Cement and Concrete Research 40 (2010) 1149–1156

Contents lists available at ScienceDirect

Cement and Concrete Research

journal homepage: http://ees.elsevier.com/CEMCON/default.asp

Leaching behaviour of mixtures containing plaster of Paris and calcium sulphoaluminate clinker

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ARTICLE INFO

Article history:
Received 12 December 2007
Accepted 17 March 2010

Keywords:
Calcium sulfoaluminate cement (D)
Ettringite (D)
Microstructure (B)
Degradation (C)
Plaster

ABSTRACT

The water resistance of plaster of Paris is limited due to the high solubility of calcium sulphate hemihydrate. In order to improve this resistance, calcium sulphoaluminate clinker was added to plaster at different contents: 30, 50, and 70%. Mortars were cast using these new binders and subjected to the Soxhlet extraction test. The presence of calcium sulphoaluminate clinker considerably reduced the quantity of sulphate extracted per day and after 5 days: from 78.9% to 89.1%. The analysis of microstructure showed the stability of ettringite during the leaching test and the refinement of the porosity.

1. Introduction

Plaster of Paris is obtained by the dehydration of calcium sulphate dihydrate. It is mainly composed of β calcium sulphate hemihydrate (60 to 80%) and anhydrite. It is used in non-structural applications (claddings, renders, and insulating boards). It presents low density and high porosity leading to good insulating performances [1]. But its water resistance is low due to its high solubility (8.9 to 9.5 $CaSO_4$ g/L) [2,3]. Moreover, the pH of the pore solution is about 7, which does not allow the use of plaster in steel-reinforced structures [4–6].

Different solutions exist to enhance the water resistance of plaster: introduction of water repellents or addition of hydraulic binders like Portland cement [7–10]. Reduced quantities of calcium sulphoaluminate clinker (20 to 50%) are efficient to get water-resistant calcium sulphates [11–13]. In the present study, calcium sulphoaluminate clinker was added to plaster to improve its water resistance. Three calcium sulphoaluminate cement to plaster ratios were studied: 30/70, 50/50, and 70/30. The leaching behaviour of mixtures containing plaster of Paris and calcium sulphoaluminate clinker and the microstructure was investigated.

2. Experimental

2.1. Materials

The plaster investigated here was mainly composed of β calcium sulphate hemihydrate: 82.3%. The mass loss observed in the thermogravimetric analysis was 5.1%, instead of 6.2% for pure β calcium sulphate hemihydrate. The chemical analysis and other properties of plaster are shown in Table 1.

The characteristics of calcium sulphoaluminate clinker (CK) are shown in Table 2. Its mineralogy was assessed by X-ray diffraction (Table 3). The amount of yeelimite, also called “Klein's compound”, was very high: 73.5%. This phase and mayenite are responsible for the development of early age strength.

When a mixture of plaster and calcium sulphoaluminate clinker is hydrated, the following hydrates precipitate: ettringite ($3CaO\cdot Al_2O_3\cdot 3CaSO_4\cdot 32H_2O$) and gibbsite [$Al(OH)_3$]. As shown in Table 4, these hydrates are little or even not soluble. Ettringite is stable in a wide range of sulphate concentration and pH [14–17].

2.2. Experimental procedures

2.2.1. Leaching test — Soxhlet test

The Soxhlet extractor is shown in Fig. 1 [18,19]. The solvent was de-ionized water heated at 100 °C. The solid material was a prismatic sample of mortar ($20\times 20\times 80$ mm^3) containing one of the different binders (Bi) and standard sand (S), in the following proportions: Bi/S = 1/3. The composition of the different binders is shown in Table 5. The water to solid ratio (W/Bi) of mortars was 0.85 in order to avoid any settlement and get normal consistency according to French standard NFP 15-402, normally referring to cement. This W/B ratio was higher than that of standard mortar (W/B = 0.5) due to the presence of plaster which needed more water for its hydration [1].

24 h after casting, the samples of mortar ($20\times 20\times 160$ mm^3) were removed from the moulds and placed in a curing chamber (20 °C; RH = 50%) for 2 weeks until constant weight. Then, they were cut in

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doi:10.1016/j.cemconres.2010.03.013

Table 1
Properties of plaster of Paris.

Chemical analysis								
Oxides	SiO_2	Al_2O_3	CaO	TiO_2	MgO	P_2O_5	SO_3	LOI
%	1.5	0.2	38.5	0.01	0.8	n.d.	49.6	9.3

Physical properties	
Average diameter of the particle size distribution (μm)	52
BET surface area (m^2/g)	4.6
Blaine specific surface area (m^2/kg)	476
Chemical properties	
Initial electrical conductivity ($mS/cm^2/cm$)	6
pH	7.7
Soluble phosphates (mg/L)	<0.1
Soluble sulphates (mg/L)	1528
Solubility in water (g/L), expressed as anhydrous $CaSO_4$ [1–3]	
20 °C	8.9
100 °C	1.72–1.8

Table 2
Properties of calcium sulfoaluminate clinker.

Chemical analysis								
Oxides	SiO_2	Al_2O_3	CaO	TiO_2	MgO	P_2O_5	SO_3	LOI
%	4.7	37.4	39.2	1.6	1.7	0.2	8.8	0.9

Physical properties	
Average diameter of the particle size distribution (μm)	16
BET surface area (m^2/g)	1.05
Blaine specific surface area (m^2/kg)	450

two equal parts to get the length (80 mm) fitting with the size of the thimble. Another series of tests occurred 28 days after casting.

When the Soxhlet extraction started, the sample was placed in the thimble after being weighed. 800 g of de-ionized water was poured in the bottom flask to be heated. Water was heated at 100 °C, evaporated and condensed in the cooling column before flowing in the central part containing the thimble. While filling the extractor, water was in contact with the mortar sample and was enriched in calcium sulphate. Once the column was entirely filled, it started to empty by priming a siphon. Then, the cycle started again. Thus, the sample was leached at a temperature of 90–100 °C by de-ionized water regularly renewed (about each half-hour), and 19 extractions occurred within a day.

The length of the test was 5 days. Every day, the leachate was only analyzed for its sulphate concentration by ionic chromatography; the flask was rinsed and 800 g of renewed de-ionized water was poured in the flask. After 5 days of leaching, the sample was removed from the thimble and dried at 20 °C and 50% RH until constant weight for one week. The mass loss due to leaching was therefore measured.

2.2.2. Compressive strength of mortars cured in water at 20 °C

As the solubility of plaster is lower at 100 °C (1.2–1.3 g/L SO_4^{2-}) than at 20 °C (6.3–6.7 g/L SO_4^{2-}), standard mortars were cast and cured in water at 20 °C. Their compressive strength was measured at different times, up to one year. Some specimens were stored in air at 20 °C and 50% RH and their strength was measured after 28 days and one year.

Table 3
Mineralogy of calcium sulfoaluminate clinker (w_t %).

Phase	Chemistry	%
Yeelimite	$Ca_4Al_6O_{12}SO_4$	73.5
Larnite	β-Ca_2SiO_4	16.1
Perovskite	$Ca_3Fe_2TiO_9$	6.9
Mayenite	$Ca_{12}Al_{14}O_{33}$	0.6
Periclase	MgO	1.7
Anhydrite	$CaSO_4$	1.0
Quartz	SiO_2	0.2

Table 4
Solubility products of different phases [14–17].

Phase	K_{sp} at 25 °C	K_{sp} at 50 °C	K_{sp} at 85 °C
Ettringite	$2.8 \cdot 10^{-45}$	$2.24 \cdot 10^{-43}$	$6.61 \cdot 10^{-41}$
Gibbsite	0.0399	0.0833	0.2249
Gypsum	$3.72 \cdot 10^{-5}$	$3.43 \cdot 10^{-5}$	$2.35 \cdot 10^{-5}$
Calcium hydroxide	$8.9 \cdot 10^{-6}$	$4.57 \cdot 10^{-6}$	$1.50 \cdot 10^{-6}$

2.2.3. Microstructure

Different techniques were used to study the influence of leaching on the microstructure of mortars: mercury intrusion porosimetry (MIP), X-ray diffraction (XRD), and scanning electron microscopy (SEM).

MIP was performed using Micromeritics Autopore III porosimeter under a maximum intrusion pressure of 400 MPa. Total porosity and pore size distribution were assessed. Pores were classified according to the distribution proposed by Brédy [20] and already used by Peysson et al. [21]:

- micropores: $\varphi < 0.1$ μm;

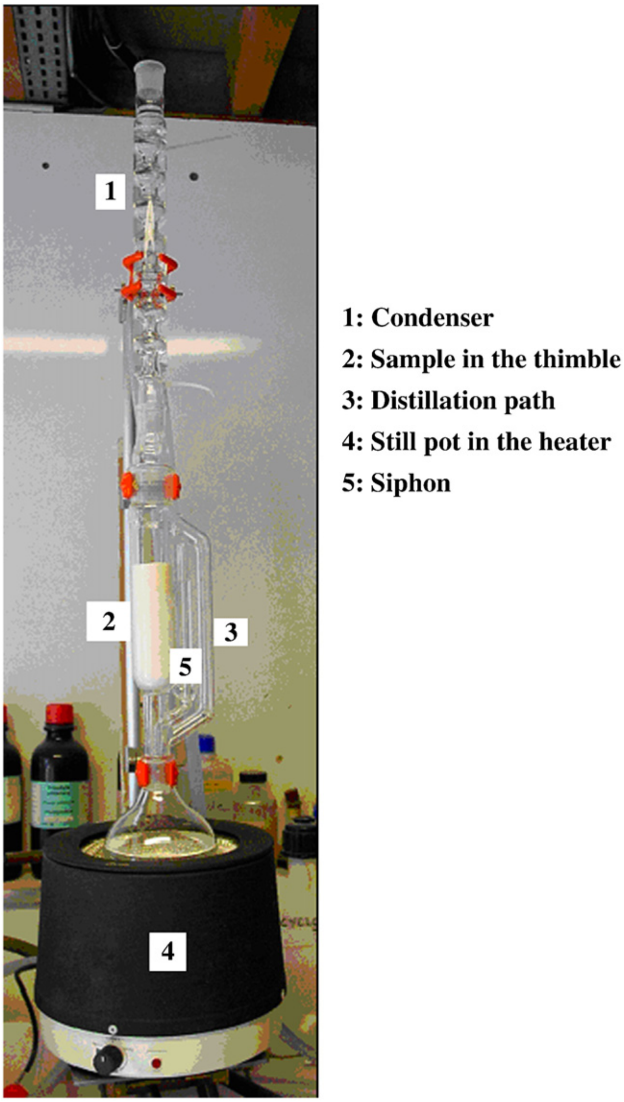


Fig. 1. Soxhlet extractor.

Table 5
Composition of binders Bi (w_t %).

Binder	Plaster	CK
B0	100	0
B1	70	30
B2	50	50
B3	30	70

- mesopores: $0.1<\varphi<0.6$ µm; and
- macropores: $\varphi>0.6$ µm.

XRD analyses were performed by a Siemens D500 apparatus operating between 5° and 70° 2θ, Cu Kα radiation, at a rate of 1°/min. The software used to exploit the results was DIFFRAC Plus Release 2001-EVA v7.

For SEM observations, a Philips XL30 ESEM-FEG instrument was used. Specimens were metallized with gold.

3. Results and discussion

3.1. Leaching behaviour of mortars

The sulphate concentration of the different leachates obtained on mortars is presented in Fig. 2, for samples prepared 24 h after casting. The higher concentration was found when mortar containing binder B0 (100% plaster) was subjected to the Soxhlet extraction: as shown in Fig. 2a, important quantities of sulphate were extracted every day (from 1110 mg/L to nearly 1800 mg/L). At the end of the test, more than 7800 mg/L of sulphate was extracted (Fig. 2b). The presence of calcium sulphoaluminate clinker considerably reduced the quantity of sulphate extracted per day and after 5 days (Fig. 2a, b). For mortars containing binders B1 to B3, the extraction kinetics was similar: higher quantities of sulphate were leached within the first two days. At the end of the test, the sulphate concentration of the solution was very low compared to mortar containing B0: 1660 mg/L for B1, 1250 mg/L for B2, and 860 mg/L for B2.

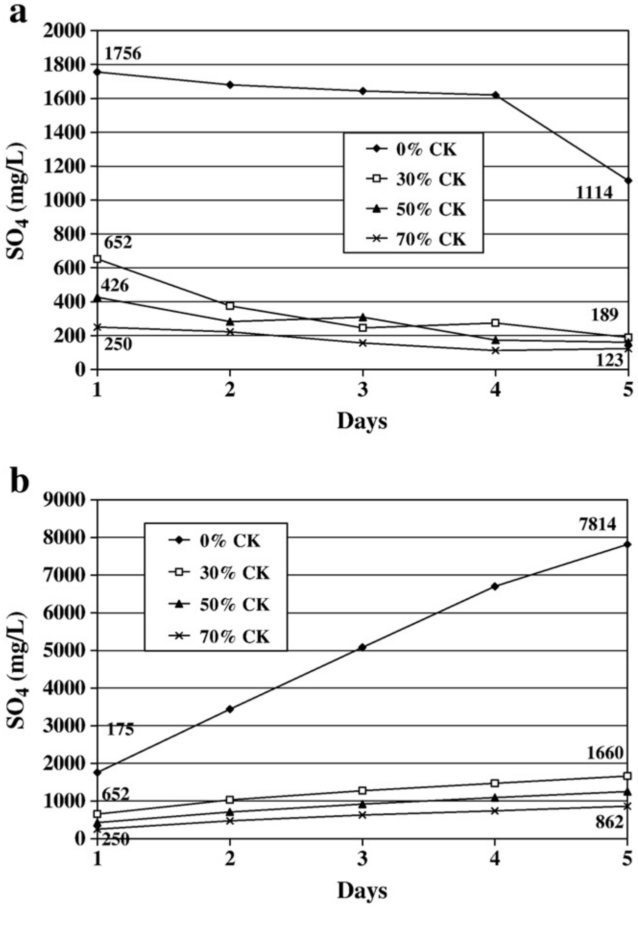


Fig. 2. Evolution of the sulphate ion concentration (samples removed at 24 h from the moulds). a — Non cumulative curves; b — cumulative curves.

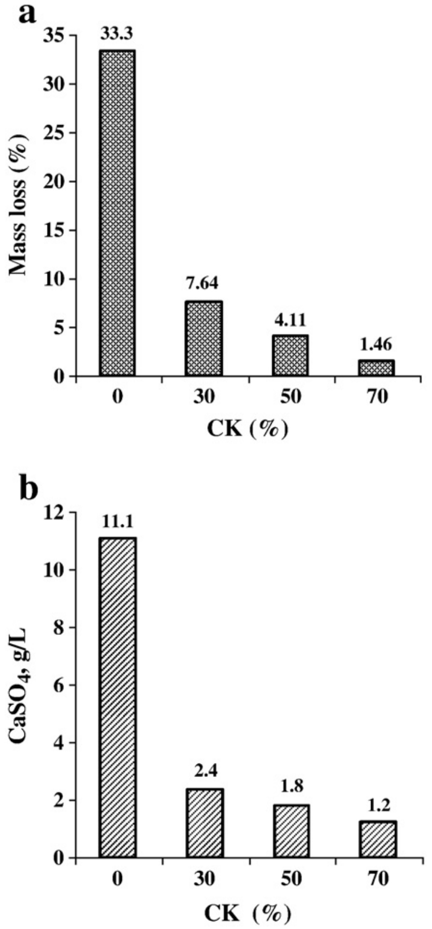


Fig. 3. Mass loss due to leaching (a) and sulphate ion concentration expressed as anhydrous sulphate (b).

Leaching of mortar containing B0 nearly destroyed the specimen: the mass loss observed was 33.3%, as shown in Fig. 3a. The mass loss reported here is relative to the whole mortar sample: sand + binder. When mortar containing B0 was subjected to the Soxhlet extraction test, grains of sand and binder were entrained outside the sample. The mass loss of mortar containing B1 (only 30% CK) was 4.4 times lower, and that of mortar containing B3 (70% CK) 22.8 times lower (Fig. 3a). Fig. 3b clearly shows that the addition of calcium sulphoaluminate clinker considerably reduced the solubility of plaster. For mortar containing B0, this solubility, expressed as anhydrous Ca SO$_4$ g/L, was 11.1 g/L. For mortar containing B1 it dropped to 2.4 g/L and 1.2 g/L for mortar containing B3.

The Soxhlet extraction test was repeated on samples removed from the moulds at 28 days (instead of 24 h) and dried at 20 °C for two weeks. The results are presented in Figs. 4 and 5. The same trends were found, as shown in Fig. 4. As the accuracy of ionic chromatography was 5%, we concluded that the same results were obtained (Fig. 5).

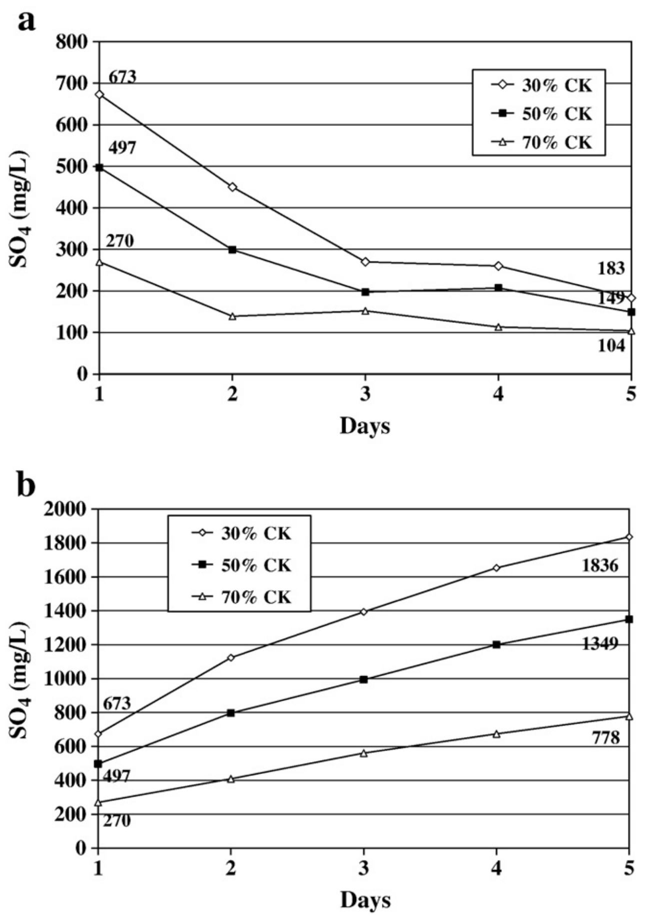


Fig. 4. Evolution of the sulphate ion concentration (samples removed at 24 days from the moulds). a — Non cumulative curves; b — cumulative curves.

3.2. Compressive of mortars at 20 °C

The compressive strength of mortars cured in water for one year is shown in Fig. 6. The strength of mortar containing B0 (0% CK) was very low, and could not be measured after 28 days of immersion in water. The specimen started to disintegrate. For other mortars, the strength did not decrease between 28 days and one year, which proved good water stability of binders containing calcium sulphoaluminate cement.

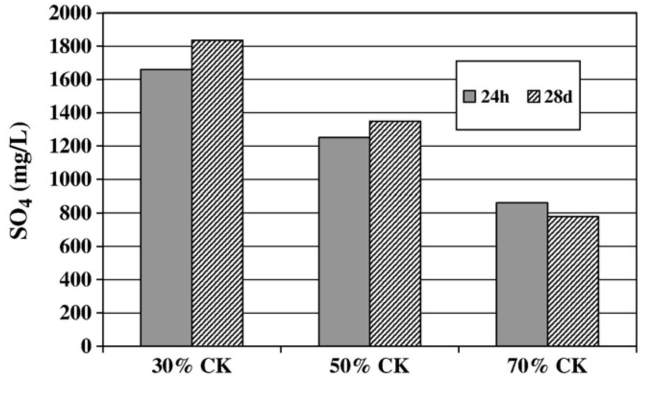


Fig. 5. Influence of the residence time in the moulds on the sulphate concentration of the leachate after 5 days of Soxhlet test.

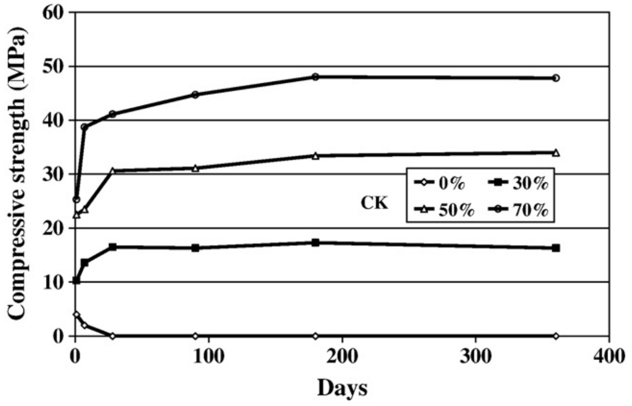


Fig. 6. Evolution of the compressive strength of mortars cured in water at 20 °C.

The strength of mortars cured either in air or in water is presented in Fig. 7. When the amount of CK was 30%, there was not any difference in strength due to curing conditions. In this case, yeelimite was quickly consumed to produce ettringite and gibbsite, according to the following equation:

$$\underset{610}{4CaO\cdot 3Al_2O_3\cdot SO_3} + 2[\underset{145}{CaSO_4\cdot 1/2H_2O}] + 37\underset{18}{H_2O} \qquad (1)$$

$$\Rightarrow \underset{1254}{3CaO\cdot Al_2O_3\cdot 3CaSO_4\cdot 32H_2O} + 4\underset{156}{Al(OH)_3},$$

in which each number under the compound formula indicates its molecular weight. To hydrate 100 g of this CK containing 73.5% yeelimite, in respect of the stoichiometry, it is necessary to use [(2 × 145/610) × 0.735] = 37.8 g of hemihydrate. Thus, the CK/hemihydrate ratio is 2.65, which is far from that present in the binder containing 30% CK: 30/70 = 0.43. Yeelimite was rapidly consumed and the hydration reaction stopped.

The results were quite different for CK = 50% or 70%. For 70%, the CK/hemihydrate was 70/30 = 2.33, which is close to that respecting the stoichiometry. Yeelimite was longer present in the mixture and the hydration continued, especially for specimens cured in water: Eq. (1) shows that 37 mol of water is needed to hydrate 1 mol of yeelimite. In air, evaporation occurred and the hydration reaction was stopped which led to lower strength. The same phenomenon appeared when CK reached 50%, but was less pronounced.

In any case, the strength did not decrease after water curing and we concluded that binders B1 to B3 behaved as hydraulic binders.

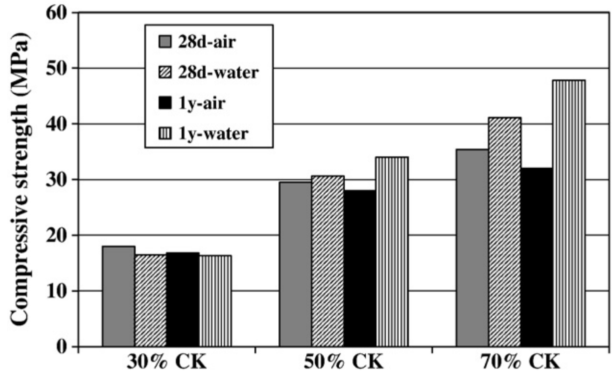


Fig. 7. Influence of the curing regime on the compressive strength of mortars.

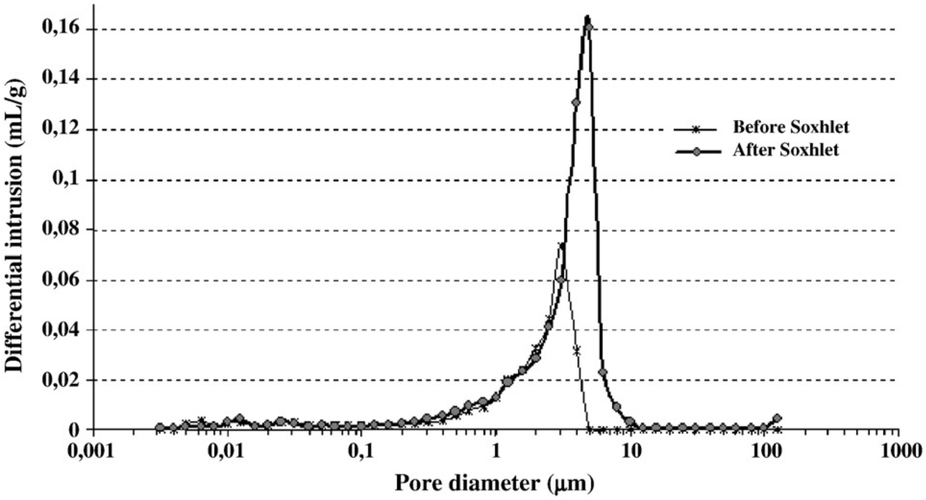


Fig. 8. Porous distribution of sample B0 before and after leaching.

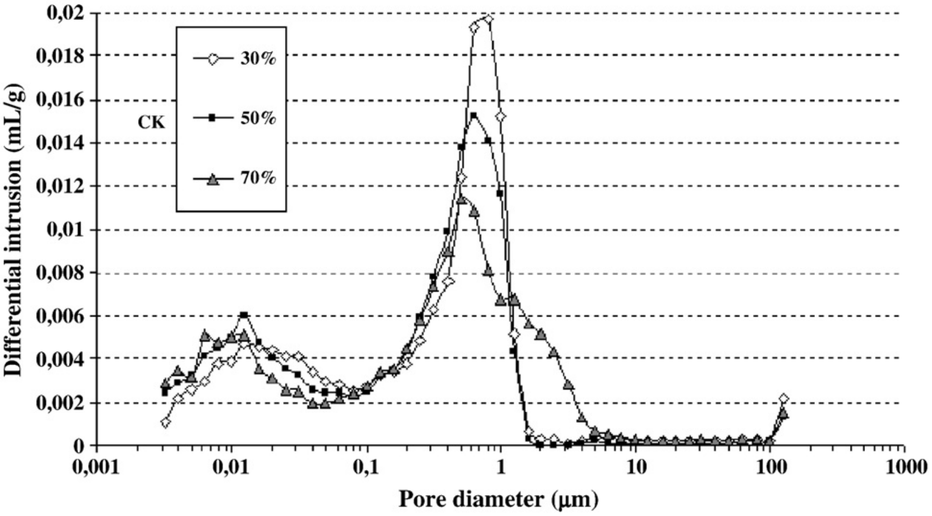


Fig. 9. Porous distribution of mortars containing calcium sulphoaluminate clinker before leaching.

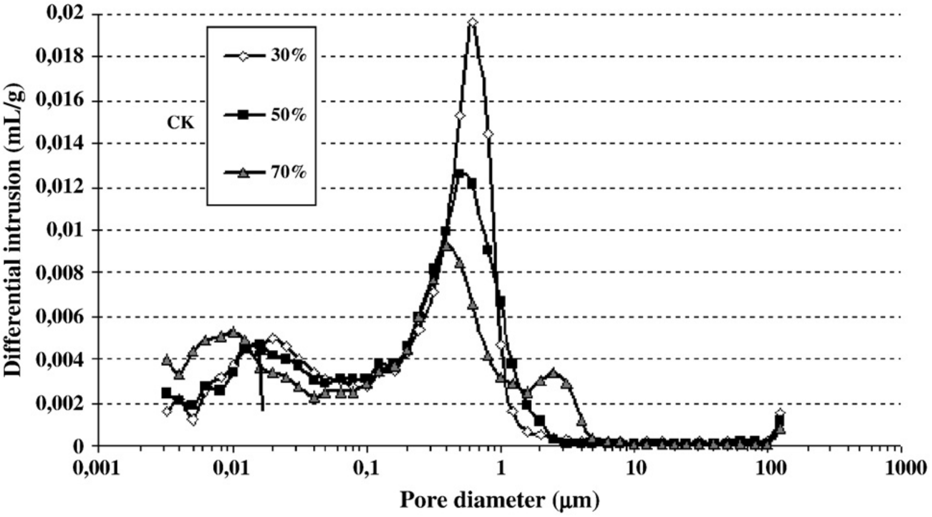


Fig. 10. Porous distribution of mortars containing calcium sulphoaluminate clinker after leaching.

Table 6
Results of mercury intrusion porosimetry.

Binder		Total porosity (%)	Average pore diameter (µm)	Macropores (%)	Mesopores (%)	Micropores (%)
B0	Before leaching	29.0	2.4	82.2	7.9	9.9
	After leaching	55.1	4.5	90.6	4.8	4.6
B1	Before leaching	29.0	0.5	39.9	27.4	32.7
	After leaching	26.8	0.4	30.5	37.6	31.9
B2	Before leaching	27.1	0.4	30	33.2	36.8
	After leaching	24.5	0.3	26.7	35	38.3
B3	Before leaching	26.9	0.4	36.1	30.8	33.1
	After leaching	23.6	0.2	23.8	34.7	41.5

3.3. Microstructure

3.3.1. Mercury porosimetry of mortars

The results are presented in Figs. 8–10, and Table 6. Fig. 8 shows that, for mortar containing B0, porous distribution was unimodal and macropores were mainly present before and after leaching: 82% and 90%, respectively. Leaching had fatal influence on mortar containing B0: total porosity increased as well the average diameter of the porous distribution and the quantity of macropores (Table 6). The capillarity of the material ($1\ \mu m < \varphi < 5\ \mu m$) was very important before leaching; the average pore diameter was 2.4 µm. This allowed an easy dissolution of the specimen and increased the average diameter of pores after leaching: 4.5 µm.

The influence of calcium sulphoaluminate clinker on the porosity of mortars is shown in Figs. 9 and 10, and Table 6:

- the pore distribution became bimodal,
- capillarity shifted to finer pores: $0.2\ \mu m < \phi < 1\ \mu m$,
- micropores appeared ($\phi < 0.1\ \mu m$), and
- the average diameter of pores was considerably reduced.

The effect of the addition of 30% calcium sulphoaluminate clinker (B1) was less marked than that of 50% and 70% addition (B2 and B3). Fig. 10 and Table 6 show that, after leaching, the values of total porosity, average pore diameter and macroporosity decreased, regardless of the quantity of calcium sulphoaluminate clinker added. These effects were more marked when the content of CK increased.

3.3.2. X-ray diffraction on pastes

Fig. 11 presents the results of XRD analyses and allows following the evolution of the intensity of some peaks (gypsum, yeelimite, and ettringite) in mixtures of plaster and CK before and after leaching. These values are the average values obtained considering each peak characteristic of the phase:

- gypsum: average value of peak intensities at $2\theta = 11.65$; 20.71; 29.11; 31.13 and 33.38°;
- yeelimite: average value of peak intensities at $2\theta = 23.75$; 33.8; and 41.3°; and
- ettringite: average value of peak intensities at $2\theta = 9.11$; 15.84; 23.35; and 40.87°.

We have not taken the main peak intensity of each phase as reference because the phases present in these materials are likely to exhibit a preferred orientation. Therefore, it seemed more accurate to take into account the average value between several peaks to describe the evolution of gypsum, yeelimite, and ettringite.

The effects of leaching on samples B1 to B3 can be summarized as follows:

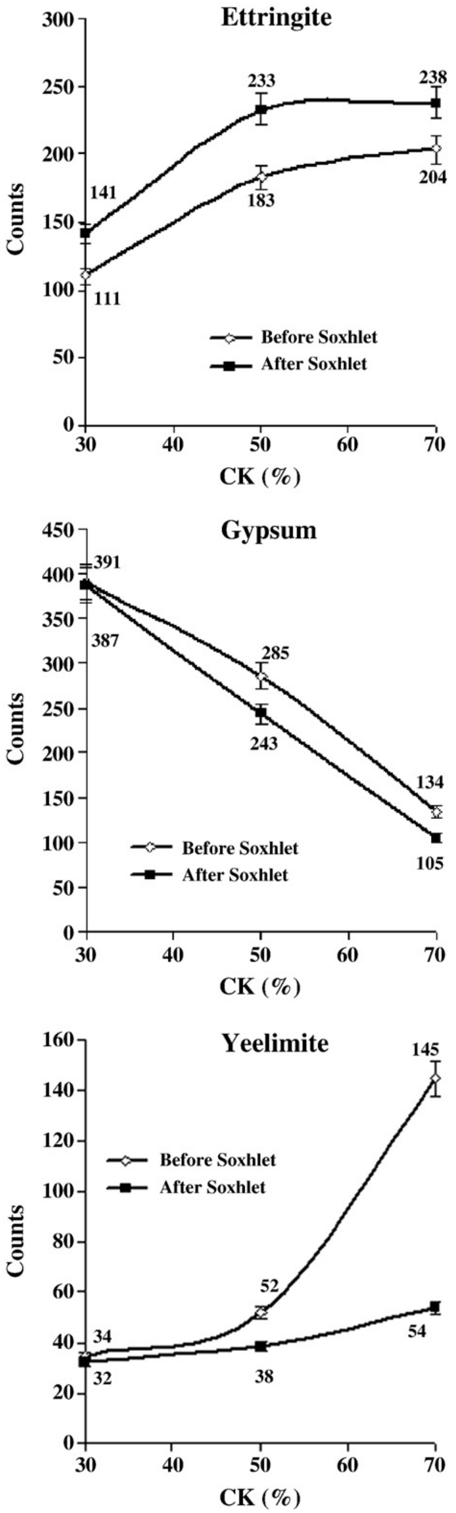
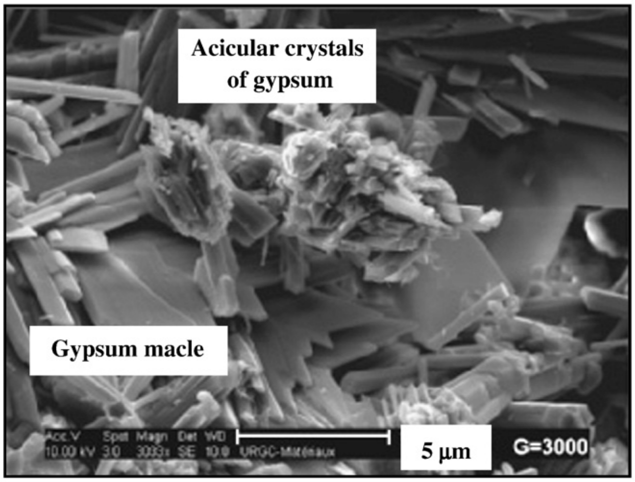


Fig. 11. Phases present in binders B1 to B3 before and after leaching.

- Gypsum was partially dissolved and therefore the intensity of the peaks decreased, especially when the amount of CK was 50 or 70%. In these cases, the difference in the average peak intensity was in the range of 17–23%, which is significant. When the amount of CK was 30%, there was no difference before and after leaching.
- The average value of ettringite peaks increased due either to new precipitation occurring during leaching or change in the degree of crystallinity. The difference between the values recorded before and after leaching was in the range of 15–25%. This was attributed to new precipitation of ettringite, and confirmed by the refinement of the porosity, as observed in Figs. 9 and 10.

a) before leaching



b) after leaching

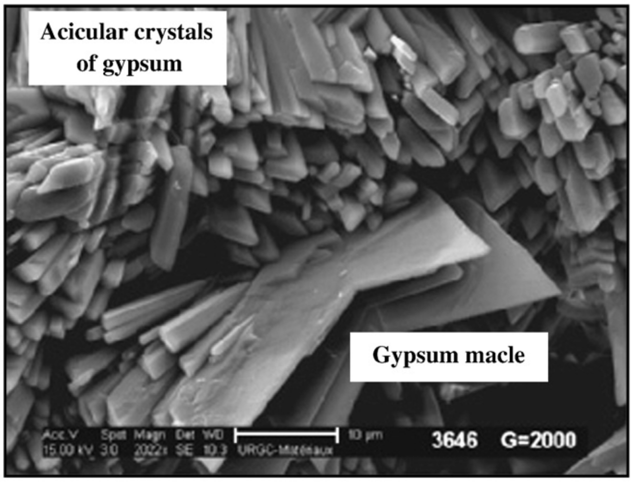


Fig. 12. SEM micrographs of B0 matrix.

– The average value of yeelimite decreased confirming new precipitation of ettringite during leaching, especially when the amount of CK was higher than 50%.

In sample B0, the average value of gypsum peaks was 1141 counts before leaching and reached 1255 counts after leaching, due to re-crystallization of the remaining gypsum after leaching.

3.3.3. Scanning electron microscopy (SEM) pastes

In sample B0 (Fig. 12), crystals occurring during the "dissolution–crystallization" process were generally acicular and their tangled network was responsible for the mechanical strength [2]. In these crystalline networks, two types of macle appeared: the penetration macle formed by the intertwining of crystals and called "swallowtail", and the contact macle [3]. A macle is defined as the association of several crystals of the same specy but differently oriented, with partial permeation. After leaching, the cross-section of acicular crystals became less distinct, while the thickness of macles increased and this type of crystallization dominated.

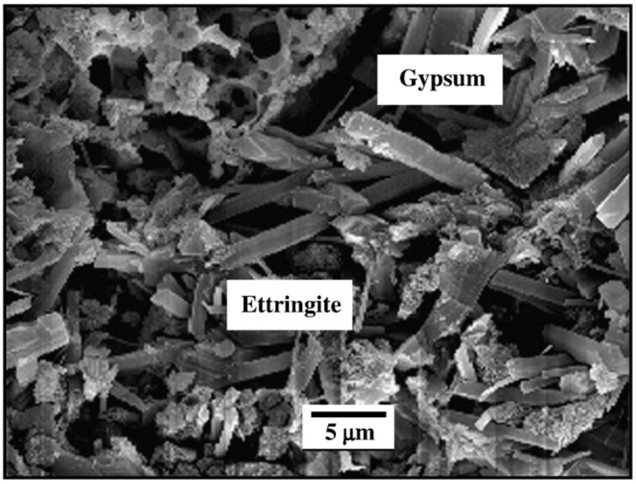
Before leaching, the addition of calcium sulphoaluminate clinker changed the morphology of crystals (Figs. 13 and 14). The presence of macles was detected in the mixture containing 30% CK (B1), while acicular crystals of gypsum were found in the mixture containing 50% CK (B2). Ettringite crystals presented different morphologies either within the matrix or in pores: thin elongated needles or short massive rods randomly distributed within the matrix. The gel-like gibbsite covered the surface of ettringite and gypsum. Some massive crystals of ettringite were observed in sample B2. In these mixtures, leaching slightly modified the microstructure (Figs. 13 and 14). The surface of gypsum crystals was less distinct; they became short and flat. Ettringite crystals remained intact, and were present as thin or massive rods. Massive crystals of ettringite were still observed in sample B2.

3.3.4. Results obtained by other scientists

Murat and Attari [22] added different clay minerals (montmorillonite, muscovite, sepiolite, attapulgite, talc, kaolinite and metakaolinite), to gypsum plaster powder before gauging. They modified some physical properties of the binder after hardening, e.g. compressive and flexural strengths, total open porosity and pore size distribution, but above all, coefficient of permeability and kinetics of water migration under variable charge. They did not perform any leaching test on their specimens.

Studies on the changes in compressive strength, split tensile strength, acid resistance, and percentage water absorption were reported for systems of plaster of Paris and plaster of Paris blends upon impregnation with monomer (MMA) followed by polymerization (*in situ*), as well as for systems of plaster of Paris-epoxy resins

a) before leaching



b) after leaching

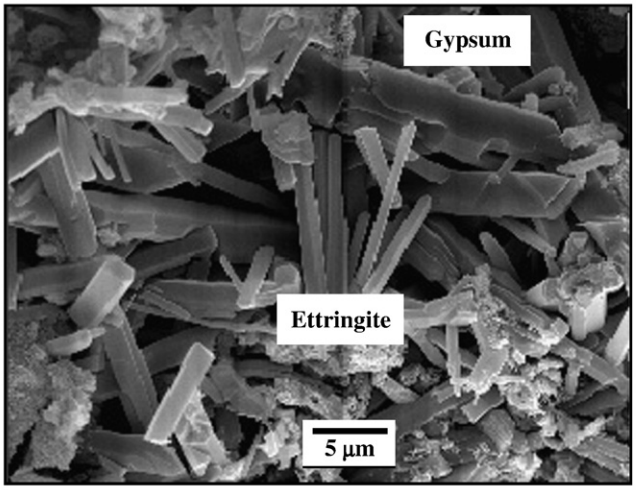
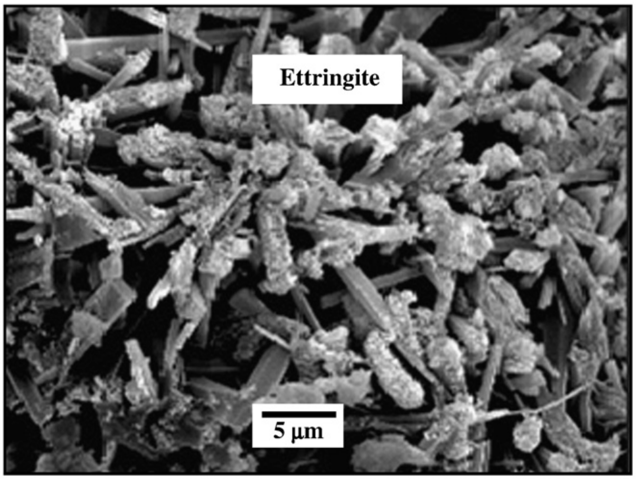


Fig. 13. SEM micrographs of B1 matrix.

a) before leaching



b) after leaching

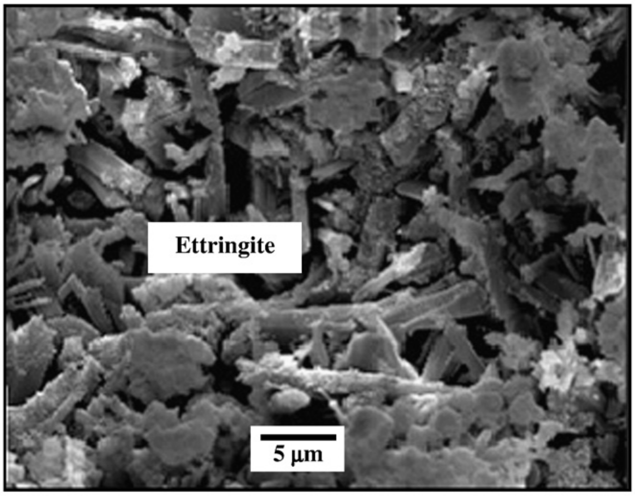


Fig. 14. SEM micrographs of B2 matrix.

with subsequent monomer impregnation [23]. In these studies, no leaching test was done.

Deng et al. [24] studied the properties of gypsum particleboard containing cement. Gypsum particleboard (GPB) has high thickness swelling (TS), high water absorption (WA), and low mechanical properties compared with cement-bonded particleboard. The properties of GPB were improved by adding cement. The experimental results showed that GPB with the added cement had good physical and mechanical properties compared with those of gypsum particleboard with no added cement. The TS and WA of gypsum particleboard with added cement were reduced by 10%. The mechanical properties of GPB, such as internal bond strength (IB), modulus of rupture (MOR), and modulus of elasticity (MOE), increased when the GPB was made with added cement. The properties of GPB improved relative to the quantity of cement added. With an increase of cement content from 5% to 10%, the TS and WA were reduced, and the IB, MOR, and MOE were increased. In contrast, the TS and WA increased and the IB, MOE, and MOR decreased when the cement content was increased from 15% to 30%. Thus the physical and mechanical properties of GPB were successfully improved when the added cement content was 10%.

Eires et al. [25] developed new composite materials based on gypsum and incorporating waste materials as granulated cork, a by-product of cork industry, and cellulose fibres, a waste of paper industry. The results obtained show that the water resistance improvement (−40%) can be achieved, above all, through the reduction of water content in paste, by the addiction of a mineral admixture, that acts also as a retarder, and by replacing the traditional casting procedure by pressure curing.

These researches show the originality of our approach: introduction of calcium sulphoaluminate cement and leaching test to prove the effectiveness of the proposed solution to improve water resistance of plaster.

4. Conclusions

The addition of calcium sulphoaluminate clinker considerably enhances the water resistance of plaster. The solubility of calcium sulphate drops from11.1 g/L to 1.2–2.4 g/L. Total porosity and average pore size diameter decrease in these mixtures. Ettringite and gibbsite precipitate and remain stable after leaching. The leaching performance increases with the amount of calcium sulphoaluminate added. The matrix becomes less porous after leaching: total porosity, average pore size diameter and quantity of macropores decrease, while proportions of meso and micropores increase.

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