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# Influences of exposure condition and sulfate salt type on deterioration of paste with and without fly ash

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## HIGHLIGHTS

- The deterioration of paste exposed to external sulfate attack is investigated.
- The influences of exposure condition and sulfate salt type are taken into account.
- The damage is evaluated by compressive strength, mass loss and Vickers hardness.
- The damage mechanisms of chemical and physical sulfate attack are analyzed.
- The influence of fly ash on different kind of sulfate attack is investigated.

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## ABSTRACT

The aim of this study is to investigate the influences of exposure condition and sulfate salt type on the deterioration features and damage mechanisms of paste exposed to external sulfate attack. Cement pastes with w/b ratios of 0.35, 0.45 and 0.55, and fly ash pastes with the w/b ratio of 0.45 were exposed to sodium sulfate solution and magnesium sulfate solution with different exposure conditions. Four exposure conditions, referenced as CI (continuous immersion), DWCI (dry-wet cycle immersion), PIAWL (partial immersion above the water level) and PIUWL (partial immersion under the water level), were designed for the exposure tests. The compressive strength, mass variation and Vickers hardness (VH) profiles of specimens were measured to evaluate the damage of the paste specimens. Scanning Electron Microscope (SEM), Energy Dispersive Spectrometer (EDS) and X-ray diffraction (XRD) were used to analyze the degradation mechanisms. As the results shown, pastes exposed to external sulfate attack with different exposure conditions suffered from different damage mechanisms, including chemical sulfate attack and physical sulfate attack. The highest damage was observed on the specimens with the PIAWL condition when they were exposed to sodium sulfate solution. While in the cases of magnesium sulfate attack, the highest damage was found on the specimens with the DWCI condition. For chemical sulfate attack, a suitable content of fly ash in pastes could increase the resistance to sulfate attack, while the addition of fly ash might accelerate the damage when the specimens suffered from physical sulfate attack.



## 1. Introduction

Sulfate attack is an important element which decreases the durability of concrete under sulfate environment. Usually, it has four acceptable primary mechanisms, i.e., ettringite, gypsum, magnesium sulfate attack and thaumasite based on the sulfate salt types, exposure conditions and the features of concrete itself [1–9].

According to whether the chemical reaction is involved, concrete deterioration caused by sulfate attack can be categorized into two aspects, called chemical and physical attack (or salt weathering), which is deeply related to environment conditions that concrete structures served [10,11]. For instance, concrete foundations of buildings constructed on sulfate-bearing ground or water often suffer from expansion damage, responsible for the formation of gypsum or ettringite, in the deteriorated parts under the water level. However, in the portions above the ground or water level (exposed to air), the surface scaling of concrete can also be observed with the presence of sulfate salt crystallizations [12,13].

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In the present testing standards of sulfate attack in concrete, such as American standards ASTM C1012 [14], ASTM C452 [15] and Chinese national standards GB2402-1981 [16] and GB/T 749-2001 [17], continuous immersion tests are most widely used, but they cannot be applied to evaluate the damage of concrete in the above-ground/water part exposed to air. Therefore, partial immersion tests for concrete exposed to external sulfate attack are thought to be more useful to evaluate the damage of concrete structures constructed on sulfate-bearing ground or water [13]. Up to now, several investigations have been done on the partial immersion tests of concrete under sulfate environment [10–13,18–21]. Nehdi et al. [18] proposed that partially immersed concrete in sodium sulfate solution could experience dual sulfate attack. The lower portion immersed in the sodium sulfate solution can suffer from chemical sulfate attack, while the upper portion can be vulnerable to physical sulfate attack. Comparing to chemical sulfate attack, physical sulfate attack often causes more serious damage to concrete because of the oversaturation of the sulfate solution that penetrated into the pores of concrete from ground or water under the action of "wick effect" [12,19]. According to the previous studies [10–13,18–23], the mechanism of damage caused by physical sulfate attack can be simplified as a "diffusion-concentration-crystallization" process, while the damage caused by chemical sulfate attack can be considered as a "diffusion-reaction-expansion" process, and both of the two types of sulfate attack are processes of damage accumulation.

From the reviews above, it is obvious that concrete structures under sulfate environment can suffer from both chemical and physical sulfate attack. Several studies have explored the associated deterioration mechanisms of concrete caused by these two mechanisms. According to Nehdi et al. [18], physical sulfate attack on the upper portion of the damaged specimens partially immersed in sodium sulfate solution was defined by SEM and EDS analyses. However, whether the chemical sulfate attack occurred on that portion remains unknown. In addition, it has been well known that a suitable content of fly ash in concrete could retard the deterioration when the concrete was in a continuous immersion condition [23,24]. Nevertheless, few studies have focused on the influences of fly ash on the resistance to sulfate attack for concrete with other exposure conditions (like partial immersion condition and dry-wet cycle immersion condition). Thus, investigations to elucidate the deterioration process and damage mechanisms of paste with and without fly ash exposed to external sulfate attack with different exposure conditions and sulfate salt type are bound to be a subject with great importance.

In the present study, the influences of different exposure conditions and sulfate salts on deterioration process and damage mechanisms of concrete exposed to external sulfate attack were investigated. Cement paste specimens were continuously immersed, partially immersed and dry-wet cycle immersed in sodium and magnesium sulfate solutions. Fly ash was added in the paste mixtures to investigate the resistance of fly ash to external sulfate attack in concrete with different exposure conditions. The compressive strength, mass variation and Vickers hardness (VH) profiles of specimens were determined for evaluating the deterioration of cement pastes with different exposure conditions. The damage mechanisms of pastes under sulfate attack with different exposure conditions were analyzed using SEM, EDS and XRD.

**Table 2**
Proportions of paste mixtures.

| Ref. No. | w/b | Cement (%) | Fly ash (%) |
|---|---|---|---|
| OPC-35 | 0.35 | 100 | 0 |
| OPC-45 | 0.45 | 100 | 0 |
| OPC-55 | 0.55 | 100 | 0 |
| FA-10 | 0.45 | 90 | 10 |
| FA-30 | 0.45 | 70 | 30 |

## 2. Experimental program

### 2.1. Materials and mixture proportions

Ordinary Portland cement and Class I fly ash were used. Table 1 lists the chemical compositions of the cement and fly ash. Table 2 shows the proportions of cement paste mixtures with and without fly ash. Pure cement paste mixtures were prepared with the w/b ratio of 0.35 0.45 and 0.55, referenced OPC-35, OPC-45and OPC-55. The referenced mixes FA-10 and FA-30 were design with a constant w/b ratio of 0.45 to investigate the influence of the addition of fly ash on deterioration of concrete exposed to external with different exposure conditions and sulfate salts. The Table 2 was designed keeping the binder weight (cement + fly ash weight) constant.

### 2.2. Specimens preparation

$40 \times 40 \times 160\ mm^3$ prismatic specimens were prepared for different exposure tests. The specimens of each paste mixture were cast and demolded after 24 h sealed curing, and the demolded specimens were immersed in limewater for 28 days. Before exposure in sulfate solutions, specimens were coated on the two end surfaces with epoxy resin, hence to make sure that sulfate attack occurs on the side surfaces. Then, the specimens for the exposure to sulfate environment were prepared.

### 2.3. Exposure conditions

According to Aye and Oguchi [23], and Haynes et al. [8,25], higher surface scaling was found for the concrete partially immersed in 5% sodium sulfate compared to that exposed to other salts such as magnesium sulfate, sodium carbonate, and sodium chloride under the same exposure conditions. Therefore, in the present study, both the performances of cement paste exposed to sodium sulfate and magnesium sulfate were investigated. The different exposure conditions investigated in this paper was listed in Table 3. For all the exposure conditions, both the performance of cement paste exposed to sodium sulfate and magnesium sulfate were investigated. The concentration of the sulfate solutions was 10 wt%. In the dry-wet cycle immersion tests, the cycle procedure was as follows: two days immersion and five days air exposure (specimens were removed from sulfate solutions to air). All the solutions were refreshed once a month. In addition, a group of control specimens were preserved in limewater for compressive strength tests. The air condition in the lab was kept with $T = 20\ ^{\circ}C$ and $RH = 60\%$.

### 2.4. Compressive strength

For the continuous and dry-wet cycle immersion tests, samples for compressive strength tests were prepared by cutting specimens with a size of $40 \times 40 \times 40\ mm^3$, from the middle part of the original specimens along the height direction. For the partial immersion tests, the related samples were cut from both the portions up and down the water level, with the same size of $40 \times 40 \times 40\ mm^3$. The mechanical properties were determined by loading on the vertical direction to sulfate diffusion, according to GB/T 50081-2002 (Standard for test method of mechanical properties on ordinary concrete) [26].

The loss ratio of compressive strength $\Delta\sigma$ (%) in the corresponding test period can be obtained by [26]

$$\Delta\sigma(\%) = \frac{\sigma_{control} - \sigma_{damaged}}{\sigma_{control}} \quad (1)$$

where $\sigma_{control}$ is the compressive strength of the control group and $\sigma_{damaged}$ is the compressive strength of the chemical damaged group.

**Table 1**
Chemical compositions of cementious materials (wt%).

| Compn. | $SiO_2$ | $Al_2O_3$ | $Fe_2O_3$ | CaO | MgO | $SO_3$ | $Na_2O$ | $K_2O$ | LoI |
|---|---|---|---|---|---|---|---|---|---|
| Cement | 21.74 | 5.32 | 3.32 | 62.62 | 1.02 | 2.18 | 0.41 | 0.77 | 1.93 |
| Fly ash | 48.45 | 30.35 | 4.98 | 3.18 | 6.38 | 0.40 | 0.61 | 1.37 | 2.79 |