Informador Técnico 82 (1) January - June 2018: 68 - 78 ISSN 0122-056X | e-ISSN 2256-5035 doi:http://doi.org/10.23850/22565035.1351

Chemical resistance of alkaline activation concrete fly ash/slag: Sulfates and acids

Resistencia química de concretos de activación alcalina ceniza volante/escoria: Sulfatos y ácidos

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Received: 02- 09 - 2017 Accepted:05-05-2018

How to cite: Valencia-Saavedra, W., Angulo-Ramírez, D. & Mejia de Gutierrez, R. (2018). Chemical resistance of alkaline activation concrete fly ash/ slag: sulfates and acids. *Informador Técnico*, 82 (1), 67-77.doi: https://doi.org/10.23850/22565035.1351

Abstract

One of the most important characteristics of concrete in addition to its mechanical properties is its durability, which is associated with the service life of structures exposed to certain conditions or environments; This property is required to define the potential fields of application and facilitate the commercialization of new cementitious materials. In this study, the chemical resistance of an alkaline activation concrete based on fly ash (CV) and blast furnace slag (ESC) in relation 80/20 was evaluated; As a reference material, a conventional cement-based concrete (OPC) was used. The compressive strength of Geo CV/ESC concrete at 28 days of normal curing was 42.9 MPa, 26 % higher than that reported by the concrete OPC. Geo CV/ESC exposed to sulfates up to the age of 180 days shows no expansion and the loss of compressive strength is not significant (2 %) compared to OPC (39 %). In the case of exposure to acids, Geo CV/ESC presents a loss of resistance of 39 % and OPC of up to 80 %. These characteristics demonstrate a greater chemical resistance of the alkaline activation concrete and therefore its potential use in aggressive environments.

Keywords: acids; geopolymers; mechanical properties; chemical resistance; sulfates.

Resumen

Una de las características más importantes del concreto en adición a sus propiedades mecánicas es su durabilidad, que se asocia con la vida en servicio de las estructuras expuestas a ciertas condiciones o ambientes; esta propiedad es requerida para definir los potenciales campos de aplicación y facilitar la comercialización de nuevos materiales cementantes. En este estudio, se evaluó la resistencia química de un concreto de activación alcalina basado en cenizas volantes (CV) y escoria de alto horno (ESC) en relación 80/20; como material de referencia se utilizó un concreto a base de cemento convencional (OPC). La resistencia a la compresión del concreto Geo CV/ESC a 28 días de curado normal fue de 42,9 MPa, 26 % superior a la reportada por el concreto OPC. Geo CV/ESC expuesto a sulfatos hasta la edad de 180 días no muestra expansión y la pérdida de resistencia a la compresión no es significativa (2 %) en comparación con OPC (39 %). En el caso de la exposición a los ácidos, Geo CV/ESC presenta una pérdida de resistencia del 39 % y OPC de hasta el 80 %. Estas características demues-tran una mayor resistencia química del concreto de activación alcalina y por tanto su potencial uso en ambientes agresivos.

Palabras clave: ácidos; ceniza volante; geopolimeros; propiedades mecánicas; resistencia química; sulfatos.

Introduction

Although ordinary Portland cement (OPC) is the material most commonly used in construction, its resistance to chemical attacks such as acids and sulfates is still a topic of interest in the scientific community. Cementitious materials can be significantly affected in the presence of acids given the acid-base reaction with the calcium hydroxide present in the pores and even the potential affectation of some of the hydration products. The sulfate attack, on the other hand, is usually caused by the exposure of the concrete to the alkali or alkaline earth sulfates that are present in the ground, in groundwater, surface water and seawater, which can enter the concrete. and react with the components of the cement matrix. This attack gives rise to the appearance of cracks, delamination, generation of expansive products, such as ettringite ($3CaO.Al_2O_3.3CaSO_4.31H_2O$) and gypsum ($CaSO_4.2H_2O$), and causes the loss of strength in concrete structures (Crammond, 2003, Irassar, Bonavetti, Trezza, and González, 2005). In recent decades, geopolitical cement has emerged as one of the possible alternatives to OPC cement due to their high resistance to early ages and their good durability properties such as resistance to acid attack and sulfates (Palomo, Grutzeck, and White, 1999). Among these, alkaline activation cement based on fly ash (CV) as a precursor has been investigated since the 1990s, with highly promising results; especially in some aggressive conditions in which concrete portland cement is vulnerable (Song, Marosszeky, Brungs, and Munn, 2005).

Geopolitical cement could be an alternative, for example, in the development of acid-resistant concrete. Davidovits, (2005); Van Jaarsveld, Van Deventer, and Lorenzen, (1997) found that alkaline activation cement based on metakaolin exhibit very low mass loss when the samples are immersed in 5 % sulfuric acid solutions for 4 weeks. Bakharev (2005b) studied the resistance of this type of material and concludes that they have better resistance than their counterparts produced with portland cement. In the same sense, Song *et al*., (2005) using accelerated tests demonstrated the greater durability of alkaline activation concretes in a 10% sulfuric acid solution for 56 days, which is also reported by other researchers (Wallah and Rangan, 2006; Thokchom, Ghosh, and Ghosh, 2009; Allahverdi and Skvara, 2005, 2006). However, the lack of standardized methods to evaluate the performance of cement in acidic environments has made it difficult to correlate the results obtained by different researchers.

Regarding the performance in the presence of sulfates, studies are scarce and most of them have been carried out in pastes and mortars and many of them at variable concentrations (Abora ***et al***., 2014; Singh, Ishwarya, Gupta, and Bhat-tacharyya, 2015). Škvára, Jílek, and Kopeckỳ, (2005) investigated the resistance to sulfates of activated CV pastes and mortars until the age of 720 days, and reported resistant increments throughout the trial, indicating that no dimensional changes or phase formation were observed expansive, it is presumed that sodium sulfate acts as an activator in the mixture. Puertas, Gutiérrez, Fernández-Jiménez, Delvasto, and Maldonado (2002), studied the behavior of steel slag mortars and mixtures of slag/CV 50/50 activated with sodium silicate and with sodium hydroxide, it is concluded that in both cases the activated mortars have high resistance to attack by sulfates, however, the samples activated with NaOH are more susceptible to degradation, which they attribute to the presence of expansive phases such as gypsum and ettringite. Ismail, Bernal, Provis, Hamdan, and van Deventer (2012), reported in activated slag pulp/50/50 CV exposed to 5 % sodium and magnesium sulfates for a period of three months, greater aggressiveness of magnesium sulfate-so attributed to the greater decalcification of hydrated calcium silicate gel and the formation of gypsum, thus coincides with the type of action that has been reported for portland cement concrete (Aye and Oguchi, 2011).

Based on the foregoing, the present study aims to evaluate the performance of alkaline activation concretes based on the binary mixture of Colombian fly ash (CV), coming from an industrial boiler, with blast furnace slag (ESC). in 80/20 proportions, after exposure to sodium and magnesium sulfate (Na_2SO_4 and $MgSO_4$) and acids acetic (CH_3COOH) and sulfuric (H_2SO_4) at age up to 180 days, and compare this behavior with the corresponding one of conventional concrete. For this, the level of longitudinal expansion and the change of mechanical resistance were determined for the case of concrete exposed to sulfates; for the samples exposed to acids, the loss of mechanical strength, the change in weight was measured and a visual inspection of the samples was carried out.

Materials and experimental methodology

Materials

In this study, fly ash (CV), a by-product of the combustion of coal in a boiler located in a paper mill, was used as the precursor of the concrete alkaline activation. The chemical composition of CV, determined by X-ray fluorescence, is presented in Table 1, where it can be seen that it is composed of approximately 56.5 % silica, alumina and iron oxides, thus fulfilling the requirement defined in the ASTM C618-12 (2012) (minimum 50 %) for fly ash type C, however its calcium oxide content is low (6.68 %) and generally this type of ash reports orders greater than 10 %. Of note is the high content of unburned (LOI = 20.67 %), a value that far exceeds that defined in the standard (6 % maximum), as well as a relatively high content of sodium oxides (7.94 %). These latter characteristics limit the use of this material as a pozzolanic addition to portland cement and justify the search for other valorization alternatives.

Table 1. Physical and chemical characteristics of the materials used

Characteristics, %	CV	ESC	Cement (OPC)
SiO_2	28,53	31,99	20,20
Al_2O_3	19,18	14,54	7,00
Fe_2O_3	8,80	1,12	4,80
Na_2O	7,94	0,23	--
CaO	6,68	46,86	58,40
S	2,71	0,82	--
MgO	2,24	1,05	--
TiO_2	1,62	0,54	--
Otros	1,65	1,03	--
LOI	20,67	1,82	9,60

Source: the authors.

Additionally, a steel blast furnace slag (ESC) was used as a partial replacement of the CV in the polymeric material, thus constituting a mixture of binary type. The slag comes from the company Acerías Paz del Río, and according to the chemical composition included in the same Table 1, it presents a quality coefficient $((CaO+MgO+Al_2O_3)/(SiO_2)+TiO_2))$ and a basicity coefficient ($Kb=CaO+MgO/SiO_2+Al_2O_3$) of 1.92 and 1.03 respectively; this allows his classification as a neutral type slag. As a reference cementing material, a Portland cement (OPC) was used, whose characteristics (Table 1) indicate that it corresponds to a highly added cement, in this case with a limestone material. As an activator, a mixture of commercial sodium silicate ($Na_2SiO_3.nH_2O$) of composition SiO_2: 32.24 % was used; Na_2O: 11.18 %; H2O: 55.85 % and industrial sodium hydroxide (NaOH) of 96.7 % purity.

Preparation of specimens and tests carried out

The alkaline activation concretes were made with the binary fly ash mixture (CV) and blast furnace slag (ESC) in 80/20 ratios, using as an alkaline activator a mixture of sodium silicate and sodium hydroxide; the dosage of the activator was adjusted to obtain molar proportions in the mixture of Si/Al=3.85 and Na/Si=0.25. For the production of the concretes a thick aggregate of siliceous type was used (maximum size: 19 mm, bulk density: 2440 kg/m^3, and absorption: 2.55 %), and a river sand as fine aggregate (modulus of fineness): 3.1, bulk density: 2510 kg/m^3, and absorption: 1.72 %). The proportion of coarse and fine aggregates was 42 % and 58 % respectively (see Table 2).

The ratio (L/S) used was 0.48. It should be noted that L represents the water content present in the mixture, in addition to that provided by the activant, and S includes the solid phase represented by the precursors (CV+ESC) and the anhydrous activator. A concrete based on OPC was used as reference material, as shown in Table 2. Geo CV/ESC samples were cured for 28 days at room temperature, with a relative humidity greater than 90 % and OPC samples They were cured under water for equal time. At the age of 28 days, and prior to exposure to sulfates, the compressive strength and the capillary suction coefficient for each concrete mixture were evaluated; the latter was determined based on the Swiss Standard EMPA-SIA 162/1 (1989).

Table 2. Proportions of components per m^3 of concrete

Mixtures	Cement (Kg)	CV (Kg)	ESC (Kg)	NaOH (Kg)	SS (Kg)	Sand (Kg)	Gravel (Kg)	Relationship L/S
Control (OPC)	400	-	-	-	-	972,72	704,39	0,48
GEO(CV/ESC)	0	320	80	28,55	158,37	972,72	704,39	0,48

Source: the authors.

The chemical resistance to attack by sulfates was carried out based on ASTM C1012-13 (2012), exposing the samples to solutions of sodium sulfate (Na_2SO_4) and magnesium sulfate ($MgSO_4$) at a temperature of 25 °C. up for a period of 180 days; the pH of the solutions throughout the study was maintained between 6 and 8. Prismatic specimens (50.8 x 50.8 x 285 mm) were used to evaluate the longitudinal expansion and cubes of concrete (50.8 x 50.8 x 50.8 mm) to determine the loss of compressive strength (ASTM C109, 2012). The solutions of sulfuric acid (H_2SO_4) and acetic acid (CH_3COOH) were prepared at a concentration of 1 M. The samples were immersed for a period of 180 days in each of the solutions; During the whole study, a pH control was carried out to avoid the neutralization of the environment. At different ages of the test, weight loss was recorded, for which the previously washed sample was subjected to a drying process at 60 °C for 24 hours, and the loss of compression resistance in cubes was determined (50, 8 x 50.8 x 50.8 mm). In addition, a visual inspection of the samples was carried out.

Results and Discussion

Physical-mechanical properties

Table 3 shows the values of compressive strength of the concretes at 7 and 28 days of curing, prior to the immersion in sulfates and acids, in which it can be seen that the Geo CV/ESC have a greater resistance to compression at 7 days of curing up to 59 % compared to control concrete (OPC). These results are consistent with those reported in the literature, where concrete activated CV and ESC bases have high resistance at early ages of curing (Fernández-Jiménez and Palomo, 2009; Gao, Yu, and Brouwers, 2015; Kovalchuk, Fernández -Jiménez, and Palomo, 2007, Nath and Sarker, 2014, Ranjbar, Mehrali, Behnia, Alengaram, and Jumaat, 2014, Ryu, Lee, Koh, and Chung, 2013). After 28 days of curing the Geo CV/ESC concretes managed to obtain resistance of 43 MPa, which exceeds that reported by the control concrete by 39 %, this behavior coincides with that expressed by other researchers (Fernández-Jiménez and Palomo, 2009; Gao *et al*., 2015), who attribute this behavior to the coexistence of the alkaline activating gels NASH and CASH. In terms of water absorption properties, Geo CV/ESC samples show a reduction in capillary absorption of 44 %, compared to OPC concrete, and the resistance to water penetration is, in turn, higher in orders of up to 70 %, this behavior is related to the more dense structure that presents the alkaline activation system and a network of pores of greater tortuosity (Valencia, Mejía de Gutiérrez, and Gordillo, 2018).

Table 3. Physical-mechanical properties of concrete before being exposed to aggressive environments

Resistance to Compression (MPa)		
Days	**Control OPC**	**Geo CV/ES**
7	21,90	34,75
28	30,93	42,92
Physical properties		
Hair absorption coefficient, k, (kg/m2 s1/2)	0,0292	0,0162
Resistance to water penetration,m, (s/m2)	1,899	3,242

Source: the authors.

Sulfate attack

Figure 1 shows the results of the expansion test carried out following the ASTM C1012 (2012) standard in reference concrete samples (OPC) and alkaline activation of Geo CV/ESC, after immersion in a solution of Na_2SO_4 and $MgSO_4$ at a concentration of 50g/L and temperature of 25 °C. The concretes immersed in the Na_2SO_4 solution show a greater expansion compared to the samples immersed in $MgSO_4$ for the two mixtures. After 28 days of exposure in the Na_2SO_4 solution, it is observed that the OPC sample shows an expansion of 0.05 %, which remains stable up to 120 days, unlike the Geo CV/ESC sample that reveals an expansion of 0.005 % (ten times lower) up to 120 days. As of this age, OPC samples showed a significant increase in the percentage of expansion (0.31 %) with respect to Geo CV / ESC samples (0.013 %) as observed up to 270 days of exposure to this medium. The greater deterioration of concrete based on portland cement is due to the reaction of sulfate with calcium hydroxide and calcium monosulfoaluminate to form gypsum and ettringite, which leads to loss of strength, expansion, appearance of cracks, and detachment of the superficial layers in concretes (Snelson and Kinuthia, 2010; Yang, Zhongzi, and Mingshu, 1996). Alkaline activated concretes, however, exhibit a different mechanism, where the main hydration products are less susceptible to attack by Na_2SO_4. (Sata, Sathonsaowaphak, and Chindaprasirt, 2012).

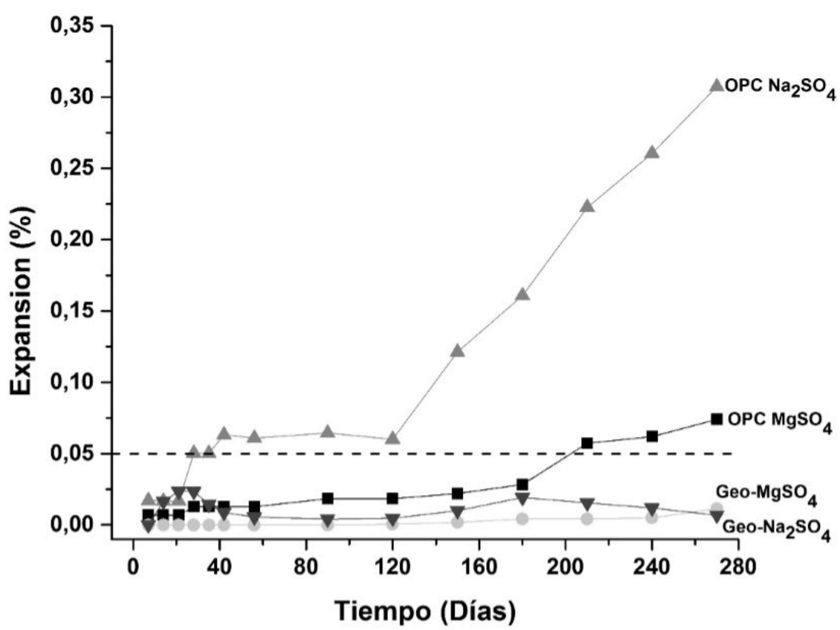

Figure 1. Graph of expansion vs Time of concretes exposed to Na2SO4 and MgSO4
Source: the authors.

The Geo CV/ESC samples immersed in the $MgSO_4$ solution did not show expansion at 21 days of exposure, unlike the control concrete which shows an expansion of 0.00 7%. The concrete Geo CV/ESC exposed in Na_2SO_4 initiate the process of expansion at early ages, different from that observed in $MgSO_4$, which begin the expansion at 120 days with a 0.0006 % expansion, at this age the OPC samples they present expansion values of 0.0184 %, tripling the expansion of the alkaline activation samples. At 270 days of exposure, the Geo CV/ESC concretes do not show a significant increase in the percentage of expansion (0.0114 %), contrary to what happened with concrete-based Portland cement (OPC) which exhibits values of relatively high expansion (0.074 %) exceeding approximately 6 times the concrete of Geo CV/ESC. In general, taking into consideration the recommended expansion limits for portland cement and cement added with slag after exposure to sulfates for a period of six months (ASTM C1012-13, 2012), it can be stated that the alkaline activation concrete Geo CV/ESC classifies as a material with high resistance to sulfates, due to its expansion of less than 0.05 % in the two media evaluated (ACI regulation 201, 2016).

Figure 2 shows the behavior of resistance to compression of the concretes of alkaline activation (Geo CV/ESC) and portland cement at 60, 120 and 180 days of exposure in the solutions of magnesium sulfate and sodium sulfate ($MgSO_4$ and Na_2SO_4). Between 60 and 120 days, the Geo CV/ESC and OPC concretes do not show resistance losses in either of the two sulphate media; and in general manage to maintain and even surpass the sample cured in water (OPC control, Geo CV/ESC control) in orders up to 12 %; This behavior in the samples of alkaline activation agrees with that obtained by Bakharev (2005a) who reported resistant increases of up to 4 %, besides this result is consistent with what was obtained in the expansion test (Maximum value of 0.05 % for the OPC samples and 0.005 % for Geo CV/ESC samples) up to 120 days of exposure. In the case of OPC, this can be attributed to the formation of expansive compounds that in the first stages could densify the matrix, while in the case of Geo CV/ESC it is attributed to the participation of the sulfates in the activation process of non-activated species which leads to new reaction compounds. After 180 days of exposure, it is observed that all the samples show a loss in the resistance, being higher for the OPC samples, this behavior can be attributed to the presence of portlandite (CH), which reacts with the SO_2 ions. give rise to ettringite, causing cracking in the structure and an increase in porosity (Mehta and Monteiro, 2006). It is observed that in the Na_2SO_4 solution, the Geo CV/ESC samples show a loss in compression resistance of only 2.65 %, which is not very significant, which agrees with the low level of expansion (0.019 %). behavior coincides with what was found by Ismail et al., (2012). On the other hand, the sample of OPC exposed to this medium presented a loss of resistance of 18.4 %, which was to be expected due to its high percentage of expansion. After 180 days of exposure in the $MgSO_4$ solution, a drop in compression resistance was observed but this time in a higher percentage, for the Geo CV/ESC samples a loss of 4.03 % was found, which it may be due to the presence of magnesium in the aggressive agent, which leads to the decalcification of the phases of the Ca-rich gel present in the alkaline activation system, causing the degradation of the system and the formation of gypsum (Ismail *et al.*, 2012; Komljenović, Baščarević, Marjanović, and Nikolić, 2013; Chindaprasirt, Paisitsrisawat, and Rattanasak, 2014). For OPC concretes, a higher resistance drop was observed, of the order of 31.87 %, this is attributed to the fact that there is a greater decalcification of the C-S-H due to the potential replacement of Ca^{2+} by Mg^{2+} to produce the hydrated magnesium silicate.

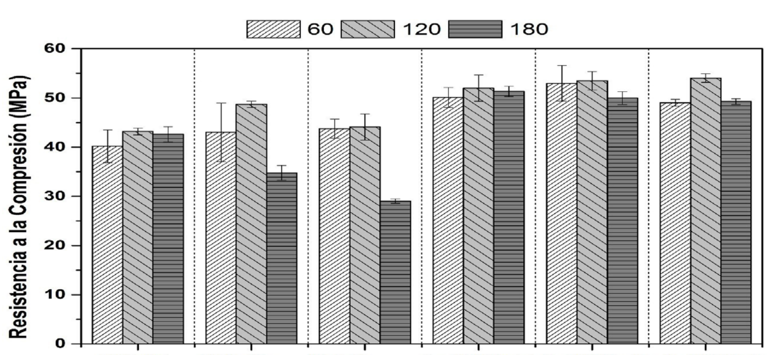

Figure 2. Resistance to compression of concrete exposed to Na_2SO_4 and $MgSO_4$
Source: the authors.

Acid attack

The exposure of the concrete to the different acid media generally leads to a deterioration that is particularly visible on the exposed surface. It is established that the reaction products are silica hydrogels, aluminum and iron oxides, and acid salts, which in the case of being soluble are removed from the solution; The generation of a decalcification process is reflected in losses in weight and physical-mechanical properties (Izquierdo, Rodríguez, and Mejía de Gutiérrez, 2015). They are identified as factors that influence the speed of attack: those related to the solution (acid resistance, calcium salt solubility, concentration, pH, diffusivity of the acid and its salt), those related to the material cement (type and amounts of cement, the nature of aggregates, permeability, curing time, and service conditions), and those that are external such as temperature (Allahverdi and Skvara, 2005). The results of the compression resistance of the samples exposed to the acids are shown in Figure 3. According to these results, the resistance values for all the concretes studied followed a pattern of loss of resistance in the two media (acetic acid (CH_3COOH) and sulfuric acid (H_2SO_4). In the Geo CV/ESC, however, the sulfates were reduced by approximately 40 % and 50 % up to 180 days of exposure in the two media (CH_3COOH and H_2SO_4), while in control concrete (OPC) the resistance was reduced by up to 82 %.

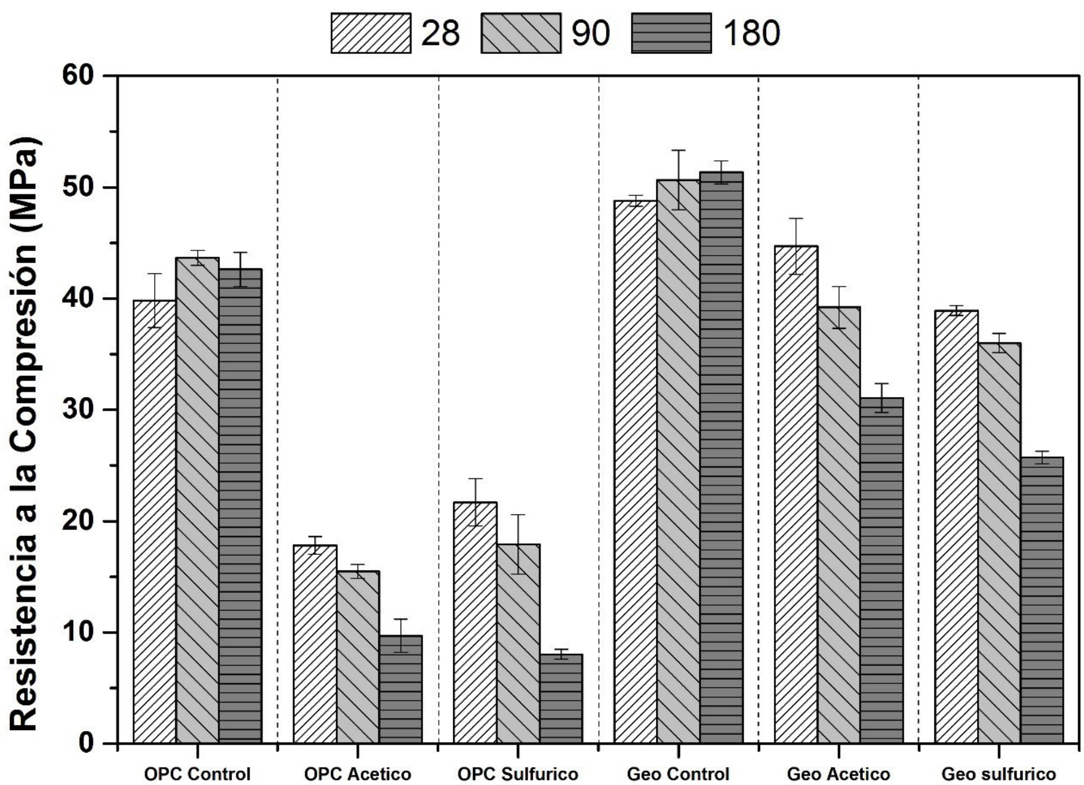

Figure 3. Resistance to compression of concrete exposed to acetic and sulfuric acid
Source: the authors.

The good performance of the Geo CV/ESC immersed in the acetic acid is highlighted, with a lower loss of resistance compared to those immersed in sulfuric acid, contrary to what happens with the OPC concretes wherein both media the samples present a very similar deterioration of the resistances. This decrease in the resistance values agrees with the loss of mass of the specimens, the concrete submerged in the CH_3COOH solution showed weight losses of around 5 % after 180 days of exposure for the Geo CV/ESC and a loss up to 14 % for control concrete. Systems immersed in H_2SO_4, however, showed a weight gain between 7 and 90 days of exposure for Geo CV/ESC samples, which is attributed to the reduced solubility of the salt formed (hydrated calcium sulfate or gypsum), at 180 days the resistant loss was 2.5 % for the Geo CV/ESC samples and 16 % for the OPC samples. It should be noted that Geo CV/ESC samples showed less weight loss in the solution of H_2SO_4.

The decrease in the mass of the concrete is attributed to the solubilization of some hydration products and the subsequent increase in porosity. This includes the decalcification process and the solubility of alumina and iron oxides (Allahverdi and Skvara, 2006). A visual examination of the specimens exposed to the acid solutions shows that the Geo CV/ESC concretes present good physical appearance until 180 days of exposure in both media (Figure 4). However, control concrete (OPC) shows a severe deterioration after 90 days of immersion in CH_3COOH and after 7 days in H_2SO_4, which coincides with its high loss of mechanical resistance and high loss of mass.

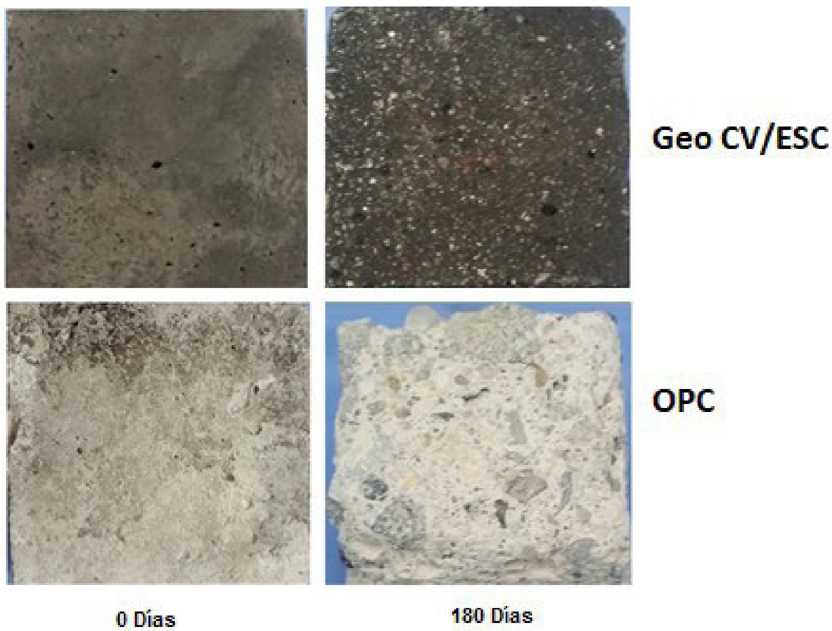

Figure 4. Concrete in sulfuric acid: Initial condition and 180 days of exposure
Source: the authors.

Conclusions

The results obtained in the present study confirm that the alkaline activation concrete evaluated (Geo CV/ESC 80/20) is less susceptible to the attack of sulfates and acids, compared to the concrete of traditional portland cement, this can be related to its greater resistance to compression, its low capillary absorption and its greater resistance to water penetration, which can cause greater resistance to attack by aggressive agents.

In the attack by sulfates, the greatest deterioration observed in the samples exposed to magnesium sulfate stands out, which indicates its greater aggressiveness. This attack results in the loss of mechanical properties attributable to the formation of new crystalline phases, particularly gypsum, in the presence of magnesium sulfate. This loss of resistance for Geo CV/ESC is 4 % at the age of 180 days of exposure to $MgSO_4$ compared to the 32 % reported by the concrete OPC. The expansion percentages of the concrete Geo CV/ESC and OPC were 0.005 and 0.062 % respectively. For concretes exposed to sodium sulphate, Geo CV/ESC shows a decrease in resistance of 2 % after 180 days, while in OPC the resistant reduction is 19 %. The percentage of expansion reported by Geo CV/ESC in Na_2SO_4 is negligible (0.0012 %) compared to that of OPC specimens (0.2604 %).

In the chemical attack by acids, it was found that the two systems show a deterioration in their properties, but the best performance of the Geo CV/ESC is highlighted, which presented the lowest loss of resistance and mass in the two exposure media acids (CH3COOH and H_2SO_4), compared to the control concrete.

Acknowledgments

The authors, members of the Composite Materials Group thank the Universidad del Valle (Cali, Colombia) and the Administrative Department of Science, Technology, and Innovation (Colciencias) for the financial support for the realization of this research work, through the project *"Construction of prototype at rural housing scale using innovative materials with low carbon footprint"*, Contract No. 0696-2016.

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