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Micro-analysis of the role of interfacial transition zone in "salt weathering" on concrete

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ABSTRACT

When concrete elements are partially immersed in the sulfate environment, researchers always attribute "salt weathering", "salt crystallization" or "physical attack" to the failure of concrete. However, there were few micro-analysis evidences to support this view. In this paper, an attempt was carried out to study whether salt weathering is really responsible for the concrete damage.

As we know, the interfacial transition zone (ITZ) between paste and aggregate plays a determining role in the performance of concrete. In this paper, we focused on the role of ITZ in "salt weathering" on concrete. Concrete specimens, made with coarse aggregate and cement paste, were partially exposed to a 5% sodium sulfate solution and a 5% magnesium sulfate solution respectively, in a controlled environment $(20 \pm 2 \,^{\circ}C, \text{ and } 60 \pm 5\% \text{ RH})$. After 8 months of exposure, a micro-analysis is performed by means of XRD, ESEM and EDS. The experimental results showed that, in the upper part of concrete above the Na₂SO₄ solution, damage initiated in the ITZ between paste and aggregate due to the formation of ettringite and gypsum. Salt crystallization cannot occur on the paste surface in the ITZ, but it was found on the aggregate surface after damage initiation due to chemical sulfate attack. On the other hand, salt crystallization could occur in the carbonated concrete. There was no trace of salt crystallization in the concrete partially exposed to MgSO₄ solution.

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1. Introduction



Review



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When masonry is partially exposed to a sulfate environment especially in the presence of sodium sulfate, salt weathering or salt crystallization is the mechanism of failure of stone or rock [1–3].

Thenardite (Na_2SO_4) or mirabilite $(Na_2SO_4 \cdot 10H_2O)$ can be identified [4–7], epsomite $(MgSO_4 \cdot 7H_2O)$ also can be found in stone pores [6,7]. A similar visual appearance can be observed on concrete elements partially exposed to this sulfate environment. So, it is often taken for granted that physical attack or salt crystallization leads to the collapse of concrete.

In the previous paper [8], the performance of cementitious pastes partially exposed to the sodium sulfate solution and magnesium sulfate solution under constant and sharply fluctuated conditions was studied in detail by means of ESEM (environmental scanning electron microscope), EDS (energy dispersive X-ray spectroscopy) and XRD (X-ray powder diffraction). However, the results showed that there was no trace of salt crystallization in the paste. On the contrary the commonly chemical sulfate attack products, such as ettringite, gypsum and brucite, were indentified to be responsible for the failure of the cementitious pastes. In this paper, further tests were performed to study the performance of concrete partially exposed to sulfate solutions.

As we know, in concrete, two main components can easily be distinguished: aggregate particles of varying size and shape, and the binding medium composed of hydrated cement paste. Concrete may be considered as a two-phase material, consisting of aggregate particles dispersed in a matrix of the cement paste [9]. The two components are linked by the interfacial transition zone (ITZ). The weak ITZ plays a particularly important and even determining role in the main characteristic of concrete [10].

Concerning sulfate attack on concrete, experimental results [11] showed that cracks developed from ITZ to bulk paste and resulted in the disintegration of cement mortar. According to the author's previous study on concrete specimens completely immersed in different sulfate solutions [12], coarse aggregates and mortar matrix were thoroughly separated along the ITZ after one year of immersion in sodium sulfate solution. After one year immersion in magnesium sulfate solution, a layer of gypsum and brucite crystals, formed in the ITZ, resulted in the collapse of concrete cores. A number of researches [13,14] also showed by means of SEM analysis that deterioration occurred first in the ITZ.

In this paper, concrete specimens were partially immersed in sodium sulfate solution and magnesium sulfate solution, respectively. Reaction products in the ITZ and in the cement paste were examined by means of ESEM, EDS and XRD. The purpose was to identify whether there were some traces of thenardite (Na₂SO₄) or epsomite ($MgSO_4$ ·7 H_2O) crystals in the ITZ and in the cement paste, and to disclose the real cause for concrete damage.

2. Experiments

2.1. Raw materials

Ordinary Portland cement (CEM I 52.5 N), complying with EN 197-1 (2000) was used in the experimental program. The chemical composition of cement is given in Table 1. Gravel with a size range of 5–16 mm and sand with the size range of 0–5 mm were used as the coarse and fine aggregates, respectively. The mixing water for concrete was tap water. Concrete specimens were exposed to a 5% sodium sulfate solution and a 5% magnesium sulfate solution by mass respectively.

2.2. Specimens preparation, curing and testing

In order to focus on the influence of the ITZ in the process of "salt weathering" on concrete and to compare the role of ITZ and the pure cement paste in the failure of concrete, concrete was just made with cement and coarse aggregate. The cement/aggregate ratio was 0.4 and the water/cement ratio (W/C) was 0.45.

The fresh concrete was cast in a 150 mm cubic steel mould, then covered with a plastic sheet and moved into a moist room. After 24 h of curing, the concrete was demoulded and stored in water at 20 ± 1 °C. At the age of 15 days, the cubic specimen was cut into smaller specimens with size of $10 \times 40 \times 150$ mm, after which they were immersed in water again fro 13 days. At the age of 29 days, the specimens were removed from the water and stored in a climate room at 20 ± 2 °C and $60 \pm 5\%$ RH for 7 days of drying. Then 10 concrete pieces were partially immersed in a container with a 5% sodium solution. The other 10 samples were immersed in magnesium sulfate solution. In both cases, the exposure time was 8 months. The solution surface was maintained constant, at the same level as the edge of the container (Fig. 1a).

2.3. Influence of carbonation

In previous tests on paste specimens [8], it was found that salt crystallization only occurred in cement paste that was completely

Chemical composition of c	ement (% by mass).

Table 1

•										
	CaO	SiO ₂	Fe ₂ O ₃	MgO	Al_2O_3	SO ₃	CO ₂	Na ₂ O	K ₂ O	Ignition loss
Cement	62.21	19.12	3.79	0.86	5.39	3.06	0.72	-	_	1.65



Fig. 1. Setups of immersion tests.

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