





Study of a repair technique in carbonated blended mortars: Electrochemical re-alkalization

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Abstract

The present article studies the influence of two pozzolanic additions, metakaolin (MK) and silica fume (SF), and the water/binder ratio (w/b) on the electrochemical re-alkalization (ER) of reinforced mortars as a technique to prevent reinforcement corrosion in carbonated concrete. Mixtures with a 10% (by weight) addition as a replacement for cement at w/b ratios of 0.45 and 0.65 were prepared. Test specimens were exposed to carbonation in a chamber under controlled conditions (1% CO₂, 65%RH, and 25°C), until reaching two levels of carbonation (50% and 100%). ER was applied for a period of 15 days using sodium carbonate as the electrolyte and a current density of 1 A/m². From the evaluated additions, MK is highlighted, which generated a resistance increase of 53.51% relative to that of the reference mixture and contributes to improvements of durability properties. Regarding the ER technique, the specimens with greater w/b ratios and partial carbonation were more effective.

Keywords: Carbonation, Electrochemical Re-alkalization, Blended Mortars, Silica Fume, Metakaolin

Estudio de una técnica de reparación aplicada a morteros adicionados carbonatados: Realcalinización electroquímica

Resumen

En este artículo se estudia la influencia de dos adiciones puzolánicas, metacaolin (MK) y humo de sílice (HS), y la relación agua/cementante (a/C) en el proceso de realcalinización electroquímica (RE) de morteros reforzados como método de prevención de la corrosión en concretos carbonatados. Especímenes con un 10% en peso como reemplazo del cemento de MK o HS y relaciones a/C de 0.45 y 0.65 se expusieron a condiciones climáticas controladas (1% CO2, 65%HR y 25°C), hasta alcanzar dos niveles diferentes de carbonatación (50% y 100%). Se aplicó RE por 15 días con una densidad de corriente de 1 A/m2 utilizando como electrolito carbonato de sodio. Los especímenes adicionados con MK y relación a/c 0.45 presentaron un incremento notable en la resistencia a compresión (53.51%) y mejores propiedades de durabilidad. Respecto a la técnica RE, los especímenes con mayores relaciones a/c y carbonatación parcial presentan una mayor efectividad.

Palabras clave: Carbonatación, Realcalinización Electroquímica, Morteros adicionados, Humo de sílice, Metacaolín

1. Introduction

Since the last decade, concrete has become the most important material in the construction field worldwide, reaching a production of approximately 1.5 to 3 tons per capita per year in the industrialized world [1]. Concrete is a ceramic material that can withstand a wide range of compressive stress. However, concrete is susceptible to cracking against other types of mechanical stresses, such as bending, traction, shear, etc. To improve its mechanical properties, reinforced concrete can be used, which is a composite material obtained from combining concrete and reinforcing steel. Reinforced concrete is widely used in the construction of buildings, bridges, tunnels, and skyscrapers [2].

Although this material is considered durable, after a certain period of time, it suffers from deterioration primarily

caused by environmental agents and service conditions, which in addition to the loss of concrete durability, contribute to the corrosion of the reinforcing steel [2,3].

Concrete is an alkaline material with a pH between 12.6 and 13.6. Under these pH conditions, steel spontaneously forms a protective passive layer. However, the latter can be destroyed by aggressive agents (chloride ions and/or carbon dioxide), thus causing its depassivation. Specifically, carbonation is a process where atmospheric CO₂ enters into the concrete and decreases its alkalinity by reducing the pH to approximately 9, and consequently, the passive layer is destabilized, which allows corrosion of the reinforcing steel to occur [2-4]. The CO₂ penetration rate depends on environmental factors and on factors related to the concrete itself; therefore, it can occur in urban and industrial Relative humidity is an important environments. environmental factor, which ranges between 50 and 70% because the diffusion of CO₂ does not occur in completely dry concretes nor in fully saturated concretes. A temperature increase can also accelerate this phenomenon. Another crucial parameter is the CO₂ concentration, which in urban environments, can reach values of approximately 0.1%. Factors associated with concrete that contribute to CO₂ propagation include an inadequate curing process, poor compaction, and high w/b ratios that generate more permeable concretes [2].

The concretes durability standards [5-7] specify the types of exposures under which a structure can be affected by carbonation-induced corrosion during its service life. These standards specify the conditions of high humidity, medium humidity, or wetting-drying cycles. There are a variety of ways to prevent concrete deterioration, such as the design of the material and control of the construction process. The incorporation of additions (pozzolanic or steel related) into concrete, such as metakaolin (MK) and silica fume (SF), which modify porosity and reduce permeability, have been studied to evaluate the resistance to CO₂ diffusion; however, the results have been controversial because in general, blended concretes are more susceptible to carbonation than non-blended concretes, and therefore, a more demanding curing process is required for the pozzolanic reactions to reach their development [2,8-12].

Other alternative methods include rehabilitation electrochemical techniques, such as electrochemical realkalization (ER) and cathodic protection, which can be applied to in-service concretes. These two techniques are based on the application of a cathodic current; for realkalization, the current is temporary, whereas for cathodic protection, it is permanent [13]. During the electrochemical re-alkalization process, a direct current of 1-2 A/m² is applied with respect to the steel surface (cathode), and an electrochemical cell is formed with an auxiliary anode; sodium carbonate is generally used as an alkaline electrolyte, and its duration lasts between 3 to 21 days [13,14]. During the treatment process, electrochemical reactions are generated on the cathode surface, producing hydroxyl ions, and therefore, the pH in the surroundings of the reinforcing steel increases. Simultaneously, the alkaline electrolyte enters into the concrete interior, which increases the pH from the surface towards the interior and acts as a collector of the

carbon dioxide present in the cementitious matrix [15]. This technique was developed at the end of the 1980s by John Miller in Norway, was patented by NORCURE ® and is primarily used in Northern Europe [14,16]. Regarding the technique efficiency, Yeih & Chang [15] reported that this treatment increases the pH values around the reinforcing steel (to approximately 11) despite the compressive resistance, elastic modulus, and adherence to the concrete decreasing linearly as the passing current and time increase. Ribeiro et al. [17] performed studies with pozzolanic cements, from where it was concluded that these cements require a greater passing charge density and longer treatment time to realkalize the entire structure. Corrêa de Araújo [18] evaluated the use of different electrolytes, such as calcium hydroxide, potassium hydroxide, and sodium carbonate, which determined that the latter two are efficient in both the structure repassivation and its re-alkalization. González et al. [19] proposed the use of potassium carbonate to maintain a more alkaline medium in the anodic region, which takes advantage of the conductive and mobility character of K+ to allow for a lower applied voltage necessary for current circulation. Tong et al. [20] demonstrated the increase of the pH around the rods after one year of application and corroborated the decrease of the corrosion activity, though the authors could not verify the repassivation of the steel rods. Bertolini et al. [16] mentioned that the application of the technique presents several potential secondary effects, such as hydrogen embrittlement, alkali-aggregate reaction, and loss of adherence, and claim that an extremely elevated current density can affect the concrete microstructure.

As shown by different published studies, ER is an effective method to solve the problem of carbonated concretes aimed at extending their useful life [15,17,19-23]. However, several authors claim that steel repassivation is not achieved, and thus, it is suggested that the technique should be used as a prevention method, i.e., before corrosion reaches the reinforcing steel [20,24].

The present research determines the susceptibility to carbonation of Portland cement mortars with and without the addition of MK and SF at a 10% proportion and evaluates the application of the electrochemical re-alkalization (ER) technique as a method to prevent corrosion of the steel embedded in specimens carbonated at different CO_2 penetration levels.

2. Experimental Procedure

2.1. Materials

Ordinary Portland cement (OPC) and two pozzolanic additions, MK and SF, were used to produce the mortars. A cement replacement percentage of 10% was chosen for each of the additions, which accounts for the results of previous investigations [2,25-27]. The chemical characterization of these materials, which was determined by X-ray fluorescence (XRF), is presented in Table 1. It should be noted that the OPC corresponds to commercial cement blended with limestone.

Table 1. Chemical composition of the MK,SF, and cement (OPC) used.

LOI ^a	SiO ₂	Fe ₂ O ₃	Al ₂ O ₃	CaO	MgO	SO_3
	(%)	(%)	(%)	(%)	(%)	(%)
8.57	21.26	4.43	5.52	54.38	4.46	1.85
1.10	49.55	0.39	47.14	0.17	1.21	0.0
1.95	93.85	1.32	2.40	0.0	0.0	0.0
	LOI ^a 8.57 1.10 1.95	LOI ^a SiO ₂ (%) 8.57 21.26 1.10 49.55 1.95 93.85	LOI ^a SiO ₂ Fe ₂ O ₃ (%) (%) 8.57 21.26 4.43 1.10 49.55 0.39 1.95 93.85 1.32	LOI ^a SiO ₂ Fe ₂ O ₃ Al ₂ O ₃ (%) (%) (%) 8.57 21.26 4.43 5.52 1.10 49.55 0.39 47.14 1.95 93.85 1.32 2.40	LOI ^a SiO ₂ Fe ₂ O ₃ Al ₂ O ₃ CaO (%) (%) (%) (%) 8.57 21.26 4.43 5.52 54.38 1.10 49.55 0.39 47.14 0.17 1.95 93.85 1.32 2.40 0.0	LOI ^a SiO ₂ Fe ₂ O ₃ Al ₂ O ₃ CaO MgO (%) (%) (%) (%) (%) (%) 8.57 21.26 4.43 5.52 54.38 4.46 1.10 49.55 0.39 47.14 0.17 1.21 1.95 93.85 1.32 2.40 0.0 0.0

^aLOI: Loss on ignition at 1000 °C.

Source: The authors

2.2. Specimen Preparation

In the present work, mortars of Portland cement (OPC) were prepared using a cement:sand proportion of 1:2.75 with two different water/binder (w/b) ratios of 0.45 and 0.65. Six different mixtures were produced: two reference OPC specimens (OPC45 and OPC65) and four specimens corresponding to the specimens with a replacement of 10% for each addition and their respective w/b ratios (MK45, MK65, SF45, and SF65). A corrugated steel rod with a diameter of 6 mm was placed in the center of several specimens. The first specimens without steel were used to evaluate the mechanical properties and durability and to monitor the carbonation process, and the specimens with steel were used for electrochemical monitoring (Fig. 1).

2.3. Testing techniques

The following properties were tested in the simple mortars: compressive strength, capillary suction [28], and electrical resistivity [29]. Tests were performed on the specimens after 28 days of curing. The carbonation test was performed on specimens with and without reinforcing steel at a curing age of 28 days inside a climatic chamber under controlled conditions of CO_2 concentration (1%), relative humidity (65%), and temperature (25°C). The carbonation front was monitored through the cross-sections of the mortars removed from the chamber at different ages and the subsequent spraying of the surface with a 1% phenolphthalein aqueous solution using the pH as the developer. Based on the results, the exposure time in the chamber was determined to obtain the carbonation levels in the reinforced specimens, a partial level (50%) and a complete level (100%). Subsequently, the rehabilitation technique was applied (re-alkalization) to each of the specimens. For the steel corrosion study, the linear polarization resistance technique was applied, Rp [30]; this test was performed using Potentiostat/Galvanostat GAMRY PC14 equipment at 0.167 mV/s with applied overpotentials of -20 to +20 mV; Ag/AgCl was used as the reference electrode material. The calculation of the corrosion current was performed by the Stern-Geary equation (1).

$$Icorr = \frac{B}{Rp} \tag{1}$$

where B is a constant that depends on the Tafel slopes, whose estimated value is 26 mV or 52 mV depending on the active or passive state of the steel, respectively. In this study, the value of B used was 0.026 V, which stimulates the corrosion active condition [31,32].



Figure 1. Design of the test sample. Source: The authors

2.4. Electrochemical Re-alkalization (ER)

The ER process was implemented according to the NACE Standard Practice SP0107-2007 [33], for which the following components were used: external anode, galvanized steel mesh: alkaline electrolyte, sodium carbonate Na₂CO₃ at 1 M; cellulose pulp placed between the external anode and the concrete surface; and current source. For this study, a current density of 1 A/m² was applied for 15 days, which corresponds to the current density used by different researchers [15,19,21,23]. Regarding the assembly, the external anode is fixed on the concrete surface, which is embedded in the alkaline electrolyte and is connected to the positive pole of the source, and the reinforcing steel is connected to the negative pole of the source. For this study, several specimens were connected to a single source; therefore, a parallel connection was set for each of them. To ensure the passage of the selected current density, a variable resistance of 1 k Ω was used to adjust the current passing through each specimen, and a resistance of 1 Ω was used to monitor the potential difference between the anode and the cathode. Therefore, the current can be verified by means of Ohm's law (Figs. 2 and 3).

Once the ER process ended, the verification of the realkalized front in the concrete was performed by applying the phenolphthalein indicator, and the electrochemical monitoring of the corrosion state in the steel was performed using Rp.



Figure 2. Schematic assembly of the experiment. Source: The authors



Figure 3. Monitoring during the re-alkalization Source: The authors

3. Results and discussion

3.1. Mechanical properties

Fig. 4 shows the compressive strength of the specimens at a curing age of 28 days for the two tested w/b ratios (0.45 and 0.65). The figure shows that the increment in the w/b ratio affects the mortar mechanical properties [34] as expected. In general terms, it is also observed that the blended mortars exhibited a greater resistance. It should be noted that in the specimens with an addition of SF at a w/b ratio of 0.65, the SF particles agglomerated, a phenomenon to which the lower resistance is attributed. The resistance reported by the test samples blended with MK after 28 days of curing should be highlighted, which represents a 53.51% difference with respect to the reported value of OPC at the same w/b ratio of 0.45.

3.2. Capillary absorption

Table 2 shows the capillary absorption coefficients K $(Kg/m^2s^{1/2})$ and the resistance to water penetration m (s/m^2) . The values show that the addition of pozzolanic materials reduces the capillary absorption coefficient (K_{SUC}) and increases the resistance to water penetration (m). The w/b ratio behaves in the opposite manner. The greatest increase of m is observed with the addition of MK, which is a 32.5% and 125% difference with respect to OPC for w/b ratios of 0.45 and 0.65, respectively. These results agree with what has been found by different researchers and with reported compressive strengths [25,35,36].

3.3. Electrical Resistivity

The electrical resistivity of the specimens was determined using the four-point method, also called the Wenner method, for which the resistivity meter, *Resipod Proceq*, was used with a distance of 38 mm between the points. Specimens with dimensions of 7.65 cm \times 15.24 cm were used. Fig. 5 shows the electrical resistivity values, which indicate that the blended specimens presented a greater electrical resistivity with respect to OPC. Noticeably, this property is increased as the w/b ratio decreases. The greatest resistivity is reported for the OPC+MK specimens with a resistivity increase of 1.93 and 2.00 times that of OPC for w/b ratios of 0.45 and 0.65, respectively. This behavior has been reported in previous investigations by a variety of authors [37-39].

3.4. Carbonation front

Fig. 6 shows the carbonation depth temporal behavior for the different concrete specimens during the exposure period of accelerated carbonation. According to the dimensions of the test samples, partial carbonation (50%) is reached at a carbonation front of 12.7 mm, whereas total carbonation is reached at 25.4 mm. From the figure, it is observed that, in general, the mixtures with a w/b content of 0.65 reach the carbonation level in less time; the effect of the w/b ratio increase was more critical for the non-blended mixture. From the evaluated additions, the better performance of MK is highlighted. In this case, under the test conditions (CO₂ 1%, RH 65%, 25°C), a carbonation depth of 50% was obtained



Figure 4. Compressive strength of the specimens Source: The authors.

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Parameters	of ca	pillary	absor	ption.

Table 2

Specimen	w/b ratio	Ksuc	m	
		$(Kg/m^2s^{1/2})$	(10^7 s/m^2)	
OPC	0.45	0.053	3.20	
	0.65	0.210	0.39	
MK 10%	0.45	0.044	4.24	
	0.65	0.135	0.88	
SF 10%	0.45	0.046	3.58	
	0.65	0.143	0.80	
Source: The outhors				

Source: The authors

after 77 and 93 hours for the mixtures with w/b ratios of 0.65 and 0.45, respectively, and a complete carbonation of the specimen occurred after approximately 235 hours with values that exceeded up to 80% more than those that were obtained with SF. For the mixtures blended with SF, an increment in the carbonation depth is observed with respect to OPC.

3.5. Electrochemical Re-alkalization

As was previously mentioned, the ER technique was applied to specimens with two carbonation levels, partial (50%) and complete (100%). The exposure times in the climatic chamber were obtained from the data in Fig. 6. The re-alkalization process efficiency was evaluated by the phenolphthalein test and the determined linear polarization resistance.

Fig. 7 shows the specimens before and after ER. A magenta coloration can be observed after the ER treatment, which is indicative of non-carbonated regions in regions that were previously colorless and is where the pH increase is deduced



Figure 5. Electrical resistivity of the mortars Source: The authors



Figure 6. Carbonation depth of the specimens exposed to accelerated carbonation (1% CO2, 65% RH, 25°C) Source: The authors

in the specimens for both carbonation levels (50 and 100%). It should be noted that the violet coloration occurs more in the central part of the specimen surrounding the reinforcing steel, which coincides with the results presented by Redaelli & Bertolini [23]. It is also observed that in the case of partial carbonation, the re-alkalization front is superior compared to that of the complete carbonation level.

The corrosion current (Icorr) was determined by the linear polarization technique, Rp, in the carbonated specimens after reaching the proposed carbonation levels (partial and complete) and in the specimens after applying ER using a depolarization time (TP) of 15 days. The corresponding results are shown in Fig. 7. Abdelaziz, Abdelalim and Fawzy [40] suggested that to obtain reliable electrochemical measurements after applying the rehabilitation electrochemical methods, it is important to give a TP because immediately after the system is disconnected, the reinforcing steel is polarized due to the electrical field generated by the treatment. In general, it is observed that the corrosion rate decreases after applying ER; however, the efficiency is superior at a greater w/b ratio. Thus, for partial carbonation, the reduction in the corrosion rate of the MK0.65 mixture is 82.5%, followed by OPC0.65 with 42.6%, and finally by SF with 16.6%. This effect is attributed to the decrease in the ohmic resistance with an increasing w/b ratio [17]. Notably, the specimens blended with MK decreased until reaching a low corrosion level after ER (Fig. 8). The specimens with the addition of SF did not present satisfactory results, which may indicate the need to increase the treatment time [17].

For complete carbonation, a greater percentage decrease of *Icorr* was observed for OPC0.65 at 59%, followed by MK0.65 at 47%, and finally SF0.65 at 27.6%. However, none of the specimens reached a low corrosion level. This result coincides with that mentioned by Miranda et al. [24], who suggested that at elevated carbonation levels, the application of ER may not be efficient; therefore, the authors recommended using this technique as a prevention method, i.e., before corrosion reaches the reinforcing steel.

4. Conclusions

According to the results obtained in the present study, the following can be concluded:

- Amongst the used additions, MK significantly influenced concrete performance when used as a cement replacement at an addition of 10% by weight. MK positively contributes to the resistance increment in orders of up to 53.5% at a w/b ratio of 0.45. Likewise, positive effects were observed when the absorption greatly decreases and the resistivity of the mixture increases.
- The mixtures blended with MK and exposed to equal conditions of CO2, RH%, and temperature require more time to carbonate compared with the mixtures with SF and OPC. Increases in the w/b ratio accelerate the process.
- Based on the electrochemical results obtained by the linear polarization resistance technique, it became evident that the application of ER generates a positive variation in the measurements of icorr. Thus, the test samples are partially carbonated with values of icorr in the range of 0.72-1.59 µA/cm2; after ER is applied, the values range from 0.2-1.02

 μ A/cm2. In the specimens with complete carbonation, the icorr decreased from 1.03-2.63 μ A/cm2 to 0.7–1.66 μ A/cm2; however, even though these values are reduced, most of the values are within the moderate corrosion region.

- The electrochemical re-alkalization (ER) processes were more effective for the partially carbonated specimens, and among these, the specimens blended with MK and with greater contents of the w/b ratio reached low corrosion levels by the end of the process.
- ER is a prevention technique more than a rehabilitation technique, as evidenced in its greater efficiency for test samples with a partial carbonation level. The application of ER increases the useful life of steel in the presence of carbonated mortars.



Figure 7. Evolution of the partial/complete carbonation front before and after ER.

Source: The authors



Figure 8. Corrosion current of the mortars subjected to complete carbonation (Carb/Complete), partial carbonation (Carb/Partial) after the ER treatment plus 15 days of TP for the partial and complete levels (ER + 15 days TP/Complete, ER + 15 days TP/Partial). Source: The authors

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