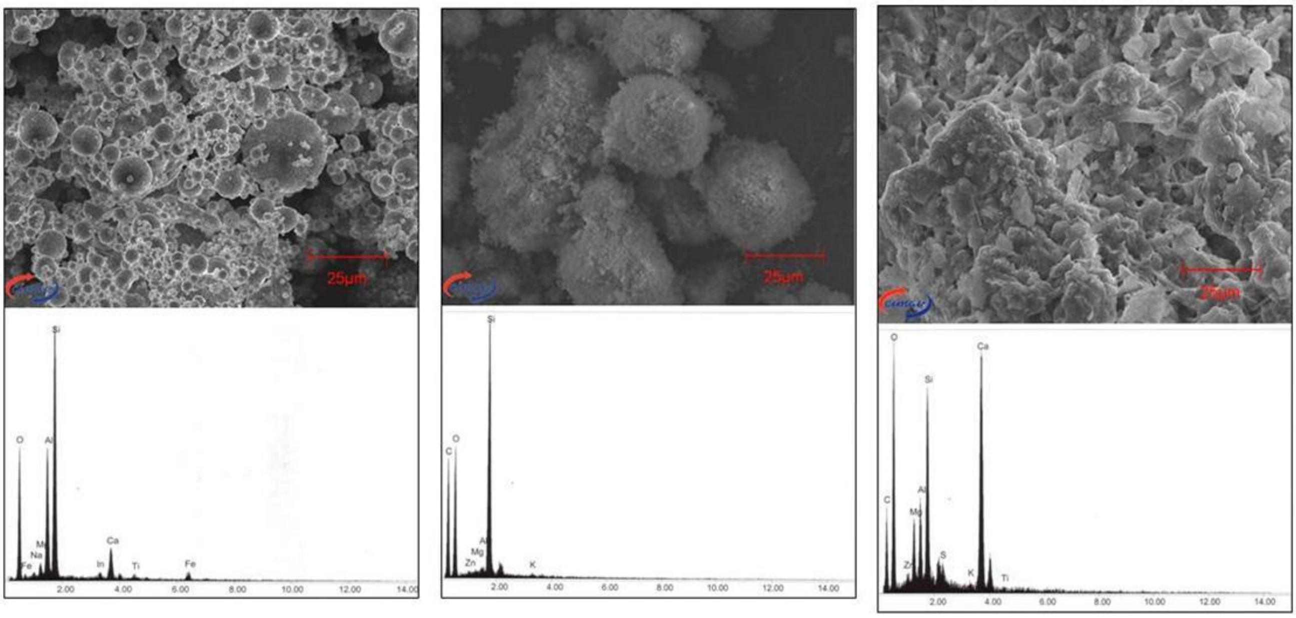

Figure. 1. SEM micrograph and EDX spectrum for a) fly ash (FA) particles. b) micro-silica (MS) particles. and c) for granulated blast furnace slag (GBFS) particles.

Doubts have been raised against the use of slag and FA in prestressed and post-tensioned concrete. Blast furnace slag contains sulfides which could catalyze the development of hydrogen atoms in the case of corrosion of prestressed steel in concrete, thus leading to hydrogen embrittlement. Tests and practical experience over 50 years have shown this concern is not justified and slag cement can be used freely [19].

It is well established that MS additions in concrete reduce the network of interconnected porosity and thereby the penetration of chlorides from the outer surface into the concrete. As was pointed out by Viden [20], the use of MS additions is a good remedy for preventing reinforcement corrosion in concrete structures exposed to sea water splashing or deicing salts. However, they have also been reported to reduce the pH of the pore solution.

Considering that reinforcement corrosion is the main cause of RCS failure [21-23], the capacity of ternary mixture mortars and concretes to passivate steel reinforcements is a very important property to guarantee the durability of RCS constructed with these materials. The passivating capacity and the permanence of the passive state once reached may depend on the nature and the dosage of the binder, as well as on environmental conditions. This paper attempts to find an experimental response to these questions, using electrochemical techniques to determine the corrosion rates of steel rebars embedded in OPC with three types of additives, FA, MS, and GBFS, immersed in a 3.5% sodium chloride (NaCl) solution.

2. EXPERIMENTAL

2.1. Materials

The present study was performed using a commercial Portland Type I cement according to ASTM C 150-02 Standard [24], and the addition of two pozzolanic materials: class F FA according to ASTM C 618-03 Standard [6], from the Carbonifera Company “Rio Escondido”, located at Piedras Negras, Coahuila, Mexico, see Figure. 1a; and MS from “Eikem Materials”, Mexico, a dust of a size 100 times smaller than Portland cement according to ASTM C 1240-05 Standard [25], see Figure. 1b. Additionally, a cementitious material: GBFS was added to the mixture, from the “Altos Hornos” Company, Monclova, Coahuila, Mexico, see Figure. 1c.

Table 1 includes the 10 mixtures designed and the reference matrix (specimen A). They were performed to achieve a resistance of 25 MPa, with a water/cement ratio of 0.45 with respect to 402 kg m^{-3} of cement. Carbon steel bars were used as reinforcements. The experiments were performed in duplicate.

Table 1. Specimens tested and composition**.**

Specimen	**Composition, %**				
	Fly-Ash (FA)	Granulated Blast Furnace Slag (GBFS)	Micro-Silica (MS)	Ordinary Portland Cement(OPC)	Labeling
A	–	–	–	100	A(100OPC)
B	5	10	–	85	B(5FA-10GBFS-85OPC)
C	5	–	10	85	C(5FA-10MS-85OPC)
D	10	5	–	85	D(10FA-5GBFS-85OPC)
E	–	5	10	85	E(5GBFS-10MS-85OPC)
F	10	–	5	85	F(10FA-5MS-85OPC)
G	–	10	5	85	G(10GBFS-5MS-85OPC)
H	10	10	–	80	H(10FA-10GBFS-80OPC)
I	10	–	10	80	I(10FA-10MS-80OPC)
J	5	–	15	80	J(5FA-15MS-80OPC)
K	–	5	15	80	K(5GBFS-15MS-80OPC)

2.2. Measurement Methods

Experiments were performed on small prismatic specimens measuring 30×15×9 cm, see Figure. 2. Two 3/8 in. (6-mm diameter) 1018 steel bars, symmetrically embedded in the prisms, were used as working electrodes during the measurements, with one of the steel bars acting as working electrode and the other as counter electrode. A copper-copper sulfate ($Cu/CuSO_4$) electrode was used as reference. An active area of 10 cm^2 was marked on the working electrodes with epoxy resin, thus isolating the triple mortar/steel/atmosphere interface to avoid possible localized corrosion attack due to differential aeration. The specimens were immersed in a 3.5% NaCl solution for a time of 13 months.

Electrode behavior over time was monitored using the following four techniques. (a) Corrosion potential (E_{corr}) values. The E_{corr} parameter may be used to define the probability of corrosion: for $E_{corr}<-0.35$ V vs. $Cu/CuSO_4$ high corrosion probability (~90%), for $-0.35 \text{ V}<E_{corr}<-0.20$ V vs. $Cu/CuSO_4$ uncertainty of corrosion, and for $E_{corr}>-0.20$ V vs. $Cu/CuSO_4$ 10% probability of corrosion [25]. (b) Linear polarization resistance (R_p) ($R_p = \frac{\Delta E}{\Delta I}$) values to calculate the steel corrosion rate from the Stern-Geary equation [27]: $i_{corr} = \frac{B}{R_p}$, applying $\Delta E \pm 20$ mV at a scan rate of 0.16 mV s^{-1}, for the B constant a value of 26 mV was adopted. The corrosion level may be defined according to the Durar Network Specification [28]: for $i_{corr}<0.1$ μA cm^{-2} passivity, for 0.1 μA $cm^{-2}<i_{corr}<0.5$ μA cm^{-2} low corrosion, for 0.5 μA $cm^{-2}<i_{corr}<1.0$ μA cm^{-2} high corrosion, and for $i_{corr}>1.0$ μA cm^{-2} very high corrosion. A general corrosion process was assumed to define the corrosion level. When pitting corrosion takes place these current density values have to be multiplied by a factor of 10 to obtain the maximum local depth; i.e. 0.3 μA cm^{-2} general corrosion is 3 μA cm^{-2} pitting corrosion [29]. An EG&G PARC potentiostat, model 273A, was utilised. Finally, (c) electrochemical noise (EN), taking 1024 points measuring one point per second. With the exception of E_{corr} all these procedures provide a quantitative measure of the corrosion rate, a parameter indicative of the corrosion activity or passivity of the steel bars.

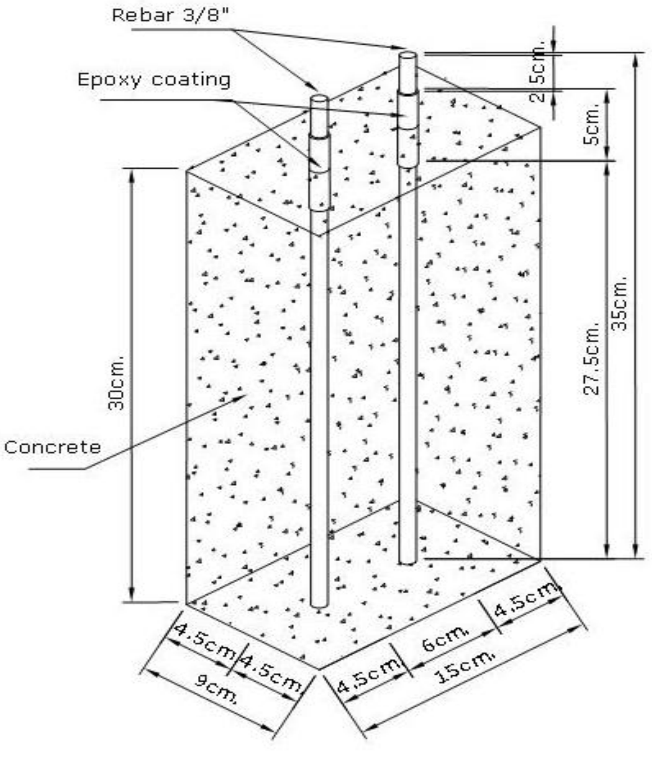

Figure. 4. Scheme of the prismatic specimen used.

The EN measurements were obtained as follow. Time records of the potential and current fluctuations under open-circuit conditions were obtained at an accuracy of ±1 μV by means of a ZRA Gill-8 potentiostat/galvanostat and a microprocessor. Voltage fluctuations between the steel bar working electrode and a $Cu/CuSO_4$ reference electrode were recorded without any external modification to the system. Current fluctuations between the two embedded steel bars in the mortar were also recorded without any external modification to the system. The effect of light on $Cu/CuSO_4$ noise was negligible, the noise level varying from –96 dB $(Hz)^{-1/2}$ in the light to –98 dB $(Hz)^{-1/2}$ in the dark. The values measured by the potentiostat at a sampling rate of 1 s (1024 points) were stored in the microprocessor. Subsequent data analyses were undertaken using the same microprocessor. Finally, the standard deviations of the potential and current fluctuations were analyzed. Electrochemical noise resistance (R_n) was obtained as a relationship between the standard deviation of the potential (SDP) and the standard deviation of the current (SDC): $R_n = \frac{SDP}{SDC}$. The localization index (LI) parameter was defined as the relationship between SDC and I_{rms}: $IL = \frac{SDC}{I_{rms}}$ according to the literature [30]. The IL parameter may be used to define the type of corrosion: for $0.001<IL<0.01$ general corrosion, for $0.01<IL<0.1$ mixed corrosion (general corrosion and pitting corrosion), and for $0.1<IL<1.0$ pitting corrosion.

3. RESULTS

Figure. 5 shows mechanical strength values for specimens A(100OPC), B(5FA-10GBFS-85OPC), D(10FA-5GBFS-85OPC), H(10FA-10GBFS-80OPC), J(5FA-15MS-80OPC), and K(5GBFS-15MS-80OPC) after 95 days of ageing in a plastic membrane according to ASTM C 156-98 Standard [31]. As can be observed the mechanical strength depends on the amount of additive added. Taking as reference specimen A, made with 100% OPC, the mechanical strength increased with the FA additive content, see specimens B, D, and H in Fig. 5. However, on specimens J and K, with 20% of the OPC replaced and containing 15% MS, the mechanical strength was 20% lower than on specimen A. The replacement of OPC by the binary mixture of FA and GBFS yielded good mechanical strength properties.

Figure. 6 shows E_{corr} values versus time for steel bars embedded in 10 mortar mixtures and the reference matrix, specimen A(100OPC), immersed in a 3.5% NaCl solution at room temperature. In general the 10 ternary mixtures and the reference matrix showed similar behavior.

According to ASTM C 876-99 Standard [26], active potentials were observed on specimens in the range from –0.550 V to –0.650 V vs. $Cu/CuSO_4$ with 90% probability of corrosion processes taking place. However, specimen B(5FA-10GBFS-85OPC) for 3-4 months of immersion and specimens H(10FA-15GBFS-80OPC) and J(5FA-15MS-80OPC) for 11-13 months of immersion showed E_{corr} values for which some uncertainty of corrosion was observed.

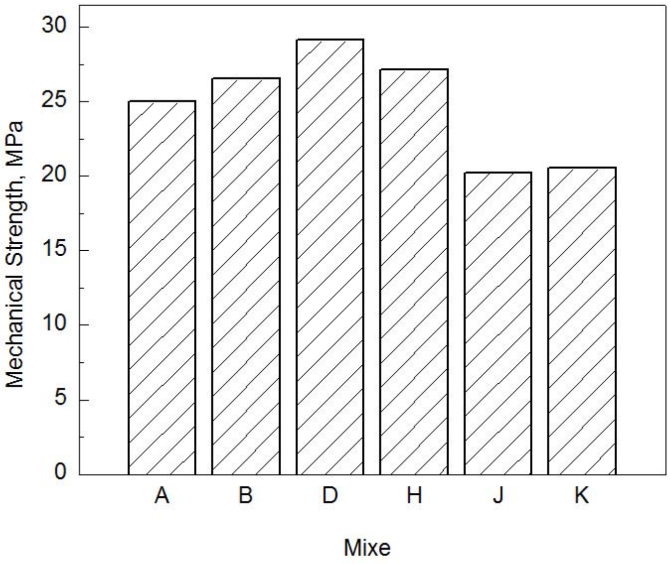

Figure. 5. Mechanical strength for specimens A(100OPC), B(5FA-10GBFS-85OPC), D(10FA-5GBFS-85OPC), H(10FA-10GBFS-80OPC), J(5FA-15MS-80OPC), and K(5GBFS-15MS-80OPC) after 95 days of ageing.

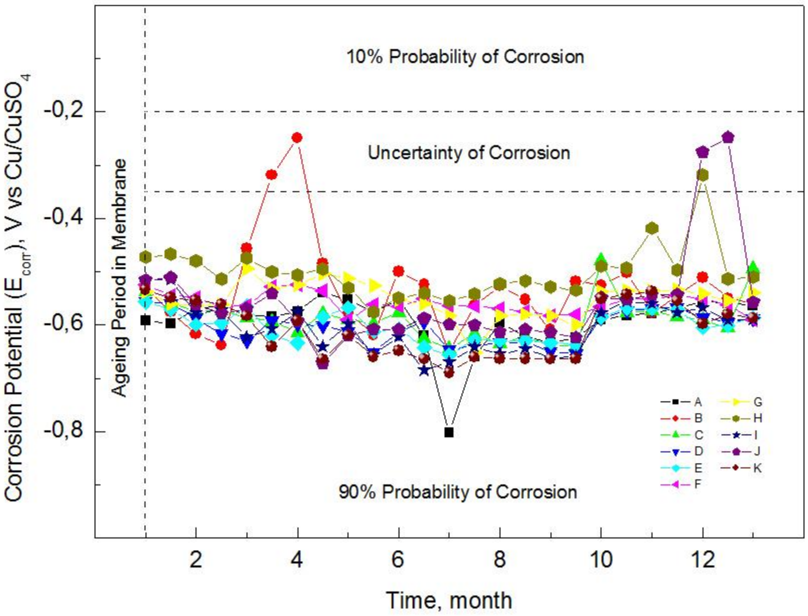

Figure. 6. Corrosion potential (E_{corr}) versus time for steel bars embedded in mortars A(100OPC), B(5FA-10GBFS-85OPC), C(5FA-10MS-85OPC), D(10FA-5GBFS-85OPC), E(5GBFS-10MS-85OPC), F(10FA-5MS-85OPC), G(10GBFS-5MS-85OPC), H(10FA-10GBFS-80OPC), I(10FA-10MS-80OPC), J(5FA-15MS-80OPC), and K(5GBFS-15MS-80OPC). The specimens were immersed in a 3.5% NaCl solution at room temperature.

Figure. 7 shows R_p values versus time for steel bars embedded in 10 mortar mixtures and the reference matrix, specimen A, immersed in a 3.5% NaCl solution at room temperature. Specimen A showed the lowest R_p value, with 0.15 kΩ cm^2 for one month of experimentation and 0.10 kΩ cm^2 for 7 months. Two differentiated groups can be observed in the tested ternary mixtures; the group with the best corrosion behavior having R_p values in the range of 0.5 kΩ cm^2 to 1.7 kΩ cm^2 while the other group has R_p values in the range of 0.2 kΩ cm^2 to 0.55 kΩ cm^2.

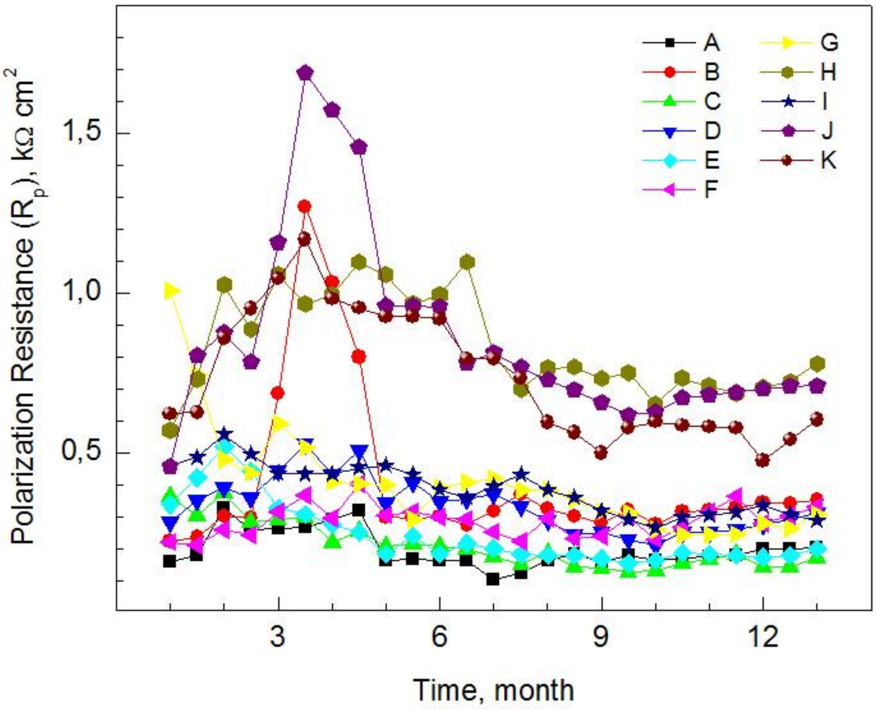

Figure. 7. Polarization resistance (R_p) versus time for steel bars embedded in mortars A(100OPC), B(5FA-10GBFS-85OPC), C(5FA-10MS-85OPC), D(10FA-5GBFS-85OPC), E(5GBFS-10MS-85OPC), F(10FA-5MS-85OPC), G(10GBFS-5MS-85OPC), H(10FA-10GBFS-80OPC), I(10FA-10MS-80OPC), J(5FA-15MS-80OPC), and K(5GBFS-15MS-80OPC). The specimens were immersed in a 3.5% NaCl solution at room temperature.

Figure. 8 shows i_{corr} and corrosion rate values, estimated from the R_p measurements, versus time for steel bars embedded in the most representative mixtures, specimens A(100OPC), B(5FA-10GBFS-85OPC), D(10FA-5GBFS-85OPC), H(10FA-10GBFS-80OPC), and J(5FA-15MS-80OPC), immersed in a 3.5% NaCl solution. Specimen A showed the highest corrosion rate, with a value of 1.8×10^{-3} mmpy for one month and 2.2×10^{-3} mmpy for 8 months of experimentation. Specimens H and J showed the lowest corrosion rate, of the order of 5.0×10^{-4} mmpy. An intermediate position was occupied by specimens B and C.

Figure. 9 shows R_n values versus time for steel bars embedded in the most representative mixtures, specimens A(100OPC), B(5FA-10GBFS-85OPC), D(10FA-5GBFS-85OPC), H(10FA-10GBFS-80OPC), and J(5FA-15MS-80OPC), immersed in a 3.5% NaCl solution at room temperature. Specimen A showed the lowest R_n. In contrast, the highest R_n value was yielded by specimen J. These results agree with the R_p values (see Fig. 7).

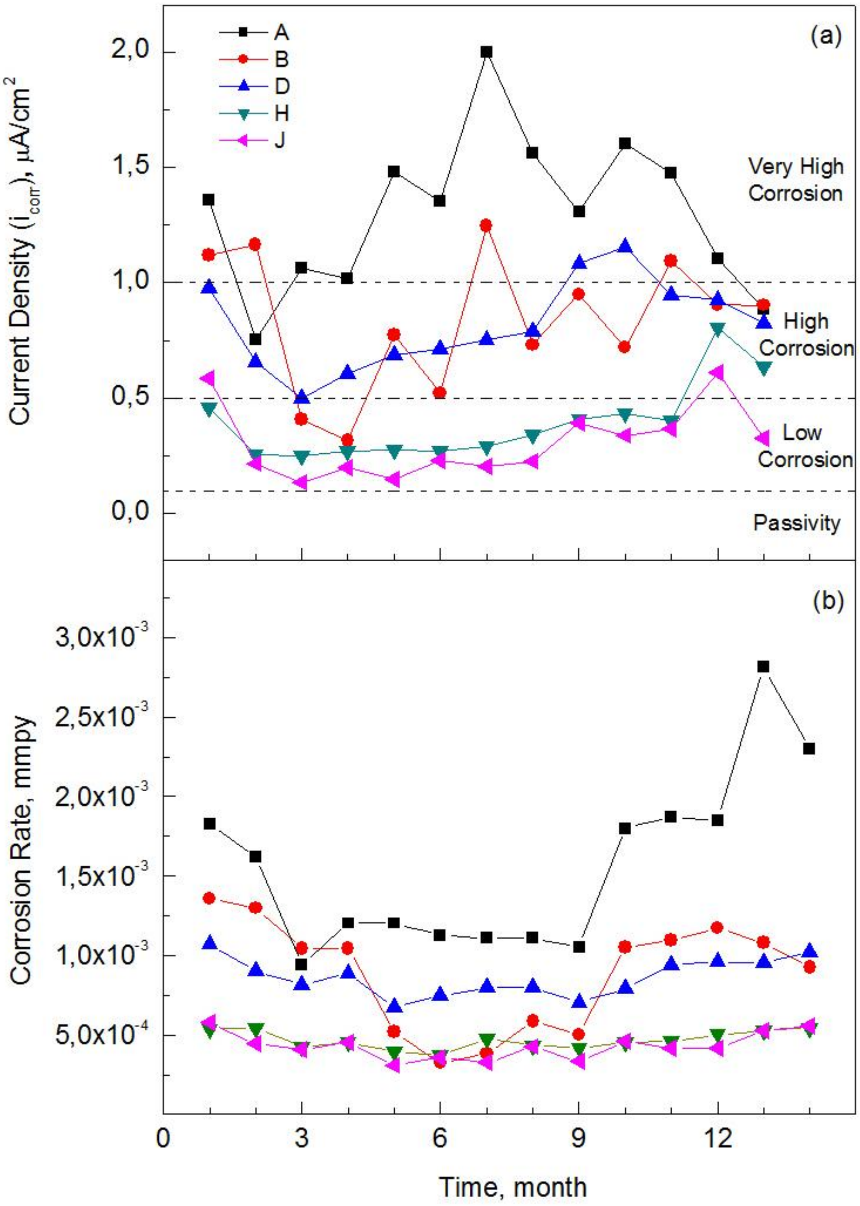

Figure. 8. (a) Current density (i_{corr}, in μA cm^{-2}) and (b) corrosion rate (in mm per year) versus time for steel bars embedded in mortars A(100OPC), B(5FA-10GBFS-85OPC), D(10FA-5GBFS-85OPC), H(10FA-10GBFS-80OPC), and J(5FA-15MS-80OPC). The specimens were immersed in a 3.5% NaCl solution at room temperature. Current density and corrosion rate values were determined using R_p measurements.

Figure. 10 shows a comparison between R_p and R_n values versus time for steel bars embedded in mortars A(100OPC) and H(10FA-10GBFS-80OPC) and immersed in a 3.5% NaCl solution. The other specimens showed similar behavior and for this reason are not included. In general, R_p and R_n methods yielded results in good agreement. High R_p and R_n values were obtained at the beginning of the experiments, probably associated with a passivation process of the steel bars, after which a progressive decrease in R_p and R_n values was observed, originated by the initiation of the corrosion process.

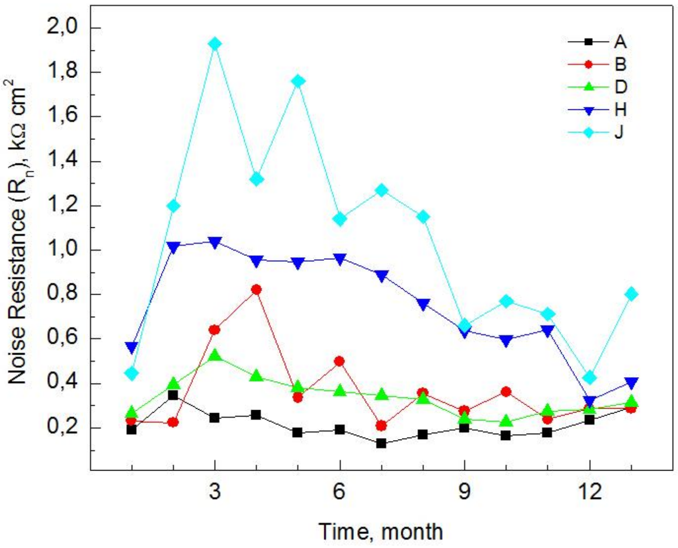

Figure. 9. Noise resistance (R_n) versus time for steel bars embedded in mortars A(100OPC), B(5FA-10GBFS-85OPC), D(10FA-5GBFS-85OPC), H(10FA-10GBFS-80OPC), and J(5FA-15MS-80OPC). The specimens were immersed in a 3.5% NaCl solution at room temperature.

4. DISCUSSION

Although the results demonstrate that ternary concrete mixtures can provide passivation to reinforcements as efficiently and permanently as conventional Portland cement, they also show that steel bar passivity depends on the substituent type and dosage used, so from a scientific, technical and economic viewpoint there is a need for more results on the subject in order to build up a data base and avoid unnecessary risks regarding the durability of reinforced structures manufactured with these new materials.

FA contains carbon particles. Carbon is more noble than steel, and it has been suggested that this may endanger service life. For concrete in which the high pH is maintained, no negative effects of coke particles are seen even at high carbon concentrations. When the pH decreases due to carbonation, a negative effect of FA cannot be excluded if combined with GBFS particles [19]. The corrosion rate calculated, in mmpy, for rebars embedded in OPC and with various FA, GBFS, and SM replacement levels are shown in Fig. 8b. From this figure it was observed that the corrosion rate for the OPC system was found to be 2.9×10^{-3} mmpy. Systems showing a corrosion rate equal to or less than 2.5×10^{-3} mmpy may be considered as improving the corrosion resistance of steel in concrete [16]. In the case of FA, GBFS, and SM systems, the corrosion rate was found to be lower at a 5-15% replacement level. A corrosion rate comparable with OPC was observed up to a 15% replacement level.

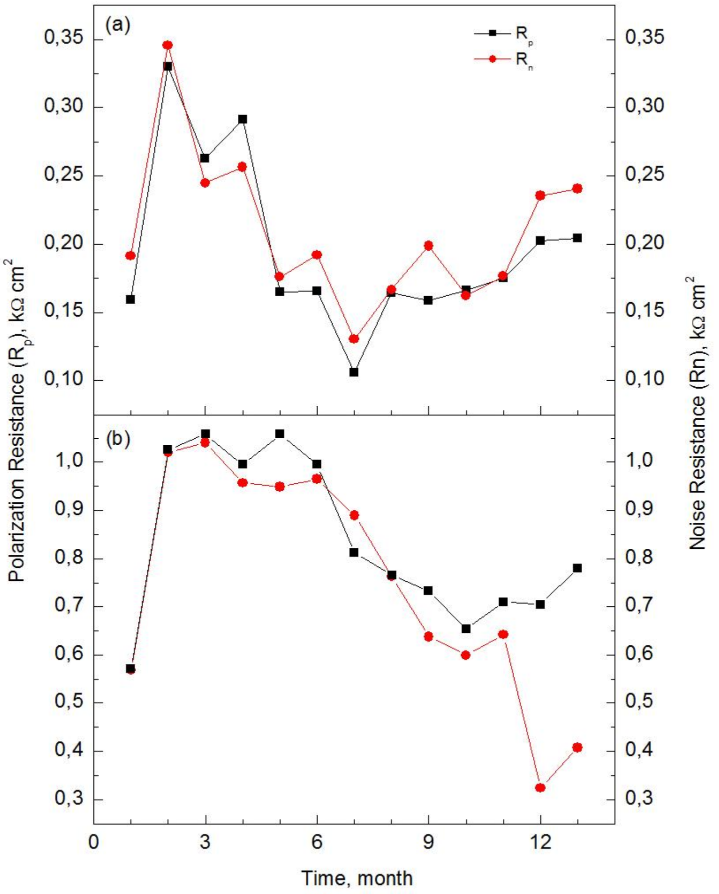

Figure. 10. Comparison between polarization resistance (R_p) and noise resistance (R_n) versus time for steel bars embedded in: (a) mortar A(100OPC), and (b) mortar H(10FA-10GBFS-80OPC). The specimens were immersed in a 3.5% NaCl solution at room temperature.

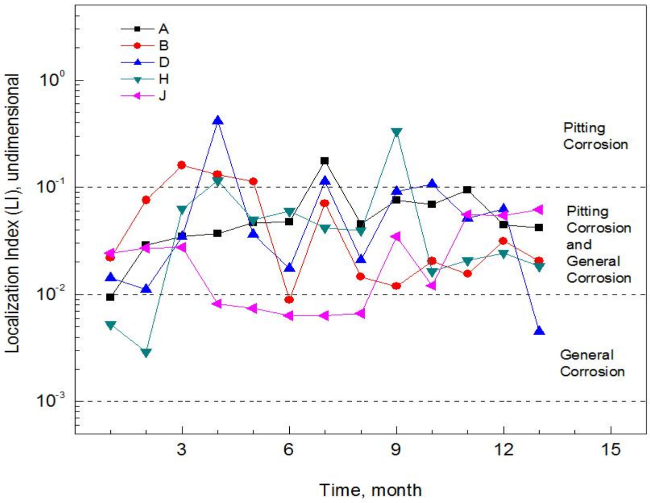

Figure. 11. Localization index (IL) versus time for steel bars embedded in mortars A(100OPC), B(5FA-10GBFS-85OPC), D(10FA-5GBFS-85OPC), H(10FA-10GBFS-80OPC), and J(5FA-15MS-80OPC). The specimens were immersed in a 3.5% NaCl solution at room temperature. IL values were determined using electrochemical noise (EN) measurements.

The improved corrosion resistance of concrete up to a 15% replacement level is due to the pore filling effect of FA and GBFS, having a small surface area of ~1 m^2 g^{-1} and ~7 m^2 g^{-1}, respectively, during the cement hydration reaction and making the concrete impermeable and thereby maintaining perfect alkalinity near the steel bar anode. Nevertheless, this explanation is less applicable to the inherent porosity of MS particles with a large surface area (~25 m^2 g^{-1}). Even though, there may be a decrease in the pH of the solution, it is expected that the increased density of FA, GBFS and also to a certain extent MS substituents inhibits the entry of oxygen and moisture that are essential for the cathodic reaction, and as a result the corrosion rate is decreased by the incorporation of FA, GBFS, and MS substituents. This corresponds with the SEM images (see Fig. 12 as an example for specimen H) as the concrete without substituents produces more attack than those with substituents.

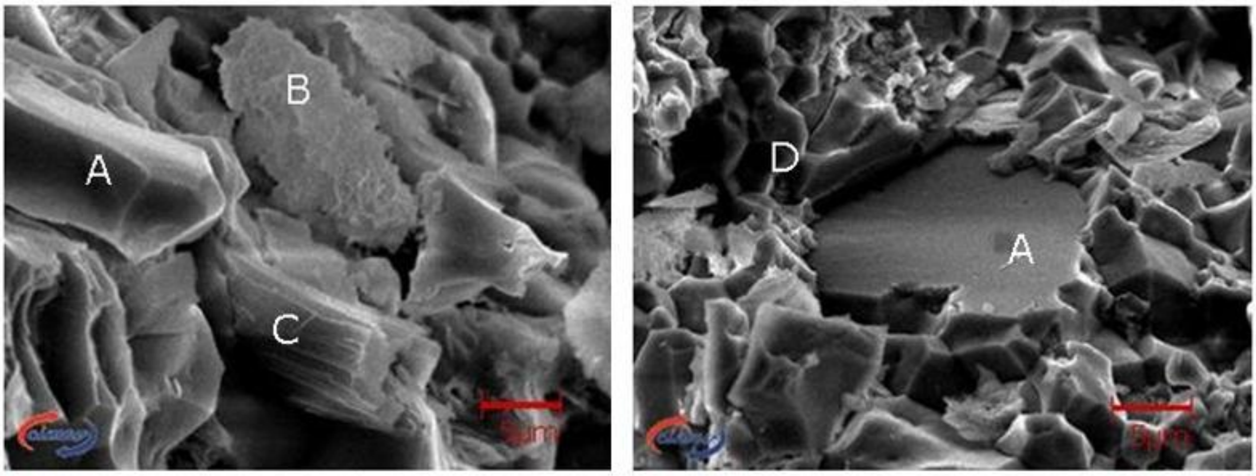

Figure. 12. SEM micrographs after 13 months immersion in a 3.5% NaCl solution for mortar H(10FA-10GBFS-80OPC). A calcite, B portlandite, C tobermorite (C-H-S gel), D granulated blast furnace slag.

5. CONCLUSIONS

Mechanical strength measurements indicated that the best properties were obtained using the mixture labeled D, replacing conventional Portland cement with 10% fly-ash (FA) and 5% granulated blast furnace slag (GBFS) substituents, achieving a 16% higher mechanical strength than conventional Portland cement. In contrast, increasing the addition of micro-silica (MS) up to 15% and with a FA or GBFS content of only 5% reduces the mechanical strength value. The best mechanical strength and corrosion behavior properties were obtained using the mixtures denominated I and J, containing 10FA-10GBFS-80OPC and 5FA-15MS-80OPC, respectively.

The steel corrosion current density, in the tested experimental conditions, yielded lower values for the ternary mixes made with micro-silica (MS) than for 100% ordinary Portland cement (OPC). In contrast, the type of attack obtained using a localization index for ternary mixes containing MS is general corrosion, which is better corrosion behavior than the other mixes which showed pitting corrosion.

The mixture with the best properties, the highest mechanical strength and corrosion resistance, was obtained by replacing OPC with 10% fly-ash (FA) and 10% granulated blast furnace slag (GBFS). The mixture containing 15% micro-silica (MS) showed good corrosion behavior and poor mechanical strength, and for this reason it is recommended for temporary remedial uses and for splashes in marine structures.

Finally, it would also seem appropriate to emphasize that these observations and conclusions cannot be expected to apply generally to Portland and fly ash, micro-silica and ground granulated blast furnace slag cement-based mortars, and the nature and integrity of the layer of solid hydration products formed in close proximity to the embedded steel (not investigated in the paper) may have an important role in controlling passivation and depassivation of the metal.

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