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Resistance of concrete against combined attack of chloride and sulfate under drying–wetting cycles

Yanjuan Chen ^{a,b,*}, Jianming Gao ^{a,b}, Luping Tang ^c, Xuehong Li ^{a,b}^a Department of Materials Science and Engineering, Southeast University, Nanjing 211189, China^b Key laboratory of Construction Materials, Southeast University, Nanjing 211189, China^c Chalmers University of Technology, Gothenburg 41296, Sweden

HIGHLIGHTS

- Durability of concrete exposed to combined solution under drying–wetting is investigated.
- Higher SO_4^{2-} contend in combined solution retarded damage for OPC under drying–wetting cycles.
- MIP, XRD and TG/DSC were used to study microstructure of interior concrete.
- DSC/TG could quantified the results from XRD-analyses of concrete.

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ABSTRACT

In this research, combined solution of chloride and sulfate was investigated for three different mixtures, including fly ash and slag. Mass change, dynamic modulus of elasticity, chloride penetration depths were tested in the different solution.

Results indicated that OPC specimens showed bigger deterioration compared specimens with fly ash and slag exposed to combined solution attack under drying–wetting cycles. Additional, higher sulfate contend in combined solution retarded damage for OPC under drying–wetting cycle.

Concerning the microstructure and deformation of samples, XRD-analyses is in accordance with TG/DSC-analyses, moreover, DSC/TG could quantified the results from XRD-analyses.

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1. Introduction

Marine environments are very aggressive to concrete structures that affect the long-term durability of concrete structures and cause huge economic loss, since sea water contains high concentrations of chlorides and sulfates. Furthermore, concretes are exposed to repeated drying–wetting cycles, such as in the marine environment of splash and tidal zone which can accelerate the deterioration of structures. A lot of research works have been reported on concretes exposed to environmental actions, such as sulfate attack [1–4], combined attack of chloride and sodium sulfate [5–7], sulfate attack under drying–wetting cycles or flexural loading [8–10], chloride attack under drying–wetting cycles [11] or flexural loading [12,13], and coupled action of flexural loading and drying–wetting [6]. However, studies of the influence of

chlorides and sulfates ions under drying–wetting cycles, which is closer to actual marine environments, are limited in the literature. This paper presents the study of degradation process of concrete exposed to combined attack of chloride and sodium sulfate and the influence of SO_4^{2-} and Cl^- under drying–wetting cycles. Mass changes and dynamic modulus of elasticity (E_{rd}) for 300 days were measured for evaluating the durability of concrete mixtures. The microstructures and corrosion products of interior concrete were analyzed using X-ray diffraction (XRD), thermogravimetric analysis and differential scanning calorimetry.

2. Materials and methods

2.1. Materials and mix proportions

Materials used in this study consisted of ordinary Portland cement (P. II 52.5 according to Chinese standard), Fly ash (FA) and grounded blast furnace slag (GBFS). The chemical and mineral compositions are summarized in Table 1. River sand with fineness modulus of 2.6 and coarse aggregate of crushed limestone with a

* Corresponding author at: Department of Materials Science and Engineering, Southeast University, Nanjing 211189, China.

Table 1
Chemical compositions and mineral compositions of used materials.

Material	Chemical compositions (wt.%)									Mineral compositions (wt.%)			
	CaO	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MgO	SO ₃	K ₂ O	Na ₂ O	LOI	C ₃ S	C ₂ S	C ₃ A	C ₄ AF
Cement	62.53	20.75	4.78	3.45	1.11	2.64	0.58	0.07	3.26	53.07	19.47	9.46	10.49
FA	5.75	51.07	30.86	5.26	2.72	1.48	1.13	0.79	2.80	–	–	–	–
GBFS	36.35	33.48	12.21	1.40	10.60	0.66	0.56	1.27	0.36	–	–	–	–

maximum size of 20 mm were used. A polycarboxylate-type super-plasticizer with a water-reducing rate of 25% by weight was used. The mix proportion is given in Table 2, where the mix code C50F30 means concrete specimen with the binder replaced by FA at the dosage of 30% by weight, while the C50K50 means mortar specimen with the binder replaced by GBFS at the dosage of 50%.

Concrete specimens were cast in steel molds of 70 × 70 × 280 mm, demolded 24 h after casting, and cured in the condition of 20 ± 3 °C and 95% relative humidity for a certain days. Except for the two opposite vertical surfaces (70 × 280 mm), other four surfaces of the specimen were covered with epoxy resin before the exposure experiments.

To examine the degradation process of concrete exposed to combined attack of chloride and sodium sulfate under drying–wetting cycles, three different paste mixtures were prepared with the same binder types as used in the concrete mixes and cured in the condition of 20 + 3 °C and 95% relative humidity until the age of testing. The paste samples were produced for X-CT, XRD and TG/DSC analyses.

2.2. Experiment programs

In the present study, combined solutions of sodium chloride and sodium sulfate were used as exposure solutions. Concrete specimens were first immersed in the exposure solutions at the room temperature for 21 h, followed by 3 h drying in the air. Then, they were dried at a temperature of 60 °C for 45 h, followed by cooling down in the air at the room temperature for 3 h. This period of three days (72 h) represents a drying–wetting cycle. This drying–wetting cycle was repeated until a specified age.

2.3. Testing parameters

2.3.1. Dynamic modulus of elasticity

The dynamic modulus of elasticity (E_{rd}) of the specimen was measured by using a nonmetal ultrasonic analyzer (NM-4A, transducer frequency is 50 KHz, and test distance is more than 100 mm), which was usually used to detect the internal defect and damage degree of the concrete specimen [13].

2.3.2. Mass change

The mass change of the specimen during a certain testing age was monitored as a possible indicator of degradation. Mass of the specimens before (m_0) and after (m_n) immersion in various aggressive environments were measured on an electronic scale with a capacity of 5 kg and an accuracy of 0.01 g. Consequently, the mass change (ΔW) was calculated as follows

$$W = \frac{m_n - m_0}{m_0} \times 100\% \quad (1)$$

2.3.3. Total chloride content

The resistance of concrete to chlorides was evaluated by the modified diffusion test in accordance with reference [14], which is a modified version of Chinese standard JJT270-98 (Chemical analysis). Powder was collected from the specimens at the different depths by the means of drilling and passed the 0.15 mm sieve. Then, powder was dried in an oven at a temperature of 105 °C for 3 h, followed by taking about 2 g powder (m) using an electronic scale (capacity of 200 g, with a precision of 0.0001 g) and mixing the powder with about 50 ml dilute nitric solution (mixed with concentrated nitric acid and distilled water by volume ratio 15:85, and the consumed volume of distilled water is V_3) for 24 h in conical flask as chloride

extraction solution. The titration solution consisted of 10 ml chloride extraction solution (V_2) and 0.01 mol/L AgNO₃ solution about 10 ml (V) with 1–2 ml glycerol. Immediately, the solution was titrated by Potassium thiocyanate (KSCN) solution until it turned to red color and kept 5–10 s, and the consumed volume of KSCN is V_1 . The extraction and titration method was repeated on every set of samples. Total chloride content (P) was calculated using Eq. (2)

$$P = \frac{0.03545(C_{AgNO_3} \cdot V - C_{KSCN} \cdot V_1)}{m \cdot \frac{V_2}{V_3}} \times 100\% \quad (2)$$

3. Results and discussion

3.1. Mass change

Fig. 1 shows the deterioration surfaces of concrete samples used for mass change and relative elasticity modulus for 200 days. Concerning the surface scaling conditions, C50 samples exposed to 5% Na₂SO₄ solution under drying–wetting showed obvious deterioration on the surface compared with the other samples (in Fig. 1(c)). However, no big differences were observed by visual inspection from the other samples.

The mass changes of different specimens are presented in Fig. 2 (5%NaCl + 5%Na₂SO₄ solution, under drying–wetting cycles). As Fig. 2 shown, OPC specimens in the corrosive condition revealed a rise in mass before 200 days, while for the C50F30 and C50K50 specimens showed mass loss trends, this finding is in accordance with the finding of Tikalsky and Carrasquillo [15]. Some researchers consider such a mass gain for OPC as associated with water imbibition during the hydration process and concrete microstructure change with time [16]. The continuous cement hydration in the specimens consumed pore fluid, leaving the specimens in an unsaturated state. It also resulted in more C–S–H, which has a large surface area to adsorb water. Therefore the specimens continued to gain mass until maximum saturation was reached. In addition, mass gain was also resulted from precipitation on the surface of pore walls and inside the pore space [17]. The mass loss for C50F30 and C50K50 were associated with acidic-type interactions between sulfate ions. The pozzolanic reaction products also lead to mass loss, for the reason that high aluminum containing fly ash and blast furnace slag, which produce alumina-bearing hydrates, such as calcium monosulfoaluminate and calcium aluminate hydrate, that are attacked by sulfate ion to form ettringite and calcium trisulfoaluminate [18]. As it can be observed, OPC specimens showed the maximum mass gain of 1.75%, whilst mass loss for C50F30 and C50K50 specimens reached maximum of 0.75% when exposed to combined attack of chloride and sulfate under

Table 2
Proportions of concrete mixture prepared.

Mix	Binder (kg/m ³)			Fine aggregate (kg/m ³)	Coarse aggregate (kg/m ³)	Water (kg/m ³)	Super plasticizer (kg/m ³)	w/b
	Cement	FA	GFS					
C50	448	–	–	673	1121	157	4.03	0.35
C50F30	314	134	–	673	1121	157	4.03	0.35
C50K50	224	–	224	673	1121	157	4.03	0.35
P35	1494	–	–	–	–	523	–	0.35
P35F30	1046	448	–	–	–	523	–	0.35
P35K50	747	747	–	–	–	523	–	0.35

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