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Evaluation of durability and mechanical properties of the cement mortar added with slag blast furnace

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Abstract

An evaluation of durability and mechanical strength in a cement mortar is presented in this work. A slag coming from steelmaking processes has been added to the mortar. This slag has similar properties to the ones of cement, and it has been added to the mortar in different proportions with two sizes of granulometry. With this addition, better properties against the action of sulfates are expected, and therefore, the presence of fracture and wear in the structures can be reduced. This allows more safety and increased life in the mortar, and the most important thing is that, this will contribute to decrease the environmental contamination because the slag used in this work is coming from the industrial waste, which is produced in big amounts in the steel sector worldwide. The studied materials are mortar cubes in which the mechanical strength has been evaluated. The dimensions of the samples are around 50×50×50 mm³ and, according to the standard ASTM C109, these cubes should be evaluated during 1, 3, 7, 28, 56 and 118 days. The expansion tests to evaluate the sulfates attack is performed according to the standard ASTM C1012, in bars of around 25×25×285 mm³. These bars were submerged in water with lime and when the bars obtained a strength bigger than 20 MPa, they were also submerged in a sulfate solution, which was prepared 24 hours earlier with a pH that should be between 6 and 8. In this way, from the point of view of strength to sulfate attack, the mortar durability is evaluated by employing techniques such as X-ray diffraction, X-ray fluorescence and scanning electron microscopy, which allow us to determine the presence of different components that are responsible for the expansion as the formation of gypsum, ettringite, and thaumasite. As a result of this research, it is evident that slag with smaller particle size shows positive behavior against the action of sulfates, thus revealing minimum expansion percentages.

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1. Introduction

Durability in concrete structures is affected by the action of aggressive agents which contain sulfate ions present in water and in soils [Cavdar and Yetgin (2006)], generating a series of chemical reactions between the sulfate ions with the calcium hydroxide and calcium aluminate to form gypsum [CaSO_4] and ettringite [$\text{Ca}_6\text{-}[\text{Al}(\text{OH})_6]_2 \cdot 4(\text{H}_2\text{O}) \cdot [(\text{SO}_4)_3 \cdot (2\text{H}_2\text{O})]$]. As a consequence, an expansion is generated, cracking and deterioration. This last is also affected by the reduction of calcium silicate hydrated gel (C-S-H) through the leaching of calcium compounds that leads to the loss of rigidity of the gel. Other reactions are presented like, thaumasite, [$\text{Ca}_3\text{Si}(\text{CO}_3)(\text{SO}_4)(\text{OH})_6 \cdot 12\text{H}_2\text{O}$], among others [Pipilikaki et al. (2009)].

The granulated slag from blast furnace, industrial subproduct coming from the iron production, is a good complement with the Portland cement (PC), and it is mainly composed of silicates and aluminas with properties that make this material to generate positive effects as the low hydration [Binici y Aksogan (2006); Siddique y Bennacer (2012); Tsai y Huang (2014)], resistance to chlorides and sulfates, reduction of permeability and hydration. The hydration of the slag from blast furnace produces a big amount of hydrated calcium silicate (C-S-H) gel and a low content of calcium hydroxide (CH) in the PC at any age, which is shown in the reduction of the capillary porosity and in the mineralogical changes [Radwan et al. (2012)]. With the increase of these hydrated products as the tobermorite gel (C-S-H) strength increases at an early age in cements, otherwise this occurs when there is presence of slag because the strength acquired in long term as the reactivity of the slag is very slow and depends on the portlandite (calcium hydroxide), which reacts with this addition to form new tobermorite gel (CSH), acquiring better results than in the cements without addition.

In this study, different proportions and particle sizes of slag were mixed with PC added Type 1. To evaluate the performance against sulphates attack, expansion and mechanical strength were measured, also measurements of X-ray diffraction, (XRD), X-ray fluorescence (XRF) and scanning electron microscopy (SEM) were performed.

2. Materials

The materials used were sand with similar characteristics to those presented by the Ottawa sand, PC type 1, produced by Argos SA, granulated slag from blast furnace (GBFS) from the steelmaker Paz del Rio (Boyacá, Colombia) and distilled water to prepare the different buckets and mortar bars. The chemical composition and physical properties according to ASTM C 188 (1995), ASTM C 204 (2004), are listed in Table 1. The granulated slag from blast furnace is ground in a ball mill and two grain sizes are obtained from standard sieves: retained material between a sieve #40 and a sieve #200 is called slag ST and retained material between a sieve #325 and a sieve #400 is called slag TAM. Here, control samples are cubes and bars produced with 100% PC.

2.1. Materials characterization

2.1.1. X-Ray diffraction (XRD)

The XRD analysis is used to identify the crystalline phases and mineralogical compound present in the samples. The XRD equipment was a PANalytical from the X'PERT PRO line equipped with a copper tube with a voltage 40 kV and a current of 40 mA in continuous mode, using the Bragg-Brentano geometry, and sweep angle (2θ) from 10° to 90° .

The XRD patterns obtained from on the samples of (PC) and granulated slag from blast furnace (GBFS) are shown in Figs. 1 and 2.

The cement diffractogram demonstrates the characteristic components of this material. A series of crystalline phases clearly appear in higher percentages. One example is C3S, which is responsible for acquiring resistance in the short term. Also evident is C2S, a component responsible for long-term resistance, and gypsum, which causes mortar to set, among other components. Furthermore, it is observed that material is amorphous between 0° and 20° of 2θ

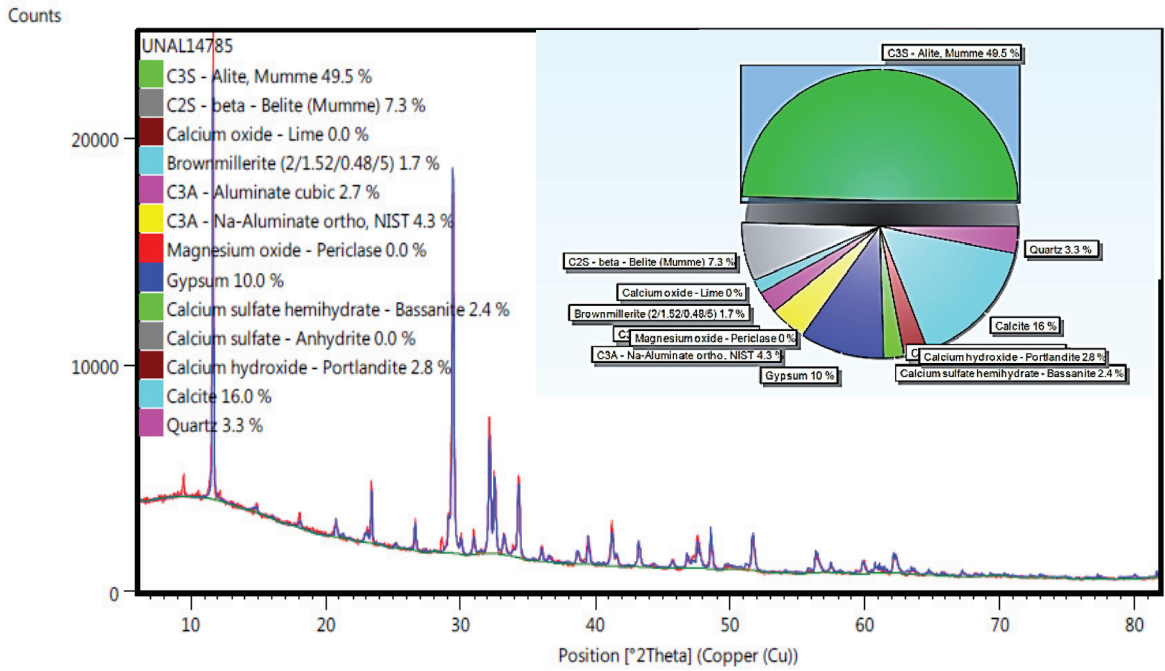


Fig. 1. XRD patterns of Portland cement.

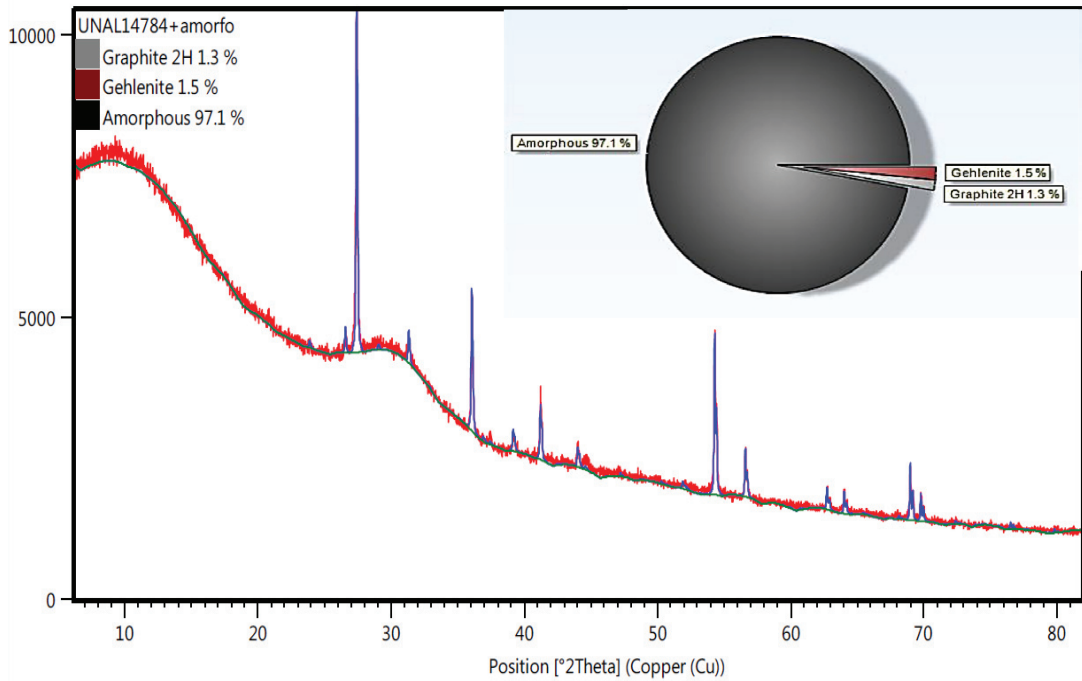


Fig. 2. XRD patterns of granulated slag from blast furnace (GBFS).

In Fig. 2, the GBFS is shown to be an essentially vitreous material as a result of the sudden and rapid cooling produced by jets of water as it exits the oven. It is mostly an amorphous material; however, it contains minimal percentages of crystalline phases.

2.1.2. Scanning electron microscopy (SEM)

The SEM technique is employed to observe the morphology and composition of different materials. The SEM equipment was a Leo 410, with a vacuum of 9.85×10^{-5} Torr, a current in the filament of 1.2 nA and an anodic voltage of 15 kV.

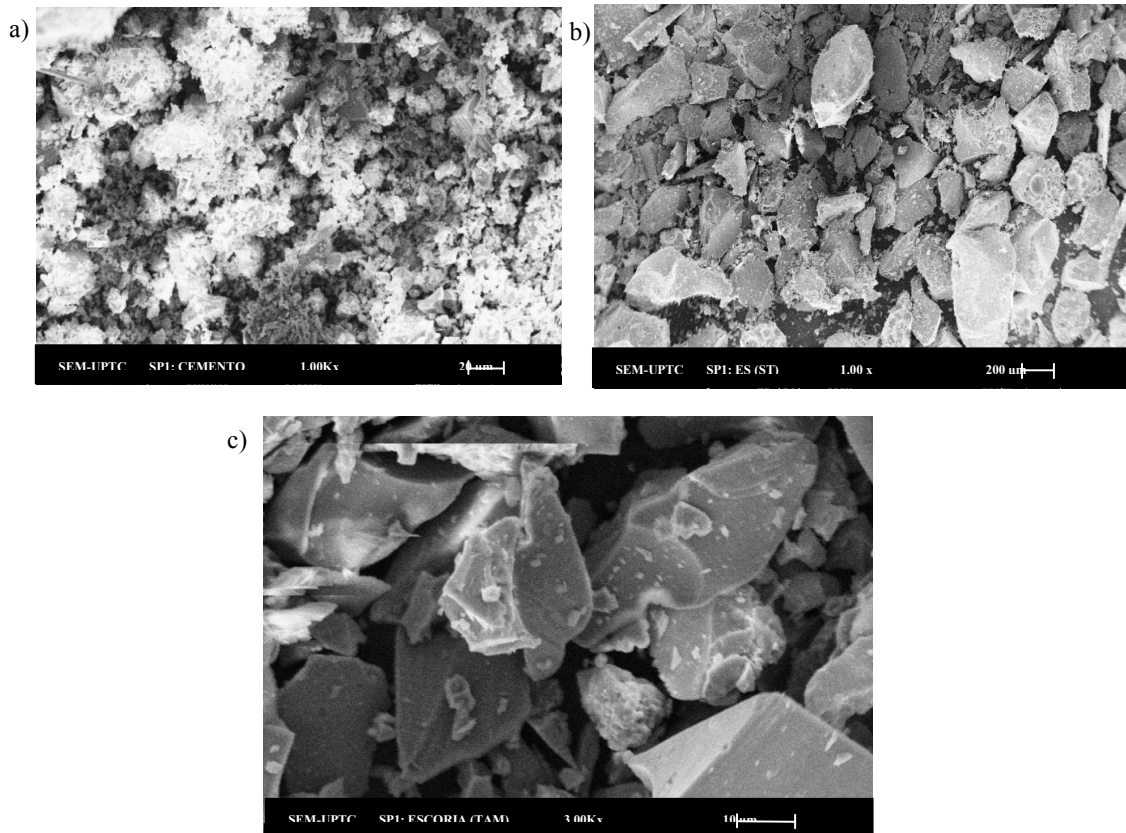


Fig. 3. SEM micrographs of (a) Portland cement (PC); (b) granulated slag blast furnace (ST); (c) granulated slag blast furnace (TAM).

As shown by the SEM micrographs in Fig. 3, it is clearly observed that the PC particles show an agglomeration of a wide variety of sizes in contrast with the ST and TAM slags, that show a heterogeneity in sizes, shapes and spacings between them.

2.1.3. X-Ray fluorescence (XRF)

The XRF is like the previous techniques, a complementary technique that is used to determine the chemical composition in a quantitative way in different materials. The analysis was performed in a PANalytical MiniPal 2, with a Rhodium tube, working at 9 kV and 0.002 mA, during 200 seconds.

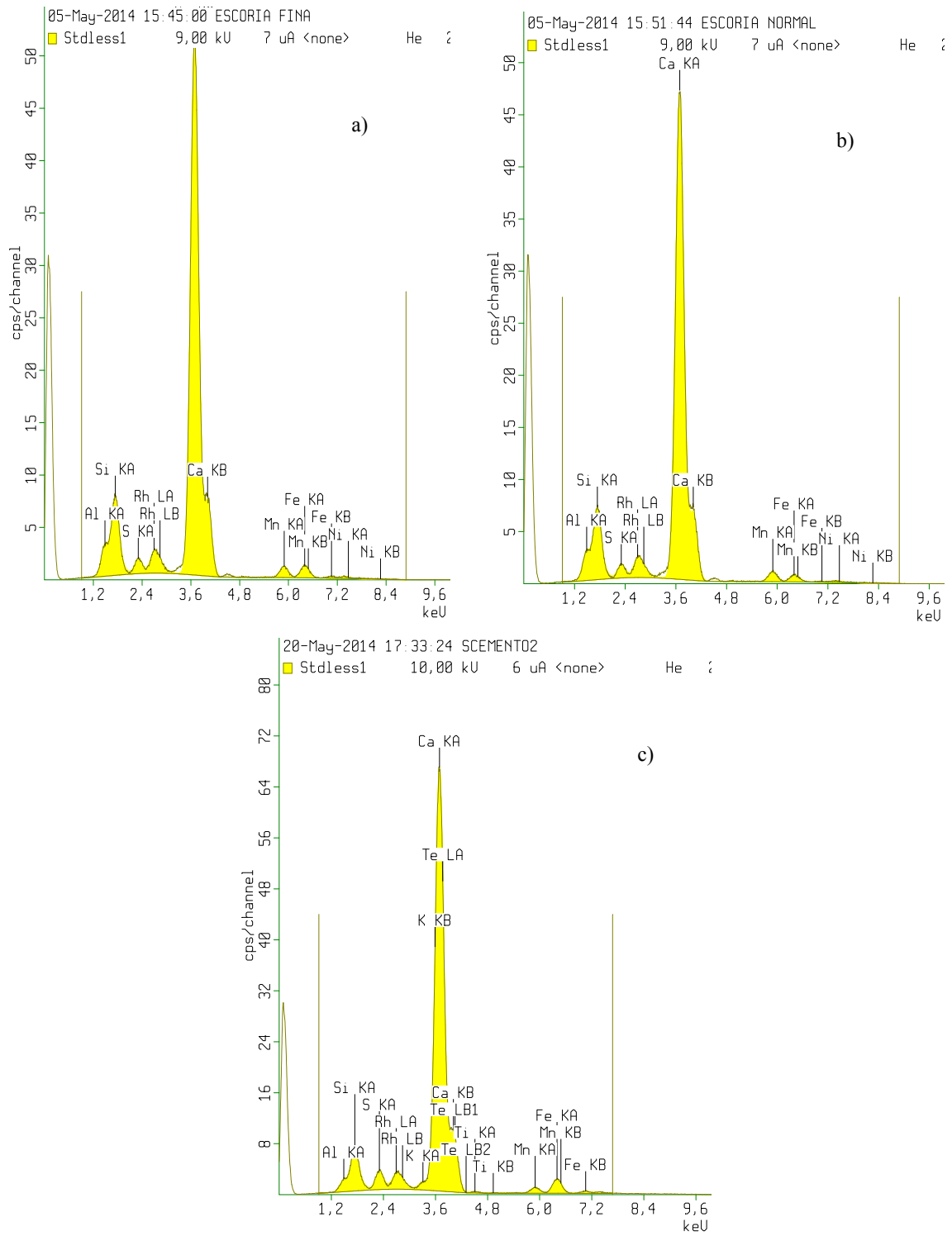


Fig. 4. XRF spectra of (a) granulated slag from blast furnace (TAM), (b) Granulated slag from blast furnace (ST) and (c) Portland cement (PC).

Fig. 4 shows that the three materials have high contents in CaO, Al₂O₃ and SiO₂ which are important to obtain mineralogical reactions and are common in the cement hydration. In addition, the high content of CaO contributes positively to obtain a good compressive strength [Cavdar and Yetgin (2010)].

Table 1. Chemical composition and physical properties of the materials.

Chemical Analysis (%)	PC	Slag TAM	Slag ST	Sand
Al ₂ O ₃	11.0	18.0	18.0	
SiO ₂	22.9	28.6	29.2	
SO ₃	3.2	1.7	1.7	
CaO	56.8	46.7	46.8	
MnO	1.1	1.8	1.9	
Fe ₂ O ₃	3.7	2.9	1.7	
NiO	--	0.6	0.9	
K ₂ O	0.78			
TiO ₂	0.27	2.91		
TeO ₂	0.6			
Physical characteristics				
Density (gr/cm ³)	2.9		2.87	2.6
Blaine Fineness. (cm ² /g)	4230			
Volume of the pore (cm ² /g)	0.002		0.003	
Pore size (mm)	16.9		25.48	
Minimum void ratio				0.52
Maximum void ratio				1
Humidity (%)				0.35

3. Experimental procedure

3.1. Mortar bars

For performing the mortar bars, the procedure described by ASTM C 1012 (2004) has been followed. In this norm, the change in the volume is evaluated in a solution of 5% of Na₂SO₄. A proportion of 2.75 of sand by 1 of cement has been used, distilled water and a relation of water/cement (a/c) of 0.48. Molds and screw terminals used comply with the requirements of ASTM C 490(2007). The bars were put in a humid chamber at a temperature of (23 ± 2) °C, for a time of 24 hours. Then, they were demoulded and cured until a strength equal or above to 20 MPa was obtained. Then, they were submerged into a solution of sodium sulfate. The bars were weekly monitored and the measurement has been taken in a comparator of lengths Humboldt MFG CO H-3250. The used mixtures are listed in Table 2.

3.2. Mortar cubes

For performing the mortar cubes, the ASTM C109 (2002) and ASTM C305 (2006) have been followed, with dimensions of 50×50×50 mm³, proportions of 2.75 of sand by 1 of cement and the use of distilled water, with an a/c relation of 0.48. The cubes were made in molds and they were left during 24 hours in a humid chamber according to ASTM C 511 (2009) at a temperature of (23 ± 2) °C. After that they were demoulded and cured until the age of failure.

Table 2. Used mixtures for cubes and mortar bars.

Mixtures	PC (%)	ST Slag (%)	TAM Slag (%)
Control	100		
20% ST	80	20	
40% ST	60	40	
60% ST	40	60	
80% ST	20	20	
20% TAM	80		20
40% TAM	60		40
60% TAM	40		60
80% TAM	20		80

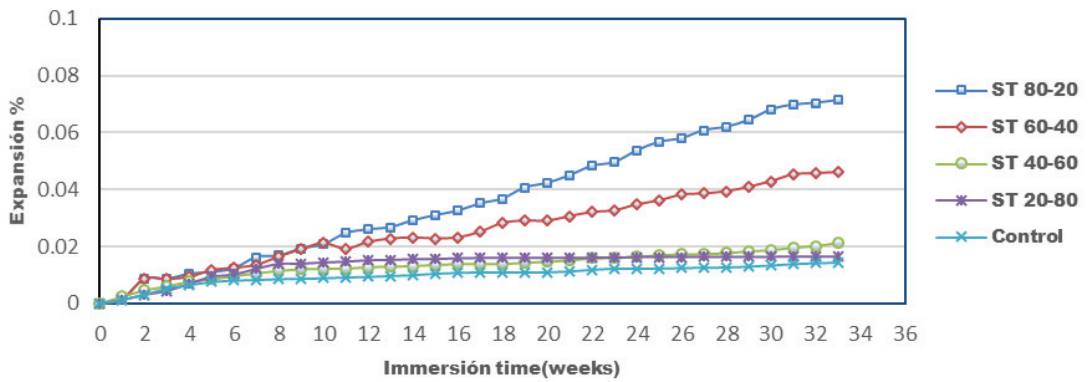


Fig. 5. Expansion of mortar bars with TAM slag and cement of control in a solution of 5% of sodium sulfate (Na_2SO_4).

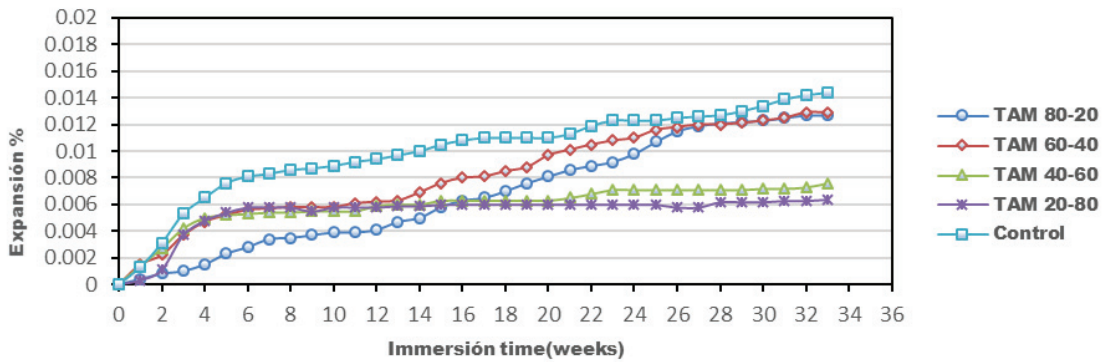


Fig. 6. Expansion of mortar bars with ST slag and cement of control in a solution of 5% of sodium sulfate (Na_2SO_4).

4. Results

4.1. Expansion

The bars made with different percentages of GBFS and different grain sizes are analyzed and evaluated in Figs. 5 and 6. In Fig. 5 the percentage of expansion vs exposure time in a solution of sodium sulfate is reported. It is seen be that the data lay within a range of 0% to 0.015%. The maximum expansion reported until week 33 is a 0.0144%, while the better minimum expansion was that obtained for with better results are revealed by the mixture with 20% of TAM slag, a value of 0.0064%.

In Fig. 6 there is a different behavior than that observed in Fig. 5 it shows an accelerated expansion in the rod made with ST slag with respect to the control bars. The maximum expansion is seen for the bars with 20% of ST slag with a value of 0.0714 %. It expands four times more than the control bars. The expansion decreases when the percentage of slag increases. As shown in Fig. 7, there is formation of ettringite, according to Giraldo and Tobón (2007), which are elongated crystals responsible for the expansive behavior of slag. In addition there is a bigger granulometry, causing a bigger porosity and bigger incidence of the sulfates.

4.2. Mechanical strength

The mortar cubes were evaluated at ages of 1, 3, 7, 28, 56, 118 days and the results are presented in the following.

In Fig. 8 the compressive strength vs time is reported. The behavior of mortar cubes with 3 different mixtures is shown: TAM slag, ST slag and control cubes, at ages of 1, 3, 7, 28, 56 y 118 days. The ST mixture with 20% of slag in all cases showed less strength, the control cubes showed higher strength except at the age of 118 days, and the

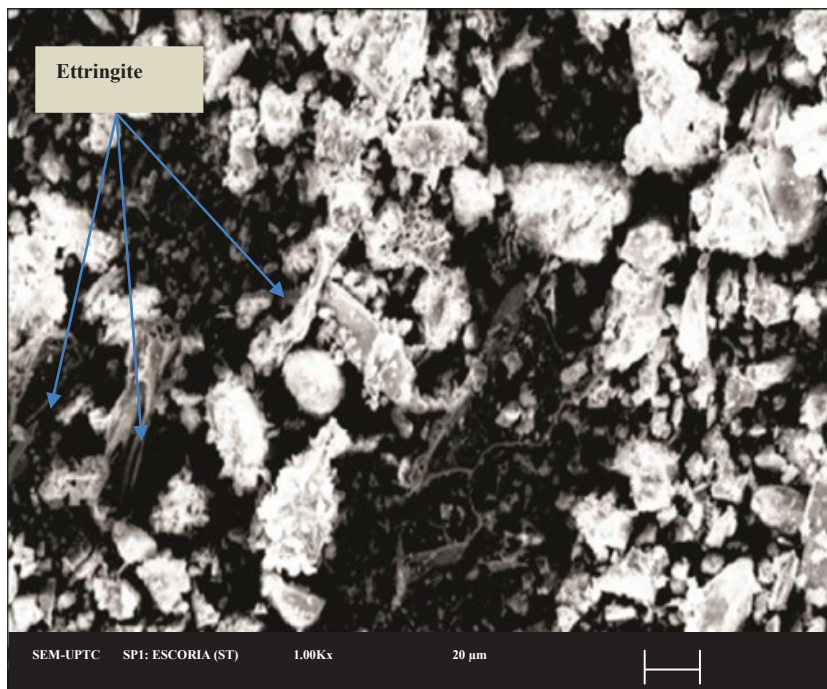


Fig. 7. SEM micrographs of granulated slag from blast furnace (ST).

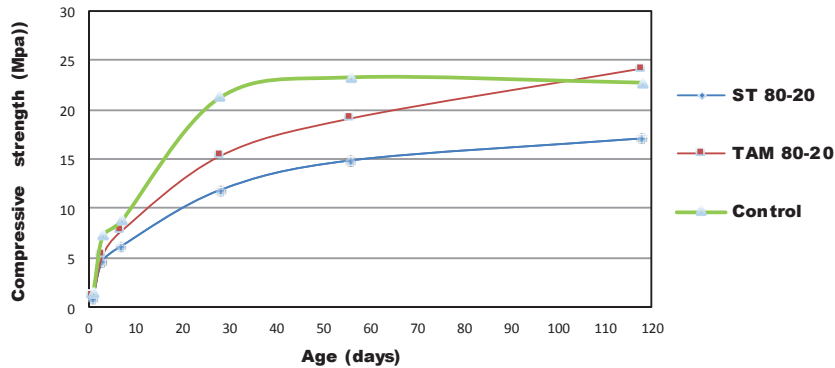


Fig. 8. Compressive strength to cubes of mortars with addition of granulated slag from blast furnace.

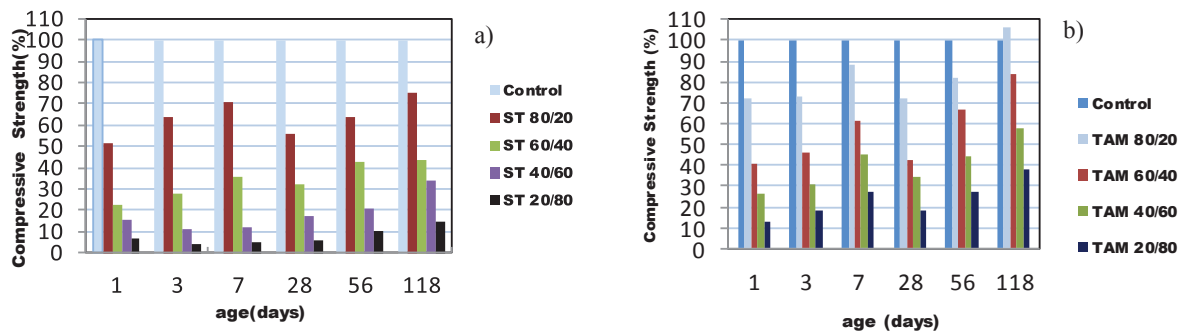


Fig. 9. Compressive strength in (%) of mortar cubes with addition of granulated slag from blast furnace. (a) TAM slag and (b) ST slag.

TAM mixture with 20% showed less strength than the control cubes except for 118 days where it showed a strength of about 24 Mpa increasing a 6%. It was also better in all strength with respect to the ST slag because the TAM slag showed a higher fineness, which allows, according to Ilker y Turhan (2007), a clear decrease in the permeability due to the pozzolanic reaction, thus improving their behavior.

In the same way, in Fig. 9 shows the results of compressive strength for different proportions of both ST and TAM slags compared with that of the control cubes for some days.

5. Conclusion

- The compressive strength increased when the grain size of the slag decreased, obtaining higher values than the control samples.
- The expansion shows better results with the addition of TAM slag regarding the control rods, showing a maximum expansion of 0.0144%.
- The ST slag did not show good results in strength because its big granulometry, which produced big voids and decreased the possible reactions that would yield a good strength.
- The ettringite is formed in a low proportion, generating an expansion in the ST rods. However, they do not increase more than 0.075%, in comparison with other materials that exceed 0.1%.

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