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Development and Properties of Sulfate-resistant and Corrosion-inhibiting Admixtures

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ABSTRACT

Sulfate and chloride-induced corrosion of concrete and steel reinforcement are the most important causes of premature failure of durability of concrete structures. To prevent damage in concrete structures, the application of sulfate-resistant and corrosion-inhibiting admixtures has proven to be an effective method. In this study, a new type of corrosion-inhibiting admixtures including organic and special inorganic components are developed and the properties of mortar mixed with them was investigated. The results show that NC-CZ series of sulfate-resistant and corrosion-inhibiting admixtures have been successfully developed. The mortar with NC-CZ has good resistance to sulfate attack, whose corrosion resistance coefficient of mortar is 1.07, meeting the standard requirement and even larger than that of moderate sulfate-resistant Portland cement. The diffusion coefficient of chloride ion at 28d decreases by 35% around. Meanwhile, the water absorption is obviously decreased. The steel bars in mortar mixed with corrosion-inhibiting admixtures don't occur rusting. By contrast, the steel bars in mortar without corrosion-inhibiting admixtures occur rusting, whose area rate of corrosion is more than 20%. This study could lead to significant benefits for durability and service life of reinforced concrete structures in China.

1. INTRODUCTION

Reinforced concrete, as a structural material, has hold the greatest application of construction, bridge, tunnel, and other engineering structures [1,2]. However, to extend the service life of existing reinforced concrete structures has been one of the major challenges, especially those exposed to marine or de-icing salts environment [2,3]. Moreover, the widespread presence of oceans, saline-alkali and brine in salt lakes also provides a rich source of chloride ions and sulfate ions for the corrosion of reinforced steels in concrete. This adversely affects the durability of the concrete structure, leading to structural damage before the designed life [3,4,5]. Thus, it is of practical significance to reduce or even eliminate the corrosion of reinforced concrete in such aggressive environments.

A wide range of methods have been employed to reduce the corrosion of reinforced steel, such as using improved materials (stainless steel), cathodic protection, steel coating, use of corrosion inhibitors, etc [6,7,8,9]. Among them, the application of corrosion inhibitors has proven to be an effective method. They are relatively low cost and easy to handle, as compared to other preventive methods for corrosion protection [3,9,10]. However, corrosion-inhibitor with single-function is far from satisfying the anti-corrosion function of present engineering applications. The compound anticorrosive and rust inhibitors have become the concern of research. They can make use of the synergistic effect between the components, obtaining the advantages of each component and overcoming the shortcomings of a single component rust inhibitor [11,12].

There exist wide coastal area and saline areas in Shandong, where the groundwater is rich in harmful substances such sulfate, magnesium salts and chloride ions. They can cause erosion of E salt and G salt of ettringite formation, and M salt erosion of Cement hydration products decomposed by Mg^{2+} and corrosion reinforced concrete. In this paper, based the fact above mentioned, a new type of sulfate-resistant and corrosion-inhibiting admixtures is developed, which was composed of rust inhibitor, compacting component, shrinkage component, corrosion ion transport inhibitor component and air entrained component. Meanwhile, the performance of mortar mixed with the admixtures was investigated.

2. EXPERIMENTAL WORK

2.1. Technical route

The main materials for preparation of sulfate-resistant and corrosion-inhibiting admixtures include reducing water component, steel rust component, crack resistant and dense component, reducing shrinkage component, inhibition ion erosion transmission component and air entraining component.

The shrinkage-reducing type polycarboxylic acid high performance water reducing agent made by our company is used as reducing water and reducing shrinkage components. The crack resistant and dense component is superfine mineral admixtures with slight expansion. Super disperse hydrophobic modified silicon nanoparticles and strong polar phosphonate ions were introduced as inhibition ion erosion transmission component. Air entraining component is supplied by the air-entraining agent from our company.

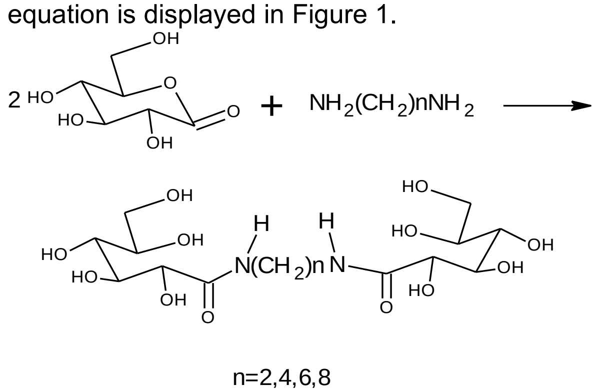
For the corrosion inhibitor component, nitrite anode corrosion inhibitor components with low cost and good effect of corrosion inhibition and glucamide or amino alcohol cathode rust inhibitor components are used to select through the steel bar salt resistance to salt water impregnation performance test. The test is carried according to " Technical specification for application of reinforcement corrosion inhibitors " (YB/T 9231).

2.2. Synthesis of new organic corrosion inhibitor

In this study, Glucose derivatives and Polyamines are used to react to synthesize glucoamides, which is named as PTAM.

The chemical reagents include D-gluconate -δ -lactone (Anhui Xingzhou Pharmaceutical & Food Co., LTD.), Organic amine, ethylenediamine, butanediamine, hexediamine, octyl diamine (Shanghai National reagent), Methanol and anhydrous ethanol (Tianjin Kermeo Chemical Reagent Co., LTD.), sodium chloride and sodium hydroxide.

The synthesis process is as follows: a certain amount of D-gluconate -δ -lactone was weighed in a 100 mL three-necked flask, and then 25~30 mL methanol was added (to make the glucolactone completely dissolved at 60℃). Heat and stir until the glucolactone is dissolved. Appropriate amount of organic amine was dissolved in a small amount of methanol, and added into the three-neck flask, which maintained reacting at 60 ℃ for 3 h, and cooled to room temperature. After that, a large amount of white powder is precipitated. Then, washing and filtration with methanol are repeated to remove unreacted organic amines. A mixture of V (anhydrous ethanol) ：V (water) =9:1 is used to wash. Finally, gluconamide rust inhibitor is obtained by drying. The equation is displayed in Figure 1.



n=2,4,6,8

Figure 1. Reaction formula of PTAM

2.3. Formulation optimization of sulfate-resistant and corrosion-inhibiting admixtures

In this experiment, the cement is Ordinary Portland cement 42.5. Fine aggregate is sand from river with fineness modulus of 2.8. Its performance indicators meet the national standard "Sand for construction" (GB/T 14684). Coarse aggregate is crushed limestone with grain size of 5~25mm. Its performance indicators meet the national standard "Pebbie and crushed stone for building" (GB/T 14685). Table 1 gives the indicators of coarse aggregate. The mix ratio of concrete is as follows: cement:sand: coarse aggregate=380:732:1098.

Table 1. Performance index of coarse aggregate

apparent density (kg/m^3)	Packing density (kg/m3)	Content of enlongated flaky particle (%)	crushing index (%)	accumulated retained percentage (%)			
				2.36 mm	4.75 mm	16.0 mm	26.5 mm
2710	1410	6.9	11.5	100	99.7	41.3	0

According to the determined technical route, after a large number of experimental studies, of various components were selected, after the initial selection of raw materials, and then select the best value of various raw materials, and then determine the best formula composition of anticorrosive rust inhibitor.

Powdery and liquid sulfate-resistant and corrosion-inhibiting admixtures are both investigated. L9(3^4) orthogonal experiment with four factors and three levels was carried out under the condition that the components of entrained air and other factors remains unchanged.

Table 2. L9(3^4) orthogonal experimental design table

Powder Number/Liquid Number	Factors			
	A	B	C	D
P1/L1				
P2/L2	A1	B1	C1	D1
P3/L3	A1	B2	C2	D2
P4/L4	A1	B3	C3	D3
P5/L5	A2	B1	C2	D3
P6/L6	A2	B2	C3	D1
P7/L7	A2	B3	C1	D2
P8/L8	A3	B1	C3	D2
P9/L9	A3	B2	C1	D3
P10/L10	A3	B3	C2	D1

Four influencing factors are respectively dense component (A), inhibition ion erosion transmission component (B), organic rust inhibitor component (C), inorganic rust inhibitor component (D). For the powdery sulfate-resistant and corrosion-inhibiting admixtures, the admixture amount is 10%, accounted for the weight of the cementitious material. For the liquid sulfate-resistant and corrosion-inhibiting admixtures, the admixture amount is 4%, accounted for the weight of the cementitious material.

2.4 Test materials and methods for performance study of mortar

In this part, the test materials used are Benchmark cement from Qufu Zhonglian Cement Co., LTD, Sulfate-resistant Portland cement P·HSR42.5 and moderate sulfate-resistant Portland cement P·MSR42.5 and six samples from the market numbered as S1~S6.

The test methods referred to include “Sulfate corrosion-resistance admixtures for concrete” (JC/T 1011), “The method for determining the choloride diffusion coefficient for cement” (JC/T 1086), “Water-repellent admixture for mortar and concrete” (JC 474) and Appendix C from “Technical specification for application of reinforced concrete anti-corrosion inhibitor” (GB/T 33803).

3. RESULTS AND DISCUSSION

3.1 Selection of corrosion inhibitor component

The corrosion inhibitor is added into experimental solution according to the designed dosage. Sodium nitrite and compound amino alcohols are respectively designated as YN and AN. The experimental results are shown in Figure 2, Figure 3, and Table 3.

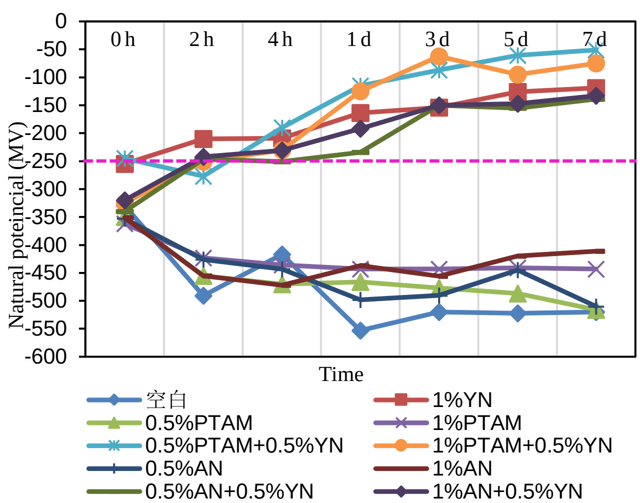


Figure 2. The curve of natural electrode potential and time

From the intuitive appearance of the steel bars and solution (Table 3), it is easy to find that steel bars in salt water corrode quickly and the solution becomes turbid without corrosion-inhibiting admixtures. With the prolongation of time, the solution becomes yellowish-brown, the reinforcement appears precipitation, and the corrosion area gradually increases. In combination with Figure 2, it can be seen that when PTAM and AN is separately added to the solution, black spots appear at the end of the reinforcement bar, which means that their effect of corrosion inhibition alone is not obvious. Meanwhile, 1%PTAM or 1%AN has little positive effect. However, adding 1%YN can inhibit corrosion, and the natural electrode potential is -119mV at 7d, greater than -250mV. While 0.5% PTAM and 05%YN are added, no corrosion on the surface of the reinforced bar occurs at 7d, and the initial natural electrode potential was -245mV, -115mV at 1d and -51mV at 7d. -115mV at 1d is equivalent to that of 1%YN mixed with solution at 7d. This means that 0.5% YN combined with 0.5% PTAM has better corrosion resistance than 1%YN combined with 0.5% PTAM. For mixing AN and YN, 0.5% AN and 0.5%YN make no corrosion on the surface of the reinforcement bar at 7d, and the natural electrode potential of 7d is - 139mV, which is 254 mV less than that of 1.0%YN mixed at 7d. It indicates that adding 0.5% AN and 05%YN to the solution is not as good as the corrosion resistance of 0.5% AN and 1%YN.

Figure 3. Comparison diagram of corroded and uncorroded steel bars

Figure 3 gives the comparison diagram of corroded and uncorroded steel bars which are in the solution for 7d, it clearly shows the good corrosion resistance of 0.5% PTAM and 0.5% YN, which is in accordance with Table 3 and Figure 2.

PTAM is characterized by polyhydroxyl structure and amide group in the molecule. Hydroxyl group can be used as an effective adsorption group in alkaline chloride medium, which can be better adsorbed on the surface of steel bar, effectively improving the corrosion and rust resistance of steel bar, while N atoms in amide group can be tightly adsorbed with steel bar, forming an effective protective film for the protection of steel bar. It shows that polyhydroxy amide compound rust inhibitor can protect steel bar well in the chlorine salt environment, greatly reduce the occurrence of corrosion of steel bar, at the same time a large number of hydroxyl is conducive to the dispersion performance of rust inhibitor in concrete. The combination of PTAM and nitrite has a certain synergistic effect on multi-component rust inhibitors. Nitrite oxidizes iron ions on the surface of steel bar, forming a dense passivation film and reducing the corrosion area. PTAM can improve the pitting effect of nitrite at a low dosage.

Table 3. Appearance of steel bar resistant to salt water impregnation

Admixtures	Phenomenon			
	1d	3d	5d	7d
/	the end of steel bar corrodes	yellow-green rusting surface increases and the solution is turbid	solution is yellow-green and rusting surface increases	yellow-green precipitation occurs and rusting surface increases
1% YN	solution and reinforcement have no changes	solution and reinforcement have no changes	solution and reinforcement have no changes	solution and reinforcement have no changes
0.5% PTAM	black spots appear on two ends	solution is turbid and corrosion changes little	solution is slightly yellow-green and corrosion changes little	solution is slightly yellow-green and corrosion changes little
1% PTAM	black spots appear on one end	solution is slightly yellow-green and corrosion changes little	solution is slightly yellow-green and corrosion changes little	solution is slightly yellow-green and corrosion changes little
0.5% PTAM+ 0.5% YN	solution and reinforcement have no changes	solution and reinforcement have no changes	solution and reinforcement have no changes	solution and reinforcement have no changes
1.0% PTAM+ 0.5% YN	solution and reinforcement have no changes	solution and reinforcement have no changes	solution and reinforcement have no changes	solution and reinforcement have no changes
0.5% AN	black spots appear on two ends	black spots appear on two ends	solution is slightly yellow-green and corrosion changes little	solution is slightly yellow-green and corrosion changes little
1.0% AN	black spots appear on one end	solution is slightly yellow-green and corrosion changes little	solution is slightly yellow-green and corrosion changes little	solution is slightly yellow-green and corrosion changes little
0.5% AN+ 0.5% YN	solution and reinforcement have no changes	solution and reinforcement have no changes	solution and reinforcement have no changes	solution and reinforcement have no changes
1.0% AN+ 0.5% YN	solution and reinforcement have no changes	solution and reinforcement have no changes	solution and reinforcement have no changes	solution and reinforcement have no changes

Table 4. compressive strength of concrete doped with powdery sulfate-resistant and corrosion-inhibiting admixtures

Number	Slump(mm)	Compressive strength(MPa)			
		3d	7d	28d	60 d
P1	80	19.8	28.9	39.2	41.2
P2	80	25.1	33.8	43.4	46.6
P3	90	26.2	35.4	44.3	45.5
P4	110	25.0	30.2	38.6	41.8
P5	120	23.7	30.0	40.2	45.2
P6	110	23.8	31.1	41.0	42.8
P7	80	26.1	33.5	42.7	44.8
P8	100	26.4	32.6	42.5	45.4
P9	90	27.0	32.1	44.4	46.3
P10	80	23.9	30.8	42.6	48.3

Table 5. Range analysis of orthogonal test for powdery sulfate-resistant and corrosion-inhibiting admixtures

Factors / Time	A	B	C	D
3d	1.24	0.67	1.47	1.96
7d	1.60	1.37	1.83	3.06
28d	1.87	1.93	2.80	2.10

Table 6. compressive strength of concrete doped with liquid sulfate-resistant and corrosion-inhibiting admixtures

Number	Slump(mm)	Compressive strength(MPa)			
		1d	3d	7d	28 d
L1	70	11.9	20.9	29.8	40.7
L2	80	12.1	22.3	30.4	41.0
L3	100	14.3	22.7	31.8	40.1
L4	85	14.2	22.9	29.7	42.2
L5	80	13.4	22.9	30.4	40.0
L6	90	13.4	23.3	31.1	39.4
L7	80	13.4	22.7	29.7	39.7
L8	70	14.8	23.9	30.7	43.1
L9	80	13.1	21.9	31.1	40.4
L10	80	13.1	22.8	29.0	41.2

Table 7. Range analysis of orthogonal test for powdery sulfate-resistant and corrosion-inhibiting admixtures

Factors / Time	A	B	C	D
3d	0.34	0.4	1.07	0.8
7d	0.36	1.86	0.1	0.7
28d	1.87	1.4	1.2	1.7

3.2. Formulation optimization of sulfate-resistant and corrosion-inhibiting admixtures

The compressive strength of concrete doped with powdery sulfate-resistant and corrosion-inhibiting admixtures and the range analysis is respectively shown in Table 4 and Table 5. It can be found the primary and secondary education of the influencing factors. Overall, D and C have greater influence than A and B. However, a different conclusion is obtained from the results of concrete doped with liquid sulfate-resistant and corrosion-inhibiting admixtures and the range analysis (Table 6 and Table 7).

Finally, the composition of the formula is determined through comprehensive consideration. The powdery sulfate-resistant and corrosion-inhibiting admixtures include two kinds, named as NC-CZ1(S) and NC-CZ2(S). The liquid one is named as NC-CZ3(L). Then, the verifying experiments are further carried out. As is shown in Figure 4. It can be seen that NC-CZ1 decreases the strength of concrete at 1d. This is due to its certain plasticizing and retarding effect. On the contrary, NC-CZ2 has no adverse effect on concrete. Besides, the strength of 3d and 7d does not decrease compared with blank concrete. The strength of 28d decreases slightly when 5% is added, and the strength grows continuously in the later period.

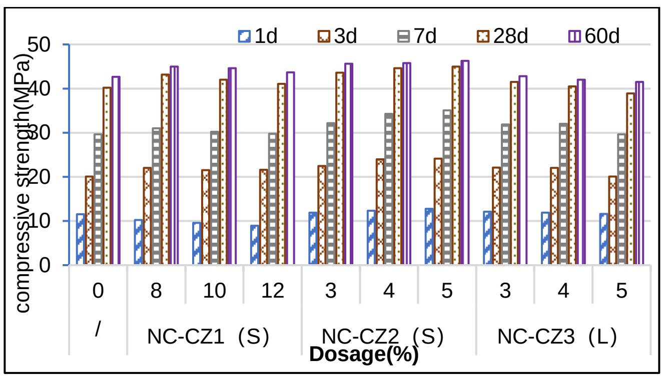


Figure 4. Influence of dosages on the compressive strength of concrete with studied admixtures

It can be concluded that three kinds of sulfate-resistant and corrosion-inhibiting admixtures are successfully developed, which are named as NC-CZ series, including powdery NC-CZ1, NC-CZ2 and liquid NC-CZ3. The corresponding normal dosage is separately 10%, 4% and 4%.

3.3. Resistance to sulfate corrosion

The sulphate resistance of developed product NC-CZ1 was compared with that of P·HSR42.5, P·MSR42.5 and similar products on the market (S1~S6). The mix ratio of mortar is as the following: cement: sand: water =1: 2.5: 0.485. The coefficient of corrosion resistance is shown in Figure 5. The P·HSR42.5 with the corrosion resistance coefficient of 1.14 has the best resistance to sulfate. The coefficient of mortar mixed with NC-CZ1 is 1.07, meeting the requirement of JC/T 1011 that is not less than 0.85. In addition, it is superior than samples from market. What's more, it is larger than that of P·MSR42.5. It is indicated that NC-CZ1 has good resistance to sulphate.

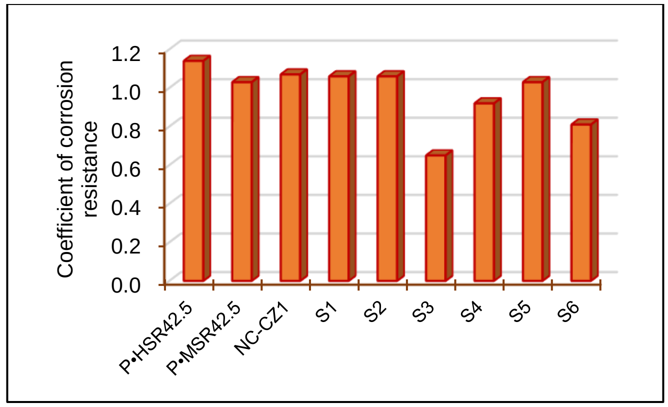


Figure 5. Coefficient of corrosion resistance of mortar mixed with corrosion inhibitors

The larger the volume of the specimen is, the denser the specimen is. At the same time, the higher the strength is, and the more difficult it is to be eroded. Therefore, specimens with larger size of 40mm×40mm×160mm are furthermore investigated. Here, the cement-sand ratio is 1:6. One day after molding, the mold is removed. The specimens are cured at 50℃ for 7 days. Then they are soaked in 15% sodium sulfate solution for 90 days to test the flexural strength ratio, which can evaluate the resistance of the specimen to sulfate erosion. The results are given in Figure 6.

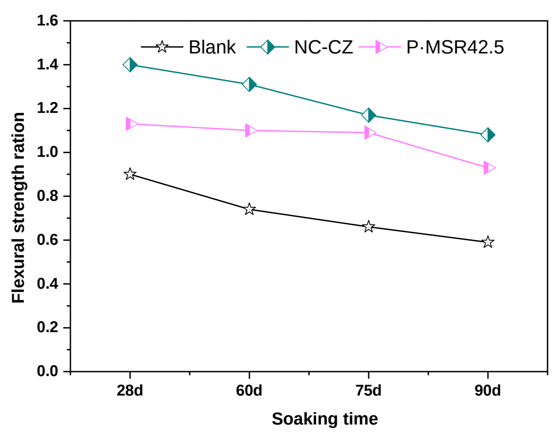


Figure 6. Flexural strength ratio of mortar at different soaking time in sulphate erosion solution

It can be seen that the flexural strength ratio decreases obviously as the soaking time elapses. The flexural strength ratio of blank specimen without corrosion inhibitors is larger than 0.85 after soaking for 28d.While after soaking for 60d, the flexural strength ratio is less than 0.85. This means it has been eroded by sulfate at 60d. The flexural strength ratio of mortar doped with NC-CZ and P·MSR42.5 is advanced. It is up to 1.31 at 60d when NC-CZ is added, and it is still 1.20 at 90d, which is higher than that of mortar with P·MSR42.5. Thus, it can be concluded that the mortar with NC-CZ has higher resistance to sulfate than that with P·MSR42.5. This is consistent with the conclusion from Figure 5.

3.4. Resistance to chloride ion erosion

The chloride diffusion coefficient at 28d can reflect the resistance to chloride ion erosion. The mix proportion and fluidity of the mortar is in Table 8. The content of doped NC-CZ is 10% and 12%. Figure 7 supplies the chloride diffusion coefficient at 28d. As is shown, compared with blank mortar, the chloride ion diffusion coefficient at 28d decreases by about 35% by adding NC-CZ1. Besides, the diffusion coefficient of chloride ion at 28d is between P·HSR42.5 and P·MSR42.5. This means that the developed sulfate-resistant and corrosion-inhibiting admixtures (NC-CZ) has good resistance to chloride ion erosion, better than moderate sulfate-resistant Portland cement P·MSR42.5, though it is inferior to Sulfate-resistant Portland cement P·HSR42.5.

Table 8. Mix proportion of studied mortar doped with different admixtures

Number	Mix proportion				Fluidity(mm)	Remarks
	C(g)	S(g)	NC-CZ1 (g)	W(g)		
T1	450	1350	0	225	220	Benchmark cement
T2	405	1350	45	220	225	
T3	396	1350	54	216	220	
T4	450	1350	0	225	225	P·MSR42.5
T5	450	1350	0	225	230	P·HSR42.5

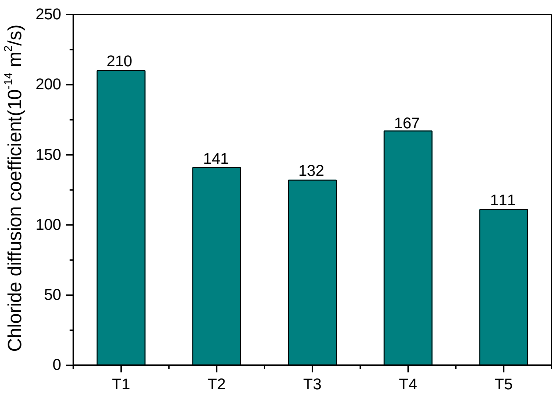


Figure 7. Chloride diffusion coefficient of studied mortar at 28d

3.5. Performance of water absorption

The properties of water absorption of mortar mixed with NC-CZ1 and blank mortar are studied. The detailed results are shown in Table 9. It is obvious that the ratio of quantity of absorbed water at 48h is 58.9% when mortar is doped with NC-CZ1. The permeability coefficient is also significantly reduced. It follows that the developed sulfate-resistant and corrosion-inhibiting admixtures can densify concrete structure, and significantly reduce the water absorption.

Table 9. Results of properties of water absorption of mortar

Dosage of NC-CZ-1 (%)	Compressive strength(MPa)	Quantity of absorbed water(g)		Ratio of quantity of absorbed water (%)		Permeability coefficient of 48h($\times10^{-9}$m/s)
	28d	24h	48h	24h	48h	
0	34.6	53	56	100	100	1.08
10	37.1	29	33	54.7	58.9	0.64

3.6. Dry and wet cold and heat cycle test of mortar sample

The mixing ratio of mortar is changed to 1:3:0.5, and the dosage of developed of NC-CZ1, NC-CZ2 and NC-CZ3 is respectively 10%, 4% and 4%. After 20 cycles, the corrosion on the surface of steel bar is checked and the percentage of corrosion area on the surface of the samples is recorded, which is shown in Figure 8. The reinforcement in the mortar without rust inhibitor easily corrodes, and the area rate of corrosion was more than 25%. Whereas, these mortar with the developed sulfate-resistant and corrosion-inhibiting admixtures don't appear corrosion. From Figure 9, the corroded and uncorroded steel bars can be clearly seen. This illustrates that the developed sulfate-resistant and corrosion-inhibiting admixtures have excellent resistance to corrosion.

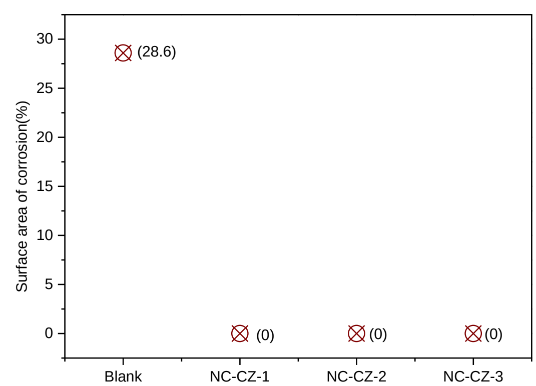


Figure 8. Corrosion of steel bar in mortar under hot and dry circulation

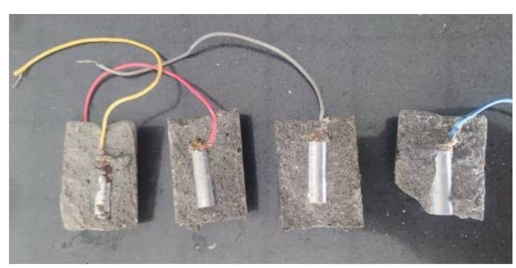


Figure 9. Corrosion of steel bar in mortar above

4. CONCLUSION

From this study, the following conclusions ca be drawn.

■ Through the orthogonal experiments, the composition of sulfate-resistant and corrosion-inhibiting admixtures is determined. NC-CZ series of sulfate-resistant and corrosion-inhibiting admixtures of sulfate-resistant and corrosion-inhibiting admixtures are successfully developed, which are powdery NC-CZ1, NC-CZ2 and liquid NC-CZ3. The corresponding normal dosage is separately 10%, 4% and 4%.

■ NC-CZ1 has good resistance to sulphate. The sulfate resistance coefficient of mortar mixed with NC-CZ1 is 1.07, meeting the requirement of JC/T 1011 that is not less than 0.85. In addition, it is superior than samples from market and larger than that of P·MSR42.5.

■ NC-CZ has good resistance to chloride ion erosion, the diffusion coefficient of chloride ion at 28d decreases by 35% around.

■ NC-CZ can densify concrete structure, and significantly reduce the permeability coefficient. Besides, the developed NC-CZ have excellent resistance to corrosion. The steel bars in mortar mixed with NC-CZ don't occur rusting.

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