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# Study of deterioration of concrete exposed to different types of sulfate solutions under drying-wetting cycles



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• Durability of concrete under different types of sulfate solutions is investigated.

• SEM, XRD and TG-DSC are used to study microstructure of concrete after corrosion.

• Large amount of corrosion products can be formed before obvious damage is occurred.

• The damage process of concrete exposed to different sulfate solutions is analyzed.

#### ARTICLE INFO

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# ABSTRACT

Properties of concrete subjected to drying-wetting cycles in different types of sulfate solutions were investigated in this paper. The corrosion solution includes three types, namely, 10% sodium sulfate solution, 10% magnesium sulfate solution and the composite solution of 10% sodium sulfate and 3.5% sodium chloride (by mass). Through the experiment, visual change, relative dynamic modulus of elastically, weight loss, compressive strength loss and the damage layer thickness of concrete were measured. To identify the products formed by sulfate attack, analytical techniques, including X-ray diffraction, scanning electron microscopy and thermal analysis were performed on the selected samples. Test results show that the deterioration degree of concrete in magnesium sulfate solution is more severe than that in the other sulfate solutions. The existence of chloride ions in the composite solution reduces the deterioration rate of concrete, and the damage degree of concrete could be inhibited effectively. Test results also show that the quantity of corrosion products in magnesium sulfate solution is higher than that in the other sulfate solutions. While, the quantity of corrosion products is the lowest when exposed to the composite solution. Furthermore, the quantity of gypsum in concrete is less than that of ettringite in test, and some of gypsum can be observed only after a certain corrosion extent. The quantity of corrosion products does not correspond well with the observations of the physical properties, and extensive amount of corrosion products can be detected before the deterioration degree of concrete increased significantly.

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### 1. Introduction

In the sulfate environment, sulfate ions, magnesium ions and chloride ions are the common corrosive ions exist widely in seawater, groundwater, salt lake and sulfate soil. Sulfate attack on cement materials is one of the most severe problems affecting the durability and service life of concrete structures in the natural climate. Sulfate attack can manifest in the form of expansion and cracking of concrete. Sometimes the expansion of concrete may cause serious structural problems. Sulfate attack can also take

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http://dx.doi.org/10.1016/j.conbuildmat.2016.04.094 0950-0618/© 2016 Elsevier Ltd. All rights reserved. the form of a progressive decrease in the strength and weight loss due to the cohesiveness loss of the cement hydration products [1,2]. In sulfate environment, the elements of concrete construction situated in water table fluctuation, tidal zone and splash zone will suffer from more complex attack because of the dryingwetting cycles, which can accelerate the deterioration of concrete.

Sulfate attack on concrete structures caused the attention of the scholars earlier. After a long period of explorative research, there already have a certain understanding to the mechanism, influence factors, evaluation methods and evaluation indexes of concrete under sulfate attack exposed to sodium sulfate solution. Many works for the damage process of concrete exposed to sulfate attack [3–6] and the coupling function of sulfate attack and



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drying-wetting cycles [7–9] such as expansion, weight loss, relative dynamic modulus of elastically (RDME) loss and the changes of mechanical properties of concrete have been reported.

However, little studies of the damage process are done on cement materials attacked by magnesium sulfate solution or the composition salt solution of chloride and sulfate ions. And it is in the little studies that most of them are conducted on cement paste or mortar. Salah et al. [10] investigated the performance of plain and blended cements exposed to magnesium sulfate solutions with varying sulfate concentrations. Siad et al. [11] investigated the effects of mineral admixture type on the behavior of selfcompacting concrete exposed to magnesium sulfate solutions over 4 years of exposure. Diab et al. [12] suggested the guidelines in compressive strength assessment of concrete modified with silica fume as results of magnesium sulfate attack. Geng et al. [13] investigated the stability of bound chlorides in chloride-contaminated cement pastes with and without fly ash and ground granulated blast-furnace slag when subjected to sodium and magnesium sulfate attack. Zhang et al. [14] studied the expansion and subsequent damage of concrete immersed in sulfate solutions of different sulfate concentrations, and mixed solutions of different sulfate and chloride concentrations. Xu et al. [15] reported the results of a study conducted to evaluate the releases of bound chlorides from chloride-admixed plain and blended cement pastes exposed to sulfate attacks. However, the role of chloride ions in the presence of sulfate solution on the deterioration of concrete is not well known. In addition, the damage mechanism of concrete exposed to dryingwetting cycles under magnesium sulfate or composite solution of sulfate and chloride environment need further studied.

In the study of microscopic-test, little literatures have been conducted concerning the quantity of corrosion products in concrete under sulfate attack, especially quantitatively compared in different corrosion layer of concrete exposed to magnesium sulfate solution or composite solution of sulfate and chloride. Santhanam et al. [16] discussed the thermal analysis results for mortars immersed in solutions of seawater and groundwater. Mathias et al. [17] investigated the amount of ettringite and gypsum using quantitative Rietveld analysis after X-ray diffraction (XRD) measurements for motar exposed to a combined sodium sulfate and sodium chloride solution. Chen et al. [18] investigated the microstructure of interior concrete by using XRD and thermal analysis exposed to combined attack of chloride and sulfate under drying-wetting cycles. Jiang et al. [19] discussed the XRD and thermal analysis results of concrete under sulfate attack exposed to freeze-thaw cycles. Chen et al. [20] studied the quantity of ettringite in concrete under the dry-wet cycling condition and sodium sulfate environment.

Furthermore, most researches have focus on raising the temperature of sulfate solution in the drying state to accelerate the damage process of concrete under sulfate attack. For example, test of specimens drying under 100 °C for 24 h was used by Sahmaran et al. [21], drying under 60 °C was used by Niu et al. [22], drying under 70 °C was used by Yuan et al. [23], drying under 80 °C was used in literatures [24,25], and drying under 105 °C for 24 h was used by Yang et al. [26]. But, the high temperature has a great influence on the quantity of corrosion products in concrete, probably altering the mechanism of sulfate attack. In the test, the drying state was natural drying in the air, which was more relevant to a real environment.

This study simulated concrete exposed to sodium sulfate, magnesium sulfate and composite solution of sodium sulfate and sodium chloride under cyclic environmental condition. Basic experimental research on the performance of concrete in different sulfate solutions was conducted based on the macroscopic and microscopic test. Visual change, weight loss, RDME loss, compressive strength loss and the damage thickness of concrete were conducted. Furthermore, the corrosion products of the concrete were distinguished and quantitatively compared by the scanning electron microscopy (SEM), XRD and the thermal analysis. The deterioration process of concrete exposed to the different types of sulfate solutions under drying-wetting cycles was also analyzed.

# 2. Experimental details

#### 2.1. Materials and mix proportions

A Chinese standard Ordinary Portland Cement (OPC) of PO 42.5R produced by the Cement Factory of Tongchuan was adopted. Grade II fly ash from the Weihe Power Station, river sand with a fineness modulus of 2.69 and coarse aggregate of crushed basalt stone with a diameter of 5–16 mm were used in the test. A naphthalene-type superplasticizer was used, and the dosage was adjusted to keep the slump of fresh mixed concrete in the range of 50–120 mm. The tap water was adopted as mix water. The chemical composition of cement and fly ash is shown in Table 1.

In this experiment, the water-binder ratio (W/B) was 0.45, and the concrete with 20 wt% replacements of cement with fly ash was used. According to the related investigations [27–29], about 20 wt % of cement is replaced by fly ash, the resistance property of concrete under sulfate attack could be improved effectively. The mixture proportion and corresponding compressive strength of the concrete are presented in Table 2.

#### 2.2. Specimens preparation and curing conditions

Concrete specimens were prepared in a 0.3 m<sup>3</sup> forced action mixer, and all the test specimens were fabricated from a single batch of concrete. The components of the concrete mixture were batched by weight, the cement was premixed with fly ash, sand and coarse aggregate before adding the water and the admixtures for 1 min. Then, the entire amount of mixing water with the dissolved superplasticizer were added and mixed for 3 min. Finally, concrete mixture was mixed for another 2 min. The concrete specimens were cast in steel moulds and compacted on a vibration table. All specimens were demolded after 24 h of casting and were cured in a condition of  $20 \pm 3$  °C and 95% relative humidity.

# 2.3. Experiment methods

In this paper, the concrete specimens exposed to sulfate solution under drying-wetting cycles up to 360 days (Fig. 1). The drying state was natural drying in the air, which was more relevant to in-service conditions. One drying-wetting cycle lasted 15 days. First, the specimens were continuously immersed in the test solution for 7 days, and then were moved into the air to dry naturally for another 8 days. In this study, concentrations (by mass) of 10% sodium sulfate solution, 10% magnesium sulfate solution and the composite solution of 10% sodium sulfate and 3.5% sodium chloride were used. For each case, 3 specimens of 100 mm  $\times$  100 mm  $\times$  400 mm prisms were used for the study of the RDME loss, weight loss and damage layer thickness of concrete. For each case, 30 specimens of  $100\,mm \times 100\,mm \times 100\,mm$ cubes were used for the study of the compressive strength loss and the analysis of attack products of concrete. All experiments were performed on three specimen replicates. The average values were used for the discussion of the test results. According to the test procedure, the deterioration of the specimens was investigated by determining the RDME loss, weight loss and compressive strength loss. The specimen was considered to be a failure if the RDME dropped to 60%, the weight loss exceeded 5% or the compressive strength dropped to 75%.

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