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Investigation of concrete exposed to dual sulfate attack

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ABSTRACT

Durability of concrete exposed to sulfates has primarily been studied on specimens fully-submerged in sulfate solutions. However, field experience shows that concrete exposed to sulfates can suffer from surface scaling above ground level due to physical attack. This damage has often been ignored and even confused with chemical sulfate attack. In this study, concrete partially-immersed in sulfate solutions and exposed to cyclic temperature and relative humidity was explored. Results show that concrete can experience dual sulfate attack. The lower immersed portion can suffer from chemical sulfate attack, while the upper portion can be vulnerable to physical attack. Lowering the water-to-binder ratio and moist-curing reduced surface scaling above the solution level, since the volume of pores was decreased. Although partial replacement of cement with pozzolans also decreased the pore volume, surface scaling increased due to increased proportion of small diameter pores and associated growth of capillary suction and surface area for evaporation.

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1. Introduction

Deterioration of rocks and stones due to salt crystallization within their pores has long been a subject of dedicated study [10]. Several studies have explored the associated deterioration mechanisms and how they can be mitigated since it is a major threat to historical monuments and building stones. However, the deterioration of concrete due to salt crystallization, or the so called physical sulfate attack, has been ignored and often confused with chemical sulfate attack [11,12,15].

According to Scherer [19] concrete can be vulnerable to damage when salt crystals grow from a supersaturated solution in its pores. This process was described as a physical attack on concrete since, unlike the chemical sulfate attack; it does not involve chemical interactions between sulfate ions and the concrete hydration products [11]. Moreover, the consequences of physical sulfate attack on concrete are different from that of chemical sulfate attack since it leads to surface degradation similar to that caused by cycles of freezing and thawing. Conversely, chemical sulfate attack usually results in expansion and cracking due to the formation of ettringite and gypsum [15].

Field experience with concrete exposed to sulfates has often shown that concrete mainly suffers from surface scaling caused by physical sulfate attack. Such damage is generally limited to the above-ground portion of concrete, while the portion embedded in the sulfate rich soil, which is directly exposed to chemical sulfate attack, was mostly found in intact condition [14,20,24]. The damage process involves capillary rise and evaporation of the ground water containing sulfates at the

above ground concrete surface, resulting in crystal growth in concrete pores and subsequent damage [11,14].

Nevertheless, current standards that evaluate the performance of concrete under sulfate attack, such as ASTM C1012 (Standard Test Method for Length Change of Hydraulic-Cement Mortars Exposed to a Sulfate Solution) and CSA A3004-C8 (Test Method for Determination of Sulfate Resistance of Mortar Bars Exposed to Sulfate Solution) only deal with the chemical aspect of sulfate attack. Indeed, they only evaluate the behavior of cement mortars when fully immersed in a sulfate solution [6,18]. This may contribute to further confusion in the assessment of concrete deterioration due to sulfates under field exposure.

Several studies have shown that the vulnerability of stones to damage by physical salt weathering depends on their pore structure [1,8,19]. For instance, stones that include higher volume of micro-pores connected with larger pores are the most vulnerable to damage by salt crystallization [1,17,23]. The presence of micro-pores increases the capillary rise and the surface area of evaporation, leading to higher supersaturation of the pore solution and subsequently more damage [17].

In the present study, concrete specimens were partially immersed in a sulfate solution to create an evaporation front, similar to that occurring in field exposure. The influence of the pore structure on the deterioration of concrete due to physical sulfate attack was investigated. Several factors that control the pore structure of concrete including the water/binder (w/b) ratio, adding pozzolanic minerals, and the curing regime were considered.

2. Research significance

There is lack of information and often conflicting data regarding the performance of concrete exposed to physical sulfate attack. Thus, in this

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study, the effects of different mineral additives, w/b ratios, along with various curing conditions on the performance of concrete exposed to severe physical sulfate attack were investigated. The findings should demystify the role of these parameters on physical sulfate attack, allowing to gain a more fundamental understanding of the associated damage mechanisms, which could enhance the durability design of concrete in sulfate laden environments and possibly prevent some of the associated litigation.

3. Experimental program

3.1. Materials and specimen preparation

Three groups of concrete mixtures with different w/b ratio (i.e. 0.30, 0.45, and 0.60) were tested. In each group, five binder types were used including: ordinary portland cement (OPC), high sulfate resisting cement (HS), OPC with 8% silica fume (SF), OPC with 25% class F fly ash (FA), and OPC with 8% metakaolin (MK). The physical and chemical properties of the cements, mineral additives, and aggregates are summarized in Tables 1 and 2. The proportions of the concrete mixtures are provided in Tables 3. For each of the fifteen concrete mixtures, standard cylinders $100 \times 200 \text{ mm} (4 \times 8 \text{ in.})$ were cast according to ASTM C192 (Standard Practice for Making and Curing Concrete Test Specimens in the Laboratory).

3.2. Mechanical properties

Compressive strength according to [4] (Standard Test Method for Compressive Strength of Cylindrical Concrete Specimens) and static modulus of elasticity according to [5] (Static Modulus of Elasticity and Poisson's Ratio of Concrete in Compression) were measured for the cured concrete cylinders partly immersed in sulfate solutions.

3.3. Curing conditions

In general, damage of concrete structures in sulfate rich soils (e.g. foundations and retaining walls) can start at an early-age before concrete is fully cured. The volume of capillary pores is typically high in concrete at earlier age compared with that of the fully cured concrete. Hence, more sulfates can penetrate into the concrete, leading to higher damage. Therefore, in the present study, both the performance of non-cured and cured concrete exposed to physical sulfate attack have been investigated. For the non-cured concrete, a group of cylinders from each mixture were exposed to the sulfate environment after 24 h

Table 1

Physical and chemical properties of various binders used in this study.

Table 2

Physical and chemical properties of fine and coarse aggregates.

Property	Coarse aggregate	Fine aggregate	
Potential alkali reactivity	0.05	_	
(mortar-bar method) (%)			
Absorption (%)	1.11	1.09	
Crushed particles (%)	68.00	-	
Flat/elongated (%)	6.00	-	
Micro-deval (A) (%)	11.00	17.00	
Soundness (freeze-thaw) (%)	2.20	-	
Soundness (MgSO ₄) (%)	3.90	-	
Specific gravity (apparent) (%)	2.73	2.73	
Specific gravity (dry) (%)	2.65	2.65	
Specific gravity (SSD) (%)	2.68	2.68	
Unit weight (kg/m ³)	1734	1512	
Materials finer than 75-µm (sieve # 200) (%)	0.90	2.10	

from casting. Another identical group of concrete cylinders from each concrete mixture was cured for 28 days in a moist room with $RH \ge 95\%$ and T = 20 °C [68 °F] before exposure to the sulfate environment. The curing was carried out according to ASTM C511 (Standard Specification for Mixing Rooms, Moist Cabinets, Moist Rooms, and Water Storage Tanks Used in the Testing of Hydraulic Cements and Concretes) [2].

3.4. Environmental exposure conditions

According to Thaulow and Sahu [21], the most commonly found salt on scaled concrete surfaces exposed to environments conducive to physical sulfate attack is sodium sulfate. Previous studies by Aye and Oguchi [6], and Haynes et al. [12,13] found higher surface scaling for 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 (i.e. similar temperature, relative humidity (RH), and sulfate concentration). In addition, Haynes et al. [12] found that surface scaling escalated drastically when the concrete was exposed to cyclic temperature and RH consisting of two weeks at temperature = $20 \degree C [68 \degree F]$ and RH = 82% followed by two weeks at temperature = 40 $^{\circ}$ C [104 $^{\circ}$ F] and RH = 31%. Therefore, all concrete cylinders were partially immersed in a 5% sodium sulfate solution and placed inside a walk-in environmental chamber with cycling temperature and RH. To accelerate the experiment, cycles were reduced to one week at temperature = 20 °C [68 °F] and RH = 82%followed by one week at temperature = 40 °C [104 °F] and RH = 31%.

Components/property	Cement Type (10)	Cement Type HS	Silica fume	Metakaolin	Fly ash
Silicon oxide (SiO ₂) (%)	19.6	22.0	95.3	52.2	43.39
Aluminum oxide (Al_2O_3) (%)	4.8	4.1	0.2	41.0	22.1
Ferric oxide (Fe ₂ O ₃) (%)	3.3	4.4	0.1	1.8	7.7
Calcium oxide (CaO) (%)	61.50	64.90	0.49	-	15.63
Magnesium oxide (MgO) (%)	3.00	1.10	0.27	-	
Sulfur trioxide (SO ₃) (%)	3.50	2.25	0.24	0.04	1.72
Loss on ignition (%)	1.90	0.70	1.99	1.10	1.17
Insoluble residue (%)	0.44	0.08	-	-	-
Equivalent alkalis (%)	0.7		-	-	-
Tricalcium silicate (C ₃ S) (%)	55	57	-	-	-
Dicalcium silicate (C ₂ S) (%)	15	20	-	_	-
Tricalcium aluminate (C ₃ A) (%)	7	3	-	-	-
Tetracalcium aluminoferrite (C ₄ AF) (%)	10	13	-	-	-
Blaine fineness (m ² /kg)	371	380	-	-	-
Autoclave expansion (%)	0.09	-0.01	-	-	-
Compressive strength 28 days (MPa)	40.9	44.8	-	-	-
Specific gravity	3.15	3.12	2.58	2.20	2.50
Time of setting (min) Vicat initial	104	225	-	-	-

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