WEP Warwick Evans Publishing

International Journal of Mechanical and Electrical Engineering
ISSN: 3005-9615 (Print), ISSN: 3005-7132 (Online) | Volume 1, Number 1, Year 2023
DOI: https://doi.org/10.62051/ijmee.v1n1.03
Journal homepage: https://wepub.org/index.php/IJMEE/index

Preparation of cementitious osmotic crystalline waterproof material and its waterproofing mechanism analysis

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ABSTRACT

Cementitious Capillary Crystalline Waterproofing materials (hereinafter: CCCW) with the advantages of good waterproof effect, green and low cost, it is widely used in the field of building waterproofing. Researchers and scholars have studied the proportioning of such materials. However, the experimental methods used in these studies are not perfect enough, therefore, this study uses a seven-factor, two-level orthogonal test method to conduct experiments and to study the waterproofing effect of CCCW coated on the substrate at different ages, and to discuss the existing waterproofing mechanism in conjunction with it. The results show that the group's homemade waterproofing agent has a good waterproofing effect, and the compressive strength is significantly increased at different ages, which suggests that CCCW can promote the hydration of cement and produce more penetrating crystals, thus blocking the capillary pores and microcracks, and making the matrix dense and waterproof.

KEYWORDS

Penetration crystallization; Waterproofing materials; Orthogonal tests; Optimal design.

1. INTRODUCTION

The problem of concrete permeability has been the focus of research in the field of construction. Concrete is a non-homogeneous brittle material, from a microscopic point of view belongs to the porous structure, consisting of tunnel-like capillaries, which is the main cause of water seepage and leakage, and secondly, the existence of cracks and capillaries allows harmful chemicals to enter into the interior of the concrete leading to concrete deterioration, causing corrosion of the internal steel reinforcement [1], which not only reduces the overall durability of the structure, but also even results in huge repair costs and major safety accidents. Therefore, whether from the point of view of economic or social benefits, the study of a waterproofing material with good waterproofing effect and low cost can ensure the integrity and stability of the building structure and reduce the maintenance cost, which is of great significance in the field of engineering and construction.

Cementitious Capillary Crystalline Waterproofing Materials (hereinafter referred to as CCCW) is a German chemist Lauritz Jensen in 1942 to solve the problem of water seepage of cement ships invented cement-based osmotic crystalline waterproofing materials, is a new type of rigid waterproofing materials made of silicate cement, quartz sand and so on as a substrate, mixed with a certain amount of active chemicals [2]. China's current building waterproofing materials are divided into rigid waterproofing materials and flexible waterproofing materials. Flexible waterproofing materials suffer from the problem of easy ageing, which leads to high maintenance costs and prevents flexible waterproofing materials from meeting the needs of certain sectors. Relative to traditional waterproofing materials, CCCW in the active substances and hydration products to generate insoluble in water needle crystals, blocking from any direction of water flow and other liquid erosion, filling the capillary pores and micro-cracks, thus improving the compactness of concrete and waterproofing to reduce the occurrence of carbonation of concrete, seepage and corrosion problems [3, 4],and it is a relatively new type of inorganic penetration waterproofing materials, in the waterproofing of concrete in the project in the display of the irreplaceable superiority.

At present, researchers and scholars have studied CCCW more, but most of them stay at the stage of the action mechanism of waterproofing, and lack of research on the optimisation of its components and mechanical properties. Therefore, this group compares the self-developed CCCW with the blank group and the waterproof materials with better market sales performance, and carries out the performance analysis and mechanism discussion, so as to provide theoretical support for its wider application in the market.

2. RAW MATERIALS AND TEST METHODS

2.1. Raw materials

Cement: P.O 42.5 ordinary silicate cement from Jiaozuo Qianye Cement Co, Ltd, whose performance data are shown in Table 1;

Sand: ISO standard sand from Xiamen Aceo Standard Sand Co;

Water: Cite the laboratory tap water to meet the requirements of the national standard requirements;

Waterproofing agent: self-made by the group. In order to understand the difference between self-developed waterproofing agents and commercially available imported products, the imported waterproofing agents with good market sales performance were selected for comparison with the group's own waterproofing agents.

Table 1. Chemical composition of cement

Composition	Al2O3	MgO	SiO2	Fe2O3	CaO	Na2O	K2O
Mass fraction/%	5.32	2.51	21.65	3.22	61.91	0.21	0.16

2.2. Test Methods

In accordance with the "cement-based osmotic crystalline waterproofing materials" (GB18445-2012) for mortar impermeability test, the use of (70 × 80 × 30) mm test moulds, moulding three groups, respectively, for the blank group, homemade group and commercially available group, to be the specimen maintenance to 27d, the specimen will be taken out and put into the room to dry naturally.

In accordance with the "Standard Test Methods for Basic Performance of Building Mortar" (JGJ/T70-2009), the mortar compressive test was carried out by using (70.7×70.7×70.7)mm triple moulds, which were put into the standard curing room to carry out the test of compressive strength after 28d of curing, and the results of the test were taken as the average of the test results of the three specimens.

In accordance with the "cement sand strength test" (GB/T 17671-1999) for mortar flexural, water absorption test, flexural and water absorption test specimens using (40 × 40 × 160) mm triple mould, into the standard maintenance room for 28d after the compressive strength test, the test results of the test results to take the average of the results of the three specimens.

2.3. Test programme

Considering the feasibility of the test, if the full-scale test method is used to optimise the proportioning, not only is it not permissible in terms of time, but it will also consume a large amount of raw materials and energy, so it is necessary to choose a test method that does not affect the results of the test but also reduces the number of tests. Orthogonal experimental design, on the other hand, is a commonly used optimisation method that can significantly reduce the number of trials, and has been experimentally verified over the years to be effective. In order to obtain waterproof materials with better waterproofing effect, the development of masterbatch is the key, in this study, orthogonal experimental design method was used to optimise the preliminary mix ratio. After testing the initial analysis of the components of the commercially available waterproofing agent to determine the amount of auxiliary components, in order to ensure that the formulated waterproofing agent work performance requirements, non-core waterproofing components of the amount of mixing is fixed as follows: ordinary silicate cement 0.3% and silica fume 0.2%, tartaric acid 0.015%, defoamer 0.1%. In order to respond as accurately as possible to the formulated waterproofing agent in the actual application of the effect of auxiliary materials added to the test groups in equal quantities.

Table 2. Orthogonal test factor level table

level	A Na_2SO_4	B Na_2SiO_3	C CaMg(CO3)2	D $Ca(HCO_2)_2$	E $C_6H_8O_7$	F MgO	G Complexing agent
1	0.3	0.2	0.2	0.2	0.1	0.1	0.1
2	0.6	0.3	0.4	0.3	0.2	0.2	0.2

3. RESULTS AND ANALYSES

3.1. 3.1 Analysis of orthogonal test results

In order to reduce the cost of raw materials and improve the performance, seven core components were selected as variables in this test to characterise the waterproofing performance of CCCW in terms of the impermeability pressure ratio. According to the seven-factor two level in Table 2, eight groups of masterbatch materials were designed, and the eight groups of masterbatch materials were ground and added with other additives such as cement to configure waterproofing materials to replace cement in equal quantities, and the baseline mortar 28d seepage resistance pressure was 0.3 MPa.

Table 3. Results of orthogonal tests for L8(2^7)

Experiment No.	A Na_2SO_4	B Na_2SiO_3	C CaMg(CO3)2	D $Ca(HCO_2)_2$	E $C_6H_8O_7$	F MgO	G Complexing agent	28d impermeable pressure ratio
1	0.3	0.2	0.2	0.2	0.1	0.1	0.1	433
2	0.3	0.2	0.2	0.3	0.2	0.2	0.2	300
3	0.3	0.3	0.4	0.2	0.1	0.2	0.2	400
4	0.3	0.3	0.4	0.3	0.2	0.1	0.1	433
5	0.6	0.3	0.2	0.2	0.2	0.1	0.2	333
6	0.6	0.3	0.2	0.3	0.1	0.2	0.1	300
7	0.6	0.2	0.4	0.2	0.2	0.2	0.1	267
8	0.6	0.2	0.4	0.3	0.1	0.1	0.2	233

Note: The impermeable pressure ratio is the percentage of the ratio of 28d impermeable pressure between the test group and the blank group. Where K1, K2, K3 the same level of the sum of the test indicators of each factor, R is the same level of each factor in the average of the maximum value and the minimum value of the difference between. The large extreme difference indicates that the factor in this level of change range caused by the difference is large, the impact on the test index is large, is the main influence factor; the small extreme difference indicates that the factor on the test results of the impact is small, is a secondary influence factor. There are two methods of analysing the results of orthogonal experiments, namely, intuitive analysis (also called the extreme difference method) and analysis of variance (ANOVA), and in this paper, we choose the extreme difference analysis method to analyse the results of orthogonal experiments, and the intuitive analysis table derived from the results of orthogonal experiments is shown in the table below.

Table 4. Visual analysis of L8(2^7) orthogonal tests

projects	A Na_2SO_4	B Na_2SiO_3	C CaMg(CO3)2	D $Ca(HCO_2)_2$	E $C_6H_8O_7$	F MgO	G Complexing agent
K1	391.5	341.5	308.3	358.3	341.5	358	358.3
K2	283.3	333.3	366.5	316.5	333.3	316.5	316.5
R	108.3	8.3	58.3	41.8	8.3	41.3	41.75
optimum level	A1	B1	C2	D1	E1	F1	G1

The optimum combination of factor levels was determined by comprehensive orthogonal test visual analysis table. Potassium aluminium sulphate has the largest extreme difference, indicating that sodium sulphate has the greatest influence on the impermeability of CCCW The most important factor affecting the waterproofing effect is sodium sulphate, followed by calcium magnesium carbonate, calcium formate and magnesium oxide, with the second highest influence of the complexing agent, the relatively small influence of sodium silicate, and the smallest influence of citric acid. Sodium sulphate can significantly improve the impermeability pressure of mortar impermeable specimens, which is due to the fact that the sulphate in sodium sulphate is able to react with the hydration products of cement such as calcium hydroxide and calcium aluminium hydrate to produce insoluble crystals of calcium sulphate and calomel and so the impermeability pressure of the mortar is improved. Sodium silicate, calcium magnesium carbonate is mainly used as a crystalline precipitant, the silicate ion in sodium silicate will penetrate into the mortar impermeable specimen void with water as a medium, and react with the calcium ions in the mortar specimen to generate crystals to block the capillary channels, thus increasing the denseness of the mortar, and the complexing agent is able to form a complex with the calcium ions, which will provide the conditions for permanent waterproofing. From the analysis, it can be seen that the optimal dosage of each active component is A1B1C2D1E1F1G1.

3.2. Effect of CCCW on the impermeability of mortar matrix

The impermeability pressure ratio is the main technical requirement index for evaluating CCCW. As shown in Table 1, waterproofing agent mixed with homemade group and commercially available group have high impermeability pressure ratio, can meet the national standard requirements, of which the homemade group 28d impermeability pressure ratio of 400%, waterproofing effect is far away from the waterproofing effect of the commercially available group, indicating that homemade waterproofing agent has a good impermeability performance and waterproofing effect, this is due to the role of CCCW in the active chemicals can be in the action of water penetrate into the interior of the substrate, and hydration This is due to the active chemical substances in CCCW can penetrate into the substrate under the action of water, react with the hydration product to generate insoluble needle crystals, blocking the internal pore channels and tiny cracks, so as to achieve the purpose of waterproofing.

Table 5. Results of seepage resistance tests

Test items	28d impermeable pressure (MPa)	28d impermeable pressure ratio (%)	The national standard requires that the impermeable pressure ratio (%)
blank group	0.3	--	
homebrew group	1.3	400	≥200%
market group	1.1	333	

3.3. Effect of CCCW on compressive and flexural strength of mortar matrix

Figure 1. shows the compressive test results of mortar specimens at different ages. Compressive strength ratio is one of the main technical requirement indexes for evaluating CCCW. The experimental results show that the 7 d, 14 d and 28 d compressive and flexural strengths of the CCCW-added group are significantly higher than those of the blank group and the commercially available group, which indicates that CCCW can improve the mechanical properties of the specimen blocks to different degrees. With Figure 1 and Table 2, it can be concluded that mixing waterproofing agent in the test block can improve the compressive strength of mortar, which is due to the fact that the sulfate in sodium sulfate in the waterproofing agent can react with the hydration products of cement such as calcium hydroxide and calcium aluminium hydrate to produce insoluble calcium sulfate and calcium alumina crystals, which can fill up the internal pore space and cracks, making the internal structure more dense, thus improving the strength of the test block. The strength of homemade waterproofing agent and commercially available imported products have good compressive effect.

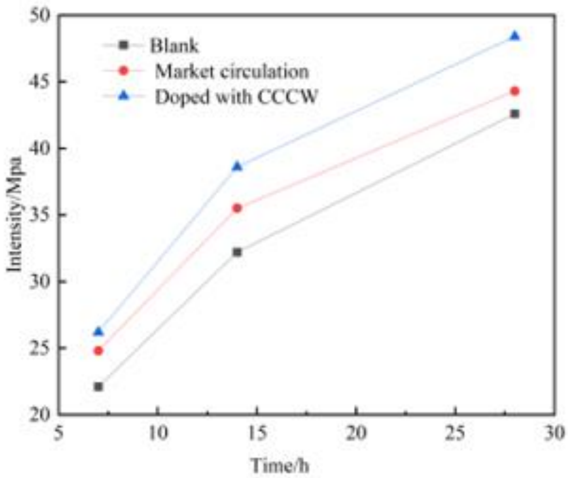

Fig. 1 Compression test results of mortar specimens at different ages

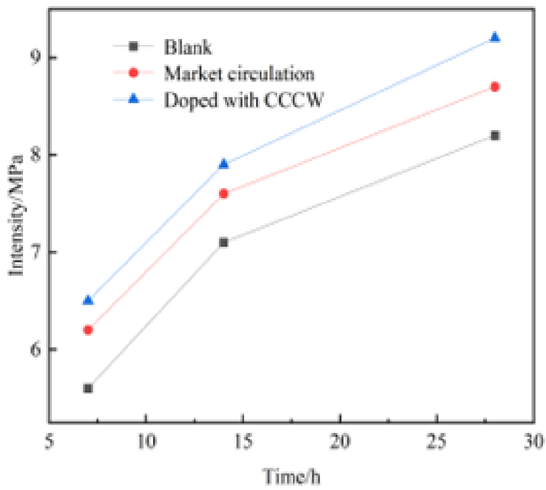

Fig. 2 Flexural test results of mortar specimens at different ages

3.4. Effect of CCCW on water absorption of mortar matrix

Figure 3. shows the water absorption changes of blank and added CCCW specimens immersed for 0, 0.5, 1, 3, 6, 12, 24 and 48 h. The water absorption of the blank and added CCCW specimens is shown in Fig. 3, whereas the water absorption of the coated specimens is shown in Fig. 4. As can be seen from the figure, with the extension of the immersion time, the water absorption rate of the added

CCCW specimens gradually tends to level off, in 48 h, with the coated specimen has reached the saturation state of water absorption, while the blank specimen is still in the state of water absorption, which indicates that the active substances play a waterproofing effect on the specimen, but no matter how long the immersion time is, the water absorption rate of the added CCCW coated specimens is smaller than that of blank specimens and commercial specimens, indicating that the active substances in CCCW play a waterproofing role on the specimens. Under the action of water, the active substance in CCCW generates needle and rod or branch and vine-like crystals, which are mainly distributed around the pores and cracks, with a compact structure, so as to enhance the self-healing ability of mortar, and these crystals may be hydrated calcium silicate or CaCO3 crystals and soluble Ca2+complexes. This shows that CCCW plays a role in all stages of cement hydration.

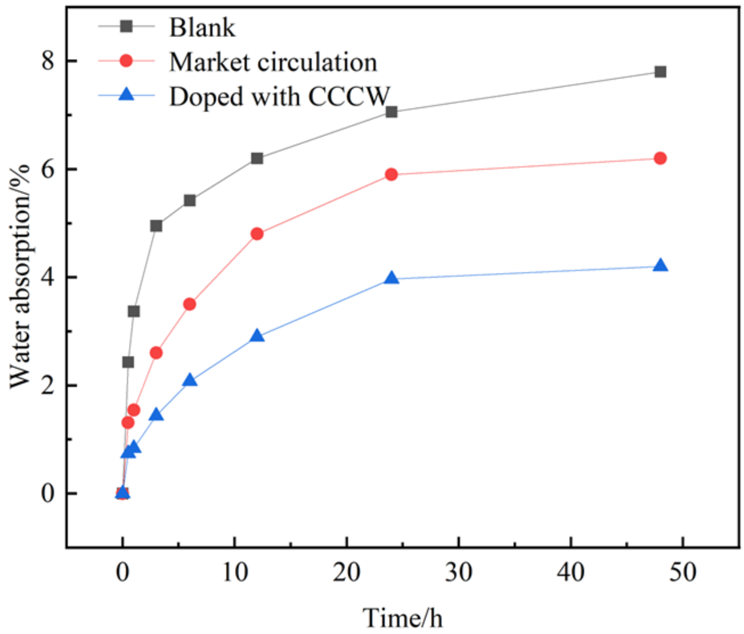

Fig 3. Test results of water absorption at different moments of mortar specimens

3.5. Exploration of CCCW waterproofing mechanism

Since the core components of CCCW have not been published, the water repellency mechanism has not been very clear [5], and researchers have conducted in-depth studies on the water repellency mechanism of CCCW [6,7]. The water repellency mechanisms generally recognised at home and abroad are divided into precipitation reaction mechanism and complexation-precipitation reaction mechanism. The precipitation reaction mechanism suggests that the active chemicals in CCCW use water as a medium to enter the concrete and react with the oxides and free lime in the pores to generate insoluble crystals, which block the pores and microcracks, improve the compactness of the concrete, and reduce the occurrence of carbonation, seepage, and corrosion [8,9], thus achieving waterproofing effects. The complexation-precipitation reaction mechanism states that active chemicals can form unstable complexes with Ca2+ in the concrete structure and then diffuse in the pores using water as a medium. When encountering unhydrated cement particles, the calcium ions in the calcium complex are seized by the more chemically stable silicate and aluminate to form a C-S-H gel and fill the pores, and the active chemical is free again and continues to diffuse in solution until it reaches the high concentration region of CH again, complexing with the ionised calcium ions once more to carry out a new round of complexation-precipitation reaction [3].

However, both mechanisms are difficult to explain the long-term waterproofing effect of CCCW alone. Although the precipitation reaction mechanism reveals the characteristics of CCCW osmotic crystallisation, the active substances have been consumed as the reaction continues, and thus cannot reasonably explain the permanent waterproofing and self-repairing properties of CCCW. The complexation-precipitation reaction mechanism, on the other hand, regards the active substance of CCCW as a catalyst and does not participate in the reaction of the complexation-precipitation cycle, which has a good resolution for the self-repairing performance of CCCW and its permanent waterproofing effect, but whether the reaction rate can meet the actual demand has yet to be studied [10]. Therefore, research scholars believe [11] that the complexation-precipitation reaction is further proposed on the basis of the precipitation reaction mechanism, and CCCW relies on its own crystalline precipitation component to react with calcium ions in the mortar structure, generating insoluble crystals to block the capillary pores, while the other component of the active substance, the complexing agent, does not participate in the reaction and promotes the hydration of the unhydrated cement, which will convert the CH into insoluble crystals with a certain strength. soluble crystals, playing a water-blocking effect.

4. CONCLUSION

Through orthogonal test, it is concluded that the waterproofing effect of the active substance is influenced by the following factors: sodium sulfate>calcium magnesium carbonate>calcium formate>complex agent>magnesium oxide>sodium silicate>citric acid and the optimal ratio of the self-fabricated waterproofing material is: sodium sulfate 0.3%, sodium silicate 0.2%, calcium magnesium carbonate 0.4%, calcium formate 0.2%, citric acid 0.1%, magnesium oxide 0.1%, and the complexing agent 0.1%. Self-assembled waterproofing materials of the performance indicators are to meet the relevant national norms, especially mortar specimens 28d impermeable pressure ratio of 400%, greater than the national standard impermeable pressure ratio of 200%.

Through the test of different mortar specimens, the results show that: for the impermeability test, compressive strength test, flexural strength test, water absorption test, CCCW have obvious performance improvement, the more popular waterproofing materials on the market is obviously better than the blank group of experimental results, but its performance improvement is not as good as CCCW.

Waterproofing materials in the penetration into the mortar specimen, its parent material in the carbonate ions, sulfate ions, silicate ions and other calcium ions in the hydration products of cement reaction, the generation of crystals and insoluble matter, blocking the pores of the mortar specimen, so that the specimen is more dense, to achieve better waterproofing effect.

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