



Accelerated sulfate attack on mortars using electrical pulse



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HIGHLIGHTS

- The effect of electrical pulse on sulfate attack was investigated.
- Electrical pulse greatly accelerated the migration of sulfate ions into mortars.
- Electrical pulse accelerated the sulfate attack on mortars.
- Electrical pulse could be a novel method for rapidly evaluating the sulfate resistance of cement-based materials.

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ABSTRACT

In this study, the sulfate attack on mortars was accelerated using electrical pulse as an external electrical field. Meanwhile, the specimens with the same mix proportions were immersed in sulfate solution as comparison. The sulfate concentration and mechanical properties, including flexural and compressive strengths, were measured to evaluate the effects of sulfate attack. In addition, the transformation of microstructural nature and phase of the specimens were evaluated by scanning electron microscope and X-ray diffraction. The results showed that electrical pulse greatly accelerated the penetration of sulfate ions into the mortars and resulted in more ettringite and gypsum formed, leading to a greater depth of sulfate reaction. In electrical pulse test, the sulfate content in $MgSO_4$ solution was greater than that in Na_2SO_4 solution, which was the opposite case in immersion test. Moreover, the electrical pulse resulted in significant mechanical strength losses in a much shorter period as compared with those in immersion test. These findings indicated that the electrical pulse accelerated sulfate attack could be a new acceleration test for evaluating the sulfate resistance of cement-based materials.

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1. Introduction

Deterioration of cement-based materials by sulfate attack is a serious problem for durability. The mechanism of sulfate attack is complex as it depends on various factors, e.g. water–cement ratio, type of cement, presence of mineral admixtures and exposure temperature, associated cation and sulfate concentration [1–6]. The best known mechanism involves the cement hydration products, particularly the hydration products of C_3A and portlandite, and sulfate ions [7,8]. The first stage is based on a diffusion–reaction phenomenon: sulfate ions diffuse into the material, and the penetrated sulfates react with portlandite to form gypsum, which can react further with the hydration products of C_3A to form ettringite. Both the formation of gypsum and ettringite are expansive. The second stage is that the increase in the expansive products results in swelling, damage and finally the strength loss of

cement-based materials. It is obvious that the degree of deterioration of cement-based materials due to sulfate attack primarily depends on the rate of sulfate ions passing through the specimen. However, the sulfate attack is a slow process because of the relative dense structure of cement-based materials, which restricts diffusion of sulfate ions. In order to obtain reliable information about the deterioration effects of sulfate attack, long test duration is always needed.

The research and application of using an electrical field to accelerate the ingress of chloride ions in concrete had been extensively reported [9–13]. Similarly, sulfate ion is an anion, it is feasible to employ an electrical field to speed up the migration of sulfate ions and then accelerate sulfate attack. However, there were few reports using electrical field to investigate the accelerated sulfate attack. Lorente et al. [14] presented a comparison of sulfate concentrations in immersion test and electrical field test, and found that the electrical field accelerated the migration of sulfate ions in concrete. Nevertheless, the sulfate products in concretes under the two experimental conditions were not compared.

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The acceleration of sulfate attack on mortars using electrical field was investigated in this study. Since the heat produced under high-voltage direct current (such as 60V in ASTM C1202-12 [15]), has a negative effect on the microstructure of cement-based materials [16], the electrical pulse with a lower voltage of 30 V was employed in this investigation. During electrical pulse, the periods with no voltage could be helpful in the heat loss. For comparison, the specimens with the same mixture were immersed in the corresponding sulfate solutions. The performance was evaluated through measurement of sulfate concentration and mechanical resistances, along with microstructural and mineralogical analysis.

2. Experimental procedure

2.1. Raw materials and mortar specimens

The cement used in this study consists of 95% of clink and 5% of gypsum by weight and their chemical composition is presented in Table 1. The specific gravity and fineness modulus of the quartz sand were 2.52 and 2.70, respectively. For the mortar mixtures, the sand: cement: water ratio was 3:1:0.5 by mass.

2.2. Electrical pulse test

As shown in Fig. 1, the specimens in electrical pulse test were cast into the middle part (40 mm × 40 mm × 160 mm) of H-shaped molds and immediately stored under a controlled condition of about 20 °C and 98% RH. Twenty-four hours later, the iron sheets used in mixing were pulled out and the specimens along with the molds were re-stored in the moist room. After 27 days, the upper surface of specimens was coated with Vaseline to prevent from carbonization and sulfate solution was added to both ends of the special molds. Two titanium rods used as electrodes for applying an external electrical field were placed with the left-hand side of cathodic compartment and the other side of anodic compartment, respectively. Fig. 2 presents the schematic drawing of electrical-pulse test. The electrical pulse cycle as external electrical field consists of a pulse of positive voltage of 30 V (20 s), followed by a period (20 s) when no voltage is applied, as shown in Fig. 3. Both sodium sulfate and magnesium sulfate solutions were used as test agents, each of them corresponding to a sulfate concentration of 33.8 g/l. The duration of the tests was varied from 15 days to 3 months depending on the quality of mortars.

In order to compare this accelerated method with the immersion test, the specimens with the same mix proportion were immersed in the sulfate solutions. The test duration was 6 months. Moreover, a controlled series immersed in water was examined as reference as well.

2.3. Sulfate concentration

The sulfate concentration profiles were tested by grinding the specimens with the grinding steps of 0.5 cm on both sides of specimens and 1 cm on the middle parts. The sulfate concentration, represented by SO₃ content (% by weight of mortar), was measured by the barium sulfate gravimetric method (chemical titration), according to a Chinese standard GB/T 50476-08 [17]. In this test method, at first 5 g of finely ground mortar sample dissolves in 100 ml of acid solution, and then sulfate is precipitated from the acid solution with barium chloride (BaCl₂). The precipitate is ignited and weighed as barium sulfate (BaSO₄) and the SO₃ equivalent is calculated. It should be noted that the measured sulfate concentration contains the sulfate in the solution and that in the solid phase.

The initial sulfate content was measured at 1.18% and it refers to the existence of sulfate in the mortar before sulfate attack, mainly from clink and gypsum in the cement. The total sulfate content refers to the sum total of external sulfate ions migrating into the specimen and the initial sulfate content. Generally, the initial sulfate content is equally distributed in the mortar specimen. Thus, the total sulfate content can reflect the amount of the penetrated sulfate ions from the external environment.

Once the sulfate concentrations at different depths are obtained, the ionic distribution profile in the specimen is plotted. In the sulfate concentration profile, the depth of sulfate can be identified and it refers to the distance from the surface of the

specimen in contact with sulfate solution to the face where the total sulfate content is almost equal to the initial sulfate content. Generally, a greater depth of sulfate means a greater depth of sulfate reaction within the specimen.

2.4. Mechanical properties

The progress of sulfate attack was evaluated by measuring the mechanical properties including flexural and compressive strengths. Relative flexural and compressive strengths were evaluated by the flexural and compressive strengths of the specimens under sulfate attack at a certain test age as compared to those of the specimens cured in water at the same test age.

2.5. Changes of microstructure and phase composition

The changes on microstructure of the specimens were examined by scanning electron microscope SEM (TESCAN VEGA 3 LMH) equipped with energy dispersive X-ray spectroscopy (EDS). Fragments of broken prisms were powdered to establish the mineralogical composition by X-ray diffraction (XRD, Phillips X'Pert, Cu K α). More details about the sample preparation and testing of the two techniques can be found in the previous study [18].

3. Results and discussion

3.1. Sulfate concentration profiles

Fig. 4 presents the sulfate concentration profile through the specimen after applying electrical pulse for 3 months. Both cathodic and anodic compartments were filled with 42.4 g/l of magnesium sulfate. The sulfate concentrations were tested from the face in contact with the cathodic compartment. The total amount of sulfate at the center (8 cm) was greater than the initial sulfate content, indicating that the sulfate reaction already occurred in the core of mortar. Note that the total sulfate concentrations close to the cathodic compartment was significantly higher than those close to the anodic compartment, which could be explained by the influence of the electrical field [15]. Sulfate ions penetrated into the specimen from both sides because of the sulfate concentration gradient between the outside sulfate solution and the pore solution. Being anions, sulfate ions would be attracted by the anodic side when the electrical field was applied. Thus, in the cathodic compartment, sulfate ions migrated inside the specimen by two driving forces: the sulfate concentration gradient and the electrical potential difference. However in the anodic compartment, the same two driving forces were competing: the sulfate concentration gradient forced sulfate ions to enter the specimen, while the electrical potential difference tended to prevent the ingress of the ions. The result of the competition was a smaller amount of the total sulfate in the vicinity of the surface of material in contact with the anodic compartment, compared to that by the cathodic side.

Fig. 5 presents the sulfate profiles during immersion and electrical pulse test after 1 and 3 months. Both cathodic and anodic compartment contained magnesium sulfate solution. The concentration profiles were also measured from the cathodic side. The specimens in the two tests had the similar sulfate-ion content distribution regularity: the amount of sulfate ions was largest near the surface and decreased with the settlement depth, indicating that the sulfate reaction propagated from the surface towards the core of the material. It was also expressed from Fig. 5 that the content and depth of sulfate in the specimen subjected to electrical pulse was markedly higher than those in immersion test. For

Table 1
Chemical composition of clinker and gypsum.

Materials	Chemical composition (wt.%)								
	SO ₃	SiO ₂	Fe ₂ O ₃	Al ₂ O ₃	CaO	MgO	K ₂ O	Na ₂ O	LOI
Clinker	2.23	19.99	2.98	4.80	61.22	3.27	0.88	0.18	3.52
Gypsum	40.61	4.47	0.36	0.99	34.05	1.84	0.23	0.08	16.87

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