

Use of sulfoaluminate cement and bottom ash in the solidification/stabilization of galvanic sludge

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Abstract

Solidification/stabilization (S/S) process can improve the physical characteristics of wastes, reduce their leaching and limit the solubility of their heavy metals. The identification of binders able to assume the fixation of contaminants is essential for the success of the technique. In this study, calcium sulfoaluminate cement was added to another waste, bottom ash, in order to treat galvanic sludge. The properties of the resultant solid matrix (MS) were determined: setting time, compressive strength and products of hydration. Solid matrix composed of 77% waste and only 23% cement presented initial setting time lower than 4 h and 28 day-strength of 6 MPa. SEM investigations showed that contaminants present in the galvanic sludge (Cr) were encapsulated in the hydrated phases and particles of bottom ash.

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1. Introduction

The solidification/stabilization (S/S) technique was developed in the 60s for radioactive waste treatment. In the last decades, it has been used in the treatment of many industrial wastes, since it is possible, through this process, to improve physical characteristics, reduce leachability and limit the solubility of their contaminants [1]. The S/S process not only improves the physical and chemical conditions of the residue, but is also cheap compared to other treatment techniques. This is possible because the binders utilized (lime, pozzolan, cement) are not expensive and the technology is based on the same materials as in building construction. In this process, the identification of binders able to assume the fixation of contaminants is essential for the success of the technique. Sulfoaluminate cement (CSA) is one of the binders used in S/S processes.

The advantages of this type of cement compared to ordinary Portland cement (OPC) are related to lower setting time, and consequently, better mechanical properties at early ages [2,3].

Such aspects are extremely relevant since the contaminants of many sludges are strong retarders of OPC hydration. Therefore, researchers choose cement of high early strength combined with accelerators [4–7].

Another advantage of sulfoaluminate cement compared to Portland cement is related to the production process which presents a lower environmental impact due to the need of lower calcining temperatures and lower emissions of CO₂ [8].

Besides the advantages aforesaid about sulfoaluminate cement and its technical performance and production process, the major advantage in the S/S process is the formation of ettringite as the main hydrated phase. This compound can incorporate some heavy metals in its structure. Trivalent ions such as Fe⁺³, Cr⁺³ and Mn⁺³ can replace Al⁺³, while bivalent ions like Mg⁺², Zn⁺², Mn⁺², Fe⁺², Co⁺² and Ni⁺² can replace Ca⁺² [9–11]. SO₄⁻² ions can be replaced by CrO₄⁻², referring to CrVI [9]. Such advantage is important because in the S/S process, even though it is difficult to obtain a physically stable solid with low consumption of cement, the biggest obstacle is to maintain heavy metals present in the waste in the solidified matrix. This difficulty is directly related to the solubility shown by many metals of amphoteric behavior like Be, V, Cr, Mo, W, Zn, B, Al, Ga, In, Si, Ge, Sn, Pb, As, Sb, Bi, Se, Te and Po [12].

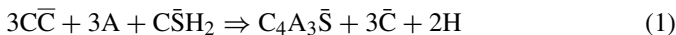
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The aim of this paper is to study the S/S of galvanic sludge (GS). GS is obtained from the chromium electroplating process and contains chromium as main heavy metal. Based on the S/S technique, a new methodology was developed to encapsulate GS based on sulfoaluminate cement and pozzolanic additions. The waste was used as produced (water content 80%). Bottom ash (BA) and sulfoaluminate cement were utilized to obtain a solid matrix (MS).

The properties of the solid matrix were evaluated by means of setting time, compressive strength, products of hydration assessed by X-ray diffraction (DRX), thermal analysis (DTA) and scanning electron microscopy (SEM).

2. Sulfoaluminate cement (CSA)

The raw materials involved in the production of sulfoaluminate clinker are bauxite, limestone and calcium sulphate [2]. The burning temperature of sulfoaluminate clinker (around 1350 °C) is lower than that required by the formation of ordinary Portland cement clinker (around 1450 °C) due to a smaller amount of limestone in the raw mix. Lower amount of limestone brings not only smaller consumption of energy during the decarbonation process, but also lower emission of CO₂ providing substantial contribution to the saving of natural resources, energy and environment. Phases of ordinary Portland cement clinker such as C₃A and C₃S (alite) do not exist in sulfoaluminate cement. In this case, the most important phase is yeelimite (C₄A₃ \bar{S}) and is generated according to the following equation [13]:



with: C = CaO; \bar{C} = CO₂; A = Al₂O₃; \bar{S} = SO₃; H = H₂O. Sulfoaluminate clinker contains about 50–70% of C₄A₃ \bar{S} . Other main phases are:

- belite (C₂S): 10–25% (S = SiO₂),
- ferrite (C₄AF or C₆AF₂): 5–20% (F = Fe₂O₃),
- mayenite (C₁₂A₇): 3–10% [8,14].

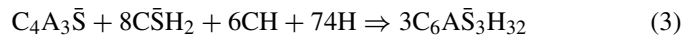
Yeelimite and mayenite fast hydrate and are responsible for setting and early age strength. Belite phase is mainly responsible for the development of ultimate strength since it slowly hydrates. C₄AF (C₆AF₂) contributes either for early or ultimate strength [15].

To obtain sulfoaluminate cement, the amount of gypsum added to sulfoaluminate clinker (CSA) can reach up to 30%, while in Portland cement this amount goes down to 5% [3]. From an environmental point of view this characteristic is also an advantage: residues containing large amount of calcium sulphate, such as phosphogypsum, can be reused in the production process. Recent studies using sulfoaluminate cement containing up to 30% phosphogypsum showed very interesting properties [16,17]. Eq. (2) shows the reaction between yeelimite (main phase of sulfoaluminate cement), calcium sulphate (usually gypsum) and water, generating ettringite and gibbsite as products of hydration [15].



The main product of sulfoaluminate cement hydration is ettringite (C₆A \bar{S} ₃H₃₂), and not C-S-H nor portlandite (CH), as in Portland cement. Ettringite is responsible for the development of early age strength [13]. The microstructure of ettringite depends on the presence of lime. According to Eq. (2), ettringite generated in absence of lime does not have expansive properties and shows high early strength.

When hydration occurs in presence of calcium hydroxide (Eq. (3)), ettringite is very expansive, and this property is used in shrinkage compensating cements [18]:



CH comes from the hydration of either belite (C₂S) or Portland cement [19]. If lime is in an unsaturated condition, ettringite will not expand and contributes to the strength of the paste [20]. According to [20], the formation of expansive ettringite depends on the alkalinity of the environment.

3. Bottom ash (BA)

BA is a by-product of the burning of coal in thermal power plants [21]. The reasons for using BA in S/S processes are based on economic, environmental and technical advantages. The first two ones are justified by the use of a waste, with no commercial value. The technical advantages are based on the physical and chemical properties of BA and its hollow nature as observed in Fig. 1. Hollow grains are able to absorb water and dissolved foreign elements. This property is very significant in S/S process of sludge with high water content. Regarding the chemical aspect, bottom ash can represent a source of alumina for ettringite formation, in the same way as fly ash [22,23].

The effect of pozzolanic additions in the expansion reactions of ettringite due to the presence of free lime in sulfoaluminate clinker was also studied [24]. In cement pastes, without addition, 5% of free lime increased expansion by 1.5–1.7 times. Addition of 8% pozzolanic material and 10% of silica reduced expansion by 1.5–2.5 times. The author concluded that this behavior occurred because free lime did not react with yeelimite but with silica and the pozzolanic material.

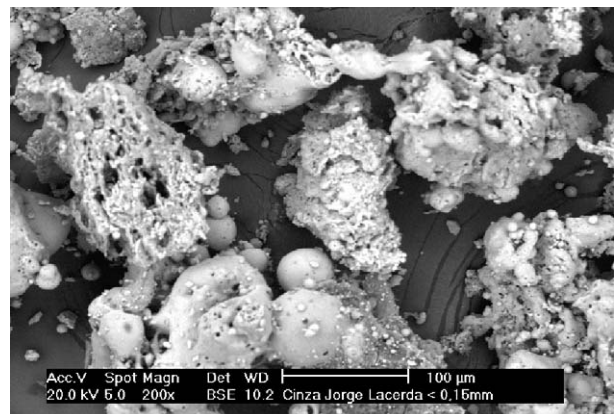


Fig. 1. SEM micrograph of bottom ash.

4. Experimental

In the present study, GS came from a Brazilian industry, in Palhoça, a city in the state of Santa Catarina. Bottom ash (generated in Santa Catarina) and sulfoaluminate cement (CSA) were the materials used in the treatment of galvanic sludge. Two formulations of CSA were used, with different quantities of clinker and phosphogypsum:

- CSA2080: 20% phosphogypsum + 80% CSA clinker,
- CSA3070: 30% phosphogypsum + 70% CSA clinker.

Table 1 shows the chemical composition of dried GS, BA and the components of sulfoaluminate cement (sulfoaluminate clinker + phosphogypsum), obtained by ICP, ICP-AES for major elements and ICP-MS for minor elements.

The water content of GS was 80%, its pH reached 9.8 and its raw density was 1.15 g/cm³. GS was dried for chemical and mineralogical analyses. The main elements present in dry galvanic sludge were silica (SiO₂), lime (CaO) and alumina (Al₂O₃). Among contaminants, the presence of chromium was significant and approximately 9%. Copper was the second main element found in GS: 3%. Nickel content was close to 1%. Other heavy metals (As, Pb, Zn) were present in smaller proportions: <120 ppm.

Figs. 2 and 3 show the X-ray diffractograms of GS, BA and sulfoaluminate cement, respectively, obtained by Rigaku Equipment, Model Mini Flex, with Cu K α radiation ($\lambda = 1.5406$). The X-ray diffraction (XRD) of galvanic sludge only showed the presence of quartz-SiO₂ (JCPDS 46-1045 card), and calcite CaCO₃ (JCPDS 05-0586card). The last one justifies the significant loss on ignition (35.4%), observed in chemical analysis. The diffractogram of BA identified quartz and mullite 2SiO₂·3Al₂O₃ (JCPDS 84-1205 card) as main crystalline phases.

Gypsum (JCPDS 03-0053 card) was the main component of phosphogypsum: 96.5%. For sulfoaluminate clinker, yeelimite C₄A₃S̄ (JCPDS 33-0256 card) was identified as the main phase, with the main ray at $2\theta = 23.6^\circ$. Phases like belite C₂S

Table 1
Chemical composition of raw materials

Elements	Dry GS	BA	Sulfoaluminate cement	
			Phosphogypsum	Clinker
Oxides (%)				
Al ₂ O ₃	5.7	26.7	0.2	31.2
CaO	20.9	0.8	32.5	43.2
SiO ₂	16.7	56.0	0.2	7.7
Fe ₂ O ₃	1.6	5.8	–	8.3
MgO	1.6	0.6	–	0.6
Na ₂ O	0.4	0.3	0.2	0.1
K ₂ O	0.6	2.6	–	0.2
P ₂ O ₅	0.7	0.2	0.2	–
TiO ₂	0.3	1.3	–	1.0
SO ₃	1.5	0.2	44.9	6.8
LOI	35.4	4.6	20.2	0.6
CO ₂ (total)	33.9	14.0	–	–
C (organic)	1.9	3.8	–	–
Minor (ppm)				
As	1.7	11	–	42.9
Ba	205	469	836	123
Cd	1.0	1.0	0.8	0.4
Cr	85460	176	7	250
Cu	29570	31	6	21
Ni	9571	43	6	39
Pb	112	70	3	8
Sr	549	124	12090	2084
Zn	110	358	15	54

(JCPDS 49-1673 card), mayenite C₁₂A₇ (JCPDS 09-413 card) and perovskite C₃FT (JCPDS 82-0191 card) were also identified. Through XDR analysis, sulfoaluminate clinker presented the mineralogical composition shown in Table 2.

The particle-size distribution of the sulfoaluminate cement components (clinker and phosphogypsum), obtained by Laser Grain Size Measurement is presented in Fig. 4a. BA was dried and separated into fractions smaller than 2 mm and the particle size distribution determined by sieving is presented in Fig. 4b.

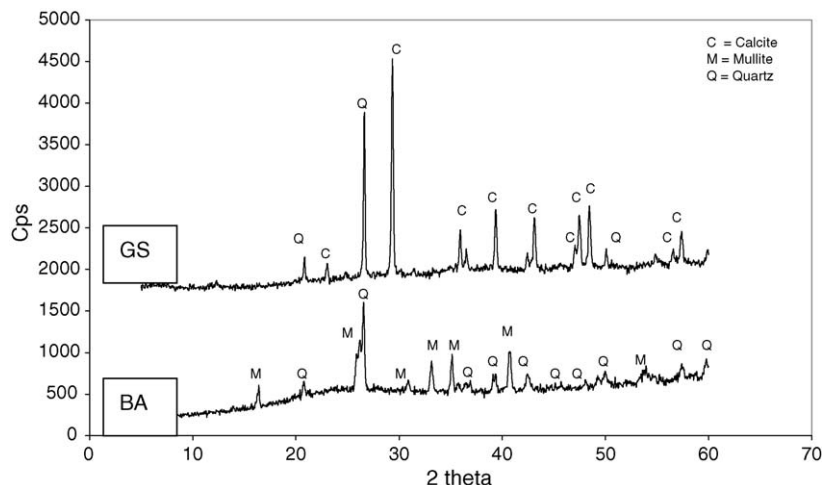


Fig. 2. X-Ray diffractograms of GS and BA.

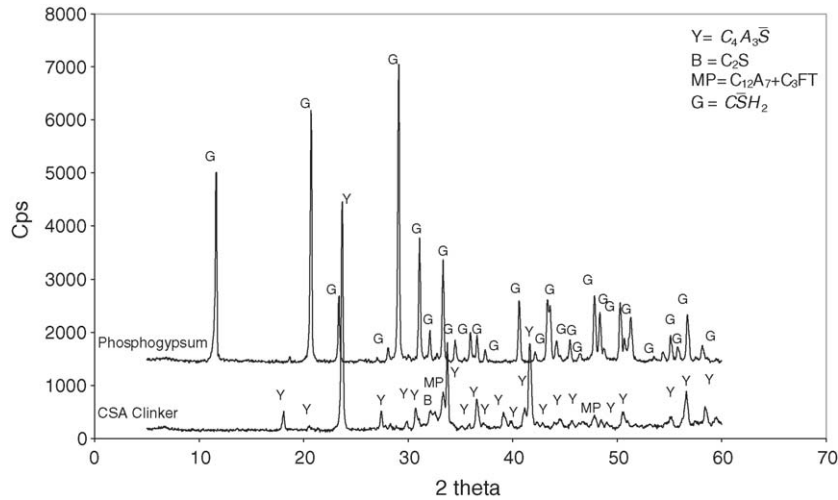


Fig. 3. X-Ray diffractograms of CSA clinker and phosphogypsum.

Table 2
Mineralogy of sulfoaluminate clinker

Phase	%
Belite	15.6
Yeelimite	66.4
Mayenite	7.1
Perovskite	9.9

The solidification of GS using CSA and BA was elaborated according to the following ratios (in mass):

$$\frac{GS}{BA} = 1$$

$$\frac{CSA}{GS(1 - h)} = 3$$

$$\frac{Water}{Cement} = 0.5$$

The GS:BA ratio (1:1) was determined by preliminary tests. Small amount of bottom ash was gradually added to galvanic sludge until it absorbed all the water contained in the sludge. The amount of water added (*w*) was sufficient to obtain good workability; the CSA/(GS(1 - *h*)) ratio considering the water contained in sludge (*h* = 80%) was fixed at 3.

Thus, the solidified matrix (MS) was composed of 38.5% GS + 38.5% BA + 23% CSA, in other words, contained 77% of waste (GS + BA).

The mixtures were prepared according to the following steps:

- BA and GS were mixed until the mixture got homogeneous and left aside for 24 h at 25 °C and relative humidity of 50%.
- After that, sulfoaluminate cement was mixed with water up to get homogeneity, adding afterwards the (GS + BA) mixture prepared the day before. The final mixture (GS + BA + CSA + water) was then mixed up to get a homogeneous material.
- The final mixtures were cast in prismatic molds (40 mm × 40 mm × 160 mm).

After 24 h, the samples were sealed in plastic bags and maintained in climate chamber (22 °C and 50% RH) until the strength tests.

The engineering properties assessed were setting time (Vicat test) [25] and compressive strength at 1, 3, 7, 28 and 90 days of age [26].

The influence of galvanic sludge and bottom ash on the hydration process of sulfoaluminate cement was also investigated by means of X-ray diffraction (XRD), thermal analysis (DTA) and scanning electron microscopy (SEM). Samples of solid matrix (MS) and reference pastes (pure CSA) prepared at *w/c* = 0.4

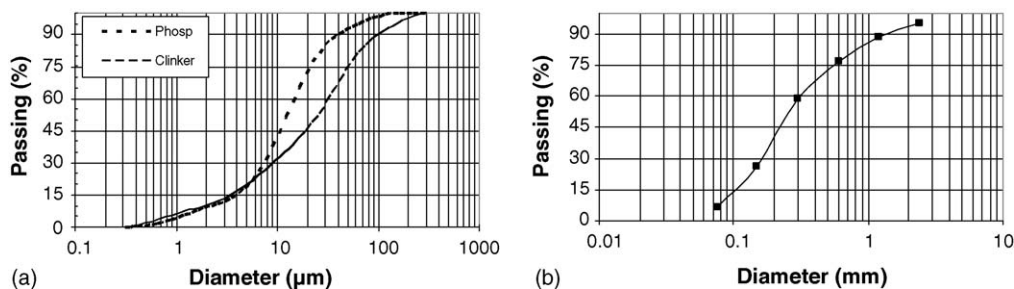


Fig. 4. Particle size distribution: (a) phosphogypsum and clinker; (b) bottom ash.

Table 3
Setting time of MS and reference samples

Type	Initial (h:min)	Final (h:min)
CSA2080_Ref	0:25	0:50
MS 2080	2:10	3:30
CSA3070_Ref	0:25	1:00
MS 3070	2:30	3:30

were cast in cylindrical molds ($\phi = 20$ mm and $h = 40$ mm) maintained in climate chamber (22°C and 50% RH) until the age of testing. Afterwards, they were demolded and dried at 60°C until they reached constant weight. The dried samples were crushed into pieces smaller than $150\ \mu\text{m}$. Unbroken samples were analysed by scanning electron microscopy (SEM), equipped with microprobe.

5. Results and discussion

5.1. Setting time and compressive strength

Table 3 shows the setting time presented by the solid matrix (MS) and plain pastes of sulfoaluminate cement (CSA). The presence of BA and GS extended the initial and final setting times by about 2 h for both formulations of CSA (2080 and 3070), though the resulted times characterized short periods, lower than 4 h.

In relation to the strength of the solid matrix (Fig. 5), it was verified that the CSA3070 formulation led to higher strength. It was also noticed that from 7 days, the strength progressed in parallel, with a difference of 2 MPa. Both matrixes presented a 28 day-strength higher than 4 MPa which is the value required for ceramic bricks with structural function (NBR 7170). The maximum standard deviation obtained was 0.6 MPa, at 28 days, for the solid matrix using the CSA3070 formulation. The shrinkage and water absorption capacity of the S/S matrix were not assessed in this preliminary study mainly devoted to the performance of CSA cement in the S/S process.

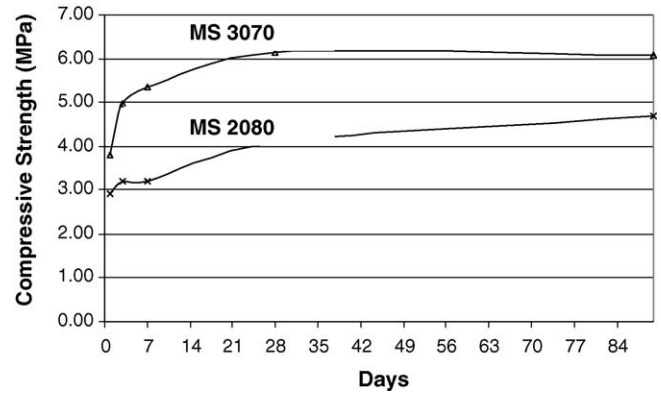


Fig. 5. Compressive strength of MS2080 and MS3070 samples.

5.2. Products of hydration

At 90 days, the presence of yeelimite (main peak at $2\theta = 23.6^\circ$) and gypsum (main peak at $2\theta = 29^\circ$) was only observed in the diffractograms of reference pastes (Figs. 6 and 7). For the solid matrix, such peaks practically did not exist (Fig. 8), which means that both of them were totally consumed. Such behavior was observed in both formulations of CSA (Fig. 8a and b).

The formation of ettringite with a main peak at $2\theta = 9^\circ$ (JCPDS 41-1451 card), was observed in reference pastes and solid matrix, with slightly smaller intensity in the last one. Such difference was more evident in the CSA 2080 formulation (Fig. 6).

The components of GS, calcite and quartz with main peaks at $2\theta = 29.3^\circ$ and $2\theta = 26.6^\circ$, respectively, were observed in the diffractograms of the solid matrix (MS). The intensity of the peak of quartz was increased by the presence of BA. Due to the presence of BA, mullite was also identified ($2\theta = 29.2^\circ$).

The formation of ettringite in reference pastes and solid matrix was also identified in thermograms (Fig. 9), starting after 2 h of hydration. In Fig. 9a and b, relative to reference pastes of CSA 2080 and CSA 3070, respectively, the peaks of ettringite

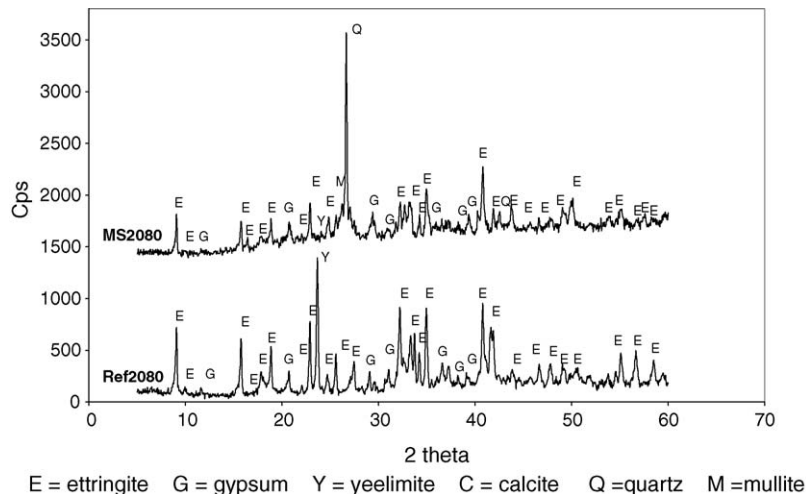


Fig. 6. X-Ray diffractograms of MS2080 and CSA2080 reference samples at 90 days.

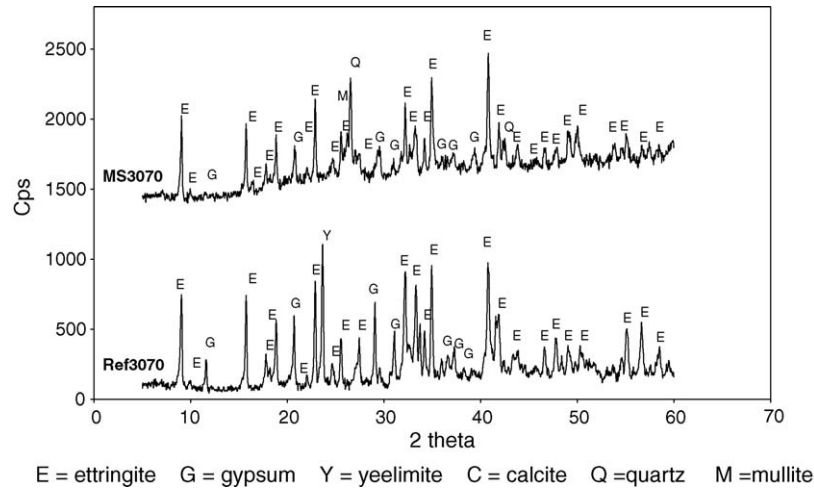


Fig. 7. X-Ray diffractograms of MS3070 and CSA3070 reference samples at 90 days.

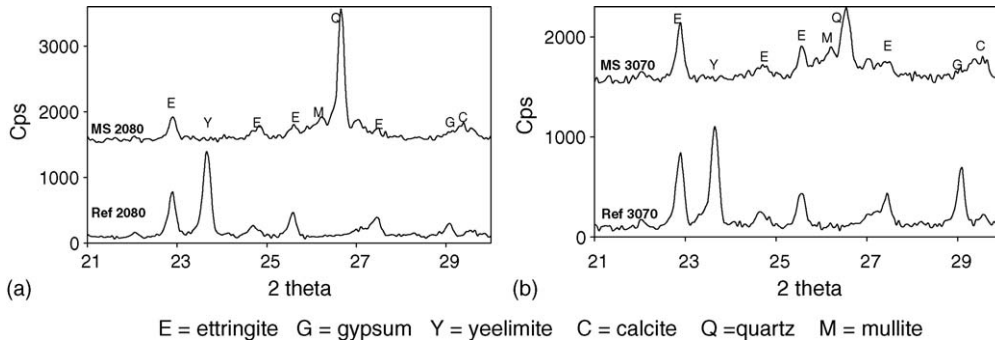


Fig. 8. X-Ray diffractograms of MS and reference samples ($2\theta = 21.0\text{--}30^\circ$): (a) CSA 2080; (b) CSA 3070.

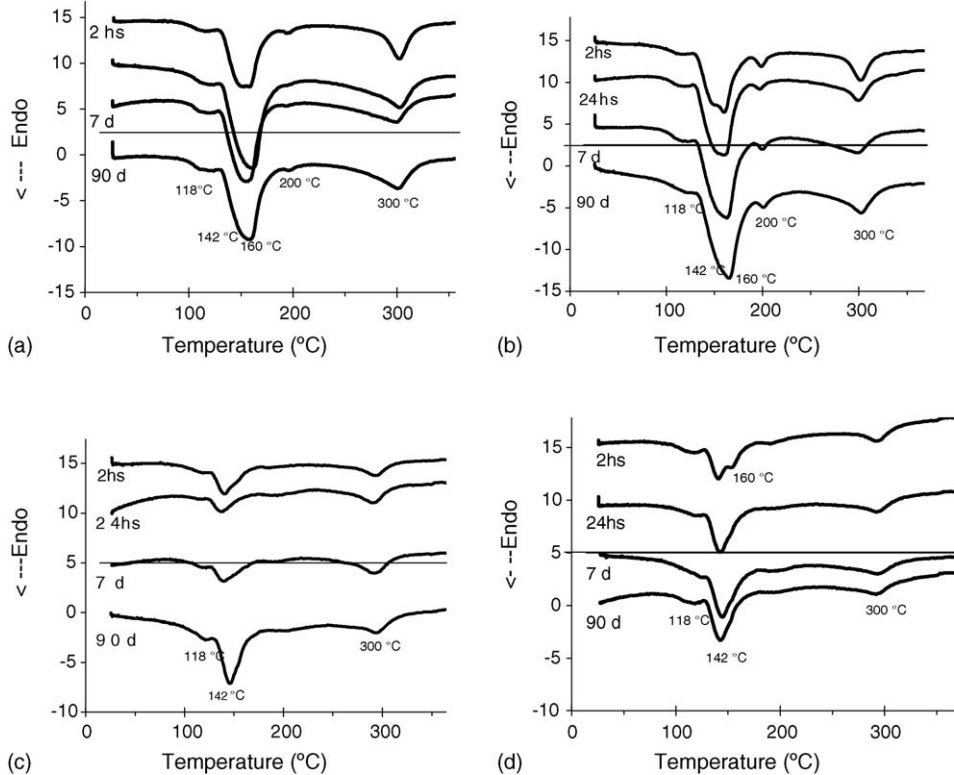


Fig. 9. Thermal analyses (DTA) of MS and reference samples: (a) Ref2080; (b) Ref3070; (c) MS 2080; (d) MS 3070.

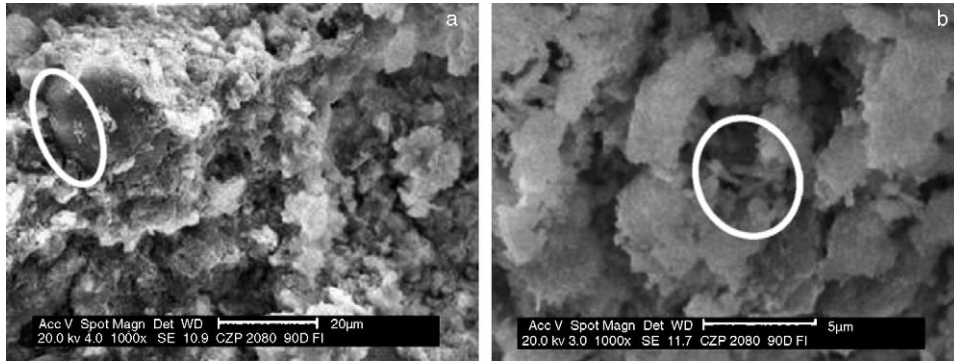


Fig. 10. SEM micrograph of MS2080 at 90 days: (a) particle of bottom ash; (b) ettringite needle.

and gypsum 160 °C overlapped due to the close temperature at which they both occur.

In CSA3070 formulation (Fig. 9b), after 2 h, it was observed that the peak of ettringite was lower than that of gypsum. This was marked by the increase of the peak at 142 °C and the reduction of the peak at 160 °C, causing the overlapping after 24 h of hydration. The second peak of gypsum (less intensive) occurred at 200 °C, and was present in both reference pastes with variable intensity.

The peaks at 118 °C and 300 °C refer to gibbsite (AH₃), in lower and higher levels of crystallization. The first peak is due

the hydration of mayenite (Eq. (4)) and the second one to the hydration of yeelimite in presence of gypsum (Eq. (2)).



Considering that yeelimite (C₄A₃S̄) and gypsum (C̄SH₂) are the main phases of sulfoaluminate cement and respecting the stoichiometry of the reactions, higher amounts of AH₃γ (300 °C) than AH₃ (118 °C) are expected, as observed in thermograms.

The presence of GS and BA resulted in considerable differences in thermograms (Fig. 9c and d). The intensities of the peaks of hydrated phases (ettringite and gibbsite, AH₃) were

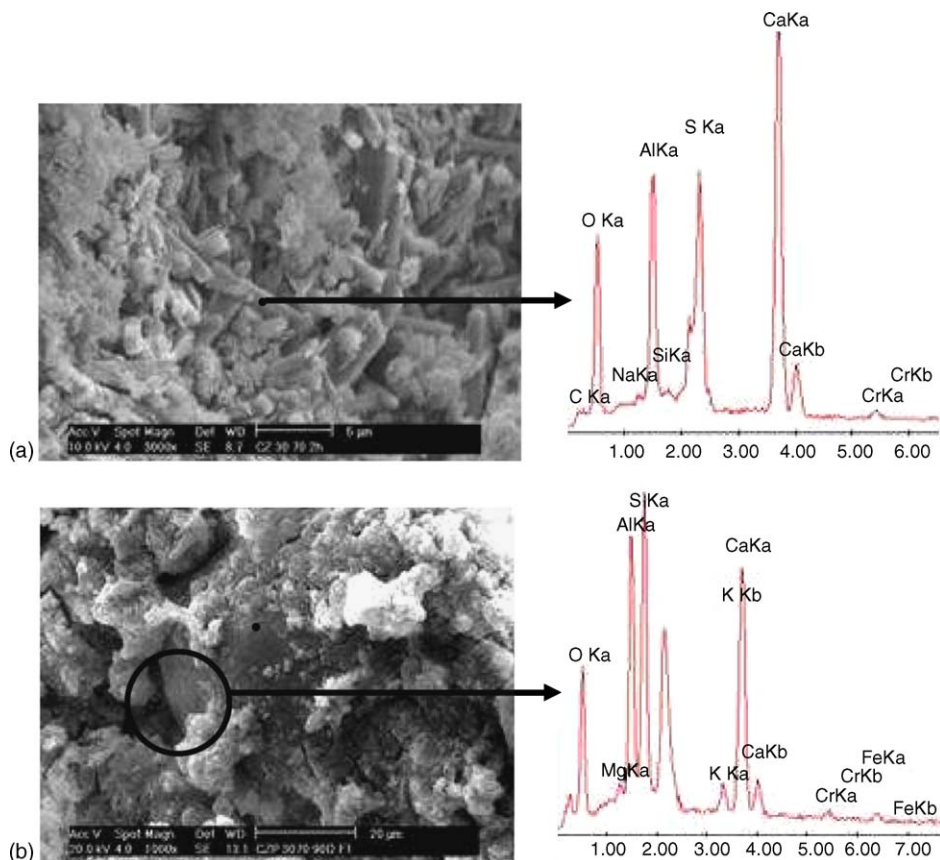


Fig. 11. SEM micrograph of MS3070: (a) ettringite needle—2 h of hydration; (b) particle of bottom ash—90 days of hydration.

less important for the solid matrix than for the reference pastes and peaks of gypsum disappeared. Gypsum was only present at early age (2 h) and for the formulation rich in calcium sulfate (CSA3070): peak at 160 °C as shown in Fig. 9d, indicating unavailability for the latter formation of ettringite.

The fact that the endothermic peak of ettringite was present in a punctual form is an important fact to highlight for the solid matrix, since an overlap of the peaks of ettringite and gypsum did not happen because the last one was totally consumed.

Fig. 10 shows the morphology of MS obtained with CSA2080 formulation, at 90 days of age. It is possible to verify the porosity of the system in both pictures. In Fig. 10a, it is also possible to see the empty space between the bottom ash grain and the cementitious system. In Fig. 10b the presence of ettringite covered by other hydrates is distinguished.

In Fig. 11a, the formation of ettringite in the solid matrix of CSA3070 at 2 h of hydration is easily identified. The EDAX analysis revealed the presence of Si, Na and Cr in ettringite. In Fig. 11b, at 90 days of age, the empty space around the grain of bottom ash is again identified. EDAX analysis punctually made over the bottom ash particle identified, among the other main components, the presence of Cr. The presence of Cr in the particle of bottom ash and ettringite shows the incorporation of chromium within the solid matrix.

5.3. Leaching tests

Leaching tests were carried out according to the Brazilian standard NBR 10005 on crushed pastes (<9.5 mm) at 28 days. MS 3070 pastes were leached in de-ionized water at a water to solids ratio of 16 for 24 h. Leachate was analyzed by ICP-MS to determine the heavy metals content. The value obtained for chromium was 0.55 ppm which is lower than the limit of NBR 10004:5 ppm. The content of all other heavy metals (Cu, Pb, Ni, Zn) was lower than 0.1 ppm.

6. Conclusions

From this limited investigation, the following conclusions can be drawn:

1. The setting time, usually extended in S/S processes is relatively short when CSA is used.
2. The values of the strength exhibited by the solid matrix, 4 MPa and 6 MPa, for CSA2080 and CSA3070 formulations were higher than the minimum value required by the Brazilian standard (NBR 7170) for structural ceramic bricks. Considering that the matrix is constituted by 77% of waste (LDG + CZP), the present performance is very interesting. If we consider the presence of phosphogypsum in sulfoaluminate cement, the amount of waste increased from 77% to 81.3 and 83.9% for CSA2080 and CSA3070 formulations, respectively, characterizing a very low consumption of CSA clinker.
3. The presence of Cr, identified by the EDAX in the needles of ettringite and over the particles of bottom ash, shows that both are able to incorporate such pollutant.

4. Leaching tests carried out on crushed samples show good retention of chromium.

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