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Deterioration and Service Life Prediction of Concrete Subjected to Freeze–Thaw Cycles in Na_2SO_4 Solution

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Abstract: To investigate the resistance of normal concrete to sulfate solution frost, concrete specimens with different water/cement (w/c) ratios, mineral admixture types, replacement ratios, and air contents were fabricated. Then, these specimens were frozen and thawed cyclically in 5% concentration Na_2SO_4 solution. As the freezing and thawing cycles proceeding, the appearance morphology, mass loss, and dynamic elastic modulus (DEM) of the specimens were observed. The service life of concrete that is subjected to the freeze–thaw cycles in Na_2SO_4 solution was calculated based on specimens' DEM losses. Results indicated that the appearance damage and mass loss of concrete along with the freeze–thaw cycles were unnoticeable until failure, and DEM losses played a controlling role in determining specimens' failure. In addition, a sudden fracture failure in the middle occurred easily in specimens with low w/c ratio. Decreasing concrete w/c ratio can slightly increase the concrete resistance to the freeze–thaw cycles in Na_2SO_4 solution, whereas incorporating fly ash or slag has almost no effect. Moreover, higher replacement ratio of fly ash or slag increases the adverse effects on concrete. Adding an air-entraining agent to concrete can significantly improve its resistance to the freeze–thaw cycles. Air content at 4.6% and 5.7% can extend the service life of concrete under the freeze–thaw cycles in Na_2SO_4 solution by more than 5 times its ordinary life span.

Keywords: Concrete, Freeze–Thaw Cycle, Deterioration, Na_2SO_4 Solution, Service Life Prediction

1. Introduction

The unique geography and harsh natural climate in the western regions of China pose challenges to concrete bridges in the area, which causes the service life of some of these structures to last for only 3 to 5 years [1-2]. Sulfate attacks from saline soil or salt lakes and freeze–thaw cycles resulting from seasonal temperature changes are the two main causes of structural damage, and sulfate attack and freeze–thaw circulation occur simultaneously most of the time.

Studies on the performance of concrete that is subjected to salt solution freeze–thaw cycles have been conducted and significant conclusions have been presented [3-6]. Mu et al. reported that various salt solutions have different effects on the frost resistance of concrete [7]. They also argued that NaCl solution causes no change in the failure mechanism of concrete freeze–thaw cycles, whereas Na_2SO_4 solution can cause freeze–thaw damage and sulfate attack at the same time. Yu et al. found significant differences in the final number of freeze–thaw cycles that concrete can withstand when subjected to different salt lake solutions [8]. They also demonstrated that some salt lake solutions promote concrete freeze–thaw damage, whereas others restrain such damage. Yuan et al. showed that, with the increase of Na_2SO_4 solution concentration, the freeze–thaw damage on concrete is gradually inhibited rather than increased [9]. Zhang et al. suggested that $MgSO_4$ solution can significantly slow down the freeze–thaw damage and failure of concrete [10], whereas Jiang et al. showed that the freeze–thaw cycles in $MgSO_4$ solution causes more damage to concrete compared with water and Na_2SO_4 solution [11].

In terms of concrete grade and raw materials, low concrete water/cement (w/c) ratio is commonly believed to increase the frost resistance of concrete [12]. However, Mu et al. found that the freeze–thaw damage rate of low-strength concrete is unaffected by the Na_2SO_4 solution, whereas high-strength concrete is damaged in advance because of the joint action of sulfate attack and freeze–thaw cycles in late period [13]. An entraining agent can improve the frost resistance of concrete to water freeze–thaw and can enhance solution freeze–thaw [12] [14]. Du et al. and Ghafoori et al. found that adding fly ash into concrete can improve its sulfate corrosion resistance [15-16]. Other researchers observed that incorporating fly ash, slag, silica fume, and other active mineral admixtures significantly reduces the frost resistance of concrete [8] [17]. Fly ash, slag, or silica fume has been widely used in preparing high-performance concrete. However, further study is needed with regard to the effects of applying mineral admixtures on the performance of concrete resistance under salt solution freeze–thaw cycles. Thus, the present study examines the performance of concrete specimens with different w/c ratios, mineral admixture types, and air contents under 5% Na_2SO_4 solution freeze–thaw cycles. The service lifespan of these specimens are estimated to provide a useful reference for engineers.

2. Experimental

2.1. Raw Materials and Mixture Proportions

Chinese-standard P·O 42.5R ordinary Portland cement produced by Huaihai Zhonglian cement factory was used. Grade I low-calcium fly ash powder and S95 slag powder were used in the test, and their cement replacement ratios (by mass) were set as 10%, 20%, and 30%. The specific chemical composition of each cementitious material is presented in Table 1.

Table 1. *Chemical composition of cement, fly ash, and slag/wt%.*

Item	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Loss on ignition
Cement	22.3	5.05	3.16	64.78	0.92	1.32
Fly ash	57.2	30.3	4.5	2.5	0.6	2.65
Slag	29.3	13.7	0.4	33.2	9.2	2.31

Natural river sand with a fineness modulus of 2.53, crushed stone with particle size of 5–20 mm, and ordinary tap water were chosen as fine aggregate, coarse aggregate, and mixing water, respectively. In addition, an air-entraining agent was used for partial specimens (PYA and PYB) to obtain different concrete air contents. A total of 12 concrete mixture proportions were designed, as shown in Table 2. The measured air contents for fresh concretes of PY0, PYA, and PYB were 1.3%, 4.6%, and 5.7%.

Table 2. *Concrete mixture proportion/kg/m^3.*

Item	Water	Cement	Fly ash	Slag	Sand	Gravel
P0.4	190	475	0	0	607	1178
P0.5	200	400	0	0	666	1184
P0.6	210	350	0	0	737	1153
F10	200	360	40	0	666	1184
F20	200	320	80	0	666	1184
F30	200	280	120	0	666	1184
K10	200	360	0	40	666	1184
K20	200	320	0	80	666	1184
K30	200	280	0	120	666	1184
PY0	190	380	0	0	623	1157
PYA	190	380	0	0	623	1157
PYB	190	380	0	0	623	1157

2.2. Test Methods

Prism specimens with a size of 100 mm × 100 mm × 400 mm were used. After demolding, the specimens were kept in a water tank for curing at 20 ± 2°C over a period of 28 days. Then, the specimens were collected and placed in an indoor natural atmosphere for another 28 days. Before the start of freeze–thaw circulation, the specimens were immersed in 5% (by weight) Na_2SO_4 solution for 4 days. In this study, “rapid freezing method” from Chinese standard on “ordinary concrete long-term performance and durability test” (GB/T50082-2009) was adopted. During the test, the specimens were frozen and thawed at 5% mass fraction of the Na_2SO_4 solution. Each freeze–thaw cycle test lasted for approximately 3.5 h, and the thawing time could not be less than one-fourth of the entire freeze–thaw cycle time. The temperature of the transformation between freezing and thawing was set at –15°C and 6°C. During the entire process of freezing and thawing, the lowest temperature of the specimen center was –18°C and the highest temperature was 10°C.

At the beginning of the freeze–thaw circulation and after every 25 freeze–thaw cycles, the specimens were collected, weighed, and tested for ultrasonic velocity using a nonmetal ultrasonic detector. The specimens were then placed back into freeze–thaw cycle boxes. When the mass loss of a specimen exceeded 5% or the dynamic elastic modulus (DEM) dropped to 60%, the specimen was considered to be a failure. Eqs. (1), (2), and (3) were used to calculate specimens' mass loss ratio α, relative dynamic elastic modulus (RDEM) P, and RDEM losses D.

$$\alpha = \frac{m_0 - m_N}{m_0} \times 100\% \tag{1}$$

$$P = \frac{v_N^2}{v_0^2} \times 100\% \tag{2}$$

$$D = 1 - P \tag{3}$$

where m_0, m_N are the mass (g) of concrete specimens before freeze–thaw circulation and after N freeze–thaw cycles, and v_0, v_N, are the longitudinal wave velocity (km/s) of concrete specimens before freeze–thaw circulation and after N freeze–thaw cycles.

3. Results and Discussion

3.1. w/c Ratio on Concrete Resistance to Freeze–Thaw Cycles in Na_2SO_4 Solution

Conventional concrete failure modes in freeze–thaw cycles can be described as follows: as the freeze–thaw cycles proceed, the grout spalls from the concrete surface, and the fine aggregate is exposed; the coarse aggregate becomes exposed and even scaled; finally, the concrete section decreases until complete failure [17]. In this study, with the action of 5% Na_2SO_4 solution freezing and thawing cycle on the specimens, sand and cement mud scaled from concrete surface also appeared. However, damage on the specimens was unnoticeable in general. When the freezing and thawing cycles reached 125 times, the specimens with w/c ratios of 0.4 and 0.5 showed a sudden fracture in the middle part, but such a phenomenon did not occur in the specimens with 0.6 w/c ratio (Fig. 1).

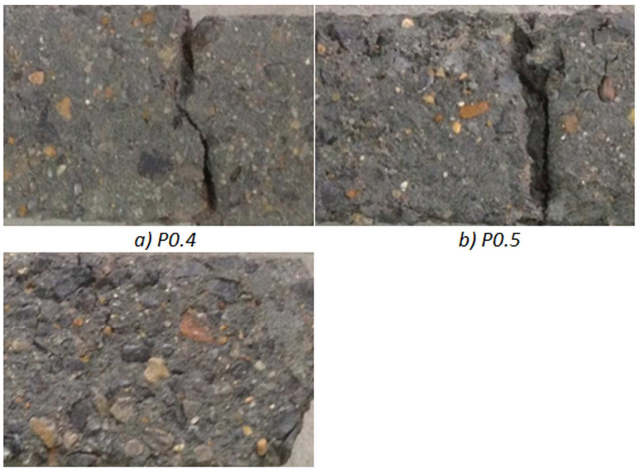


Figure 1. *Appearance of specimens with different w/c ratios after 125 freeze–thaw cycles.*

Sulfate solution has better compressible plasticity than water and chlorine salt solution after freezing [7]; thus, as the freeze–thaw cycles proceed, the phenomena of concrete surface damages are not serious. However, with the continuous accumulation of sulfate solution invasion and salt crystallization [14], specimens subjected to the Na_2SO_4 solution freeze–thaw cycles are more prone to failure characterized by a sudden fracture, especially in the case of concrete specimens with low w/c ratio and high strength. These findings have also been reported by other researchers [13] [18]. The mass loss and RDME development of specimens with three kinds of w/c ratios along with the freeze–thaw cycles are shown in Figs. 2 and 3.

As shown in Fig. 2, the mass of all specimens increased slightly at the beginning, and then decreased thereafter. However, three kinds of specimens did not have serious mass loss until 125 freeze–thaw cycles and did not reach the failure criterion of 5% weight loss. For specimens with 0.4 w/c ratio, the mass loss had a negative value until 100 freeze–thaw cycles. The mass loss and appearance damage of the specimens were consistent, which suggested that concrete specimens do not have serious mass loss until the occurrence of failure.

As shown in Fig. 3, the RDEM of concrete decreased gradually with the freezing and thawing cycles; the decrease was slow at the initial stage (25 to 50 cycles), and then it accelerated. The RDEM of the specimens with 0.6 w/c ratio dropped to 63.2% after 50 freeze–thaw cycles, which was close to the failure criterion. The RDEM values of the specimens with different w/c ratios dropped to 60% or lower after 75 freeze–thaw cycles, which reached the failure criterion. Until this point, the mass losses of the specimens were extremely low. Notably, the RDEM of concrete is the control index used to determine the damage caused by the Na_2SO_4 solution freeze–thaw cycles.

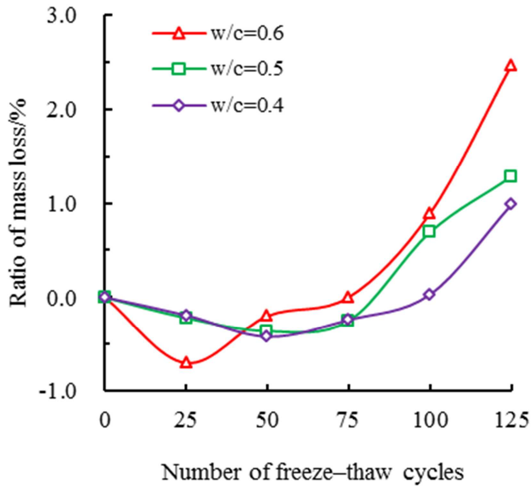


Figure 2. *Mass loss of concrete with different w/c ratios.*

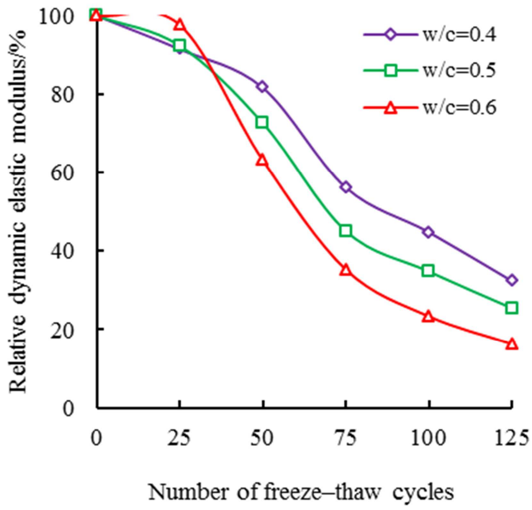
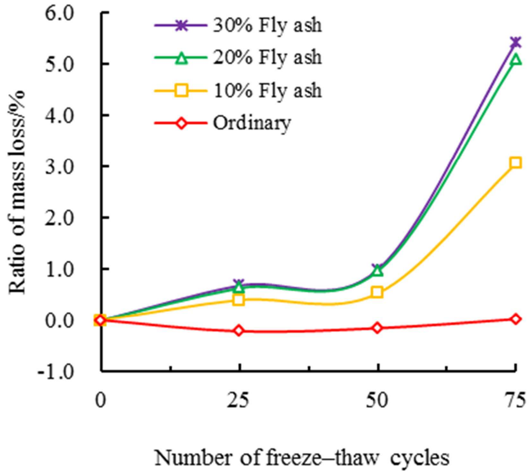


Figure 3. *RDEM of concrete with different w/c ratios.*

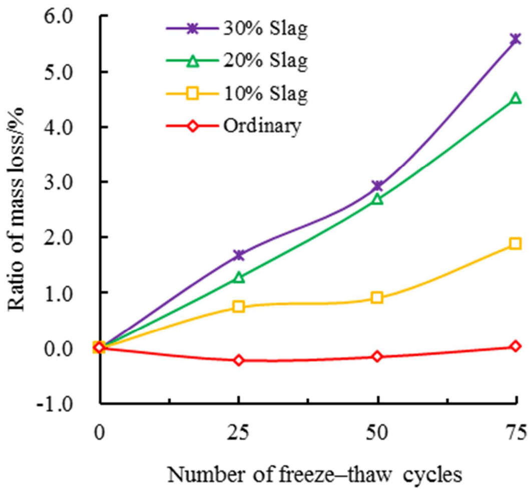
At the same time, with the same freeze–thaw cycle, the specimens with higher w/c ratios usually suffered more mass loss and RDEM loss except at the initial stages. This finding was consistent with the results of many previous studies [8] [13] [18]. Lower concrete w/c ratio often corresponds to higher concrete strength and density, which is beneficial in resisting freeze–thaw cycle damage. However, in another aspect, lower concrete w/c ratio causes significant brittleness, which results in sudden fracture failure in the middle part of the specimen.

3.2. Mineral Admixtures on Concrete Resistance to Freeze–Thaw Cycles in Na_2SO_4 Solution

After the addition of fly ash or slag, the surface spalling of the specimens, which results from the freezing and thawing cycle becomes more obvious than that in ordinary specimens (no fly ash or slag), and the specimens with higher replacement ratio usually exhibit more serious surface damage. Figs. 4 and 5 present the mass loss and RDME development of the specimens with two mineral admixture types subjected to the freeze–thaw cycles.

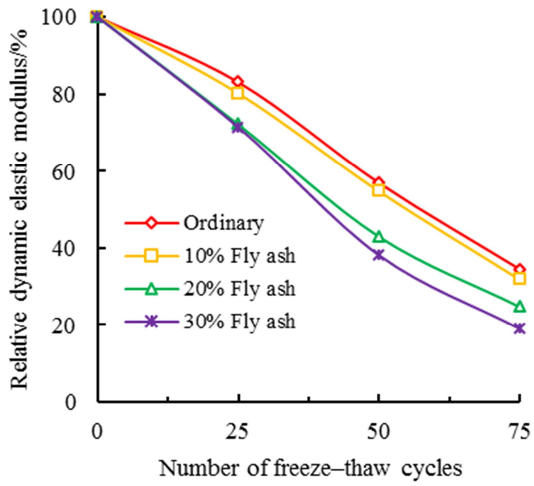


a) Fly ash specimens

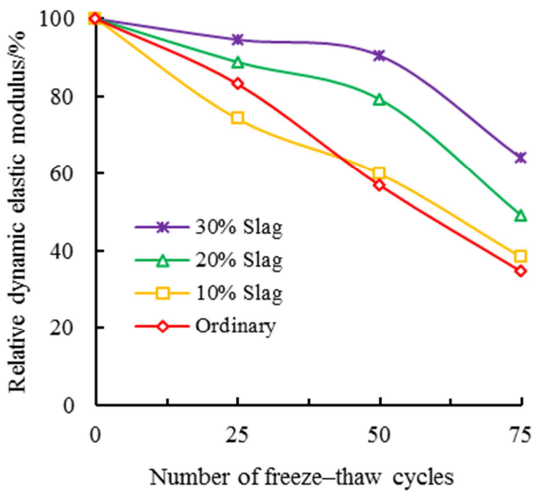


b) Slag specimens

Figure 4. *Development of mass loss of mineral admixture specimens.*



a) Fly ash specimens



b) Slag specimens

Figure 5. *Development of RDME of mineral admixture specimens.*

As shown in Fig. 4, the mass losses of either fly ash specimens or slag specimens were significantly higher than those of ordinary specimens at the same freeze–thaw cycle, and the specimens with higher cement replacement ratios usually have greater mass losses. As shown in Fig. 5, the RDME of fly ash and slag specimens showed a gradual decline with the progress of the freeze–thaw cycles. However, some differences were observed between fly ash specimens and slag specimens. With the same freeze–thaw cycle, the RDME values of fly ash specimens were all lower than those of the control specimens, and these values dropped below 60% at the period of 50 freeze–thaw cycles. By contrast, the addition of slag improved the RDEM of concrete. The specimens with slag tended to have higher RDME than the control specimens. In addition, the higher the replacement ratio is, the greater the improvement is.

The addition of fly ash and slag is believed to reduce the early strength of concrete because of relatively low pozzolanic activity. The pozzolanic hydration reaction of fly ash and slag is a lagging reaction. The failure mechanisms of concrete under Na_2SO_4 solution frost are mainly due to freeze–thaw damage and salt crystallization damage; the resistance to sulfate chemical corrosion of fly ash and slag has no effect. Hossack and Thomas [19] studied concrete with mineral admixtures for long-term immersion experiments in 5% Na_2SO_4 solution at different temperatures. They also found that the addition of fly ash can improve corrosion resistance to sulfate at higher temperatures (10°C, 23°C), whereas nearly no effect is observed at lower temperatures (5°C, 1°C). Therefore, fly ash and slag can reduce the concrete resistance to sulfate freeze–thaw cycles. Meanwhile, the pozzolanic activity of slag is slightly higher than that of fly ash. Accordingly, the addition of slag has slightly lower adverse effects on concrete than that of fly ash. Other researchers have confirmed this phenomenon [8] [18]. In general, fly ash or slag is not recommended for application in concrete members used in the environment of sulfate freeze–thaw cycles.

3.3. Air Content on Concrete Resistance to Freeze–Thaw Cycles in Na_2SO_4 Solution

Compared with the specimens that had no air-entraining agents, which showed surface mortar peeling and stone exposure phenomenon at a very early time, the specimens of PYA and PYB did not show any sign of damage until 200 freeze–thaw cycles. Remarkably, even after 300 freeze–thaw cycles, the surface changes of these specimens remained small, and the specimens did not show serious spalling. The mass loss and RDEM development of concrete with air-entraining agent are presented in Figs. 6 and 7.

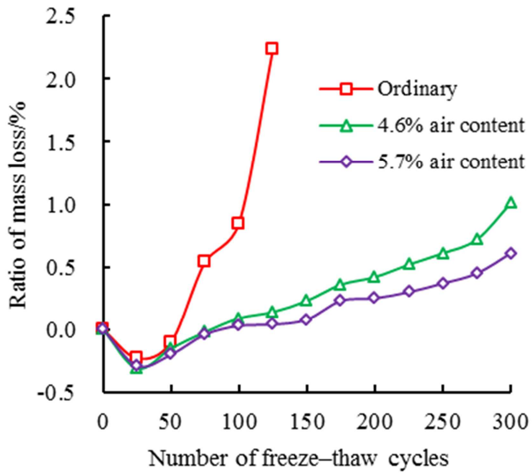


Figure 6. *Mass loss of concrete with different air content rates.*

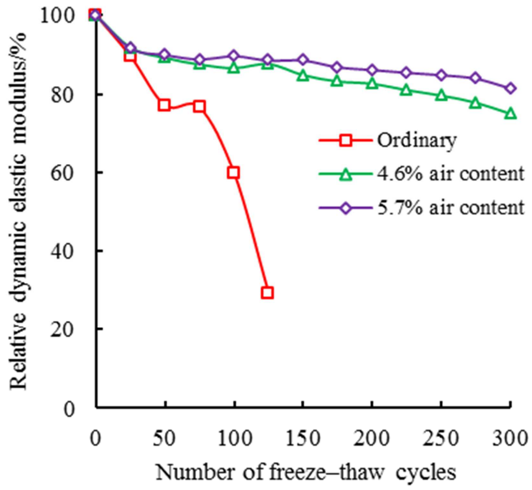


Figure 7. *RDEM Development of concrete with different air content rates.*

The mass loss of concrete along with the Na_2SO_4 solution freeze–thaw cycles decreased after the addition of an air-entraining agent, as shown in Fig. 6. After 125 freeze–thaw cycles, the mass loss ratio of the specimens without the air-entraining agent was 2.24%, whereas the mass loss ratios of the specimens with 4.6% and 5.7% air content were only 0.14% and 0.04%, respectively. The mass loss ratios of these two air-entrained specimens dropped to 94% and 98% compared with that of ordinary concrete, and were still very small even after 300 freeze–thaw cycles. For the loss of RDME, the value dropped to 59.8% after 100 freeze–thaw cycles for the ordinary concrete, which was lower than the specified failure criterion of 60%. At this point, the RDEM losses for specimens with 4.6% and 5.7% air content were 86.6% and 89.6%, which increased by 45% and 50% compared with those of ordinary specimens. Even after 300 freeze–thaw cycles, the RDEM losses for the air-entrained specimens remained above 80%. Therefore, adding certain air-entraining agents can effectively improve the resistance of concrete to Na_2SO_4 solution freeze–thaw cycles.

The air-entraining agent brings a number of uniformly distributed small bubbles into the concrete; these bubbles block the channel that the sulfate solution passes through and optimize the internal pore structure of the concrete [11]. As a result, the PYA and PYB specimens exhibit good resistance to sulfate solution freeze–thaw cycles. This finding is consistent with those reported by other researchers [12] [14] [18].

The compressive strengths of the PY0, PYA, and PYB specimens after 28 days were 53.4, 46.3, and 43.3 MPa. Concrete compressive strength declined after the addition of the air-entraining agent, and higher air content usually increases the concrete strength loss [17]. Compared with non-air-entrained concrete, concrete with 4.6% and 5.7% air content respectively showed a 13.3% and 19% decrease in compressive strength after 28 days. Therefore, to ensure both compressive strength and resistance to salt solution frost, the air-entraining agent should not be added abundantly to the concrete.

3.4. Service Life Prediction of Concrete Under Freeze–Thaw Cycles in Na_2SO_4 Solution

According to the conclusion obtained in section 3.1, the RDEM loss of concrete often plays a controlling role in the failure modes of concrete subjected to Na_2SO_4 solution freeze–thaw cycles. Thus, a relationship can be established between RDEM loss D and ultimate number of freeze–thaw cycles NF. This relationship can be used to predict the service life of concrete that is subjected to sulfate frost. With the adverse effects of adding fly ash or slag on concrete performance to resist Na_2SO_4 solution freeze–thaw cycles, the regression equations are made only for normal concrete and air-entrained concrete in Figs. 3 and 7 based on the least squares principle. These regression equations are listed in Table 3.

Table 3. *Regression equations of concrete RDEM loss D and freeze–thaw cycle number N.*

Item	Regression equation	R^2
P0.4	$D=-5.3\times10^{-7}N^3+1.1\times10^{-4}N^2+3.8\times10^{-4}N$	0.99
P0.5	$D=-7.4\times10^{-7}N^3+1.4\times10^{-4}N^2+6.5\times10^{-4}N$	0.99
P0.6	$D=-7.0\times10^{-7}N^3+1.3\times10^{-4}N^2+1.6\times10^{-4}N$	0.99
PYA	$D=-3.0\times10^{-8}N^3+1.4\times10^{-5}N^2+2.5\times10^{-3}N$	0.96
PYB	$D=-3.0\times10^{-8}N^3+1.4\times10^{-5}N^2+2.3\times10^{-3}N$	0.90

As shown in Table 3, the correlation coefficients R^2 of all regression equations are more than 0.90. This value indicates that the regression equations are reliable. The number of rapid freeze–thaw cycles that concrete can bear before failure can be calculated based on the regression equations in Table 3 and the failure criterion D=0.4. Li et al. suggested that 1 rapid freeze–thaw cycle for concrete in the laboratory is nearly equal to 12 freeze–thaw cycles for concrete in the natural environment [20]. Moreover, the relationship between the predicted service life of concrete T and the maximum number of rapid freeze–thaw cycles N_F can be calculated using the following equation:

$$T = \frac{BN_F}{kM} \tag{4}$$

where B is the coefficient of rapid freeze–thaw experiment, namely, the number of 1 rapid freeze–thaw cycle equivalent to the natural freeze–thaw cycles, which is generally set at 12; k is the safety factor for concrete structure design, which is commonly set at 1.5; and M is the annual average number of freezing and thawing cycles of concrete structures in the natural environment. According to [20], the value of M in the northwest region of China can be set at 118.

The expected service life of concrete under sulfate freeze–thaw cycles can then be calculated. The predicted service life of concrete with w/c ratios of 0.4, 0.5, and 0.6 is equivalent to 5.1, 4.3, and 4.0 years, respectively. The average life span is 4.47 years. Decreasing the w/c ratio from 0.6 to 0.4 can extend the expected service life by 27.5%. The average life of concrete with 4.6% and 5.7% air content is approximately 25 years, which is nearly 5.6 times that of the non-air-entrained concrete. Therefore, adding the air-entraining agent can significantly extend the service life of concrete in a sulfate frost environment.

4. Conclusions

The following conclusions are obtained from the present experimental study:

1) The appearance damage and mass loss of concrete subjected to freeze–thaw cycles in Na_2SO_4 solution are usually unnoticeable until failure, and the DEM losses often play the controlling role to determine the failure of the specimens. A sudden fracture failure in the middle occurs easily for specimens with low w/c ratio. In general, decreasing the w/c ratio of concrete can slightly increase its resistance to Na_2SO_4 solution freeze–thaw cycles.

2) Adding fly ash or slag reduces the concrete resistance to Na_2SO_4 solution freeze–thaw cycles, and a higher replacement ratio increases the adverse effects. In particular, the adverse effects of slag powder are slightly less than those of fly ash. Therefore, adding fly ash or slag into concrete is not recommended for the environment of Na_2SO_4 solution freeze–thaw cycles.

3) Adding an air-entraining agent to concrete can significantly improve the resistance to Na_2SO_4 solution freeze–thaw cycles. The concrete with 4.6% and 5.7% air content has a service life that is 5 times longer than that of ordinary concrete under the Na_2SO_4 solution freeze–thaw cycles.

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