



THE DETERIORATION OF ADMIXTURE CONCRETE IN A SULPHATE ENVIRONMENT

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The deterioration mechanism of concrete with mineral admixtures subjected to drying-wetting cycles in a 10 % sodium sulphate solution was studied. Plain concrete without fly ash addition, concrete with 10 %, 20 % and 30 % fly ash addition, and a concrete with 20 % fly ash and 30 % slag addition were used. Through the experiment, the weight loss, the relative dynamic modulus of elastically (RDME), the compressive strength loss and the damage layer thickness (H_f) of the concrete were measured. Furthermore, scanning electron microscopy, X-ray diffraction and thermal analysis were used to investigate the corrosion products of the concrete. The test results show that the ultrasonic velocity in the damage layer decreases and the H_f of the concrete increases as the corrosion time grows, indicating that the deterioration degree of the concrete increases. There is a significant relationship between the RDME and the H_f after 210 d. The deterioration degree of the concrete decreases with an increase in the fly ash content. However, the sulphate resistance property of the concrete significantly decreases with 30 % fly ash content. When mixed with 20 % fly ash and 30 % slag, the sulphate resistance property of the concrete is the best.

INTRODUCTION

Sulphate widely exists in the world; sulphate attack is one of the most aggressive environmental degradation effects that affect the durability and service life of concrete structures. The deterioration of concrete caused by a sulphate attack can manifest in the form of expansion and cracking of the concrete. Sometimes the expansion of the concrete may cause serious structural problems. A sulphate attack can also take the form of a progressive decrease in the strength and loss of mass due to loss of cohesiveness of the cement hydration products [1, 2].

For concrete in the fluctuating groundwater table splash and tidal zone, drying-wetting cycles can accelerate the deterioration of the concrete [3-5]. Based on the mechanism of the sulphate attack, the deterioration of the concrete in the sulphate environment is a process from outside to inside. The one-side non-destructive testing method can be used to detect the damage layer of the concrete, which allows for the valid prediction of the deterioration degree of the concrete. Naffa et al. [6] showed that it is possible to detect and characterise concrete cover degradation using a high-frequency ultrasound. Wang et al. [7, 8] suggested that ultrasonic penetration testing can be used to locate the interface between the solid and corroded concrete. Niu et al. [9] obtained that the damage to the concrete under a sulphate attack can be determined by using simple ultrasonic testing methods. According to the literature [10, 11], the $H_{\rm f}$ of the concrete exposed to a freeze-thaw environment could be detected based on ultrasonic detection methods.

Incorporation of mineral admixtures such as fly ash, slag and silica fume has often been used to improve the sulphate resistance property of concrete. The quantity of tricalcium silicate in cementitious materials decreases by using mineral admixtures in concrete. This method can partially consume calcium hydroxide through the pozzolanic reaction and reduce the formation of corrosion products [12-14]. However, the types and content of mineral admixture have different effects on the deterioration of the concrete. The deterioration mechanism of the concrete with mineral admixtures in a sulphate environment also needs further research.

The present study simulated a sulphate attack on concrete exposed to a sulphate solution under cyclic environmental conditions, the drying state was natural drying in the air, which is more relevant to the real environment. The weight loss, the RDME change, the compressive strength loss and the $H_{\rm f}$ of the concrete with the addition of different mineral admixtures were conducted. Furthermore, the corrosion products and the damage mechanism of the concrete were discussed by using microscopic tests.

EXPERIMENTAL

Materials

A Chinese standard ordinary Portland cement (OPC) of PO 42.5R produced by the cement factory of Tongchuan was used. The slag was produced by the Powder Engineering Research Institute in Xi'an University of Architecture and Technology. Grade II fly ash from the Weihe power station, river sand with a fineness modulus of 2.69 and coarse aggregate of crushed basalt stone with a diameter of 5 mm to 16 mm were used in the test. A naphthalene-type superplasticiser was used, and the dosage was adjusted to keep the slump of the fresh mixed concrete in the range of 50 mm to 120 mm. Tap water was used as the mix water. The chemical composition of the cement, fly ash and slag is shown in Table 1.

Table 1. The chemical composition of the OPC and fly ash.

Constituent (wt. %)	SiO ₂	Al ₂ O ₃	CaO	MgO	SO ₃	Fe ₂ O ₃
OPC	21.66	5.13	64.37	1.06	2.03	5.25
Fly ash	49.02	31.56	4.88	0.83	1.2	6.97
Slag	33.15	12.91	40.00	6.75	0.12	2.80

Preparation of the sample

In this experiment, the water binder ratio was 0.45 and the sodium sulphate solution with a concentration (by mass) of 10 % was used. Five different binders, namely 100 % OPC, 90 % OPC + 10 % fly ash, 80 % OPC + 20 % fly ash, 70 % OPC + 30 % fly ash, and 50 % OPC + 20 % fly ash + 30 % slag (denoted as F0 %, F10 %, F20 %, F30 % and F20 % + S30 %, respectively) were used in this study. All specimens were demoulded after 24 h of casting and cured in a condition of 20 ± 3 °C and 95 % relative humidity until the age of testing. The mixture proportion of the concrete is given in Table 2.

Test method

Test procedure

At the age of 90 days curing, the concrete specimens were exposed to the sulphate solution under drying-wetting cycles up to 360 days. One wet-dry cycle lasted 15 days. First, the specimens were continuously immersed in the test solution for 7 days, and then were

Table 2. The mix proportion of the concrete.

moved into the air to dry naturally for another 8 days. Specimens of $100 \times 100 \times 400$ mm prisms were used for the study of the weight loss, the RDME and the $H_{\rm f}$ of concrete. Specimens of $100 \times 100 \times 100$ mm cubes were used to study the compressive strength loss and the analysis of the microstructure of the concrete. According to the test procedure, the deterioration of the specimens was investigated by determining the RDME, weight loss and compressive strength loss.

Weight loss

According to the GB/T50081 method [15], Equation 1 was used to calculate the weight loss:

$$\Delta W_{\rm n} = \left[(W_0 - W_{\rm n}) / W_0 \right] \times 100 \tag{1}$$

where ΔW_n is the weight loss of the specimens after every 30 days of exposure (%), W_0 is the average weight of the concrete specimens before the sulphate attack (kg) and W_n is the average weight of the concrete specimens after every 30 days of exposure in the sulphate solutions (kg). RDME loss

The dynamic modulus of elasticity (E_d) was determined by Equation 2 [16]. The RDME was the ratio of the E_d value to the initial E_d value after every 30 days of exposure, which was determined by Equation 3.

$$E_{\rm d} = \frac{(1+v)(1-2v)\rho V^2}{1-v}$$
(2)

$$RDME = \frac{E_{dn}}{E_{d0}} = \frac{V_n^2}{V_0^2}$$
(3)

where V is the ultrasonic speed (m·s⁻¹), E_{d0} is the dynamic modulus of elasticity of the concrete before the sulphate attack (GPa), E_{dn} is the dynamic modulus of elasticity of the concrete after every 30 days of exposure (GPa), ρ is the density of the specimen (kg·m⁻³) and ν is Poisson's ratio.

Compressive strength

The measurement of the compressive strength property was conducted according to the GB/T50081 [17] method. Equation 4 was used to calculate the compressive strength:

$$f_{\rm cc} = F/A \tag{4}$$

where f_{cc} is the compressive strength (MPa), F is the maximum load (N) and A is the area of the cube loading face (mm²).

NO.	Water-binder ratio	Cement (kg·m ⁻³)	Fly ash (kg·m⁻³)	Slag (kg·m⁻³)	Sand (kg·m⁻³)	Aggregate (kg·m ⁻³)	Water (kg·m ⁻³)	Superplasticiser (%)
F0 %	0.45	355	_	_	585	1300	160	0.5
F10 %	0.45	319	36	_	585	1300	160	0.5
F20 %	0.45	285	70	_	585	1300	160	0.5
F30 %	0.45	249	106	_	585	1300	160	0.5
F20 % + S30 %	0.45	178	35	142	585	1300	160	0.5

Damage layer thickness

The $H_{\rm f}$ is detected by using a high accuracy nonmetal ultrasonic analyser according to CECS 21:2000 [18] and literature [19]. The prism-side of a 100×400 mm prism was selected as the test surface, as shown in Figure 1. The transmitting transducer A is placed on the surface, and then a receiving transducer B moves along the concrete surface to record the corresponding time of the sound propagation. The test distance l between A and B is 50 mm, 75 mm, 100 mm, 150 mm, 200 mm and 250 mm, respectively.



Figure 1. Setting the transducer.

At first, the receiving transducer *B* close to the transmitter will only sense the top layer (the damaged layer of the concrete), but as the distance increases, the influence from the lower layer (the sound concrete) is felt. When the distance between the two transducers reaches a certain range l_0 , the ultrasonic propagation time from *A* to *B* through the damage layer and the sound concrete is equal to the time propagated only in the damaged layer as shown in Figure 1. These are:

$$\frac{l_0}{V_{\rm f}} = \frac{2\sqrt{h_{\rm f}^2 + x^2}}{V_{\rm f}} + \frac{l_0 - 2x}{V_{\rm a}}$$
(5)

$$H_{\rm f} = \frac{l_0}{2} \sqrt{\frac{V_{\rm a} - V_{\rm f}}{V_{\rm a} + V_{\rm f}}} \tag{6}$$

where x is the horizontal projection of the ultrasonic propagation path through the damaged layer (mm), V_f is the ultrasonic velocity in the damaged layer (km·s⁻¹); V_a is the ultrasonic velocity in the sound concrete km·s⁻¹), l_0 is the distance from A and B when the ultrasonic pulse hits the interface between the damaged layer and the sound concrete (mm).

The experimental results are plotted in a time vs. distance plot, which will be a straight line similar to Figure 2. It is shown that l_0 is the intersection of the two straight lines. The regression equations of *l*-*s* in the damaged layer and in the sound concrete, by using the linear regression method, are as follows:

$$l_{\rm f} = A_{\rm f} + V_{\rm f} t_{\rm f} \tag{7}$$

$$l_{\rm a} = A_{\rm a} + V_{\rm a} t_{\rm a} \tag{8}$$

where l_f is the distance before l_0 in Figure 2, such as l_1 and l_2 (mm); l_a is the distance after l_0 , such as l_3 , l_4 and l_5 (mm); t_f is the time corresponding to l_f before l_0 , such as t_1 and t_2 (µs); t_a is the time corresponding to l_a after l_0 , such as t_3 , t_4 and t_5 (µs); A_f and A_a are the intercept of the two lines, respectively; V_f and V_a are the slope of the two lines, respectively.

Therefore, l_0 can be written as:

$$l_{0} = \frac{A_{1}V_{a} - A_{2}V_{f}}{V_{a} - V_{f}}$$
(9)

and $H_{\rm f}$ can be calculated by Equation 6.



Figure 2. The relationship of the travel time and the test distance.

Characterisation of the sample

To identify the products formed by the sulphate attack, a scanning electron microscopy (SEM) equipped with an energy dispersive spectroscopy (EDS) detector, and X-ray diffraction (XRD) (Cu-K α) were used. In addition, thermogravimetric analysis (TG) and differential scanning calorimetry (DSC) was also used to analyse the corrosion products.

RESULTS AND DISCUSSION

The weight loss of the concrete

As can be seen from Figure 3, the weight loss of the concrete specimens exposed to a sulphate attack and drying-wetting cycles exhibits three distinct stages: a decreased stage, a steady stage and an increased stage. The weight loss for all the specimens is less than 1 % after 360 d. The weight loss of F20 % is seen to be less than those of F10 % and F30 %. When exposed to 300 days, the weight loss of F30 % is higher than that of F10 %. Compared to the concrete by adding the single fly ash, the weight loss of F20 % + S30 % presents a minimum increase. The test results also show that the weight loss of F0 % is inconspicuous compared with that of F20 % + S30 %. Moreover, the weight loss of F0 % is significantly lower than that of the concrete by mixing in the fly ash.



Figure 3. The weight loss of the concrete.



Figure 4. The RDME of the concrete.

The RDME of the concrete

As can be seen from Figure 4, the behaviour of the RDME of the concrete exposed to the sulphate solution under the drying-wetting cycles also exhibits three distinct periods: a decreased period, a steady period, and an accelerating decreased period. After 360 days, the RDME of F0 %, F20 % and F30 % are 22.3 %, 18.8 % and 27.2 %, respectively. The deterioration of the concrete is seen to increase with an increase in the fly ash content. However, the sulphate resistance property of the concrete is significantly decreased with the addition of 30 % fly ash. The degradation rate of F30 % becomes much faster after 270 days compared with F0 %. The concrete with 30 % fly ash did not achieved a greater resistance under the sulphate attack. The RDME of F20 % + S30 % decreases slowly in period III. It is shown that F20 % + S30 % has the best performance in terms of the resistance of the concrete in the sulphate environment. The reason is that two kinds of mineral admixtures reasonably added and with the proper amount of water reducing the admixture is used to refine the pore structure. The compactness of the concrete is obviously improved, thus lowering the diffusion coefficient of the sulphate ions under the drying-wetting cycles.

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The compressive strength of the concrete

Figure 5 shows the results of the compressive strength change of the concrete exposed to the sulphate solution. The compressive strength loss exhibits the following three periods: an increased period, a decreased period and an accelerating decreased period. As can be seen from Figure 5, the compressive strength of F0 %, F20 % and F30 % decreased by 14.8 %, 12.7 % and 16.6 % after 360 days, respectively. The compressive strength loss of the concrete is seen to increase with an increase in the fly ash content. The test results also show that the compressive strength loss of F0 % is 16.2 %, indicating the degradation rate is lower than that of F30 %. However, the compressive strength loss of F20 % + S30 % presents a minimum decrease of only by 9.7 %.



Figure 5. The relative compressive strength of the concrete.

The damaged layer of the concrete

The damaged layer of the concrete is not obvious at the beginning of the corrosion time. As the damage degree increases, it could be accurately calculated after 210 days in the test. The results agree with those suggested by Zhang et al [20], which showed that a larger error was observed for ultrasonic penetration testing when the $H_{\rm f}$ was small. The temporal variation of the $V_{\rm f}$ and the $H_{\rm f}$ are given in Figure 6 and Figure 7, respectively.

As can be seen from Figure 6, the decrease rate of $V_{\rm f}$ is seen to reduce with an increase in the fly ash content. But the decrease rate of $V_{\rm f}$ is considerably increased when the fly ash content reached 30 %. The change in $V_{\rm f}$ of F10 % is inconspicuous compared with that of F30 %. However, the $V_{\rm f}$ of F30 % is lower than that of F10 % after 300 days. The $V_{\rm f}$ of F20 % + S30 % presented the slowest reduction, it only decreased by 9.7 %.

As seen from Figure 7, the $H_{\rm f}$ increases with an increase in the corrosion time, which exhibits the opposite trend compared with $V_{\rm f}$. The results for the $H_{\rm f}$ change indicate a dependence on the fly ash content. The $H_{\rm f}$ decreases when the appropriate amount of fly ash content is used. But the $H_{\rm f}$ is largest when the content reaches

30 %, indicating that F30 % covers the most aggressive corrosion. When the $H_{\rm f}$ of the concrete is thicker and the $V_{\rm f}$ is lower, indicating that the compactness reduces and the deterioration degree of concrete increases. The results are in a good agreement with what was obtained in the literature [10, 20], which showed that an increase in $H_{\rm f}$ reduces the density of the concrete when exposed to a sulphate attack or freeze-thaw cycles.

Figure 8 shows the relationship between the RDME



Figure 6. The $V_{\rm f}$ of the concrete.



Figure 7. The $H_{\rm f}$ of the concrete.



Figure 8. The relationship between the RDME and $H_{\rm f}$.

and $H_{\rm f}$ of the concrete. It is shown that the temporal variation of $H_{\rm f}$ is in good agreement with the RDME after 210 days. There is a significant relationship between them. Obviously, the $H_{\rm f}$ of the concrete increases with a reduction in the RDME, indicating that the damage to the concrete increases. Therefore, the deterioration degree of the concrete could be estimated effectively by measuring the $H_{\rm f}$.

The microstructures of the concrete

Figure 9 shows the microstructure observation of the concrete under a sulphate attack and drying-wetting cycles. As shown in Figure 9a, needle-like crystals can be observed in the concrete pores. As the corrosion time grows, the amount of needle crystals continuously increases. The pores are almost completely filled up by needle-like corrosion products as shown in Figures 9b and 9c. Moreover, the microcracks in the pores can be clearly observed from the micrographs. The EDS spectra showed that the needle crystals consist of the elements Al, Si, S and Ca, which indicated that the needle crystals are ettringite (Figure 9d).



a) SEM micrograph



b) SEM micrograph

Figure 9. The SEM micrographs (a, b) of the needle crystals in the concrete. *(Continue on next page)*

As shown in Figure 10a, short columnar crystals can be observed in the interface transition zone between the paste and the aggregate in the concrete specimens. The EDS spectra indicated that these crystals are gypsum, which detected the elements Ca and S (Figure 10b). When the expanding pressure generated by the ettringite

and gypsum exceeds the tensile strength of the concrete, cracks occur.

In the test process, some petals shape crystals can be observed by using SEM as shown in Figure 11a. It was confirmed to be thenardite by using EDS (Figure 11b). The results show that the concrete specimens are attacked



Figure 9. The SEM micrographs (c) and EDS spectrum (d) of the needle crystals in the concrete.



a) SEM micrograph







by corrosion products in the sodium sulphate solution during the wetting cycles. Furthermore, the concrete specimens also suffer from the crystallisation pressure of the thenardite crystals during the drying cycles.

XRD analysis

Figure 12 shows the XRD patterns of F20 % under a sulphate attack and drying-wetting cycles. The results show that the peaks selected for the qualitative analysis of these phases are ettringite at 9.06° 20, gypsum at 15.86° 20, calcium hydroxide at 18.02° 20, quartz at 20.82° 20 and calcium carbonate at 29.36° 20. In these diagrams, quartz is mainly formed by the composition of the sand. Calcium carbonate is formed by the carbonisation reaction in the atmospheric environment during the dying cycles. The ettringite and gypsum peaks in the concrete are strong. This phenomenon demonstrates that they are the main corrosion products. Obviously, the concentrations of gypsum and ettringite increase with the corrosion time, and the content of gypsum in the concrete is less than that of ettringite in the test. Moreover, the concentration of calcium hydroxide gradually decreases along with the corrosion time growth.



Figure 12. The XRD patterns of F20 %.

Thermal analysis

Figure 13 presents the TG-DSC curve of the degradation product of F20 % exposed to the sodium sulphate solution subjected to drying-wetting cycles. According to the decomposition temperature corresponding to the heat absorption peak in the DSC curves, the chemical product is identified. Moreover, the weight loss is shown simultaneously in the TG curves. There are three typical endothermic peaks that lie in the ranges 90 - 110 °C, 130 - 140 °C, and 430 - 450 °C. These three temperature stages correspond to the dehydration and decomposition of ettringite, the dehydration and decomposition of gypsum, and the decomposition of calcium hydroxide, respectively [21,22]. It is further proved that ettringite and gypsum are the main corrosion products in the concrete.



Figure 13. The TG-DSC curves of the deterioration products of F20 % after 90 days.

CONCLUSIONS

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

- When the different types of concrete exposed to drying-wetting cycles in the sodium sulphate solution, the changing process of the weight loss of concrete can be classified into three stages: a decreased stage, a steady stage and an increased stage. The RDME of the concrete exhibits three distinct periods: a decreased period, a steady period, and an accelerating decreased period. The compressive strength loss of the concrete exhibits the following three periods: an increased period, a decreased period and an accelerating decreased period, a decreased period and an accelerating decreased period. The degradation rate of the concrete greatly accelerated after 210 d.
- As the corrosion time grows, the $V_{\rm f}$ decreases and the $H_{\rm f}$ of the concrete increases, indicating that the deterioration degree of the concrete increases. There is a significant relationship between the RDME and the $H_{\rm f}$ of the concrete after 210 d. By measuring the $H_{\rm f}$ of the concrete, the deterioration degree of the concrete could be effectively estimated.
- The addition of a mineral admixture in the concrete could improve the sulphate resistance property of the concrete when exposed to the simultaneous action of a sulphate attack and drying-wetting cycles. The damage to the concrete decreases with an increase in the fly ash content. However, the sulphate resistance property of the concrete is significantly decreased when the fly ash content reaches 30 %. When mixed with 20 % fly ash and 30 % slag, the sulphate resistance property of the concrete is the best.

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