

Accelerate techniques to evaluate the corrosion susceptibility of steel in blended mortars exposed to chloride environment

Técnicas aceleradas para evaluar la susceptibilidad a corrosión de aceros embebidos en morteros con adiciones minerales expuestos a cloruros

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Abstract

This paper studies the corrosion behavior of reinforced Portland cement mortars without addition and with additions of metakaolin (MK) and silica fume (SF). The proportion of the addition was 10% as cement replacement. Specimens with and without reinforcement were prepared. First, the compressive strength, absorption, porosity, and chloride permeability were determined. The reinforced mortar specimens were exposed to chloride (NaCl 3.5%). Two techniques of accelerated corrosion, wetting-drying cycles and impressed voltage were applied. In both cases was used water as reference. The progress of the corrosion process in the steel was accomplished by using electrochemical linear polarization resistance (LPR). The results showed that the additions increased the compressive strength of OPC mortar and contributed positively to reduce the chloride permeability. The results of corrosion show the same tendency regardless of the techniques used. The best performance corresponds to the mixture containing MK, followed by SF and OPC. The corrosion of the specimens OPC is reduced up to 90% when MK is used as addition. In this study, we suggest to use the impressed voltage technique due to the short time to obtain corrosion results.

Keywords: Metakaolin; silica fume; blended mortars; chloride attack; corrosion.

Introduction

The armed concrete is one of the most important materials in the construction sector; while the concrete is a ceramic material that withstands squeezing efforts,

its combination with structural steel gives resistance to traction, torsion, cut and flexion. The main problem is that this composite material is the corrosion of steel armors that according to its advance causes losses to mechanical properties and can lead to an structural failure. In general, corrosion is caused by the interaction of aggressive environments, in its majority in presence of chloride ions and/or carbonation.

The attack by chlorides to the concrete can come by two main reasons: the first, the chloride ions can be present inside the concrete mix (e.g contaminated aggregates, sea water or contaminated, cement and/or additives with high contents of chlorides); in the second, the chlorides once penetrate the concrete they distribute as linked chlorides and free chlorides. The first correspond to those that are chemically linked when reacting with the tricalcic aluminate (C3A) present in the paste thus forming calcic chloroaluminates, composites known as "Friedel salt" ($3\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot\text{CaCl}_2\cdot 10\text{H}_2\text{O}$); on the contrary, the free chlorides spread themselves until reaching the steel where they accumulate until achieving a critical concentration, which has the capacity to destroy a passive layer of steel and start the corrosive process (Aguirre and Mejía, 2013).

In this sense, the concrete durability standards specify the classes of exposition in which an structure in service can be affected by the presence of the chlorides (NTC5551; EN206-1), and include the induced corrosion by the chlorides coming from the sea water (structures exposed to the environment or to the immersion to salt water), and the induced corrosion by chlorides of different origin than the marine (humectation-dry cycle, specifically bridge structures, pavements and parking zones). In general the mobility of the chloride ions inside the concrete is related with its permeability; thus some important factors to be in account are: the water/cement relation, the type and cement proportion and the curing process (Song *et al.*, 2008; Güneysi *et al.*, 2007). Besides, the presence of additions (pozzolanic and steel) in the concrete, such as metakaolin (MK) and silica fume (SF), modify the porosity and reduce the permeability, thus improving the resistance to chloride penetration in the concrete, though there are addition limit percentages in which the increase in the water demand could affect the (Angst *et al.*, 2009; Aguirre and Mejía, 2013; Song *et al.*, 2010).

MK is a pozzolanic of the aluminosilicate kind highly reactive obtained by the calcination of the kaolinitic clay at temperatures between 500°C and 800°C (Mejía de Gutiérrez *et al.*, 2000; Mejía de Gutiérrez *et al.*, 2004; Shi *et al.*, 2012; Kannan and Ganesan, 2014). The

addition of MK to the concrete, in orders from the 10% to 15%, increased the compression and flexion resistance and positively contributed to the (Sabir *et al.*, 2001; Courard *et al.*, 2003; Ramezaniyanpour y Jovein, 2012; Poon *et al.*, 2006; Siddique y Klaus, 2009; Parande *et al.*, 2008, Kim *et al.*, 2007, Güneysi *et al.*, 2013; Keleştemur and Demirel, 2015). The silica fumes are a sub product of the silicon and ferrosilicon industry; the reduction of high purity quartz to temperatures superior to 2000°C produce various silica fumes which oxidize and condense to form small amorphous silica particles. SF has a silica content of 85% and 9%, fine particle size (0,1 to 0,5 µm) and a great specific surface (Siddique, 2011, Shi *et al.*, 2012). Saojeng and Weiting (2013), assert that when using the SF as a replacement for cement the concrete's permeability is reduced and the probability of reinforce steel, particularly in chlorides presence. (Khan and Siddique, 2011). To reach high compression resistances and low chloride spread coefficients it is suggested a use between 5% and 10% of the cement's (Zhang y Ba, 2013; Shekarchi *et al.*, 2009; Farahani *et al.*, 2015).

The current research was scoped in evaluating the susceptibility to corrosion by chloride attack in armed mortars of Portland cement, without and with addition of the MK and SF in a 10% proportion, using accelerated techniques of lineal polarization with analysis method. The objective was, besides comparing the performance in the two additions in chlorides presence, verify the effectiveness of the accelerated techniques of inclusion in the aggressive agent as a characterization method in added cements.

Experimental procedure

Materials

For the study portland cement (OPC) and two pozzolanic additions, one aluminum kind, MK and other siliceous kind, SF. It was taken in consideration the optimum percentages of MK and SF proposed by different researches (Aguirre y Mejía, 2013; Torres-Agredo *et al.*, 2011; Song *et al.*, 2010; Siddique *et al.*, 2009); this work was chosen with a unique percentage of 10% in replace of cement for each one of the mentioned pozzolans. The chemical characterization of these materials, determined by the X-Ray fluorescence (XRF), is presented in the Chart 1. It is worth to mention that the used cement contains chalk addition in its formulation, due to this a high percentage of loss by ignition (LOI) is seen; this cement distributes itself commercially as gray cement for its general use (OPC).

Chart 1. Chemical composition of the used material

Material	LOI (%)	SiO ₂ (%)	Fe ₂ O ₃ (%)	Al ₂ O ₃ (%)	CaO (%)	MgO (%)	SO ₃ (%)
OPC	8.57	21.26	4.43	5.52	54.38	4.46	1.85
MK	1.10	49.55	0.39	47.14	0.17	1.21	0.0
HS	1.95	93.85	1.32	2.40	0.0	0.0	0.0

Source: The authors

Preparation of specimens and test techniques

For the presented study, portland cement (OPC) mortars were prepared in sand: cement proportion pf 1:2, 75, with a relation water/cementing of 0.55. In total three mixtures were done: of reference (OPC), and with substitution of 10% in each addition (10MF, 10SF). The prototypes were elaborated with and without corrugated steel of 6mm diameter, thus the test tubes without steel were used to evaluate the mechanical characteristics and the mortar's durability with embedded steel were used for the corrosive tracking (Figure 1). The mechanical resistance of the mortars was evaluated in 28 to 19 curing days and the durability properties such as absorption and porosity (ASTM C642), capillary suction (ASTM C1585) and chloride permeability (ASTM C1202) were determined in prototypes with 28 curing days. The prototypes built in the same curing age were exposed to chlorides (solution accues the 3.5 of NaCl) using two accelerated techniques humection-dry and printed voltage; these techniques are equivalent to those of natural diffusion and accelerated chloride diffusion (Andrade and Buják, 2013).

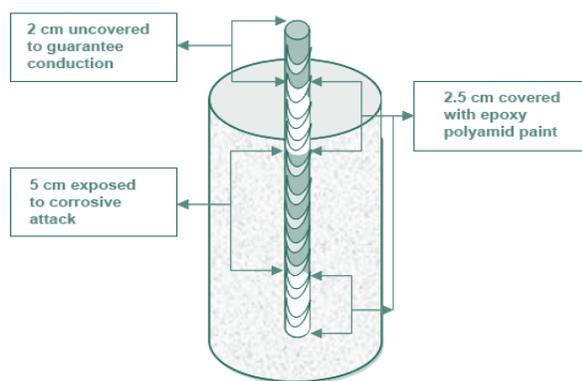


Figure 1. Built mortars scheme
Source: The authors

The corrosion tracking was held with an electrochemical technique known as lineal polarization, LPR or Rp (ASTMG 59), which is a non-destructive technique. The test was done in a *Potenciostat/Galvanostat GAMRY PC14*

equipment at 0.167 mV/s, that applied overpowers of -20 to +28 mV; as reference electrode Ag/AgCl was used. In the corrosion current calculation the Stern-Geary ($I_{corr} = B/R_p$) was used, where B is a dependent constant of the tafel slopes, whose estimated values are 26 mV or 52 mV depending in the active and passive state of the steel. In this study the "B" value used was of 0,026 V that simulate the active corrosion condition (Andrade and González, 1978; Andrade and Alonso, 1996).

Results and discussion

Mechanical properties and the mortar's durability

The resistance to compression at the age of 28 and 90 days of curing tests results (Figure 2) evidenced that the mortars with pozzolanic additions present a higher resistance that the ones without addition (OPC); the resistance rate was calculated defined as the relation between added mortar and the OPC concrete without addition. At 28 curing days the mechanic performance of the mix is highlighted with 10% MK, representing in a 51.9% regarding to OPC. The total volume of permeable pores present values that fluctuate between 19.60% and 21.08%, being added prototypes, though less permeable, the diminution is only 1.48% for the SF and 0.87 for the MK regarding the OPC test tube. The obtained results by capillary suction showed that when adding 10% MK in replace with portland cement, the effective porosity decreased in 12.3% in comparison to the OPC test tube; same way the added test tube with 10% of SF achieved to decrease capillary absorption in 35%. The capillary absorption coefficients were even a 31% lesser than the one corresponding to the OPC. The permeability test to chlorides done with MK and SF mortars reported values considered as very low permeability (≤ 1000 coulombs) regarding the ASTM C1202 standard. The test tube with 10% SF presented a load decrease of 19.6% and the test tube with the 10% MK with a 31.4% reduction; this better performance of the MK can be attributed to the pore's refinement and the matrix densification, forming Friedel salt that links chloride ions preventing its diffusion to the reinforcement bar. In general, these results show the same tendencies to the reported in previous research (M. de Gutiérrez *et al.*, 2000; Torres-Agreto *et al.*, 2011; Rao, 2001; Siddique y Klaus, 2009; Kim *et al.*, 2007; Ramezaniapour and Jovein, 2012; Hassan *et al.*, 2012; Badogiannis *et al.*, 2015a).

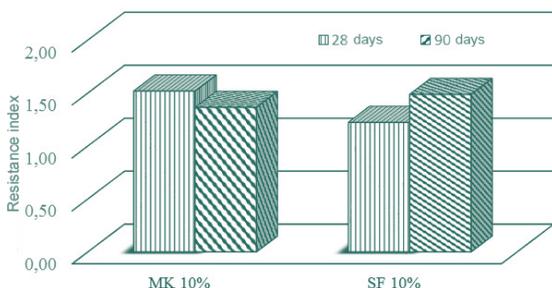


Figure 2. Resistance to the compression at 28 and 90 days
Source: The authors

Corrosion tests

Accelerated technique of humectation-dry cycle (H-D)

In the humectation/dry (H-D), the prototypes that were put completely immerse in an aqueous solution of NaCl at 3.5% by a week and forwardly are exposed to a dry outdoors at room temperature by another week; this way each H-D cycle were of two weeks, equivalent in total to 98 days of exposition. At the end of each cycle H-D was applied was applied to the electro-chemical LPR was done, based on which the current corrosion (I_{corr}) was calculated; this essay was used equally to the exposed test tubes at the reference environment, which was safe water. In the Figure 3 I_{corr} values at the end of each exposition cycle; the exposition cycle 0; the cycle 0 represents the initial state where the test tubes haven't been exposed to the H-D test.

It is observer that in the two exposition ways, water and NaCl 3.5%, present an increasing corrosion tendency with the elapsing of the cycles, however in the corrosive way that simulates the marine (NaCl solution), the increase of the current is superior compared to the reference environment. It is worth to mention that the test tubes added with pozzolanic, the negative effect in the two environments is less pronounced, what says that the contribution of the used additions to the best material presence of chlorides. The test tubes contain a 10% MK replacement of cement that report values less than $0.5 \mu A/cm^2$ at the end of cycle 7, while the 10% of SF and OPC report values of $2.5 \mu A/cm^2$ and $5.0 \mu A/cm^2$ respectively. The MK's performance is attributable, besides the densification of the structure, to its high content of amorphous character aluminum that allows forming the Friedel salt and this way avoid the penetration of chloride ions thus reducing the corrosion speed (M. de Gutiérrez *et al.*, 2000; Vejmelková *et al.*, 2010; Badogiannis *et al.*, 2015b). Considering the limit values in the corrosion current in Char 2, it can be affirmed that the

corrosion level at the end of the H-D test for the prototypes immersed in the despicable water and the added 10% with the MK and immersed in NaCl is low; in the remaining level, corrosion is high.

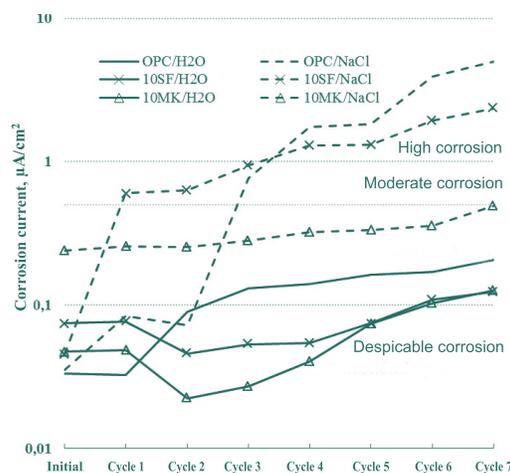


Figure 3. Corrosion current of submitted specimens to H-D
Source: The authors

Chart 2. Corrosion current criteria

I_{corr} ($\mu A/cm^2$)	Nivel de Corrosión
≤ 0.1	Despicable
0.1 – 0.5	Low
0.5 – 1	Moderate
> 1	High

Source: The authors

Technique with printed voltage (PV)

The technique denominated as printed voltage (PV) is an accelerated corrosion technique that indirectly gives information about the permeability characteristics of a concrete (Topçu and Bogă, 2010). The chloride ions material entry is accelerated through a process of application of a voltage from an external source the steel bar of the reinforce prototype is constituted in a working electrode and connects itself through to the positive terminal of the source, while a counter electrode, that can be a steel plate acts as an anode and the stainless steel foil as a cathode, at the same time the NaCl solution is the electrolyte. Although the different authors use a voltage that may vary between 2V and 55VV (Ha *et al.*, 2007; Diab *et al.*, 2011; Horsakulthai *et al.*, 2011; Topçu and Bogă, 2010), this study was based in the NT BUILD 356 standard in which is recommended a constant 5v voltage. Along the test a change was registered in the current and the necessary time was identified to evidence the fracture of the prototype (Figure 4). A maximum time in

the OPC test tube of 2270 minutes (Approx. 38 hours) and for the test tube with 10% SF it was 3625 minutes (Approx. 60.5 hours) that means 60% superior, which confirms the mortar's performance with addition.

As well as the initial current like the appearing time in the crack are considered factors in the technique to define the higher resistance to the chloride penetration and the susceptibility to the steel corrosion, due to that the consequence of the volume of the corrosion products, can be between 2.5 and 6 times the steel's, a crack is produced at the same time the current's increase is appreciated. When the start of the vertical fissure is produced to inferior values than those of the current, it is considered that the propagation speed of the corrosion is very slow; same way the a small current at the beginning of the test is an indicative that the resistance offers the entry to chloride ions coming from the exterior. In both cases these factors match with the reported for the added cements. This technique has been applied to the study of additions such as fly ash, ironworks slags, rice seed's ash, among others (Ha *et al.*, 2007; Horsakulthai *et al.*, 2011; Topçuandy Bogă, 2010; Bogă y Topçu, 2012; Ferraro *et al.*, 2012).

Diab *et al.* (2011) applied the PV technique to concretes with and without MK and SF addition in percentages up to 25% in replacement of the cement, using voltage variables in a 15 V and 55 V ranges report a higher resistance to corrosion for the added with 15% of MK; regarding the applied voltage affirm that an increase is translated in a lesser appearing time of the crack. Basunbul *et al.* (1994) y Saraswathy and Song H-W (2008) compare this technique with the permeability of the chloride (ASTM C1202) in mortars, with or without addition, and affirm that the two techniques present adequate sensibility to evaluate coating materials

Combined technique

For the application of the combined technique (VP + H-D), departing from the previous results it was defined the required time for the exposition of all the test tubes at BP, this was of 1580 minutes (Approx. 26 hours). Finished this process it was submitted the prototypes to 3 H-D cycles. In the Figure 4 it's observed the state of the armors at the end of the test with two evaluated mortars, OPC (Figure 4a and 4b) and the 10% of the MK (Figure 4c and 4d), such as it was appreciated in the stereomicroscope MODEL GX41F. Here is evidenced a higher degree of deterioration in OPC.

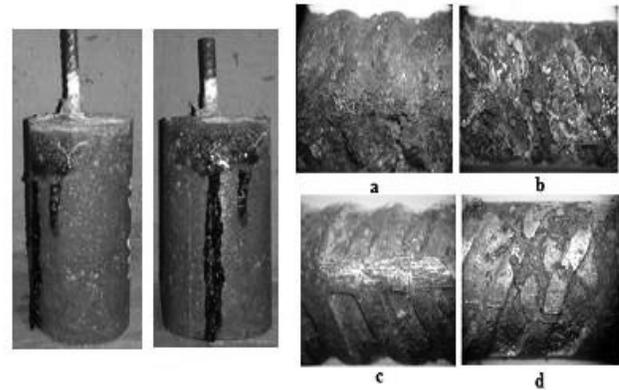


Figure 4. Prototypes with steel reinforcement after the exposure to chlorides (Printed voltage)
Source: The authors

In the Figure 5 the values of the corrosion current are shown after the application of this printed voltage technique (PV), in every case there is a meaningful increase of the current regarding the initial one. For the OPC this increase is 4.5 times while for the 10% SF and the 10% MK is 1.8 times. When comparing the different mixtures that the MK appreciates it is located in the despicable corrosion levels. In the exposed specimens to the H-D cycles, it is observed that at the end of the third cycle that the I_{corr} of the 10% of Mk and 10% of SF is a 20% and 40% respectively reported by OPC. This shows the first case, where the applied technique answers positively to evidence the higher or lesser susceptibility of the process mix to the corrosive process (Güneyisi *et al.*, 2013), pair that the best performance of the added mixes, specially added with MK.

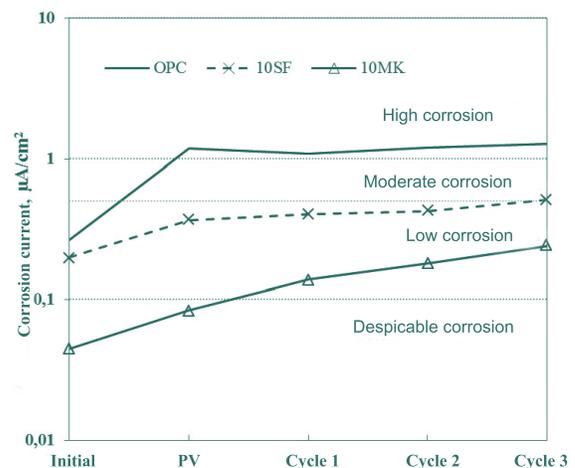


Figure 5. I_{corr} ($\mu A/cm^2$) in the NaCl solution. VP and VP+H-D
Source: The authors

Conclusions

Based on the obtained results in this project, it is shown that the pozzolanic additions in the cement improve in a relevant way the mechanical and durability properties, thus the performance resistance to the chlorides in the built mortars.

Of the evaluated additions, it is highlighted the mechanical performance with the 10% MK, represented in a 51.9% increase regarding OPC. In the same mixture it presents a better performance in the study of corrosion, surpassing even the behavior of the mixtures with SF addition.

Of the accelerated applied techniques for studying the corrosion in presence of chlorides it can be concluded that the accelerated humectation-dry (H-D) as well as the printed voltage (PV), allow to evidence the susceptibility at the corrosive process of the specimens; however the printed voltage (VP) technique presents an advantage when throwing results in lesser time. The combination of VP and the H-D process was equally effective to evidence the susceptibility in each evaluated material in the presence of chloride.

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