Influence of Super Absorbent Polymer on the Sulfate Resistance of Cement Mortar

Meiyan Hang¹, Yubin Yang¹, Minghui Jiang¹*, Chenggong Wang², Teng Cheng¹, Hao Wang¹ and Gangming Zhou³

¹ School of Civil Engineering, Inner Mongolia University of Science and Technology, Baotou, 014010, China;
² Guangdong Business and Technology University, Guangdong, 526238, China;
³ Beijing Tieke Shougang Rail Technology Co., Ltd, Beijing, 102206, China.
Email: 15310062855@163.com

Abstract. Structures are easily corroded in the Salt Lake areas of China, especially in sulfate solution. This study was intended to settle the problem of sulfate corrosion failure of concrete structures, the influences of different contents of super absorbent polymer (abbreviated as SAP) on the working performance, mechanical properties, corrosion resistance and expansion performance of cement mortar were studied. The mechanism of SAP in mortar was analyzed and studied by SEM. The results showed that although SAP could slightly decrease the fluidity and strength of cement mortar, but it could remarkably improve the coefficient of resistance erosion of specimens and the inflation coefficient of cement paste. When the content of SAP was 0.3%, the sulfate corrosion resistance and expansion performance of specimens showed the best (the coefficient of resistance erosion and inflation coefficient of mortar specimens were 0.95 and 0.97, respectively). Besides, SAP could release much water in the hydration process, form irregular holes, and increase the porosity of mortar specimens. There would more hydration products generated and filled in the pores during the hydration process, thereby improving the sulfate resistance of mortar specimens. Therefore, this research provides theoretical guidance and basis for the study of sulfate corrosion damage of concrete structures in the future.

Keywords. Super absorbent polymer (SAP), mechanical property, corrosion resistance, expansion property, microscopic mechanism.

1. Introduction
Concrete structures are widely used in engineering structures because of their superior material properties such as convenient material selection, low cost and low energy consumption [1-5]. However, under the circumstance of harsh environments, concrete structures are vulnerable prone to corrosion damage, resulting in a large number of durability problems [6, 7]. It’s reported that the erosion damage of concrete caused by sulfate erosion accounts for about 2.5 trillion dollars annually worldwide [8], which not only caused great economic losses, but also hindered the further development of infrastructure construction [9-11]. Therefore, it is necessary to attach great importance to the sulfate attack damage of concrete structures, thus improving the endurance quality of concrete structures.

A large number of experiments showed that the incorporation of Super absorbent polymer into concrete could effectively promote the characteristics of concrete, thereby improving the working life
of concrete structures [12, 13]. Super absorbent polymer (abbreviated as SAP), which was a kind of intensity hydrophilic polymer compound with 3d network structure. It showed the excellent water absorption and demoulding properties [14,15]. In addition, it could also markedly postpone the decrease of internal humidity of concrete, inhibit spontaneous contraction, promote hydration reaction of cement at 7 days and 28 days, thereby improving hydration degree of the cement [16-20]. Hasholt et al. [21] researched that SAP could accelerate hydrolysis reaction of cement, form hydrated calcium silicate gel, thus enhancing the density of concrete structures. Therefore, SAP is extensively applied in architecture industry to improve the self-healing ability of concrete, reduce concrete cracking, improve the rheological properties, mechanical properties and frost resistance of concrete, et al. Justs et al. [22-24] found that SAP could enhance water retention and alleviate the decrease of internal relative degree of humidity, thereby reducing the shrinkage of concrete in dry environment. Beushausen [25] concluded that SAP did not have much effect on the strength of specimens, but it could significantly improve the durability of specimens. However, most of the studies payed attention to the influence of SAP on the mechanical properties of mortar, the study of SAP on the sulphate resistance performance of mortar was less. Therefore, it is necessary to investigate the influence of SAP on the sulfate resistance of mortar.

Aiming to solve the problem of concrete corrosion damage, compared with sulfate-resistant silicate cement mortar, SAP were 0.1 %, 0.3 %, 0.5 % of the mass of cementitious materials mixed into the mortar in this study. The influences of different dosages of SAP on the working property, mechanical properties, corrosion resistance, expansion performance and microscopic mechanism of cement mortar were investigated. Finally, the results of this research could provide theoretical guidance and basis for the study of sulfate attack failure of concrete structures in the future.

2. Experimental Program

2.1. Experimental Materials
In this study, 42.5 grade ordinary Portland cement, sulfate resistant Portland cement was used, respectively, both of cement were produced by Shandong Chongzheng Special Cement Co., Ltd. The physical properties of the two kinds of cement were listed in table 1. Besides, natural sand was used in this study. The preservatives were mainly composed of nano-silica, which meet standard demands and its dosage was 10% of the weight of cementitious materials. The water absorption principle diagram and microscopic morphology diagram was depicted in figure 1, figure 2, respectively. Different concentrations (3%, 5%, respectively) of Na2SO4 were used as the erosion solutions. Tap water was used mixing water, which meet the requirements of JGJ63-2006.

| Table 1. The Chemical analysis of P.O 42.5 and P.HSR 42.5 cement. |
|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|
| Types           | Fineness (45μm) (%) | Normal consistency (%) | Surface area (m²/kg) | Setting time/min | Folding/Compressive strength / MPa |
| P-O 42.5        | 4.4              | 27.6             | 384              | 135             | 5.0/34.9        |
| P-HSR 42.5      | 4.2              | 28.2             | 325              | 216             | 4.7/27.1        |

3 days 28 days
2.2. Methods

2.2.1. Working Performance and Mechanical Properties Experiment. The natural sand with the size of 0.075mm~2.36mm was selected. The mixing proportions of cement: sand: water was 450: 1350: 225, the content of preservative was 10% of the quality of cementitious material, and the content of SAP was 0%, 0.1%, 0.3% and 0.5% of the cementitious material, respectively, the following experiments of the content of preservative and SAP was the same. The working performance and mechanical properties experiment were measured according to the relevant provisions detailed in the GB/T 17671-1999 and T0506-2005. The fluidity of mortar was tested at first, then the specimens measured 40mm ×40mm ×100mm in size, all of specimens were cured in the room temperature for twenty-four hours and demolded, maintained in fresh water with temperature of 20±1°C. Ultimately, the strength of specimens at different ages were measured.

2.2.2. Sulfate Resistance Coefficient Experiment. The medium-sized natural river sand with the size of 0.5mm~ 1.0 mm was applied in this research, the ratio of cement: sand: water was 300: 750: 150, the dimensions of 10mm ×10mm ×60mm were formed, and first placed in molds and demoulded after curing at 20 °C for 24h ±2 h. Moreover, the specimens were put in 50°C±1°C water curing 7days, and then allocated randomly into 2 categories; one category was cured in fresh water for 28 days, another group was maintained in 3% Na₂SO₄ erosion solution for 28 days; finally, the flexural strength of specimens was tested according to the relevant provisions of GB/T 749-2008, then calculated the corrosion resistance coefficient of specimens.

2.2.3. Expansion Performance Experiment. Since cement mortar contained fine aggregate with uneven distribution, the test results of expansion rate of cement mortar were not accurate. Therefore, in this paper, cement paste was used to replace cement mortar to test the expansion property of cement paste. The expansion performance experiment of cement was carried out according to the relevant provisions of JC/T 313-2009. Firstly, the initial length (L₁) of the cement specimen was tested, and the length (Lₓ) of the specimen at a certain age after curing was tested. The expansion rate (Eₓ) of the cement specimen at a certain age was calculated according to equation (1). Then, the expansion coefficient E of each group was calculated according to JC/T 1011-2006 and equation (2). In the equation, Eₓ represents the expansion rate of the specimen in fresh water for 7 days and 5 % Na₂SO₄ erosion solution for 28 days.

\[ E_x = \frac{L_x - L_1}{250} \times 100 \]  
\[ E = \frac{E_{xr}}{E_x} \]
2.2.4. Microstructural Analysis. For analysis the influence of SAP on hydration products of cement mortar, in this research, the grinder was applied to grind the surface substances on the supericies of specimens. The specimen with a size of 5mm × 5mm × 5mm was slightly beat with a steel drop, and the hydrothermal reaction was terminated by soaking the specimen in anhydrous ethanol until 7 days. The specimen was dried in vacuum about seven days; meanwhile, the specimen was sprayed gold on specimens after post-drying. The 3 days and 28 days microscopic morphologies of each group of specimens were tested.

3. Consequence and Analysis

3.1. Working Performance and Mechanical Properties

Ma [26, 27] found that SAP would obviously change the mortar’s fluidity. This paper, through the experiment, the fluidity of the J group, K group, 0.1%-SAP group, 0.3%-SAP group, 0.5%-SAP group was 195mm, 190mm, 185mm, 175mm, 160mm, respectively. It obtained that the mortar’s fluidity was significantly lower than the J and K group under the circumstance of incorporating SAPs; meanwhile, with increasing the amount of SAP, the fluidity of mortar gradually decreased; moreover, compared with J group, the fluidity of mortar with different SAPs decreased by 5.13%, 10.26%, 17.95%, respectively. The primary reason for the decreased in fluidity might be that the molecular structure of SAPs was network structure, when incorporating SAP into mortar, SAP would absorb partial water in mortar specimens and enwrap water in its network structure, reducing the free moisture in the system, and then increasing the plastic viscosity between cement slurry and sand, therefore, resulting the fluidity of mortar to decrease.

![Figure 3](image3.png) **Figure 3.** The flexural strength image.  
![Figure 4](image4.png) **Figure 4.** The compressive strength image.

The strength of mortar specimens was depicted in figure 3 and figure 4, respectively. It can be seen that the strength of each group of specimens were gradually increasing with the increase of age; nevertheless, the strength of specimens of group K and incorporated SAP group are lower than those of group J, and the strength of mortar specimens incorporated with SAP was gradually decreasing with SAP content increasing. Because group J involved preservatives, the constituents in preservatives would fill some pores, decrease the porousness of mortar specimens, and thus enhancing the specimens’ strength [28]. The group K didn’t contain preservatives, so the density of mortar was lower than that of group J, and the strength was also lower than that of group J. For the mortar specimen incorporated with SAP, with the hydrothermal reaction of cement, when the relative humidity of the mortar specimen decreased, the internal and external osmotic pressure of SAP increased [29]. SAP would release a lot of water, so that the size of SAP particles decreased gradually, leaving a lot of pores in the mortar specimen, increasing the porosity of the mortar specimen, resulting in the bonding force between SAP and mortar decreased gradually, when the mortar specimen was compressed, the stress would be concentrated here, thus promoting the development of micro cracks, therefore, decreasing the specimens’ strength. As the deepening of cement hydration, when the relative humidity inside the mortar specimen decreased, SAP would release water, accelerate the cementitious materials
producing the secondary hydrothermal reaction, and continue generating more products which would tamping the internal specimens’ holes, therefore, enhancing the degree of compaction, also further improving the mechanical properties of the later mortar specimen [27, 30].

3.2. Coefficient of Resistance Erosion
The flexural strength and coefficient of resistance erosion of each group were depicted in figure 5 and figure 6. Both of figures showed that the flexural strength of the K group and the specimens with different SAP contents in clear water was lower than that of the J group, and the flexural strength in 3% Na₂SO₄ solution was almost higher than that of the J group, while the flexural strength of the specimens added different SAP contents was just below the K group. The corrosion resistance coefficient of mortar specimens in K group and SAP group with different contents were greater than 0.83 in J group, indicating that K group and SAP group with different contents could significantly improve the corrosion resistance coefficient and sulfate resistance of mortar specimens. When the content of SAP was 0.3%, the corrosion resistance coefficient was 0.95, indicating that the mortar specimen with this content showed the best anti-sulfate erosion effect. The main reason was that the hydration degree of K group cement was high, and lots of hydration products were blocked the mortar specimen to prevent Na₂SO₄ solution from entering the mortar specimen, so the sulfate corrosion resistance was better. The incorporation of different content of SAP group was due to SAP would absorb the free water in the system, SAP volume expansion occurred, blocking part of the pores inside the mortar specimen, preventing the erosion solution into the mortar specimen, so as to improve the early sulphate resistance performance of specimens. In addition, with the deepening of cement hydration, when the percentage of moisture the mortar specimen degraded, SAP would release water, so that cement hydration generated a large number of hydration products (ettringite crystals, etc.), filling the pores inside the mortar specimen, preventing Na₂SO₄ solution from entering the mortar specimen, thus its sulfate resistance [31].

![Figure 5. The flexural strength image.](image1)

![Figure 6. The corrosion resistance coefficient image.](image2)

3.3. Expansion Coefficient
The expansion rates of different group at each age were tested in this paper. The expansion ratio of mortar specimens was illustrated in figure 7. The figure showed that every specimen showed different degrees of expansion. The expansion rate of J group specimens in fresh water and 3% Na₂SO₄ erosion solution increased slowly, but the expansion rate of specimens in erosion solution was less than fresh water group. The expansion rate of specimens in K group increased slowly after curing for 3 days, and the expanding rate of specimens in corrosive solution was almost greater than that in fresh water. The expanding rate of specimens added different contents of SAP increased rapidly within 3 days to 7 days, while the expansion rate of specimens increased slowly after curing for 7 days. The expansion rate of specimens in corrosive solution was lower than that of specimens in fresh water. When SAP was 0.3%, the expansion rate of mortar specimens in fresh water and corrosive solution was the largest.
According to the test data, the expansion coefficient of J group, K group, 0.1% SAP group, 0.3% SAP group and 0.5% SAP group was 0.82, 0.96, 0.87, 0.97 and 0.94, respectively, which showed that the expansion performance of 0.3% SAP group was the best. The reason was that although the cement specimen of J group was mixed with a amount of preservatives, the cement specimen had a certain expansion in fresh water, but in the sulfate erosion environment, the cement specimen of group J would suffer from sulfate erosion damage, so the expansion performance of the cement specimen of group J was poor. Although the expansion rate of cement specimens in group K in fresh water and sulfate was smaller than that in group J, the expansion coefficient was much larger than that in the reference group. Because the hydration degree of sulfate-resistant silicate cement in group K was relatively high, which would accelerate the cementitious materials hydration to generate a large number of hydration products block the internal pores of cement specimens, so the expansion performance was good; besides, the incorporation of SAP could improve the hydration degree of cement, and promote the hydration of cement to generate lots of hydration products to make the specimen volume expansion; the reciprocity between preservatives and SAP promoted the expansion of cement specimens, thereby improving the expansion performance of cement specimens. The relationship between the expansion rate of cement specimens and the curing age was fitted by Origin software. The fitting results were in line with the equation (3). The fitting curves and fitting parameters were shown in figure 7 and table 2, respectively. The errors were greater than 0.93, indicating that the fitting effect was good. Therefore, equation (3) could be used to predict the variation of the expansion rate of each group of cement specimens with the curing age.

\[ y = a - \frac{b}{(1+c+x)^d} \]  

(3)

Table 2. The fitting parameter of expansion ratio.

| Group | Curing method | a    | b     | c     | d     | R²    | Note                                      |
|-------|---------------|------|-------|-------|-------|-------|-------------------------------------------|
| J     | FW            | 0.075| 109.689| 22674.361| 1.184 | 0.938 |                                          |
|       | FW+ES         | 0.074| 550.889| 58725.680| 1.085 | 0.933 |                                          |
| K     | FW            | 0.016| -0.007| 28.565| -3.598| 0.999 | FW indicates cured in freshwater. FW + ES indicates that it is maintained in fresh water for 7 days and then in 3% Na_2SO_4 solution until the specified age. |
|       | FW+ES         | 0.062| 0.031| -0.017| -0.164| 0.995 |                                          |
| 0.1%  | FW            | 0.160| 0.118| 0.706| 3.646| 0.999 |                                          |
| SAP   | FW+ES         | 0.118| 0.102| 3.803| 2.963| 0.992 |                                          |
| 0.3%  | FW            | 0.128| 0.079| 0.197| 0.977| 0.999 |                                          |
| SAP   | FW+ES         | 0.110| 0.069| 0.504| 1.107| 0.987 |                                          |
| 0.5%  | FW            | 0.128| 0.090| 0.667| 2.365| 0.997 |                                          |
| SAP   | FW+ES         | 0.242| 0.316| 735.845| 12.498| 0.979 |                                          |
3.4. Microscopic Analysis

Considering the above test results, when the SAP content was 0.3 %, the corrosion resistance of mortar specimen showed the best. Therefore, the hydration mechanism of mortar specimens in J group, K group and 0.3 % SAP group were shown in figure 8. According to figure 8 (a), amount of floccular C-S-H gels, Ca(OH)\textsubscript{2} crystals and small amount of needle-like ettringite were formed in J group. Figure 8(b) showed that amount of floccular C-S-H gels, small amount of needle-rod ettringite and Ca(OH)\textsubscript{2} crystals were generated by hydration of group K cement. Figure 8(c) showed that lots of long needle bar ettringite, flocculent C-S-H gel and little Ca(OH)\textsubscript{2} crystals were generated by hydration of cement in 0.3 % SAP group, the cement matrix surface covered with C-S-H gels. The reason was that the composition of preservatives in group J would be filled into the pores on the surface of mortar, making the cement mortar denser, so the strength of group J was higher; the content of Ca(OH)\textsubscript{2} in K group was relatively low, which was mainly due to the reaction between part of Ca(OH)\textsubscript{2} and sulfate in erosion solution to form gypsum, and the gypsum expands after crystallization, when the expansion pressure exceeded a certain limit, the specimen would be corroded by gypsum-type sulfate, so the strength of mortar specimens in K group was significantly lower than that in J group, the reaction equation was as shown in equations (4). While 0.3 % SAP group contained preservatives, and the composition of preservatives would block some pores of cement mortar, which would improve the strength of mortar. However, due to the incorporation of SAP, SAP would provide sufficient water for cement hydration reaction, and promote the reaction of mineral composition C\textsubscript{3}A in cement with gypsum to produce more ettringite, the reaction equation for which was as listed in equation (5) [32]. The formation of ettringite would reduce the specimens’ strength. Therefore, the strength of specimens in the 0.3 % SAP group was less than J group, but slightly higher than that in the K group.

\[
\text{Ca(OH)}_2 + \text{Na}_2\text{SO}_4 + 2\text{H}_2\text{O} \rightarrow \text{CaSO}_4 \cdot 2\text{H}_2\text{O} + 2\text{NaOH} \quad (4)
\]

\[
3\text{C}_3\text{A} + 3 (\text{CaSO}_4 \cdot 2\text{H}_2\text{O}) + 12\text{H}_2\text{O} \rightarrow 3\text{C}_3\text{A} \cdot 3\text{CaSO}_4 \cdot 32\text{H}_2\text{O} \text{(abbreviated as AFt)} \quad (5)
\]

Figure 8 showed the micro-morphologies of mortar specimens after curing for 3 days.

![Figure 8](image)

**Figure 8.** The micro-morphologies of mortar specimens after curing for 3 days.
mortar specimens were extruded, which promoted the development of microcracks, and ultimately led to the decrease of the strength of mortar specimens.

Figure 9. The micromorphology diagram of mortar specimens after curing for 28 days.

4. Conclusions
(1) SAP could significantly reduce the free water in the system, thereby reducing the fluidity of mortar.
(2) SAP would slightly reduce the strength of mortar specimens. When SAP was 0.5 %, the strength reduction was the most significant.
(3) SAP could significantly improve the coefficient of resistance erosion of specimens. When SAP was 0.3 %, the coefficient of resistance erosion of specimens was the largest and the sulfate resistance effect showed the best.
(4) When SAP was 0.5 %, the expansion coefficient of specimens was higher than that of J group and K group, but when the content of SAP was 0.3 %, the expansion rate of mortar specimens in fresh water and corrosive solution was the largest, indicating that the expansion performance of mortar specimens was the best.
(5) SAP could enhance the cement’s early hydration degree, improve the hydration of cement to generate a large number of needle bar ettringite, release much water in the hydration process, form irregular holes, and increase the porosity of mortar specimens. Cement hydration would generate more hydration products blocked the pores, thereby improving the sulfate resistance of mortar specimens.

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