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Deterioration mechanism of plain and blended cement mortars partially exposed to sulfate attack

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HIGHLIGHTS

- Durability of cement mortars partially exposed to sulfate attack was investigated.
- The upper part mainly suffered physical attack, while the low part suffered chemical attack.
- The water-soluble SO_4^{2-} contents of cement mortars were measured.
- SEM, MIP, XRD and X-CT were used to investigate the microstructure changing.

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ABSTRACT

The degradation of cement-based materials partially exposed to sulfate solution are dramatically serious than under ordinary full immersion. To research the damage regime of cement-based materials partially exposed to sulfate attack, the resistance of drying (upper) portion and immersed (lower) portion of both the plain cement mortars as well as fly ash (FA) and grounded blast furnace slag (GBFS) modified cement mortars partially immersed in 10% Na_2SO_4 solution was investigated in this paper. The mass change, relative dynamic modulus of elasticity, compressive/flexural strength and water-soluble SO_4^{2-} contents were monitored to show the damage progress. SEM/EDS, XRD, MIP and X-CT were used to investigate the microstructure changing.

The research results indicated that the deterioration of cement mortars partially immersed in sulfate solution is not only caused by chemical sulfate attack, but also by physical sulfate attack. The upper part of specimens mainly suffer physical sulfate attack, as well as chemical sulfate attack, while the lower part only suffer chemical sulfate attack. Furthermore, fly ash (FA) have better effect than grounded blast furnace slag (GBFS) on improving the resistance of cement mortars to partially immersed in sulfate solution.
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1. Introduction

Concrete structures in South China Sea region and salt lakes of west China deteriorated significantly, so it is very important to indicate the degradation mechanism of cement-based materials partially immersed in salt solution. In the past years, many research works had been reported on cement-based materials partially exposed to sulfate solutions [1–6], but are still not reach an agreement on the deterioration regimes.

Sulfate attack can be categorized as chemical attack and physical attack, due to its different degradation regimes. Chemical sulfate attack is considered a complex physicochemical process. The sulfate ions penetrate into cement-based materials, directly react with the cement hydration products-calcium hydroxide (CH) and form harmful products (such as ettringite and gypsum) [7–9], resulting in softening, expansion, cracking, spalling and disintegration of cement-based materials. The damage caused by the crystallization of sulfates in the pores near the drying surface of cement-based materials without chemical reactions was referred to as "physical salt attack" by the American Concrete Institute (ACI) Committee 2011 (Durability of Concrete) [6]. The physical sulfate attack also calls sulfate salt crystallization or sulfate salt weathering, resulting in progressive surface scaling and flaking of cement-based materials. For instance, the conversion of anhydrous sodium sulfate (thenardite, Na_2SO_4) to the hydrous (mirabilite, $Na_2SO_4 \cdot 10H_2O$) form is associated with 314% volumetric expansion. In the field of concrete durability, the issues of physical sulfate attack and chemical sulfate attack cannot be distinguished clearly. Such

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as Nehdi [10] investigated plain and blended concrete partially immersed in 5% Na_2SO_4 solution and exposed to cyclic temperature and RH consisting of one week at temperature = 20 °C and RH = 82% followed by one week at temperature = 40 °C and RH = 31% and indicated that the degradation of the upper part of concrete was caused by the physical sulfate attack, resulting from the transformation of the formed mirabilite to thenardite. By contrast, Liu [11] researched the cement and cement + fly ash (25% dosage) paste partially immersed in different sulfate solutions and results showed that chemical sulfate attack occurring in the high concentration sulfate pore solutions of upper part of concrete in contact with air was likely the cause of the worse deterioration of the upper part. So the deterioration mechanism of concrete partially exposed to sulfate should be determined.

There are several factors affecting sulfate exposure, such as the water-to-cement ratio, mineral admixtures (fly ash, grounded blast furnace slag, silica fume), surface treatments and the environmental temperature and humility. However, contradictory conclusions about these factor are still exist. Mehta [12] pointed out the mineral additions could increase the deterioration of concrete partially exposed to sulfate attack due to pore size refinement by mineral additions promoted the capillary rise of the solution in the concrete, giving by the following equation [13,14]:

$$h = \frac{2\gamma_{LV}\cos\theta}{rg\rho} \quad (1)$$

where h (m) is the height of capillary rise, γ_{LV} (N m^{-1}) is the liquid/air inter-facial energy, θ is the contact angle, r (m) is the pore radius, g (kg/m^3) is the gravitational acceleration, and ρ is the density of solution. On the contrary, Bassuoni [15] showed that fly ash can improve the resistance of concrete to physical sulfate attack, ascribing to the pozzolanic effect of fly ash on improving the pore structures and discounting the continuity of capillary pores.

There are conflicting data as well the contradictory theory regarding the degradation progress of cement-based materials partially exposed to sulfate attack. Thus, in this paper, fly ash and grand graduate blast slag modified cement mortars partially immersed in 10% Na_2SO_4 solution were investigated to verify the effect of mineral mixtures on the sulfate exposure. Moreover the drying and immersed portions of specimens were compared to determine the degradation mechanism of cement mortars partially exposed to sulfate solution.

2. Experimental program

2.1. Materials and methods

In this study P I 52.5 Portland cement meeting the requirements of GB175-2007 standard was used. Class-I fly ash (FA) and grounded blast furnace slag (GBFS) complying with GB/T 1596-2005 and GB/T 203-2008 were used. The physical properties and chemical compositions of Portland cement, FA, and GBFS are given in Table 1. River sand (<5 mm) with fineness modulus of 2.6 was used. The mixture proportions of mortars are summarized in Table 2. In addition, MO means mortars with Portland cement, MSO represents 50% replacement of Portland cement with GBFS and MFO means 30% replacement of Portland cement with FA.

Table 1
Physical properties and chemical compositions of cement, FA and GBFS.

CaO	64.47	5.75	36.35
SiO_2	20.87	51.07	33.48
Al_2O_3	4.87	30.86	12.21
Fe_2O_3	3.59	5.26	1.40
MgO	2.13	2.72	10.60
SO_3	2.52	1.48	0.66
K_2O	0.65	1.13	0.56
Na_2O	0.11	0.79	1.27
Physical properties			
Specific gravity/(g/cm^3)	3.11	2.35	2.82
Blaine fineness/(cm^2/g)	3689	4000	4600

The mortars specimens were mixed in a mechanical mixer and cast in cuboid molds of 40 × 40 × 160 mm. Mortar specimens were demould after placed in lab for 24 h, then cured for 60 days at a temperature of 20 ± 2 °C and 95% of relative humidity.

2.2. Partially sulfate exposure

Following standard curing, mortar specimens were placed in the laboratory for 24 h to remove excess water from the surface of specimens and keep the surfaces of the mortars sufficiently dry to provide a uniform basis of comparison. Then their initial data were detected before sulfate exposure, such as compressive/flexural strength, mass, dynamic modulus of elasticity (E_{rd}) and also some samples were extracted from initial mortars for mercury intrusion technique (MIP) as well X-ray diffraction (XRD) researches. Following that mortars were partially immersed in plastic containers with 10% sodium sulfate solution up to 60 mm (nearly one-third of the total height of specimens). In order to minimize the evaporation of sulfate solution, each lid of plastic containers was cut out six 40 × 40 mm openings, which were equal to the size of mortars, as shows in Fig. 1. The drying portion was exposed to a room condition with 35 ± 1 °C and 50 ± 5% relative humidity, which was provided by controlled environmental chambers. The high concentration of sulfate solution, relative high temperature and low humidity were selected to accelerate the sulfate attack. Moreover, this temperature and humidity were in accord with the South China Sea region. In order to keep the solution level up to 60 mm height during this experiment, sulfate solution was frequently replenished in plastic container and also replaced every month to maintain the solution concentration and PH value.

2.3. Testing scheme

During the partially exposure, the mortar specimens were monitored (mass, E_{rd}) every month. After exposure, salt efflorescence and debris were carefully removed from the surface of mortar specimens by a nylon brush. Then the specimens were placed in laboratory with room condition for 6 h to remove the excess water from the surface of specimens. Subsequently, the mass and E_{rd} were determined. The mass of the specimens before (M_o) and after (M_n) partially immersed in sulfate solution were measured on an electronic scale (capacity of 5 kg and an accuracy of 0.01 g) . Consequently, the mass change (W_1) was calculated as follows:

$$W_1 = \frac{M_n - M_o}{M_o} \times 100\% \quad (2)$$

The relative dynamic modulus of elasticity (E_{rd}) of the specimen under partially exposure was measured by a nonmetal ultrasonic analyzer (NM-4A, transducer frequency is 50 kHz) every month.

The flexural and compressive strength of specimens were also an important indicator of mortars degradation. The MO specimens fractured after partially exposure for 6 months, so the flexural and compressive strength of all mortar specimens were detected after erosion for 6 months. Moreover, the upper part and immersed part of specimens were under different deterioration regimes, so both the upper and immersed portions of specimens were determined after 6 months exposure. The strength of mortars before (S_o) and after (S_n) partially immersed in sulfate solution were measured on universal testing machine. Consequently, the strength change (ΔS) was calculated as follows:

$$\Delta S = \frac{S_n - S_o}{S_o} \quad (3)$$

To identify the penetration of sulfate ions in mortars, the water-soluble SO_4^{2-} contents were determined by ultra-violet and visible spectrophotometer (UVPC). The powder sample was collected from both the drying and immersed portions of specimens and passed the 0.15 mm sieve. 2 g powder were taken by an electronic scale (capacity of 200 g, with a precision of 0.0001 g) after the powder was dried in an oven at a temperature of 60 °C to achieve constant weight. Following that the 2 g powder dissolved in 50 ml distilled water and stored for 24 h. Then the whole solution were filtered through slow filter paper and 25 ml of them were put into colorimetric cylinder (capacity of 50 ml). The extraction solution was titrated by diluted hydrochloric acid (2.5 mol/l), and added 10 ml $BaCl_2$-PVA mixture as dispersant, then diluted with distilled water to 50 ml, followed by oscillation 2–3 times and standing for 5 min. Then solution was extracted into cuvette and compared with the blank solution. The wavelength used was 440 nm [16].

3. Results and discussion

3.1. Visual inspection

Visual appearance of MO, MFO, MSO specimens partially exposed to 10% Na_2SO_4 solution are shown in Fig. 2. As Fig. 2(a)

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