



# Durability of concrete containing fly ash and silica fume against combined freezing-thawing and sulfate attack



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## HIGHLIGHTS

- Sulfate resistance of concrete with fly ash (FA) & silica fume (SF) is investigated.
- The effects of FA and SF replacement level under the combined freezing-thawing and sulfate attack are analyzed.
- Sodium sulfate concentration under the combined freezing-thawing and sulfate attack are analyzed.
- Interaction between freezing-thawing and sulfate attack to concrete with fly ash (FA) & silica fume (SF) is discussed.
- SEM analysis of concrete subjected to freezing-thawing cycles in sulfate solutions were conducted.

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## ABSTRACT

Durability of concrete containing fly ash (FA) and silica fume (SF) against combined freezing-thawing and sulfate attack was studied in this paper. Concretes with w/b of 0.38 and 0.33 containing FA (i.e. of 10%, 15% and 25% by weight) and SF (i.e. of 5%, 8% and 11% also by weight) as partial replacement of Portland cement (PC) were exposed to 5% and 10% sodium sulfate solution under freezing-thawing cycles. The performance, including deterioration resistant coefficient of compressive strength, relative dynamic elastic modulus (RDEM) and microstructure, of concretes were evaluated after being subjected to certain freezing-thawing cycles in sodium sulfate solution. It was found that when exposed to 5% sodium sulfate solution, both FA and SF can improve concrete's resistance to sulfate attack and in comparison SF performed better than FA. Concrete deterioration was attributed to the interaction between freezing-thawing and sulfate attack. As for concrete without any admixture, its resistance against combined freezing-thawing and sulfate attack increased up to 125 freezing-thawing cycles and then decreased. The replacement level of 25% FA and 5–8% SF both by weight led to significant improvements in the resistance of concrete against combined freezing-thawing and sulfate attack. 10% sodium sulfate solutions more obviously improved freezing-thawing resistance of concrete with 25% by weight FA replacing OPC than 5% sodium sulfate solutions, while 5% and 10% sodium sulfate solution had the similar improvements in freezing-thawing resistance of concrete with 8% by weight SF replacing OPC.

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## 1. Introduction

Durability and service life of concrete mainly depends on many environmental factors, such as carbonation, sulfate attack, alkali-aggregate expansion [1–5], heating-cooling cycles, freezing-thawing cycles, wetting-drying cycles, reinforcement corrosion etc. [6]. Sulfate attack is one of the most common and severe

factors to concrete in service, which can be found at various regions throughout the world such as Northwest China, southern California in the USA, Arabian Gulf region, Japan, Australia and Alpine area [7–14]. Sulfate attack to concrete is a complicated physical and chemical process, which includes physical salt attack due to salt crystallization and chemical sulfate attack by sulfate from certain sources external to concrete [15].

In the case of chemical sulfate attack, sulfates of sodium, potassium, calcium, or magnesium in soil or dissolved in groundwater or seawater in the vicinity of concrete structures enter concrete, attack the hardened cement paste and increase the potential of

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deterioration. Free lime ( $\text{Ca}(\text{OH})_2$ ), calcium aluminate (C3A) and ferroaluminate phases in cement are the main determining compositions influencing sulfate resistance of concrete [16,17]. As it is well known, the two recognized chemical reaction products of cement hydration are ettringite and gypsum. The formation of ettringite results in an increase in solid volume, leading to expansion and cracking if expansion is restrained. The formation of gypsum can lead to softening and loss of concrete mass and strength [15,16,18,19].

The mechanism of physical salt attack is not fully understood and some discussions on it are presented in literature [20–22]. The ACI [15] considers that sodium sulfate, sodium carbonate and sodium chloride dissolved in groundwater transport through the moist concrete and then concentrate and precipitate at the exposed surface. Only salt crystallization such as thenardite and mirabilite rather than the chemical sulfate products such as ettringite and gypsum are identified in physical salt attack, which differs from the scenario of chemical sulfate attack. Cases of concrete damaged by physical sulfate attack are sometimes misinterpreted as by chemical sulfate attack [23]. As reported by Skalny [24], complete separation of physical and chemical sulfate attack is probably technically impossible and may cause more confusion. However, in real environments, concrete durability is influenced by factors acting in a combined and possibly synergistic physical and chemical manner. Therefore, it is significant to study concrete performance under combined deteriorating factors to obtain sufficient information on concrete durability.

Yu et al. [25] investigated freezing–thawing durability of concretes under the attacks of a combination of external flexural stress and chemical solution. Their experimental results indicated that the freezing–thawing resistance of concrete was visibly reduced at the presence of flexural stress. Ganjian [26] revealed that different deterioration mechanisms make SF or GGBS cement to be more vulnerable in the magnesium sulfate bearing seawater particularly within tidal zone under wetting and drying cycles. Zivica [3] studied the influence of compression stress on sulfate corrosion rate of cement mortar and confirmed that the applied compression stress up to 60% of ultimate strength of mortar significantly slowed down this rate and the inhibition effect by decreasing bound  $\text{SO}_3$  content and the destructive phases, as ettringite and gypsum generated in concrete/cement paste attacked was connected. Mathias [27] pointed out that chloride penetration increased when the sulfate content increased at short immersion periods for OPC concrete and the presence of chlorides had a mitigating effect on the sulfate attack. According to Jiang [28], freezing–thawing cycles and sulfate attack affected each other and the deterioration of concrete with 20% by weight FA replacing OPC attacked by magnesium sulfate led to the most aggressive deterioration subjected to freezing–thawing cycles. The shotcrete durability under combined frost and sulfate attack was investigated [29] and the results revealed that ordinary shotcrete was more durable under the combined action of freezing–thawing cycles and sulfate ion attack than that of the ordinary concrete with the same mixture. Steel fiber reinforced shotcrete had the best durability performance in frost and sulfate resistance.

Investigations on frost and sulfate resistance of concrete had also been conducted in concrete science and engineering community, but the effects of sodium sulfate on the deterioration of concrete with FA and SF under combined freezing–thawing and sulfate attack was limited in literature. In this research, deterioration resistant coefficient of compressive strength, relative dynamic elastic modulus (RDEM) and Scanning Electron Microscopy analysis (SEM) of concrete subjected to sulfate attack and freezing–thawing cycles in water and in the sulfate solutions were conducted. The influence of FA and SF replacement level and

sodium sulfate concentration under the combined action of freezing–thawing and sulfate attack was also analyzed.

## 2. Experimental program

### 2.1. Materials

P.O.42.5R OPC conforming to Chinese standard GB175–2007 and similar to the 42.5 R Portland cement conforming to EN197–1:2009, was used for preparing concrete in this research. The physical and mechanical properties of the cement are listed in Table 1. Table 2 presents the chemical composition of FA and Table 3 the performance index of SF used in this study as partial replacement of OPC. Crushed limestone aggregates were used as coarse aggregates and washed mountain sand as fine aggregates. The fineness modulus of fine aggregates was tested according to Chinese standard JGJ52–2006 (similar to ASTM C136–01) and the results are tabulated in Table 4. Tap water was used for mixing concrete. A commercially available water reducer (i.e. SM) was used to keep concrete slump between 80 and 100 mm. Sodium sulfate anhydrous with 99% purity were used for making sulfate solutions as the sulfate attack source.

### 2.2. Mix proportion and specimen preparation

The actual mix proportions in terms of  $1\text{ m}^3$  concrete for the mixtures investigated in this study are given in Table 5. In order to investigate the effect of FA and SF on the resistance of concrete to sulfate and freezing–thawing attack, two groups of concrete mixtures were prepared with w/b ratios of 0.38 and 0.33, respectively. In each group, concrete mixtures with three different FA contents (i.e. of 10%, 15% and 25% by weight of cementitious materials (i.e. OPC + FA + SF)), three different silica fume contents (i.e. of 5%, 8% and 11% also by weight of cementitious materials) as partial replacement of OPC were prepared and tested.

Concrete mixtures were prepared by a single horizontal-axis forced mixer. After moulded, concrete specimens were placed in the curing room with temperature of  $(20 \pm 5)^\circ\text{C}$  for 24 h. Then they were demoulded and immersed in limewater with temperature of  $(20 \pm 2)^\circ\text{C}$  for another 27 days. For each mix, a total of 24 specimens were prepared, among which 15 specimens had the dimensions of  $100 \times 100 \times 100\text{ mm}^3$  for measuring compressive strength and resistance to sulfate attack, while the remaining 9 specimens with the dimensions of  $100 \times 100 \times 400\text{ mm}^3$  were prepared for assessing freezing–thawing resistance. For each test, the results presented were the average of the values obtained from three specimens under identical conditions.

### 2.3. Sulfate resistance assessment

For assessing sulfate resistance of concrete, cubic specimens with the dimensions of  $100 \times 100 \times 100\text{ mm}^3$  cured standardly for 28 days were immersed in designated solution (i.e. 5% sodium sulfate by wt%). Conforming to Chinese standard GB/T 50082–2009, which is similar to ASTM C1012–04 but it should be noted here that ASTM C1012–04 protocol only includes immersion but does not have drying–immersion cycle, a standard drying–immersion cycle lasted for 24 h and was proceeded as following: first the cubic specimens were immersed in the designated sodium sulfate solution for 15 h at  $(25–30)^\circ\text{C}$ ; then they were taken out from the solution and dried in air for 1 h; subsequently the cubic samples were heated up to  $(80 \pm 5)^\circ\text{C}$  to dry for 6 h and followed by cooling down in air at  $(25–30)^\circ\text{C}$  for another 2 h. PH value of the solution was measured once every 15 cycles and it was found

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