



# Influence of early sulfate exposure on the pore network development of mortars



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## HIGHLIGHTS

- Aggressive curing leads to alterations in the pore network from 7 curing days.
- XRD tests attribute the alterations to the precipitation of ettringite and gypsum.
- The pore characteristics influence the precipitation pattern of expansive phases.
- Laboratory tests should be performed in similar exposure conditions to the reality.

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## ABSTRACT

The objective of this paper is to evaluate the influence of sulfate exposure on the pore network development of several Portland cement matrices. MIP, XRD and SEM analysis were performed at different ages in samples exposed to sulfates after 2 days of casting. Results suggest that patterns of precipitation of the expansive products are linked to the degree of refinement of the pore network. During early stages of exposure, large pores concentrate a higher proportion of the expansive product formed. At later stages, precipitation evolves towards finer pore sizes.

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## 1. Introduction

External sulfate attack (ESA) has been recognized as a complex degradation phenomenon that may cause severe damage in cement based materials [1,2]. The stresses generated by precipitation of expansive phases (ettringite and gypsum) in the pore network are reflected on the macro-scale in the form of expansions and cracking. After decades of extensive research, important aspects such as the precipitation pattern of expansive products are not fully understood. Because of that, codes have adopted simplified approaches to mitigate ESA in real structures. Moreover, numerical models to predict the damage include simplifications regarding the precipitation of expansive products, requiring

several fitting parameters to correctly reproduce the effects of ESA [3,4].

The uncertainties in fundamental issues might be explained by the fact that most of the research conducted in this field is focused on the dimensional variation and cracking, which are the macro-scale response of the precipitation of expansive phases in the pore network. However, according to recent publications [5–7], to correctly assess the potential damage is key to understand the relationship between ettringite formation and pore network. When it comes to evaluate the damage generated, K. Tosun et al. [5] suggested that the location and morphology of the ettringite precipitation may be more relevant than the total quantity of ettringite formed. In fact, the crystallization pressure theory [6,7] states that only expansive products precipitated in small pores are likely to cause damage. Unfortunately, few experimental studies focusing on these aspects are found in the literature due to limitations in the techniques available to characterize the precipitation in the pore structure of the cement-based materials.

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Another important issue hardly ever evaluated is the early sulfate exposure. In reality, the source of external sulfate ions is usually found in sulfate-rich soils and underground waters in contact with concrete. Therefore, ESA is especially significant in underground structures like foundations, tunnels or waste containers. Due to their massive size, these structures are usually built *in situ*, hence being exposed to sulfates since casting. However, most studies about the ESA in laboratory rely on testing of specimens cured several days in lime water prior immersion in the aggressive solution with sulfates.

To illustrate this, Table 1 summarizes some of the recent studies on the ESA performed in laboratory, including data on the mineral admixtures used and the age at which the specimens were exposed to the sulfate solution. Even though it only represents a small part of studies published, the table indicates that in most of them specimens are cured at least 28 days before sulfate exposure. If mineral admixtures are used, this non-aggressive curing may be extended to 90 days. This contrasts with the reality found in most structures, which are subjected to sulfates shortly after casting.

Despite the influence of pore characteristics on the precipitation of expansive phases and the importance of early sulfate exposure, there is little or no research focused on the combination of these aspects. The objective of this study is to assess the pore network development and estimate qualitatively the pore ranges at which the alterations might occur in specimens subjected to early sulfate exposure. Since these aspects are expected to vary depending on the initial pore size distribution of the material, five mortars with different pore systems were evaluated: one without any mineral admixture (reference), three with mineral admixtures (fly ash, silica fume or limestone filler) and one with air-entrainer. In addition to that, the influence of two different sulfate concentrations representative of field and laboratory conditions were evaluated.

The evolution of the pore structure was assessed by mercury intrusion porosimetry (MIP). Although this technique may be affected by several experimental factors [20,21], it is one of the few that may provide an overview on the pore distribution. Moreover, a qualitative comparison should provide valuable information regarding the influence of early sulfate exposure and highlight possible differences on the precipitation patterns depending on the characteristics of the material. X-ray diffraction (XRD) combined with Rietveld analysis was performed to evaluate the presence of expansive phases at the end of the curing period. Scanning electron microscopy (SEM) was also performed to assess the influence of the external sulfate concentration and the degree of refinement of the matrix on the morphology and crystallinity of the ettringite formed.

The results obtained shed light on important issues related with the ESA. It provides a different view on the evolution of the attack, suggesting possible explanations for the alteration patterns observed in the porous network over time. The conclusions derived from this study may help to further understand the phenomenon behind the ESA, being useful for future development of prediction models and guidelines.

## 2. Experimental

### 2.1. Materials and mixture characteristics

Table 2 shows the properties of the Portland cement and mineral admixtures used in this work. Chemical composition of Portland cement (CEM I 52.5 R) was determined by X-ray fluorescence (XRF) and phase compositions were calculated using Bogue equations. Due to the presence of ferromagnetic particles, elemental compositions of the fly ash and silica fume were determined by ICP-MS. The fly ash used is equivalent to ASTM C618 class F. A low-calcium fly ash was chosen for its little or no self-cementing properties and to avoid a supplementary addition of the crystalline phases that may be present in high-calcium fly ashes, such as  $C_3A$  or  $C_4A_3S$ . Siliceous sand with maximum particle size of 2 mm was chosen for all binders.

Table 3 shows the composition of the 5 matrices included in the study, which intends to simulate the typical composition of the mortar that surrounds the coarse aggregate in a conventional concrete applied in foundations. In certain applications, mineral admixtures are used to replace a fraction of the cement, which reduces the  $C_3A$  content of the binder and its expansive potential. However, in this work, the additional volume from the mineral admixtures was fully compensated by a reduction in the content of sand. This way, the content of cement remained the same for all mixtures, ensuring a constant amount of  $C_3A$  per unit of volume and similar potential expansion. Although the water/cement ratio remained constant for all compositions at 0.45, the addition of mineral admixtures caused changes to the water/binder ratio. This allows the assessment of the alterations introduced by early sulfate exposure in the pore system over a wide range of different pore size distributions. Values of water/binder ratio for each composition are included in Table 3.

The mixing procedure defined in UNE-EN 196-1:2005 was adopted, assuming small modifications to cover the specificities of the admixtures added and the cylindrical molds used. In the case of compositions with mineral admixtures, the latter was first thoroughly mixed with cement prior to adding water. GLENIUM ACE

**Table 1**  
Age of sulfate exposure in a selection of recent publications

Authors	Year	Additions				Age of exposure [days]
		FA <sup>a</sup>	SF <sup>b</sup>	LF <sup>c</sup>	Sl <sup>d</sup>	
M.T. Bassuoni, M.L. Nehdi [8]	2009	x	x	x	x	56
T. Schmidt et al. [9]	2009			x		28
B. Lothenbach et al. [10]	2010					28
T. Aye, C.T. Oguchi [11]	2011	x	x			28
El-Hachem [12]	2012					3 and 28
W. Kunther et al. [13]	2012	x	x	x	x	28
J. Gao et al. [14]	2013	x			x	60
W. Müllauer [15]	2013					28
Zhang et al. [16]	2013					28
I. Sirisawat et al. [17]	2014	x		x		28
J. Stroh et al. [18]	2015	x			x	28
Yu et al. [19]	2015				x	90

<sup>a</sup> Fly ash.

<sup>b</sup> Silica fume.

<sup>c</sup> Limestone filler.

<sup>d</sup> Slag.

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