



# 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.

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## 1. Introduction

Plaster of Paris is obtained by the dehydration of calcium sulphate dihydrate. It is mainly composed of  $\beta$  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<sub>4</sub> 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  $\beta$  calcium sulphate hemihydrate: 82.3%. The mass loss observed in the

thermogravimetric analysis was 5.1%, instead of 6.2% for pure  $\beta$  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·Al<sub>2</sub>O<sub>3</sub>·3CaSO<sub>4</sub>·32H<sub>2</sub>O) and gibbsite [Al(OH)<sub>3</sub>]. 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 (20×20×80 mm<sup>3</sup>) 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 (20×20×160 mm<sup>3</sup>) 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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**Table 1**  
Properties of plaster of Paris.

Chemical analysis								
Oxides	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	CaO	TiO <sub>2</sub>	MgO	P <sub>2</sub> O <sub>5</sub>	SO <sub>3</sub>	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 <sup>2</sup> /g)								4.6
Blaine specific surface area (m <sup>2</sup> /kg)								476
Chemical properties								
Initial electrical conductivity (mS/cm <sup>2</sup> /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 <sub>4</sub> [1–3]								
20 °C								8.9
100 °C								1.72–1.8

**Table 2**  
Properties of calcium sulfoaluminate clinker.

Chemical analysis								
Oxides	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	CaO	TiO <sub>2</sub>	MgO	P <sub>2</sub> O <sub>5</sub>	SO <sub>3</sub>	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 <sup>2</sup> /g)								1.05
Blaine specific surface area (m <sup>2</sup> /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<sub>4</sub><sup>2-</sup>) than at 20 °C (6.3–6.7 g/L SO<sub>4</sub><sup>2-</sup>), standard mortars were cast and cured in water at 20 °C. Their compressive strength was measured at

**Table 3**  
Mineralogy of calcium sulfoaluminate clinker (w<sub>t</sub> %).

Phase	Chemistry	%
Yeelimite	Ca <sub>4</sub> Al <sub>6</sub> O <sub>12</sub> SO <sub>4</sub>	73.5
Larnite	β-Ca <sub>2</sub> SiO <sub>4</sub>	16.1
Perovskite	Ca <sub>3</sub> Fe <sub>2</sub> TiO <sub>9</sub>	6.9
Mayenite	Ca <sub>12</sub> Al <sub>14</sub> O <sub>33</sub>	0.6
Periclase	MgO	1.7
Anhydrite	CaSO <sub>4</sub>	1.0
Quartz	SiO <sub>2</sub>	0.2

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

Phase	K <sub>sp</sub> at 25 °C	K <sub>sp</sub> at 50 °C	K <sub>sp</sub> at 85 °C
Ettringite	2.8 · 10 <sup>-45</sup>	2.24 · 10 <sup>-43</sup>	6.61 · 10 <sup>-41</sup>
Gibbsite	0.0399	0.0833	0.2249
Gypsum	3.72 · 10 <sup>-5</sup>	3.43 · 10 <sup>-5</sup>	2.35 · 10 <sup>-5</sup>
Calcium hydroxide	8.9 · 10 <sup>-6</sup>	4.57 · 10 <sup>-6</sup>	1.50 · 10 <sup>-6</sup>

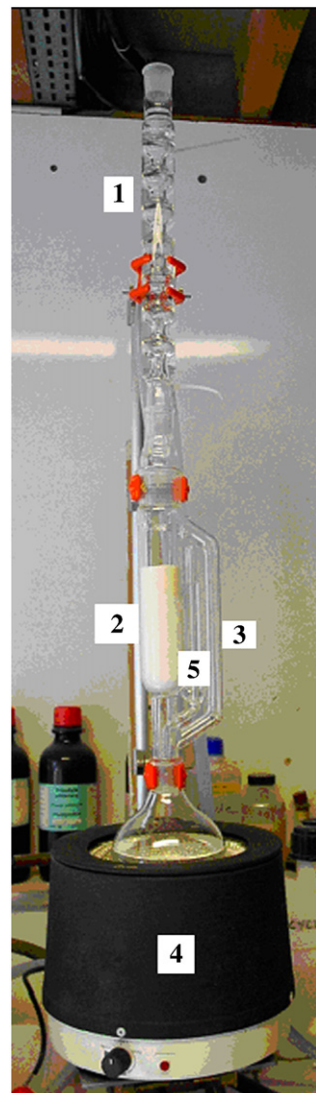
different times, up to one year. Some specimens were stored in air at 20 °C and 50% RH and their strength was measured after 28 days and one year.

### 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:  $\varphi < 0.1 \mu\text{m}$ ;



**1: Condenser**  
**2: Sample in the thimble**  
**3: Distillation path**  
**4: Still pot in the heater**  
**5: Siphon**

**Fig. 1.** Soxhlet extractor.

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