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Resistance to acid corrosion of blended cements mortars with spent fluid catalytic cracking (sFCC) catalyst

Resistencia a la corrosión ácida de morteros de cementos adicionados con catalizador de craqueo catalítico gastado (sFCC)

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Abstract

Spent fluid catalytic cracking (sFCC) catalyst is a residue that is produced in high volumes by the petrochemical industry and has shown a high potential to be used as a supplementary cementitious material (SCM). This article assesses the sFCC effectiveness as a SCM in mortar based on ordinary Portland cement (OPC), as well as its chemical resistance when the materials are immersed in sulfuric (H_2SO_4), acetic (CH_3COOH) and hydrochloric acid (HCl). The performance of the sFCC was compared to other commercially available mineral additions, such as silica fume (SF) and metakaolin (MK). The results show a mechanical strength increase of up to 17% when the sFCC was included, compared to a control mortar without mineral additions. Immersion in H_2SO_4 during 90 days led to the formation of gypsum and a loss of chemical resistance up to ~25% in mortars with MK and sFCC. In the samples immersed in HCl, and particularly those made with sFCC and MK, Friedel's salt was identified due to the ingress of Cl and the subsequent reaction with the monosulfoaluminates formed during cement hydration. Immersion in acetic acid led to a loss of strength of up to 57% and 37% in mortars with OPC and sFCC, respectively. Immersion in HCl leads to a stronger attack for blended systems, followed by CH_3COOH and H_2SO_4. The mortar with 20% of sFCC exhibits the best performance, especially in the presence of sulfuric acid.

Keywords: Spent fluid catalytic cracking catalyst, blended mortars, acid attack, chemical resistance

Resumen

El catalizador de craqueo catalítico gastado (sFCC) es un residuo producido por la industria petroquímica y ha demostrado una elevada potencialidad como adición puzolánica en mezclas de cemento Portland (OPC). El presente artículo evalúa la eficacia del sFCC como adición mineral activa en morteros de cemento Portland, así como la resistencia al ataque químico cuando los materiales producidos son inmersos en ácido sulfúrico (H_2SO_4), acético (CH_3COOH) o clorhídrico (HCl). Se llevó a cabo una comparación del desempeño del sFCC como adición frente a adiciones comercialmente disponibles, tales como humo de sílice (SF) y metacaolín (MK). Los resultados revelan un incremento en las resistencias mecánicas de hasta un 17% en morteros con sFCC en comparación a un mortero referencia sin adición (OPC). La inmersión en H_2SO_4 durante 90 días condujo a la formación de yeso y pérdida de resistencias mecánicas de hasta un ~25% en las mezclas con MK y sFCC. Para las muestras inmersas en HCl y especialmente las constituidas con sFCC y MK, se identificó Sal de Friedel, como consecuencia del ingreso de Cl- y posterior reacción con los monosulfoaluminatos formados durante la hidratación del cemento. La inmersión en ácido acético condujo a pérdidas de resistencia de hasta un 57% para OPC y 37% para morteros adicionados con sFCC. De esta manera, la inmersión en HCl promueve el mayor ataque para los sistemas adicionados y posterior deterioro, seguido del CH_3COOH y H_2SO_4. Se destaca el mejor desempeño del mortero con 20% sFCC, en particular en presencia de ácido sulfúrico.

Palabras clave: Catalizador de craqueo catalítico gastado, morteros adicionados, ataque ácido, resistencia química

1. Introduction

Cement is one of the inputs of highest demand in the construction industry with a production of ~3.7 Gt/year (Van Oss, 2013). However, even though the cement industry has a major economic impact, its production accounts for 5-8% of greenhouse gas emissions globally, because obtaining a ton of cement releases approximately 0.89 t of CO_2 (Damtoft et al., 2008). The use of mineral additives as a partial replacement of OPC has been one of the most effective solutions for reducing the ecological footprint caused by cement (Imbabi et al., 2012). To that effect, various natural minerals, by-products, and industrial waste products have been evaluated, all of which have shown satisfactory results in technical, economic, and environmental terms (Mehta and Monteiro, 2006; Siddique and Klaus, 2009; Salas et al, 2009).

Standardization of some industrial by-products such as granulated blast furnace slag (GBFS), silica fume (SF) and fly ash (FA), has helped to define the physicochemical properties required for use in the production of concrete (Lothenbach et al, 2011). However, it is estimated that its availability will not be able to meet the demand of ~6 Gt/year of cement by 2050 (CEMBUREAU, 2012). Therefore the use of other types of industrial waste, including spent fluid catalytic cracking catalyst (sFCC), continues to be evaluated.

sFCC is a residue that comes from the petrochemical industry for the refining of crude oil whose output is ~400 kt/year (Zornoza et al., 2008) and has been listed as an inert solid residue which may be used (Martinez et al., 2013). sFCC is a material with crystallinity with amorphous phases due to the partial destruction of the structure during catalysis (Chen et al., 2004) with a chemical composition based in Al_2O_3 (37-46%) and SiO_2 (47-58%) (Paya et al., 2003; Izquierdo et al., 2013a). Several authors have reported the effectiveness of sFCC in properties of durability of blended concrete (Izquierdo et al., 2013b; Zornosa et al., 2008; Torres et al., 2013; Pacewska et al., 2000). However, there are no reports regarding the effect on resistance to acidic media, which for some applications is widely recommended.

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This paper presents the study of the behavior of blended mortars containing 10% and 20% of sFCC exposed to sulfuric acid (H_2SO_4), acetic acid (CH_3COOH), and hydrochloric acid (HCl) at a concentration of 0.1 M. H_2SO_4 was considered as this can be formed by the oxidation of sulfides, particularly pyrite present in the soil, or due to aerobic bacteria attacks in wastewater or the presence of acid rain in industrial atmospheres (Li et al., 2009; Allahverdi and Skvara, 2000a). Acetic acid can occur in association with agricultural products or food decomposition processes (Allahverdi and Skvara, 2000; De Belie et al., 1996).

2. Experimental methodology

2.1 Materials

A waste product of spent fluid catalytic cracking (sFCC) catalyst was used, which was supplied by an oil industry in Colombia. The sFCC efficiency was compared with silica fume (SF) supplied by Sika Colombia and a Metamax® metakalin from Basf (MK). The samples were produced with a Portland cement (OPC) (ASTM C150). The physicochemical characteristics of the utilized materials are presented in Table 1.

2.2 Preparation of specimens and tests conducted
2.2.1 Pastes and mortars

Blended and non-blended cement (OPC) pastes and mortars were prepared. The blended sFCC was incorporated at 10% and 20% of replacement of cement weight (10FCC, 20FCC), with MK and SF in proportion of 10% (10MK, 10SF). In the pastes, a ratio of water/binder (a/C) of 0.35 was used, and in the mortars, 0.50. The pastes were cast into

30×60 mm cylindrical molds, and for the mortars, cubic specimens of 50×50 mm were used. Curing was performed under water at room temperature. For the production of mortars, a fine siliceous aggregate (Ottawa sand) was used.

2.2.2. Inmersion in acidic media and tests

Solutions of hydrochloric acid (HCl), acetic acid (CH_3COOH) and sulfuric acid (H_2SO_4) were prepared at a concentration of 0.1 M; the pH was 1.0, 2.9, and <1.0 respectively. The specimens were immersed for a period of 180 days in each of the solutions; throughout the study, a pH control was carried out to avoid neutralizing the environment. At different ages of the test, weight loss in specimens immersed in each solution was registered, for which the previously washed specimen was subjected to a drying process at 70°C for 24 hours. The change in compressive strength after 7, 28, and 90 days of immersion was determined. In addition, structural changes in each of the pastes immersed for up to 180 days, previously crushed, washed and filtered with acetone, were evaluated using:

- X-ray diffraction (XRD): a PanAnalytical X'Pert diffractometer was employed using $Cu\alpha_1$ radiation with a step size of 0.020° in a 2θ range between 8 and 60°.
- Thermogravimetric Analysis (TGA/DTG): a TA Instruments SDTQ600 thermobalance was used up to 1000°C with a heating rate of 10°C/min in an atmosphere of N.
- Scanning Electron Microscopy (SEM): An FEI Phenom 2005 scanning electron microscope was used in low vacuum mode with an accelerating voltage of 5 kV.

Table 1. Characteristics of utilized materials

Chemical composition and physical characteristics	sFCC	MK	HS	OPC
SiO_2	44.13	53.00	>90.00	19.43
Al_2O_3	46.06	43.80	-	4.00
Fe_2O_3	0.81	0.43	-	3.61
CaO	0.52	0.02	-	64.46
MgO	-	0.03	-	1.52
K_2O	0.11	0.19	-	0.39
TiO_2	0.67	1.70	-	0.34
Loss on ignition (950°C)	6.50	0.46	6.00	2.58
Density (g/cm³)	2.63	2.48	2.01	3.15
Average particle size (µm)	16.15	1.20	0.20-0.50	23.0

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3. Results and discussion

3.1 Physical-mechanical characterization

Table 2 shows the compressive strength values of mortars produced after 28 and 60 days of curing, in which it can be seen that replacing 10% of cement with sFCC increased strength at 28 days of curing up to 17% compared to the non-blended control mortar (OPC). Meanwhile, MK utilization led to an increase of 11%. These results are consistent with those reported in literature, where the spent fluid catalytic cracking (sFCC) catalyst exhibits comparable pozzolanic properties and in some cases, properties superior to those of MK (Soriano et al., 2013; Allahverdi et al., 2011; Torres and Torres 2010; Payá et al., 2013). The increase in compressive strength with the use of sFCC is attributed to its high reactivity with the $Ca(OH)_2$ released during cement hydration and subsequent formation of products like calcium silicate hydrate (C-S-H), calcium aluminosilicates hydrate (CASH) and calcium aluminate hydrate (CAH) (Izquierdo et al., 2013a). However, the degree of substitution of cement by sFCC has no direct relationship with the mechanical performance, as mortars made with 20% of sFCC (20FCC) exhibited a smaller increase in the compressive strength (~7%) compared to the corresponding 10FCC. The high specific surface area of the sFCC, because of the presence of zeolite structures, may contribute to the increased demand for water during mixing and therefore have a negative effect on the mechanical performance of the material.

Mortars made with silica fume (SF) showed a resistance up to ~8% less at 28 days of curing compared to the control mortar (OPC). The SF is listed as a pozzolanic addition of high reactivity because it is made with spherical particles of sub-micrometric size (0.1-0.5 μm) that occupy the empty spaces between the grains of cement (filler effect) and react quickly with portlandite to form C-S-H type products. These two effects contribute to obtaining high mechanical strength, especially at an early age of curing. However, commercially available SF is subjected to a densification process during collection in order to facilitate handling and transport. The material obtained is composed of agglomerates of SF particles (in some cases greater than 100 μm) that are not readily destroyed during kneading and hence SF microparticles are not homogeneously dispersed. The presence of such agglomerates embedded in hydrated cement paste leads to a decrease in the effectiveness of SF and hence low strength increases (Diamond, 2004; Rodriguez et al., 2012).

The rates of pozzolanic activity of blended mortars with sFCC and MK, included in Table 2 and evaluated based on ASTM C311, largely outweigh the ASTM C618 specification (75%), confirming the pozzolanic nature of these additions. It should be noted that ASTM C1240 requires an activity index of approximately 105% at age 7 days of curing for SF, which the used blend does not meet, as can be seen in Table 2. Previously reported studies of thermogravimetry confirm the high reactivity of sFCC (Izquierdo et al., 2013a).

3.2 Inmersion of mortars in acidic media

Exposure of mortar specimens to different acidic media generally leads to a deterioration that is particularly visible on the exposed surface. It is established that products of reaction are, in addition to silica hydrogels, aluminum and iron oxides, and acid calcium salts, which in the case of being soluble are removed from the solution, generating a decalcification process reflected in losses in weight and physical-mechanical properties (Pavlik and Uncik, 1997; Allahverdi and Skvara, 2000b). They are identified as factors that influence the speed of attack: those related to solution (acid resistance, solubility of the calcium salt, concentration, pH, diffusivity of the acid and its salt), those related to cementing (type and amounts of cement, aggregate nature, permeability, curing time, and conditions), and those that are external such as temperature (Allahverdi and Skvara, 2000b).

Acidic media used in this study, HCl and H_2SO_4, are considered strong acids, while CH_3COOH is a weak organic acid (pKa = 4.756). It should be noted that the latter is generally used in acid leaching tests because it represents the decomposition of organic matter in landfills (Shi and Stegemann, 2000).

Mortars immersed in the HCl solution exhibited weight losses of around 6% after 90 days (Table 3). Meanwhile, immersion in CH_3COOH led to weight loss of less than 1.5% for each of the materials. Systems immersed in H_2SO_4, however, exhibited weight gain in some cases, which is attributed to the reduced solubility of the salt formed compared to calcium chloride and calcium acetate that are highly soluble (Allahverdi and Skvara, 2000b). It should be noted that the 10MK and 10SF exhibited the greatest loss of mass in HCl and CH_3COOH. The decrease in the mass of the mortars is attributed to the solubilization of some hydration products and the subsequent increase in porosity. This includes the decalcification process and the solubility of the alumina and iron oxides (Allahverdi and Skvara, 2000a), which occurs preferably at a pH below 3.0 as in the case of this study

Table 2. Compressive strength of produced mortars

Mortar	Pozzolanic activity index (ASTM C311)	Compressive strength (MPa)	
		28 days	60 days
OPC	--	45.09 ±0.81	50.61 ±0.73
10FCC	1.17	52.61 ±0.58	52.86 ±0.37
20FCC	1.07	48.46 ±0.53	52.82 ±2.71
10MK	1.11	50.21 ±2.09	52.66 ±0.35
10SF	0.92	41.40 ±1.12	41.40 ±1.13

Table 3. Weight loss of mortars at 90 days of immersion

	Weight loss (%)		
	HCl	H_2SO_4	CH_3-COOH
OPC	5.77	-0.24	0.64
10FCC	5.85	0.16	0.82
20FCC	6.02	1.22	0.51
10MK	6.77	-0.80	1.23
10SF	6.50	-0.21	1.15

In general, immersing the different mortars in acidic media leads to a decrease in compressive strength. Figure 1 shows the compressive strength indices calculated from the ratio of the values obtained in mortars immersed in acid for time t (R_{Ct}) and the initial strength of mortars (R_{Ci}). The greatest loss of strength was recorded for the OPC mortars immersed in CH_3-COOH (Figure 1A) with values of up to ~7% at 90 days of immersion, while 10MK mortar showed the best resistance to attack with strength losses of ~31%, followed by 20FCC. The 10FCC mortars exhibited greater strength loss compared to those with a higher degree of substitution (20FCC). For each of the materials, significant decreases in strength were identified (>14%) from 7 days of immersion. This acid is considered highly corrosive, even more so than nitric acid, and has a pH of about 3.0 where dissolution of aluminous species are increased (Shi and Stegemann, 2000).

Mortars immersed in HCl (Figure 1B) have losses of strength of up to ~12% with 7 days of immersion. After 90 days in HCl, there were no significant differences observed in the mortars studied, except 10FCC, which showed the greatest loss of strength (~54%). Mortars immersed in H_2SO_4

(Figure 1C) reported a loss of compressive strength of up to 25%, making it the acidic medium with the weakest attack. The highest rate of strength obtained for this medium is closely related to the lowest weight loss and the formation of less soluble calcium salts. Other studies have reported strength loss of up to 50% after OPC mortars were exposed to the same concentration of sulfuric acid for 5 years (Li et al., 2009) and 34% in the presence of HCl (De Ceukelaire, 1992).

Overall, the effect of the type of acid on the rate of deterioration of the mortar has the following order of aggressiveness from highest to lowest: $HCl>CH_3COOH>H_2SO_4$. This confirms that the aggressive effect is higher in those acids with soluble calcium salts, and among these, the strongest acid (Marck et al., 2013). This result corresponds with those obtained by Pavlík (1994) in Portland cement pastes. Regarding the type of addition, it should be noted that there are controversial results in relation to the effect of the additions in the behavior of the OPC acids (Pavlik and Uncik, 1997). This study highlights the most negative effect of all the evaluated additions in the presence of CH_3COOH.
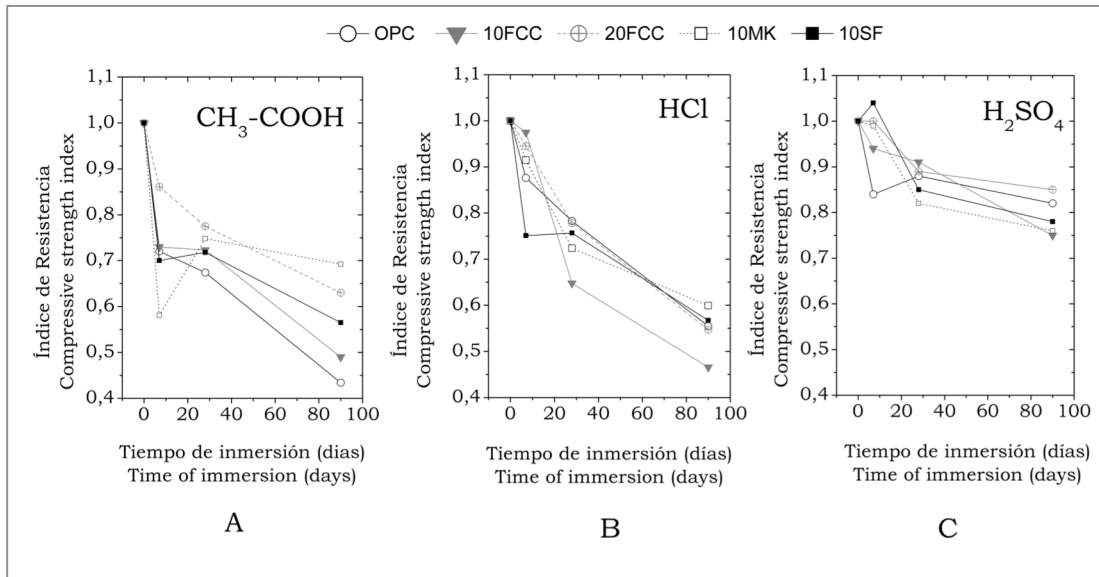
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Figure 1. Index of loss of strength of mortars exposed to acidic media

3.3 Microestructural characterization

3.3.1 X-ray diffraction

Figure 2 shows the diffractograms of the referenced pastes, exposed to various acids for 180 days of immersion. The evaluated pastes showed the characteristic peaks of the hydration products of cement, portlandite ($CaOH_2$; CH, PDF#01-078-0315), ettringite ($Ca_6Al_2(SO_4)_3(OH)_{12}.26H_2O$; AFt; PDF#03-0013-0350), calcium monosulfoaluminate hydrate ($Ca_4Al_2O_6(SO_4)\cdot14H_2O$; AFm, PDF#00-042-0062), calcium aluminate hydrate ($Ca_3Al_2O_6\cdot xH_2O$; C-A-H, PDF#00-002-0083), and calcium silicate hydrate ($2Ca_3Si_2O_7\cdot3H_2O$, C-S-H, PDF#00-003-0548). In the same way, the characteristic phases of anhydrous cement were identified, such as alite (Ca_3SiO_5; C_3S, PDF#00-042-0551) and belite (Ca_2SiO_4; C_2S, PDF#01-083-0464). In all cases, calcite ($CaCO_3$, PDF#00-024-0027) was identified as a result of carbonation during sample preparation. The samples made with additions exhibit a greater deviation from the baseline due to the presence of C-S-H gels formed during the pozzolanic reaction and a lower intensity in the peaks of CH.

The intensity of the characteristic peaks in portlandite in samples immersed in HCl suffers a slight decrease by dissolution of $Ca(OH)_2$ in the acid medium and subsequent formation of calcium chloride ($CaCl_2.6H_2O$). This compound was not identified by XRD because of its high solubility (279.3g/100g water) and therefore is easily leachable without the formation of microcracks that may affect the internal structure. This leaching has a significant effect on mass loss, as observed in Table 3, and the increase in capillary porosity that facilitates a significant drop in mechanical resistance (Figure 1). Also, a portion of $CaCl_2\cdot6H_2O$ formed by acid attack can react with hydration products rich in Al_2O_3, and depending on the Ca/Al ratio leads to the formation of Friedel's salt, especially in the presence of sFCC and MK, as identified in XRD (Figure 2) ($3CaO\cdot Al_2O_3\cdot CaCl_2\cdot10H_2O$, PDF#00-035-0105). These results are consistent with other studies in which the formation of Friedel's salt is attributed to the reaction of Cl present in the acid solution that has entered with the monosulfoaluminates generated during cement hydration (Miyamoto et al., 2013).

Immersion in H_2SO_4 leads to the formation of gypsum ($CaSO_4\cdot2H_2O$, PDF#01-074-1433) from the interaction of portlandite with H_2SO_4 via the reaction: $Ca(OH)_{2(S)}$ + $H_2SO_{4(aq)} = CaSO_4\cdot2H_2O_{(s)}$. This leads to a change in the geometry and pore size distribution within the material. The reduced solubility of gypsum salt (0.24g/100g water) results in less weight loss during immersion (Table 3). However, by increasing the salt content, internal stresses are generated in the material, especially in the aggregate/hydrated paste interface, which is consistent with strength losses recorded in Figure 1.

By contrast, in the presence of acetic acid, the formation of $Ca(CH_3COO)_2.H_2O$ salt takes place. Like the salt generated by HCl, it has high solubility (34.7g/100g water) and is not visible by XRD.

3.3.2 Scanning electron microscopy

Figure 3 shows micrographs of OPC, 10FCC, and 10MK pastes immersed in H_2SO_4 for 180 days, in which deterioration in the area that was in direct contact with the acid is clearly observed. The acid attack is identified by the appearance of microcracks traveling into the sample and increased porosity. Although the gypsum crystals are not identified, mainly due to the low magnification of the images, its presence and growth generates increased internal stresses that lead to the formation of microcracks shown in the micrographs. Considering that in the images, the areas that have the greatest open porosity from the attack exhibit a darker hue, it can be identified that 10MK has the highest deterioration, coinciding with the largest loss of strength (Figure 1).
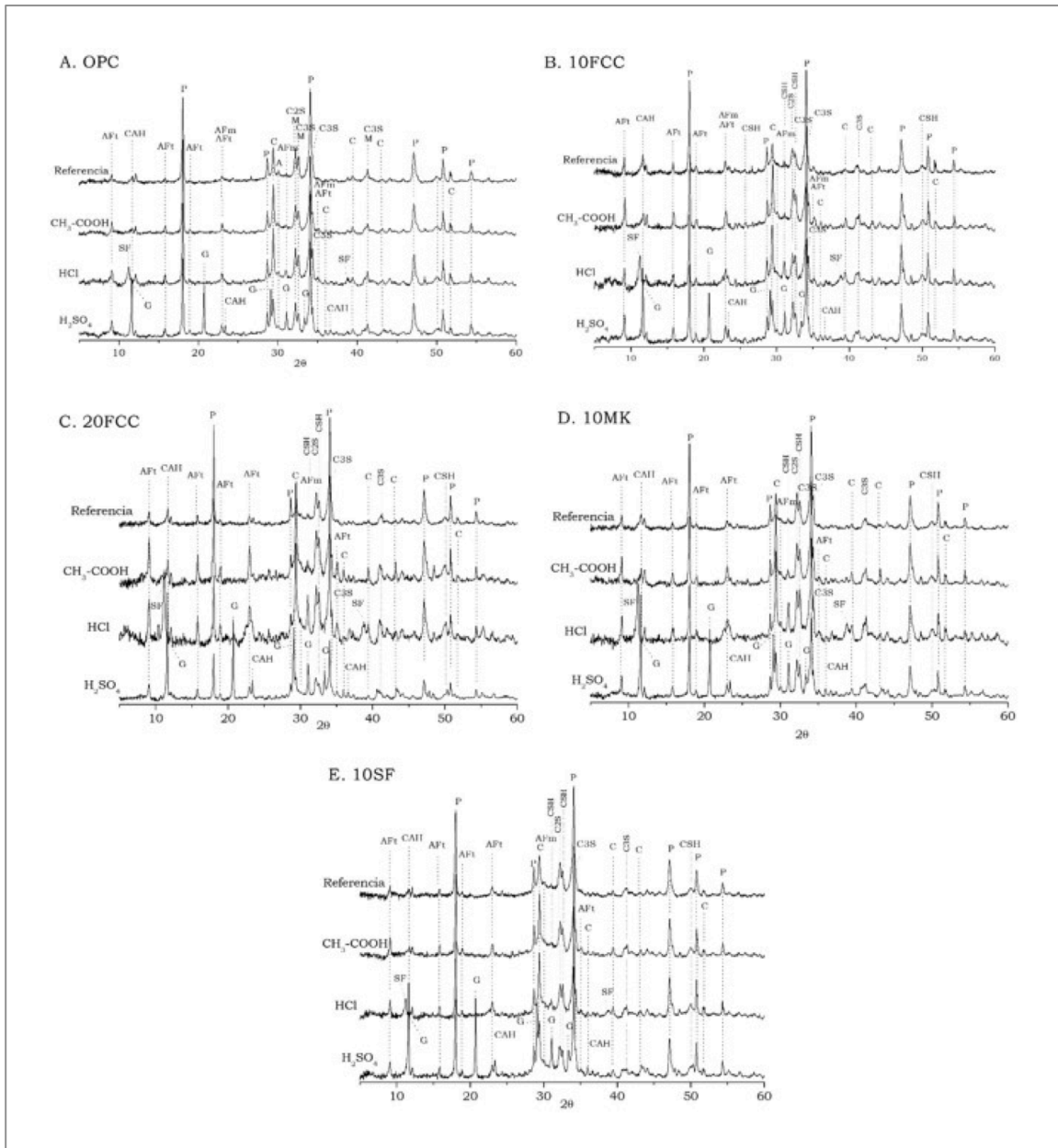
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Figure 2. *Diffractograms of pastes immersed in acids after 180 days. AFt: ettringite; CAH: calcium aluminate hydrate; P: portlandite; AFm: monosulfoaluminate; G: gypsum; C2S: dicalcium silicate; C3S: tricalcium silicate; SF: Friedel's salt; C: calcite*
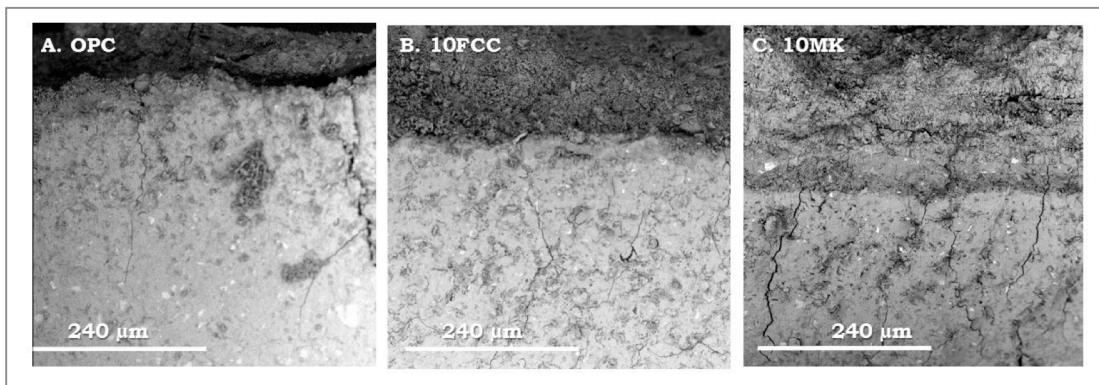

Figure 3. *Micrographs of pastes immersed in sulfuric acid after 180 days*

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4. Conclusions

The high reactivity of sRCC when incorporated into mortar mixtures leads to increased strength and structural densification through the pozzolanic reaction and formation of more stable and chemically resistant products. Increases in strength by incorporating sFCC were reported up to 17% compared to a non-blended control mortar. Immersion in acetic acid for OPC mortar led to a reduction in compressive strength of up to 57%, while mortars blended with sFCC reported declines of 37%. Microstructural analysis of the specimens immersed in HCl showed the formation of Friedel's salt, especially in those systems made with MK and sFCC. Meanwhile, mortars immersed in sulfuric acid reported losses in strength of up to 25% due to the formation of gypsum crystals that contributed to increased internal stresses. Overall, the rate of deterioration of the mortars evaluated presents the following order of aggressiveness from highest to lowest, $HCl>CH_3COOH>H_2SO_4$. The mortar blended with 20% of FCC has acceptable chemical resistance for use in the acidic media tested, with results comparable to MK in the presence of HCl and CH_3COOH; its best performance is in the presence of sulfuric acid.

5. Acknowledgments

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