Durability of FCC-blended mortar exposed to sodium and magnesium sulfate

Durabilidad de morteros adicionados con FCC expuestos a sulfato de magnesio y sulfato de sodio

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Abstract

This paper presents a comparative analysis of the deterioration of mortars containing 10% and 20% of a fluid catalytic cracking residue (FCC) in the presence of sodium sulfate (Na_2SO_4) and magnesium sulfate (MgSO_4) at 5% (concentration 50g/L). Cement without addition (reference cement), and blended with 10% metakaolin (Cem-10MK) and 10% silica fume (Cem-10SF) were used as reference materials. The longitudinal expansion and loss of compressive strength at 392 days of exposure were evaluated. Additionally, microstructural analysis was performed using X-ray diffraction (XRD) and scanning electron microscopy (SEM) to identify the generated products. The results indicate that the residual strength of mortars blended with 10% and 20% of FCC after 360 days of immersion in the Na_2SO_4 solution is 18.5% higher than the value reported by the standard mixture, and 55% and 39% higher with respect to that reported for the mixtures containing 10% MK and SF. Mortars containing 10% of FCC in the presence of Na_2SO_4 report a loss of resistance of approximately 10%, while in MgSO_4 it was 38% lower than the values of other evaluated mixtures. In general, MgSO_4 is a more aggressive solution than Na_2SO_4 . Ettringite and gypsum were identified as products from the reaction with sulfates.

Keywords: Fluid catalytic cracking residue, chemical attack, sodium sulfate, magnesium sulfate

Resumen

En este trabajo se presenta un análisis del deterioro de morteros adicionados con 10 y 20% de un residuo de catalizador de craqueo catalítico (FCC) en presencia de sulfato de sodio (Na_2SO_4) y sulfato de magnesio (MgSO_4) al 5% (concentración de 50g/L). Como materiales de referencia se utilizaron cemento portland sin adición (Cem.Ref), adicionado con 10% metacaolín (Cem-10MK) y con 10% de humo de sílice (Cem-10HS). Para evaluar el desempeño frente a sulfatos se utilizó la norma ASTM C1012 (2012), determinándose la expansión longitudinal y la pérdida de resistencia a la compresión hasta edad de 392 días de exposición. Adicionalmente se efectuó el análisis microestructural por medio de difracción de rayos X (DRX) y microscopía electrónica de barrido (SEM), para identificar los productos generados. Los resultados indican que la resistencia residual de las mezclas adicionadas con 10 y 20% de FCC luego de 360 días de inmersión en la solución de Na_2SO_4 son un 18,5% superior al valor reportado por la mezcla patrón y un 55% y 39% superior con respecto al de las mezclas conteniendo 10% de MK y HS respectivamente. Los morteros FCC10% en presencia de Na_2SO_4 reportan pérdida de resistencia del orden del 10% e inmersos en MgSO_4 del 38%, valores inferiores al resto de mezclas evaluadas. Cabe anotar, que el MgSO_4 es una solución más agresiva que Na_2SO_4 para todas las mezclas evaluadas. Como productos de la reacción con los sulfatos se identificaron etringita y yeso.

Palabras clave: Residuo de catalizador de craqueo catalítico, ataque químico, sulfato de sodio, sulfato de magnesio

1. Introduction

One of the main performance characteristics of concrete, besides its mechanical properties, is its durability, which can be associated to the service life of the structure once it is exposed to specific conditions or environments (ACI 201.2R, 2008; Aguirre et al., 2013). In general terms, the durability of concrete depends on the materials used, mixture design, execution on site and curing, as well as on the environment's aggressiveness based on parameters such as relative humidity, temperature and contaminants (NTC 5551; ACI 201.2R, 2008).

The durability factors can be: physical, chemical, biological and structural (Aguirre et al., 2013). Among the chemical factors, carbonation, chloride attacks and sulfate attacks are worth mentioning (ACI 201.2R, 2008). However, according to their mechanism, sulfate attacks can also be

classified as physical. The chemical action is based on a series of chemical reactions between sulfate ions and hydration products or cement compounds and the physical action is a consequence of the continuous crystallization of sulfate salts under certain environmental changes, which generate pressure on the pore walls and lead to the concrete's expansion (Skalny et al., 2003). Sulfates react with the hydrated paste and produce expansive ettringite ($3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 3\text{CaSO}_4 \cdot 32\text{H}_2\text{O}$) and gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$); nevertheless, they can also produce thaumasite ($\text{CaSiO}_3 \cdot \text{CaCO}_3 \cdot \text{CaSO}_4 \cdot 15\text{H}_2\text{O}$) (Crammond, 2003; Irassar et al., 2005). The latter requires calcium silicate sources, sulfate and carbonate ions, excess humidity, and preferably low temperature. It should be noted that, currently, most cements at global level contain limestone as a filler in variable proportions, which, from the sulfates' aggressiveness perspective, is a critical factor to be considered (Tsilivilis et al., 2002). The ettringite formation increases the volume of solids, which leads to expansion and checking, while the formation of gypsum can cause concrete softening and loss

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of resistance (ACI 201.2R, 2008; Atahan and Dikme, 2011). The magnesium sulfate attack can also generate brucite (magnesium hydroxide, $Mg(OH)_2$).

This deterioration of the Portland cement compounds is attributed to the presence of portlandite (CH), which is the hydration product most vulnerable to sulfate attacks (Mehta and Monteiro, 2006). The use of additional materials such as pozzolan and slag contribute to the sulfate resistance of mortars and concretes, especially due to their reaction to portlandite and its consequential reduction in the system. Furthermore, pozzolans generate pore size reduction, which in turn decreases the permeability and reduces the entry of aggressive ions (Lothenbach et al., 2011; Sideris et al., 2006; Al-Akhras, 2006; Karakurt and Topçu, 2011). Some researchers state that, although the chemical effect can be reduced, from the physical point of view the materials with fine pore sizes can be affected during the crystallization of salts (Aye and Oguchi, 2011).

A pozzolanic-type of material is the catalyzer used from the Fluid Catalytic Cracking (FCC), which is a residue from the petrochemical industry derived from crude oil refinement during the production of gasoline and other fuels (Tseng et al., 2005; Hsu et al., 2001; Payá et al., 2009; Dweck et al., 2008; Izquierdo et al., 2013). Bukowska et al. (2004) studied the behavior of mortars with addition of FCC in the presence of Na_2SO_4 , and report a lower resistance to sulfates for cements with addition. These authors attribute the samples' high checking level to the accumulation of expansive corrosive products at a depth close to the surface, especially on the corners and edges of the elements. The use of X-ray diffraction (XRD) in the mortars immersed in sulfates report the presence of thernardite, formed by sodium sulfate decahydrate used to prepare the sulfate solution. Although in the TG test at 170°C they report a significant increase in the weight loss of mortars containing 20% FCC, attributed to the action of sulfate ions and the formation of gypsum or ettringite, these components could not be confirmed through XRD, possibly due to insufficient crystalline structures or small amount of minerals present. In previous studies, the same researchers report a

compressive strength decrease, compared with mortars without addition (Pacewska et al., 2000). They compare these results with other studies performed on cements with additions (Torres et al., 2013; Al-Amoudi, 2002; Atahan and Dikme, 2011).

This paper deals with the behavior of Portland cement mortars blended with 10% and 20% FCC exposed to magnesium sulfate and sodium sulfate. Moreover, and for comparison purposes, mortars with addition of MK and SF at 10% are studied.

2. Materials and experimental procedure

2.1 Materials

In the study, a catalyst residue from the FCC process was used, supplied by a Colombian petroleum company; the physical and chemical characteristics are listed in Table 1. This Table shows that the FCC is mainly composed of alumina and silica, in the order of 90% and with an average particle size of 16.15 μm . Other materials used in the study, besides ordinary Portland cement (OPC), is a silica-type of addition, silica fume (SF) and a alumina-type of addition, metakaolin (MK), whose characteristics are included in the same table and are used for comparison purposes against the FCC addition. Figure 1a shows an image from a scanning electron microscope (SEM), which illustrates that particles have, in general, a highly porous spherical shape. The XRD diffractogram of Figure 1b indicates that the material is partially amorphous, the crystalline phases correspond to a hydrated sodium aluminosilicate of zeolitic nature similar to the faujasite (F) with formula $Na_2[Al_2Si_{10}O_{24}].nH_2O$, (peaks $2\theta = 6.19^\circ, 15.6^\circ, 23.58^\circ$) (Su et al., 2000; Tseng et al., 2005), and in a lesser proportion, the presence of kaolinite (K) and quartz (Q) can be evidenced. For comparison purposes, we include the MK and SF diffractograms that allow appreciating that these materials have mainly an amorphous nature.

Table 1. Chemical and physical characteristics of the materials used

Characteristics	FCC	MK	SF	OPC
Chemical Composition, %				
Silica oxide, SiO_2	44.13	53.00	>90.00	19.43
Aluminum oxide, Al_2O_3	46.06	43.80	-	4.00
Iron oxide, Fe_2O_3	0.81	0.43	-	3.61
Calcium oxide, CaO	0.52	0.02	-	64.46
Magnesium oxide, MgO	-	0.03	-	1.52
Potassium oxide, K_2O	0.11	0.19	-	0.39
Titanium oxide, TiO_2	0.67	1.70	-	0.34
Loss on ignition, LOI	6.50	0.46	6	2.58
Physical Properties				
Density (Kg/m^3)	2.63	2.60	2.01	3.15
Average particle size (μm)	16.15	1.20	0.20-0.50	23.00

Figure 1. (a) SEM micrograph and (b) XRD of the FCC spent catalyst

2.2 Tests

Pastes and mortars were prepared with ordinary Portland cement (OPC), which was replaced by a FCC spent catalyst in a proportion of 10% and 20%. Moreover, and as a reference material, pastes and mortars were prepared with addition of metakaolin (MK) and silica fume (SF) in a 10% proportion. A water/binder ratio (w/B) of 0.35 was used for the pastes, while for the mortars the w/B ratio was 0.5 and the cement-aggregate ratio of 1:2.75; the aggregate used in this study was sand from Ottawa. After curing the mortars for 28 days in saturated limewater, their performance in the presence of sulfates was evaluated, based on the ASTM C1012 standard (2012); the samples were exposed to sodium sulfate (Na_2SO_4) and magnesium sulfate (MgSO_4) solutions at 5% (50 g/L concentration) and the pH of the solutions was controlled between 6 and 8 and the temperature remained constant (25°C) throughout the exposure time.

The dimensional changes were registered, evaluating the longitudinal expansion of prismatic tubes measuring 25x25x285mm and the loss of compressive strength (ASTM C109) in cylindrical samples of 30mm

diameter and 60mm height, until the age of 392 days of exposure. A visual inspection of the samples was also made, and the microstructural analysis was performed through X-ray diffraction (XRD) and scanning electron microscope (SEM). The XRD test was made using an X'Pert MRD Diffractometer (PanAnalytical), radiation $\text{CuK}\alpha 1$ was used, the copper tube worked at 45 Kv and 40 mA, and data was recorded in a range from 8° to 60° (2θ) for 30 min. The SEM analysis was carried out on a JEOL electron microscope, Ref: JSM-6490LV, high vacuum (3×10^{-6} torr).

3. Results and discussion

3.1 Expansion tests

Based on ASTM C1012 (2012), Figure 2 shows the results obtained from the expansion test applied to cement mortars without addition (reference cement), and blended ones with 10% and 20% FCC, 10% MK and 10% SF in replacement of cement, exposed to sodium sulfate and magnesium sulfate solutions.

Figure 2. Expansion chart vs time of exposed mortars to a. Na_2SO_4 and b. MgSO_4

Mortars with and without addition immersed in the Na_2SO_4 solution present a similar expansion percentage (lower than 0.01%) until the exposure age of 196 days, which is lower compared to that reported by mortars immersed in MgSO_4 . As of day 196, in mortars immersed in Na_2SO_4 , the expansion degree starts to increase until the final test age (392 days). At 196 days, mortars with 20% and 10% FCC additions report expansions of 0.001% and 0.002% respectively; these results are very close to those obtained for the other mixtures assessed, including the standard one (reference cement). At the final test age (56 weeks), the samples with reference cement show a strong increase in their expansion percentage (0.074%), in the same way as the samples with 10% MK (0.031%); on the other hand, mortars blended with 20% FCC, 10% SF and 10% FCC report values of 0.0%, 0.004% and 0.008% respectively, which in general terms are 90% lower than those reported by the reference cement.

Samples immersed in the MgSO_4 solution present low expansion percentages up to 49 days of exposure, including cement with 10% MK, which reports 0.0040%. From that age on, there is an upward trend, indicating that the deterioration process in the presence of MgSO_4 is faster than in the presence of Na_2SO_4 , which is consistent with the solution's aggressiveness level according to the type of cation therein (Al-Amoudi, 2002). However, at the end of the test (392 days) the expansion percentage does not exceed 0.02% in any of the evaluated mixtures. It should be highlighted that mixtures containing 20% FCC show the best performance in this test, followed by those containing 10% SF and 10% FCC.

The best performance of mortars with 20% FCC addition in the MgSO_4 and Na_2SO_4 solutions at 392 days of exposure can be attributed to the higher consumption of portlandite and a greater refinement of the pore structure, as a consequence of pozzolanic reactions (Bukowska et al., 2003; Bukowska et al., 2004; Torres et al., 2013).

3.2 Visual inspection

During the samples' exposure to Na_2SO_4 and MgSO_4 , a visual inspection was made as well as a photographic record of the progress regarding the deterioration process of mortar bars made of cement without addition (reference cement) and with addition of 10% FCC, 20% FCC, 10% MK and 10% SF, after 203 and 392 days of immersion (Figure 3).

The beginning of the physical deterioration of the samples exposed to the MgSO_4 solution occurs at 203 days of exposure (Figure 3b). It is possible to see spalling at the bar edges and part of the surface, particularly in the mixtures with added 10% MK and 10% FCC. In the samples immersed in the Na_2SO_4 solution (Figure 3a), no deterioration is observed at this exposure age; this behavior is consistent with the results of the expansion tests (Figure 2). After 392 days of exposure to these solutions (Figure 3c and 3d), the samples exposed to MgSO_4 still show greater deterioration (highly irregular edges) compared with those exposed to Na_2SO_4 (Al-Amoudi, 2002). In this last solution, we observe a slight spalling at the bar edges and the appearance of cracks, a deterioration known as onion peeling (Torres J. et al., 2008). This deterioration is more evident in the mortars with reference cement and 10% MK, which presented higher expansion percentages (Figure 2).

Figure 3. Mortars exposed to a. 203 days in Na_2SO_4 , b. 203 days in MgSO_4 , c. 392 days in Na_2SO_4 , and d. 392 days in MgSO_4

3.3 Compressive Strength

Figure 4 shows the compressive strength results of cement mortars without addition (reference cement) and with different additions cured under water for a period of 28 days and subsequently exposed to solutions of magnesium and sodium sulfates (Na_2SO_4 and MgSO_4) at 60, 180 and 360 days.

In general, the compressive strength of the reference-cement mortars immersed in both solutions decreased when the time of exposure was increased, and a greater loss of resistance can be observed in the samples exposed to MgSO_4 . At 360 days of immersion in Na_2SO_4 and MgSO_4 , the loss of resistance was 20.14% and 40.63% respectively. This loss of resistance at 360 days was equally observed in the mixtures with MK and SF, and it was even higher than in the reference cement; thus, in Na_2SO_4 it was 41.48% and 20.80% and in MgSO_4 it was 63.72% and 48.07% respectively. On the other hand, the mixtures blended with FCC at 10% and 20% present a better performance. The 10% FCC samples in the presence of Na_2SO_4 report loss of resistance of approximately 10% and 38% when immersed in MgSO_4 ; these values are lower than for the rest of the mixtures, including the reference one.

It is important to note that the resistances reported by the mixtures with additions of 10% and 20% FCC after

360 days of immersion in the Na_2SO_4 solution are 18.5% higher than the value reported by the standard mixture and 55% and 39% in relation to that reported by the mixtures containing 10% MK and SF respectively. Unlike the reference-cement mortars, in mortars blended with FCC the negative effect of the presence of sulfates is first noted after 180 days of exposure, which is consistent with other studies (Torres et al., 2013).

On the contrary, in the MgSO_4 solution, from the beginning of the exposure to this aggressive environment a decrease in the compressive strength values is observed in all samples exposed, and this effect is higher at 60 days of immersion for samples blended with 10% SF, followed by the reference cement. The loss of resistance is more significant in the case of the reference cement, with a strength loss value at 180 days almost twice as the value reported for mortars blended with 10% FCC.

As can be observed, the sulfates' aggressiveness depends on the type of sulfate; in this respect, it is suggested that calcium sulfate is less aggressive than sodium sulfate and that the latter is less aggressive than magnesium sulfate, which is consistent with what has been reported herein (ACI 201.2R, 2008; Al-Amoudi, 2002; Aye and Oguchi, 2011).

Figure 4. Compressive strength of mortars exposed during 28, 60, 180 and 360 days to sodium and magnesium sulfates

3.4. Mineralogical Characterization

In order to evaluate the nature of the products generated by the reaction of cements with and without addition immersed in Na_2SO_4 and MgSO_4 solutions, pasted were prepared and exposed to this environment for 180 days. Afterwards, the XRD method was used to obtain the diffractograms illustrated in Figure 5. Here it is possible to appreciate the reduction of the peak corresponding to portlandite (CH), particularly in the mixtures blended with 20% FCC. This effect is due to the portlandite consumption in

the pozzolanic reaction. As for new compounds generated in the reaction with sulfates, there is ettringite (E) and gypsum (Y) in all samples exposed, where the highest peaks are evidenced in the samples immersed in the MgSO_4 solution. The diffractogram of the sample containing 20% FCC shows a higher peak of ettringite followed by those blended with 10% MK and 10% FCC. Calcium-aluminate type of compounds in the cementitious material, such as C_4AH_{13} , C_2ASH_8 and C_3AH_6 , generate a greater proportion of ettringite in the presence of sulfate ions due to their high alumina content.

Additionally, Figure 6 shows the SEM micrographs for reference-cement pastes blended with 10% FCC and exposed for 360 days to Na_2SO_4 and MgSO_4 solutions. These micrographs confirm the presence of the reaction products found in the X-ray diffraction analysis.

In the reference paste (Figure 6a and 6c), small needles indicate the formation of ettringite, while pastes blended with

FCC indicate (Figure 6b and 6d), besides a larger amount of this phase, the presence of monosulfate crystals is appreciated. The SEM micrographs confirm a bigger deterioration of the samples immersed in the MgSO_4 solution (Figure 6c and 6d). The increased checking is consistent with the lower compressive strength results for the samples immersed in MgSO_4 (Figure 4).

Figure 5. X-Ray diffractograms of the pastes after 180 days exposed to a. Na_2SO_4 and b. MgSO_4 . E: Ettringite; M: Monosulfate; Y: Gypsum; CH: Portlandite

Figure 6. SEM micrographs of pastes exposed for 360 days. a) Reference cement in Na_2SO_4 , b) Reference cement with 10% FCC in Na_2SO_4 , c) Reference cement in MgSO_4 and d) Reference cement with 10% FCC in MgSO_4 . (E: Ettringite; M: Monosulfate)

4. Results

The results obtained from this study allow concluding the following:

- The evaluated additions present $< 0.10\%$ in one year, when assessed individually in the mixture with Portland cement using the ASTM C1012 standard (2012), a condition required to qualify pozzolans as resistant to sulfates (ACI 201).
- In general, the loss of resistance is higher in all mixtures evaluated with and without addition in the presence of $MgSO_4$, compared to those immersed in Na_2SO_4 .
- Cement mortars without addition (reference cement) report a loss of resistance at 180 days almost twice at the one reported by mortars blended with 10% FCC.
- At 360 days of immersion in Na_2SO_4 , the residual strength of the mixtures blended with 10% and 20% FCC are 18.5% higher than the value reported by the standard mixture and a 55% and 39% higher in relation to what was reported for the mixtures containing 10% MK and 10% SF.

- The increased chemical resistance of mortars blended with 10% FCC is highlighted, because in the presence of Na_2SO_4 they report a loss of resistance of approximately 10% at 360 days of exposure, and approximately 38% when immersed in $MgSO_4$; these values are lower than those reported by the other evaluated mixtures.
- The main products identified in the cement pastes exposed to sulfates were ettringite and gypsum.

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