Deterioration of mortars exposed to sulfate attack under electrical field

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Abstract: The damage process of plain and blended cement mortars subjected to sulfate attack under electrical field was investigated in this paper. The deterioration of mortars was investigated by measuring the loss of compressive strength. To identify the changes of microstructure and mineral phases after the test, scanning electron microscopy, energy dispersive spectroscopy and X-ray diffraction analysis were performed on the selected samples. The results indicated that compared with sulfate attack alone, the combination of sulfate attack and electrical field accelerated the damage process of mortars, which can be explained through two different mechanisms. On the one hand, Ca^{2+} ions directionally moved and then leached out from mortars under the electrical field, leading to the dissolution of portlandite as well as the decomposition of C-S-H gel at the later stage. On the other hand, the electrical field accelerated the migration of sulfate ions and then they reacted with hydration products to form massive ettringite and gypsum, which resulted in the microcracks and strength loss of mortars. Moreover, the deterioration of the mortar blended with fly ash was still visible in spite of its better chemical resistance. The strength loss of limestone powder incorporated mortar increased in comparison with Portland cement mortar as a result of an increase in porosity.

Key words: Deterioration; Sulfate attack; Electrical field; Compressive strength; Microstructure

1. Introduction

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1 External sulfate attack is one of the key durability issues for cement-based materials and it has
2 been investigated extensively for many decades. The entering sulfate ions can react with hydrated
3 cement composites to produce gypsum and ettringite, which can cause expansion, cracking and
4 spalling.

5 There are many reports on the damage process of concrete exposed to single sulfate attack
6 through the sulfate immersion tests [1-5]. Nevertheless, there has often been the different
7 performance of concrete in immersion test from that observed in a field exposure. As a result,
8 researchers have also investigated the deterioration mechanism of concrete exposed to sulfate
9 attack couple with other parameters existing in the field, such as drying-wetting cycles [6-8],
10 flexural loading[6,7,9-11] and freeze-thaw cycles [8,12].

11 In the last decade, the urban rail transit systems have been developing rapidly in China and
12 other developing countries. In these systems, stray current, which originates from the electrical
13 railway line and then flows through the reinforced or prestressed concrete, could accelerate the
14 deterioration of concrete structure. Bertolini et al. [13] investigated the corrosion behavior of steel
15 in concrete in the presence of stray current, reporting that stray current initiated corrosion on
16 rebars embedded in cement-based materials and remarkably increased the corrosion rate on
17 corroded reinforcement bars in the chloride-contaminated concrete. Solgaard et al. [14] also found
18 that stray current was able to promote corrosion initiation on steel fibers embedded in mortar, and
19 the corrosion rate showed a significant increase in the specimens with 4% and 6% of chloride by
20 mass of cement after the exposure to stray current. Saito et al. [15] studied the leaching tests on
21 different mortars using an electrochemical method, indicating that electrical field accelerated the
22 dissolution of Ca^{2+} ions from the mortar in contact with water, leading to the increase of water

1 permeability and reduction in compressive strength of specimens. However, to the authors'
2 knowledge, the effect of stray current or electrical field on sulfate attack induced deterioration of
3 concrete has not been investigated so far, although sulfate attack is one of the main deterioration
4 mechanisms of underground infrastructures.

5 In this study, a low-frequency pulsed electrical field was applied on mortar specimens exposed
6 to sulfate solution, to simulate the effect of stray current. The coupling effect of sulfate attack and
7 electrical field on the microstructure and compressive strength of mortars was investigated, and
8 the corresponding mechanism was analyzed. In addition, the influence of fly ash and limestone
9 powder on the damage of mortar was evaluated.

10 **2. Experimental details**

11 *2.1 Materials and mix proportions*

12 The Portland cement (PC) used in this investigation consisted of 95% of clinker and 5% of
13 gypsum by weight. Fly ash (FA), limestone powder (LP, 95 wt.% CaCO_3) and siliceous sand with
14 the fineness modulus of 2.70 were used. The chemical composition of clinker, gypsum, FA and
15 LP is shown in Table 1. The mix proportions of mortar are given in Table 2. 30% FA and 30% LP
16 were used as a replacement of cement by weight in the FA-30 and LP-30 mortar mixtures,
17 respectively. In all mixtures, the binder: sand: water ratio was kept constant as 1:3:0.5 by weight.

18 Each of PC, FA-30 and LP-30 mortars has 10 groups of specimens (3 specimens in each group):
19 3 groups for water immersion, 3 groups for sulfate immersion and 4 groups for combined test of
20 sulfate attack and electrical field. Values of compressive strength and SO_3 concentration reported
21 in this paper are all average over three specimens.

22 *2.2 Experimental procedures*

1 Generally, the dimension of mortar specimens is 40 mm × 40 mm × 40 mm for compressive
2 strength measurement in Chinese standard. However, the specimens in this work need to be
3 clamped for better cutting before SO₃ concentration measurement and the length of the clip is
4 about 20 mm. Therefore, the dimension of specimens in this paper is determined to 40 mm × 40
5 mm × 60 mm.

6 Mortar mixtures were cast into the middle part of special molds (as shown in Fig. 1). 24 hours
7 later, the plates were moved out and then the specimens along with molds were cured in a
8 condition of 20±2 °C and above 95% of relative humidity.

9 After additional 27 days of curing, the upper surface of specimens was coated with Vaseline to
10 prevent from carbonation. Then, a 5% Na₂SO₄ solution and a 2.8% NaOH solution were added
11 into the cathodic and anodic cells, respectively. The combined action test of sulfate attack (SA)
12 and electrical field (EF) is shown schematically in Fig. 2. The principle and idea of the
13 experimental device design were derived from the measurement of chloride diffusion coefficients
14 in a migration test using electrical field. In consideration of stray current's non-stationary nature
15 [13], a low-frequency pulsed electrical field was employed in this investigation. The voltage
16 applied was 30 volt, and the pulse cycle was 40 seconds during which both the circulation and
17 interruption periods were 20 seconds [16]. For comparison, the specimens with the same mixture
18 were immersed in the corresponding sulfate solution (namely sulfate attack alone) and water,
19 respectively. It should be noted that, under the combined condition, only one of the end surfaces
20 (40mm × 40mm) was exposed to sulfate solution. Therefore, one surface (close to sulfate solution)
21 of the specimen was also exposed in immersion test, while the other five surfaces were sealed by
22 Vaseline.

2.3 Test methods

2.3.1 Sulfate and calcium concentrations

After slicing and grinding the samples, the sulfate-ion content, represented by SO_3 content (wt.%, by weight of mortar), was determined by barium sulfate gravimetric method (chemical titration), according to GB/T 50476-2008 [17].

It should be noted that the measured sulfate content represents the total content, which refers to the sum total of external sulfate ions migrating into the specimen and the initial sulfate content.

The initial sulfate content is the sulfate existing in a mortar from the raw materials before the test.

They were measured at 1.18%, 0.99% and 0.84%, respectively for PC, FA-30 and LP-30 specimens. Thus, the migrated sulfate concentration can be calculated as the difference between the measured total and initial sulfate content.

The CaO content in mortars and the dissolved Ca^{2+} ions concentration into the cathodic compartment were measured by EDTA titration method, according to GB/T 176-2008 [18] and GB/T 15452-2009 [19], respectively.

2.3.2 Microstructure analysis

The surface part (0-20 mm depth from the exposed surface) of selected samples was vacuum dried at 50 °C for 48 h, and then coated with gold, finally examined by scanning electron microscopy (SEM, TESCAN VEGA 3 LMH with an acceleration voltage of 20 KV) equipped with an energy dispersive spectroscopy (EDS) detector. The average Ca/Si molar ratio of C-S-H gel was determined from 20 different measurement points through EDS analysis. More details about the measurement strategy can be found in the previous studies [20-22]. In addition, X-ray diffraction (XRD, PANalytical Empyrean system using $\text{CuK}\alpha$ radiation, operating at 60 kV and 55

mA) was performed on powder samples (particle size $\leq 75\mu\text{m}$) to identify any compounds formed during the test.

2.3.3 Compressive strength

The damage of mortar specimens was investigated by measuring the relative compressive strength, which was calculated as follows:

$$R = f_{\text{attack}} / f_o \times 100\%$$

Where, R was the relative compressive strength; f_{attack} was the average compressive strength of specimens after the test, MPa; f_o was the average compressive strength of specimens cured in water for the same period, MPa.

3. Results and discussion

3.1 SO_3 concentration

Fig. 3 shows the migrated sulfate content of PC mortar after immersion and combined exposures. After 90 days of immersion, the specimen showed a clear increase of SO_3 near the exposed surface, and the sulfate depth and maximal SO_3 content was about 1.0 cm and 0.84%, respectively. Nevertheless, the sulfate content and depth after 30 days of combined exposure were significantly greater than those in immersion exposure. Up to 90 days, the SO_3 content progressed further into the sample with a maximal SO_3 concentration around 2.84%. The clear difference of SO_3 profiles between immersion and combined conditions indicated that the electrical field markedly sped up the migration of sulfate ions into the specimen.

The migrated sulfate contents of FA-30 and LP-30 specimens during the test are plotted in Figs. 4 and 5, which followed a similar trend to that of PC specimen.

The SO_3 content profiles of PC, FA-30 and LP-30 specimens under combined exposure after 90

days are further compared in Fig. 6. FA-30 mortar showed reductions in sulfate depth and content compared with PC mortar. This was attributed to that the pore refinement process occurred in FA-30 mortar due to the pozzolanic reaction of fly ash [23-25], which had a certain limitation for the penetration of sulfate ions. Additionally, there was higher sulfate content in the mortar blended with 30% limestone fillers (LP-30). Marzouki et al. [26] reported that the porosity of mortars (W/B=0.5) gradually increased along with a number of limestone powder content from 0 wt.% to 30 wt.% present in cement due to the increase of effective water/cement ratio and the negligible activity of limestone powder. Consequently, sulfate ions were easier to penetrate into LP-30 specimen in comparison with PC specimen [27].

3.2 CaO and dissolved Ca^{2+} content

Fig. 7 presents the CaO content collected from the surface (0-20mm depth from the exposed surface) of specimens before and after 90 days of the test. The contents of CaO before the test were 12.92%, 10.04% and 14.72%, respectively for PC, FA-30 and LP-30 mortars. After immersion in Na_2SO_4 solution, all specimens showed a slight reduction in CaO content due to the diffusion of Ca^{2+} ions in pore solution into the external sulfate solution. However, under the combined condition CaO content decreased more significantly. For example, the remained CaO contents in PC, FA-30 and LP-30 mortars were 10.34%, 8.04% and 11.84%, respectively. This was due to the fact that Ca^{2+} ions in pore solution moved from anode towards cathode under electrical potential gradient and then leached out from mortar into sulfate solution of the cathodic compartment. The degraded region induced by leaching of Ca^{2+} ions near the cathodic side surface under electrical field has been found in Ref. [15].

Therefore, the cumulative quantity of dissolved Ca^{2+} ions in the cathodic solution together with

sulfate attack and the electrical field was further measured as shown in Fig. 8, and they increased with time. The reduction of leached Ca^{2+} ion quantity from FA-30 specimen was observed compared with PC specimen. This was attributed to that the denser structure due to the pozzolanic reaction of fly ash slowed down the leaching of Ca^{2+} ions. In contrary, in comparison with PC specimen, the amount of leached Ca^{2+} ion from LP-30 specimen was greater due to the larger porosity [28].

3.3 Ca/Si ratio of C–S–H gel

Fig. 9 shows the Ca/Si molar ratio of C–S–H gel obtained from the surface part of mortars under different conditions after 90 days. In water curing, the average values of Ca/Si ratios were 1.76, 1.35 and 1.64, respectively for PC, FA-30 and LP-30 mortars. Comparatively, there was no significant variation in the Ca/Si ratios after immersion in sulfate solution. However, the Ca/Si ratios of PC, FA-30 and LP-30 mortars under the combined action decreased to 1.48, 1.25 and 1.38, respectively.

There is a chemical equilibrium between the main hydration products ($\text{Ca}(\text{OH})_2$ and C–S–H) and their component ions in pore solution, as described by the following chemical reactions (1) and (2) [29].

When Ca^{2+} ion concentration in the pore solution decreases, $\text{Ca}(\text{OH})_2$ dissolves to supply additional Ca^{2+} ions in order to maintain equilibrium. As soon as a large amount of $\text{Ca}(\text{OH})_2$ has dissolved, C–S–H gel starts to dissolve to provide Ca^{2+} ions. In this study, Ca^{2+} ions moved rapidly to the cathodic side under electrical field, leading to a reduction of its concentration in the

1 pore water. This accelerated leaching process led to the decrease of Ca/Si molar ratio in the
2 surface layer. Moreover, the decrease degree of Ca/Si ratio in FA-30 specimen was found to be
3 lower than that in PC and LP-30 specimens due to the reduction of leached Ca^{2+} ions as shown in
4 Fig. 8.

5 *3.4 SEM observation*

6 SEM was used to examine the microstructure of mortars subjected to different exposures.

7 Microstructure of samples after 90 days of immersion in sulfate solution is shown in Fig. 10. A
8 few needle-like ettringite crystals can be observed in the pores and cracks.

9 Fig. 11 presents the microstructure of PC mortar exposed to combined condition after 90 days.
10 As shown in Fig. 11a, a large amount of columnar crystals were noted in the surface part. Careful
11 observation of the EDS spectrum showed that the crystals consisted of the elements S, Ca and O
12 (Fig. 11e), which indicated that the crystals were gypsum crystals. At the near-surface part (about
13 10-20 mm depth from the exposed surface), gypsum crystals were also observed (Figs. 11b and c),
14 along with fine needle shaped crystals (Fig. 11d). The EDS spectrum of these needle crystals
15 indicated that they were ettringites, which detected the elements Al, S, Ca and O (Fig. 11f).

16 SEM examinations of FA-30 and LP-30 mortars subjected to combined exposure after 90 days
17 are shown in Figs. 12 and 13. Similarly, a large number of gypsum crystals were formed in the
18 surface region, while gypsum and ettringite crystals coexisted in the near-surface part.

19 *3.5 XRD analysis*

20 Fig. 14 shows the XRD patterns of PC samples subjected to different exposure conditions after
21 90 days. In immersion condition, a high intensity of portlandite as well as low intensities of
22 ettringite and gypsum were exhibited. This was because of that the quantity of penetrated sulfate

ions was not sufficient to convert more portlandite into ettringite and gypsum. Moreover, the ettringite and gypsum peaks were strong under the combined condition, for which a large number of sulfate ions migrated into the specimen and then reacted with portlandite to form more ettringite and gypsum. The reaction with sulfate as well as the dissolution induced by leaching of Ca^{2+} ions resulted in a significant drop in portlandite concentration.

Fig. 15 presents the XRD patterns of FA-30 samples subjected to different exposure conditions after 90 days. The intensity of portlandite in FA-30 mortar cured in water was lower than that in PC mortar due to the dilution effect of fly ash, as well as its pozzolanic reaction which consumed some portlandite. Few ettringite crystals were detected in immersion condition, while a significant amount of ettringite and gypsum were formed in the combined exposure condition. As discussed earlier, the less permeable of FA-30 mortar due to pozzolanic effect resulted in the reduction of sulfate content (Fig. 6). Thus, the formation of gypsum and ettringite in FA-30 mortar was lower than that in PC mortar. Meanwhile, the portlandite peaks could not be detected through the XRD analysis.

The XRD patterns of LP-30 samples subjected to different exposure conditions after 90 days are shown in Fig. 16. The portlandite peaks in LP-30 samples cured in water decreased compared with PC samples because of a reduction in cement quantity. Similarly, a few gypsum and ettringite were observed in immersion condition. Besides, the concentrations of sulfate corrosion products, especially gypsum, in LP-30 mortar subjected to combined condition were higher than those in PC mortar due to the increase of migrated sulfate ions as seen in Fig. 6.

3.6 Compressive strength

Fig. 17 shows the relative compressive strength of mortars exposed to immersion and combined

1 conditions. In immersion condition, the compressive strength of specimens increased after 90 days
2 due to the pores being filled by sulfate products (ettringite and/or gypsum). In the literature,
3 similar observations were also found by other researchers [30-31]. After 180 days, the relative
4 compressive strength of PC, FA-30 and LP-30 mortars decreased to 97.3%, 102.2% and 96.7%,
5 respectively. This indicated that the damage process of mortar under sulfate attack alone was fairly
6 slow. However, the relative compressive strength was lower than 100% after 30 days of combined
7 exposure. The specimens exhibited a significant reduction in compressive strength with time,
8 indicating that the combined sulfate attack and electrical field sped up the deterioration process of
9 mortars. For instance, the relative compressive strengths of PC, FA-30 and LP-30 mortars were
10 76.5%, 86.2% and 70.1% by 90 days, respectively.

11 According to the previous analysis, the rapid deterioration of mortars subjected to the combined
12 condition can be explained by two primary mechanisms. On the one hand, the electrical field
13 accelerated the dissolution of portlandite in a mortar by increasing Ca^{2+} ions moving speed in the
14 pore solution, as well as the decomposition of C-S-H gel in the later stage. On the other hand, a
15 large amount of external sulfate ions migrated into mortar under the potential gradient and then
16 reacted with cement hydrates, particularly portlandite and the hydration products of C_3A , to
17 produce expansive ettringite and gypsum in large amounts, which resulted in swelling, cracking
18 and strength loss of mortars.

19 Fly ash can increase the chemical resistance of mortar. However, the compressive strength loss
20 of the mortar blended with fly ash was still remarkable. In addition, the increase in porosity
21 resulted in a greater strength reduction of the mortar blended with limestone powder.

22 **4. Conclusions**

From the present study, the following conclusions can be drawn:

1) Electrical field resulted in the dissolution of portlandite and decomposition of C–S–H gel due to an accelerated leaching process.

2) Electrical field accelerated the sulfate attack induced deterioration, as it facilitated the ingress of sulfate ions into the matrix of mortar and the formation of expansive gypsum and ettringite.

3) The combined sulfate attack and electrical field accelerated the damage process of mortars compared with the single sulfate attack.

4) The deterioration of the mortar blended with fly ash was still notable, despite its chemical resistance increased. The deterioration degree of limestone powder incorporated mortar was greater than that of Portland cement mortar due to the increase in porosity.

Except for the mix proportions of mortar, there are other influencing factors under the combined condition, such as the concentration of sulfate solution as well as the voltage and type (i.e. direct current and pulse) of electrical field, so the further research is required.

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Table 1 Chemical composition of clinker, gypsum, FA and LP.

Constituent (wt.%)	Clinker	Gypsum	FA	LP
SiO ₂	19.99	4.47	45.08	2.50
Al ₂ O ₃	4.80	0.99	29.11	0.60
Fe ₂ O ₃	2.98	0.36	16.12	0.36
CaO	61.22	34.05	3.41	54.03
SO ₃	0.23	40.61	0.76	0.01
MgO	3.27	1.84	0.49	0.54
Na ₂ O	0.18	0.08	0.88	0.08
K ₂ O	0.88	0.23	1.05	–
Loss on ignition	3.52	16.87	3.10	41.57

Table 2 Mix proportions of mortar

Mixture	Binder	Water–binder ratio	Sand–binder ratio
PC	100%PC	0.5	3.0
FA-30	70%PC+30%FA	0.5	3.0
LP-30	70%PC+30%LP	0.5	3.0

Fig. 1. Special mold.

Fig. 2. Schematic drawing of combined action test of sulfate attack and electrical field.

Fig. 3. Migrated SO₃ content distribution in PC mortar after the test.

Fig. 4. Migrated SO₃ content distribution in FA-30 mortar after the test.

Fig. 5. Migrated SO₃ content distribution in LP-30 mortar after the test.

Fig. 6. Comparison of migrated SO₃ content in PC, FA-30 and LP-30 mortars after 90 days of combined condition.

Fig. 7. Comparison of CaO content in mortars before and after 90 days of the test.

Fig. 8. Cumulative quantity of dissolved Ca^{2+} ions under combined condition.

Fig. 9. Ca/Si ratio of C-S-H gel in mortars after 90 days of different exposures.

Fig. 10. SEM of the mortars immersed in sulfate solution after 90 days: (a) PC, (b) FA-30, and (c) LP-30.

Fig. 11. SEM and EDS analysis of PC mortar subjected to sulfate attack under electrical field after 90 days: (a) Surface part, (b) Near-surface part, (c) Magnification of Area A in Fig. 11b, (d) Magnification of Area B in Fig. 11b, (e) EDS of columnar crystals in Fig.11a, and (f) EDS of needle crystals in Fig.11d. (G-gypsum, E-ettringite.)

Fig. 12. SEM of FA-30 mortar subjected to sulfate attack under electrical field after 90 days: (a) Surface part and (b) Near-surface part.

Fig. 13. SEM of LP-30 mortar subjected to sulfate attack under electrical field after 90 days: (a) Surface part and (b) Near-surface part

Fig. 14. XRD patterns of PC mortar under different exposure conditions after 90 days.

Fig. 15. XRD patterns of FA-30 mortar under different exposure conditions after 90 days.

Fig. 16. XRD patterns of LP-30 mortar under different exposure conditions after 90 days.

Fig. 17. The relative compressive strength of mortars under immersion and combined conditions.