Study on the repair effect of cement-based self-healing microcapsules under different curing conditions

Chenghui Wu, Junhu Li*, Yinghao Yang, Hang Lei

Jinhua Polytechnic, Zhejiang, 321017, China

285014418@qq.com*

Abstract. Microcapsules were prepared by in situ polymerization with an average particle size of 198μm. The self-healing properties and mechanical properties of microcapsule cement sand adhesive specimen were measured by the strength recovery rate. And the self-healing properties of self-healing cement substrates with different microcapsule contents (0%, 1.5%, 3%, 4.5% and 6% of cement mass) under dry-culture and wet-culture restoration and curing conditions were studied. The results show that under the conditions of dry and wet culture, the flexural strength and compressive strength of the specimen decrease with the increase of microcapsule content; The strength recovery rate of dry culture specimens under different microcapsule dosages was 106.60%~124.29%, and the strength recovery rate of wet culture specimens was 98.74%~110.12%, and the wet culture repair effect was significantly lower than that of dry culture self-repair effect. When the amount of microcapsules is small, the self-healing performance of self-healing slurry under dry and wet culture shows the same law, and the performance is improved with the increase of the dosage, and the peak is reached when the dosage is 4.5%, which is the optimal dosage, and the repair effect is subsequently reduced.

Keywords: Cement-based material; microcapsule; self-repair; mechanical property

1 Introduction

The heterogeneity and brittleness of cement-based materials make it easy to produce microcracks on the surface and inside, forming channels for harmful substances to invade and gradually decay, affecting the strength and durability of cement-based materials[1-2].

Based on the principle of bionics, self-healing is a current research hotspot, among which microcapsule technology is extremely promising[3-4].Microcracks are repaired by incorporating microcapsules into the matrix, triggering cyst wall rupture when cracks occur, releasing internal capsule core repairers[5]. Sidiq et al.[6] prepared polyurethane-coated sodium silicate microcapsules, which successfully repaired the crack of 0.203 mm, and the mechanical properties of cement substrate were effectively improved. Li et al.[7] synthesized urea-formaldehyde resin/epoxy resin microcapsules and

H. Bilgin et al. (eds.), *Proceedings of the 2023 5th International Conference on Civil Engineering, Environment Resources and Energy Materials (CCESEM 2023)*, Advances in Engineering Research 227,
https://doi.org/10.2991/978-94-6463-316-0_13

successfully repaired the crack of 0.416mm in seawater environment. Pu Qi et al.[8] prepared microcapsules by in-situ polymerization method, which had excellent performance, and it was found that the higher the temperature during curing, the better the self-healing performance. Wang Xianfeng et al.[9] mixed epoxy resin microcapsules into cement mortar, and found that when the dosage was in the mortar range of 3%~6%, the repair rate gradually increased. Jin Chengyang et al.[10] prepared self-healing microcapsules by round pot granulation method to characterize the repair effect by permeability test, and found that when the crack width was fixed, the higher the microcapsule content, the better the repair effect.

At present, domestic and foreign research mainly focuses on the self-healing effect and mechanism of action of microcapsules, and there are few studies on self-healing and maintenance conditions. In this test, the in-situ polymerization method was used to synthesize urea-formaldehyde resin/epoxy resin microcapsules. The experimental study of sand rubber specimens under different self-healing conditions of dry and wet culture was carried out, and the repair effect of urea-formaldehyde resin/epoxy resin microcapsules in mortar under different self-healing curing conditions was discussed.

2 Experimental design

2.1 Microcapsule preparation

Microcapsules were prepared by in-situ polymerization method[10].The resin/epoxy resin microcapsules, which were white powder, as shown in Figure 1. The microscopic morphology of SEM microcapsules is shown in Figure 2, which is spherical particles with a rough surface and flocculent substances, which are easy to trigger the release of core material when cracks appear in the cement matrix [11-12]. The particle size distribution of microcapsules observed by laser particle size distributor is shown in Figure 3, with an average particle size of 198 μm.

Fig. 1. Microcapsule

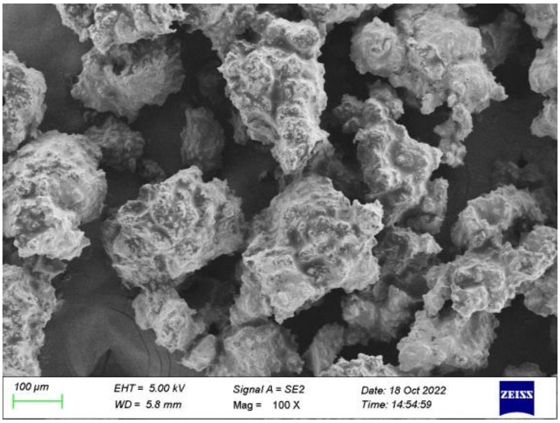

Fig. 2. SEM photo

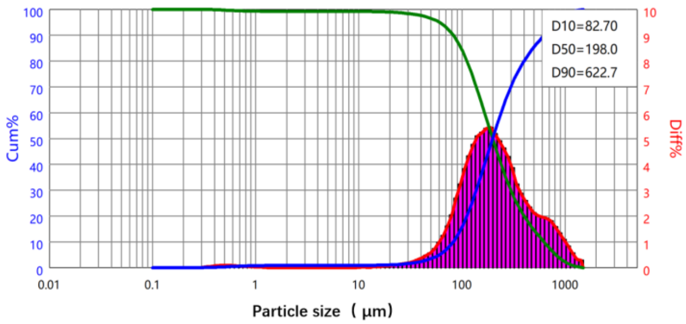

Fig. 3. Microcapsule size distribution

2.2 Mortar mix ratio

The mortar mixing ratio is water: cement: sand = 1:2:6, and the microcapsules are incorporated according to the percentage of cement mass, for example, Z1.5 represents that the microcapsule content is 1.5% of the cement mass. The combination of each test group is shown in Table 1, and the curing agent content is 50% of the mass of microcapsules.

Table 1. Mortar mix ratio of each test group.

Constituencies	water/g	cement /g	Standard sand /g	Microcapsules /g	Curing agent /g
Z0	225	450	1350	0	0
Z1.5	225	450	1350	6.75	3.375
Z3	225	450	1350	13.5	6.75
Z4.5	225	450	1350	20.25	10.125
Z6	225	450	1350	27	13.5

2.3 Test piece forming and test design

According to the matching ratio of Table 1, cement mortar specimens are made, the size is 40mm×40mm×160mm, and the standard curing is 28d Each group of specimens will be designed into four categories: A, B, C, and D. Taking the Z0 group as an example, 2 groups, that is, 6 specimens, were prepared, and after the flexural test, 12 small specimens (size of about 40mm×40mm×80mm) were obtained, divided into 4 categories, and 3 small specimens in each category were used to test the compressive strength of mortar specimens under each category. The repair conditions for each category are shown in Table 2 below. The remaining groups are the same as the Z0 group.

Table 2. Classification of CL0 specimens.

category	name	remark
A	Z0-A	measure the initial compressive strength of the specimen.
B	Z0-B	measure residual compressive strength after pre-injury.
C	Z0-C	measure repair compressive strength of dry culture curing after pre-injury.
D	Z0-D	measure repair compressive strength of wet culture curing after pre-injury.

Note: Dry culture is maintained for 7 days in the general indoor environment, the temperature is 20±2 °C, and the relative humidity is 60%±5%; Wet culture is remediated and cured for 7 days for immersion in water.

Studies have shown that the material matrix will be damaged by 60%σmax compressive stress, and microcracks will occur inside[13]. In order to fully induce the microcracks of the matrix to ensure the performance study of the subsequent self-healing mortar, a compressive stress of 70%σmax is applied to cause pre-damage to the self-healing mortar.

The experimental design is as follows: the category A specimen is loaded at a speed of 2.4±0.2kN/s, and its failure load is measured as the initial compressive strength, which is recorded as f_o (MPa); Category B,C,D specimens are loaded to 70%f_o and continue to act for 1min before unloading. After unloading, the Category B specimen is loaded to the specimen at the same speed to fail, and the residual compressive strength value after pre-damage is determined, which is recorded as f_u(MPa); After 7d maintenance and repair of Category C and D specimens according to Table 2, compressive strength tests were carried out, and the compressive strength values of dry and wet maintenance were obtained respectively, which were recorded as f_{dc} (MPa) and f_{wc}(MPa).

2.4 Definition and characterization of strength repair effects

The self-healing effect of mortar compressive strength is evaluated by strength recovery rate, and the calculation method is as follows[14-15]:

$$\eta_{odc,x} = \frac{f_{dc}}{f_o} \times 100\% \tag{1}$$

$$\eta_{owc,x} = \frac{f_{wc}}{f_o} \times 100\% \tag{2}$$

$\eta_{odc,x}$, $\eta_{owc,x}$ respectively, the strength recovery rate of pre-damaged mortar specimens with x% microcapsule content after drying curing and immersion curing after 7d self-repair (%).

3 Self-healing effect analysis

3.1 Analysis of compressive strength and flexural strength

Figure 4 shows the change of flexural strength and compressive strength of mortar specimens with the amount of microcapsules. With the increase of microcapsule content, the strength value showed a decreasing trend. When the amount of microcapsules is small (not more than 1.5%), the flexural strength has little effect; However, with the increase of microcapsule content, the flexural strength decreased significantly, and the largest decrease was at 3%, which decreased by 12.94% compared with 1.5% content. However, with the increase of microcapsule content, the flexural strength decreased significantly, and the largest decrease was at 3%, which decreased by 12.94% compared with 1.5% content. However, under the dosage of 3%~6%, the change range of flexural strength is small, the maximum is 5.47%. After being incorporated into microcapsules, the overall compressive strength decreased significantly. When the microcapsule content was 1.5%, the compressive strength decreased the most, reducing by 24.64%.

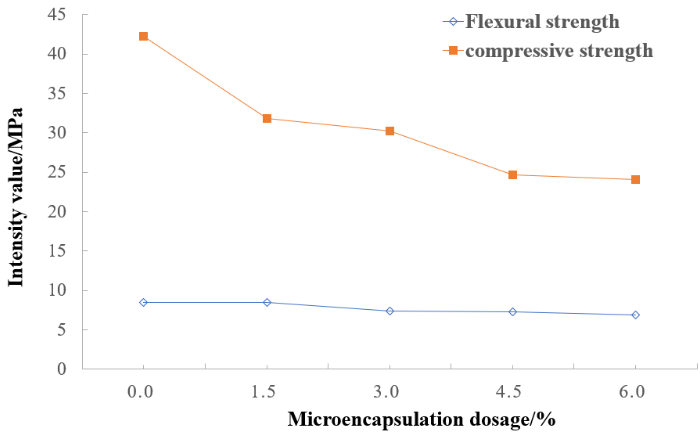

Fig. 4. Effect of different microcapsule dosages

In view of the decrease in the strength of self-healing cement mortar after incorporation of microcapsules, from the aspect of density, the density of microcapsules is small (less than water), and the overall density of self-healing cement mortar specimens decreases with the increase of dosage, resulting in a decrease in the flexural and compressive strength of microcapsule specimens. From the analysis of the microcapsule itself, the average particle size of the self-made microcapsule in this project is 198μm, which is much larger than the particle size of cement and other materials, also its own mechanical strength is very low[16], and the proportion of other substances is reduced after the self-made microcapsule being incorporated into the matrix, but it cannot provide sufficient strength, which becomes a defect in the mortar matrix. Therefore, when the more microcapsules are contained, the more defects inside the self-healing mortar matrix, which affects the flexural and compressive strength of the material.

3.2 Study on the repair rate of self-healing mortar

Taking the microcapsule content as the abscissa and the recovery rate as the ordinates, the line chart of self-repair effect under dry and wet culture conditions was drawn, see Figures 5.

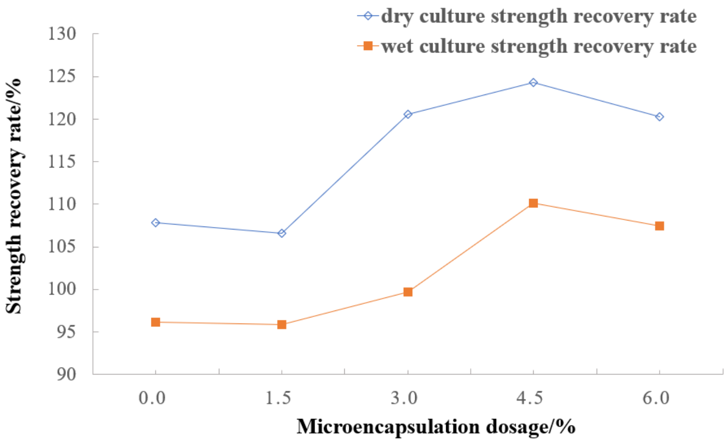

Fig. 5. Mortar recovery rate on mechanical properties of mortar

It shows that the incorporation of microcapsules under dry-culture conditions makes cement mortar have obvious self-healing effect, and the recovery rate of dry-rearing strength of mortar under each dosage is all greater than 105%. For the Z0 control group, the recovery rate of dry strength also reached 107.82%, indicating that the mortar itself had a certain self-healing ability, and the continued hydration of unhydrated cement particles inside the specimen and the continuous carbonization crystallization of the hydration product calcium hydroxide provided subsequent strength. However, when the microcapsule content was 1.5%, the recovery rate of self-healing mortar was 106.60%, which was lower than that of the Z0 test group. And when the dosage was

4.5%, the strength recovery rate of dry cultivation intensity reached a maximum of 124.29%. Then, with the increase of the dosage, the recovery rate of dry nourishment strength decreased, because the microcapsule was ruptured after the matrix was damaged, and the internal epoxy resin flowed out to form a cavity, which further expanded the filling defect of the microcapsule, the mechanical properties of the mortar were reduced, and the repair effect was weakened, but the recovery value was still much higher than that of the Z0 control group.

The strength recovery rate curve of wet culture in Figure 4 shows that cement mortar mixed with microcapsules also has a certain repair effect in a water conservation environment with a temperature of 20°C ±2°C. When the content of microcapsules is not greater than 1.5% of the quality of cement, the repair effect of microcapsules is not enough to make up for the defects brought by them, and the recovery rate is low to 98.74%; When the content of microcapsules was 4.5% and 6%, the recovery rate of mortar moisture strength reached more than 100%, which was 110.12% and 107.47%, respectively. The trend of the strength recovery rate curves of wet culture and dry culture was high under different microcapsule dosages, but the recovery rate curve of wet culture intensity was significantly lower than that of dry culture. This is due to the fact that after the self-healing mortar specimen is damaged, microcracks are distributed on the surface and inside. When the specimen is immersed in water, the moisture in the surface and internal microcracks is in a state of fullness, although it can provide water for the subsequent hydration of cement, but the continuous infiltration of water in the cracks affects the development form of cement hydration products and the filling effect of the cracks, so that the complete closure of the cracks is blocked.

4 Conclusion and outlook

(1) The incorporation of microcapsules and curing agents with an average particle size of 198μm will have a significant impact on the flexural and compressive strength of mortar, and the flexural and compressive strength values will decrease with the increase of the dosage.

(2) The self-healing cement mortar mixed with microcapsules has a good repair effect, and the compressive strength value is significantly enhanced compared with the damaged specimen.

(3) The curing conditions have a great influence on the repair effect of self-healing cement mortar, and the self-healing effect of the substrate under dry curing conditions is significantly better than that of the substrate cured by immersion in water.

(4) For the restoration effect of mortar, when the microcapsules dosage is 4.5%, the strength recovery rate of dry and wet culture is maximized, which is the optimal dosage.

(5) The microcapsules in this project are limited to one damage repair of the self-healing matrix. When the matrix is damaged in the same position during subsequent use and work, it will be difficult to achieve self-repair again. Therefore, it is necessary to carry out secondary repair related research on materials and repair mechanisms in the future.

Acknowledge

A Project Supported by Scientific Research Fund of Zhejiang Provincial Education Department (Y202043252,Y201941554), and 2020 Zhejiang Province Construction Scientific Research Project(2020K138)

References

1. SIDIQ A,GRAVINA R,GIUSTOZZI.Is concrete healing really efficent? a review[J]. Construction and Building Materials,2019(205):257-273.
2. Khaliq W,Ehsan M B. Crack healing in concrete using various bio influenced self-healing techniques[J]. Construction and Building Materials, 2016,102(1):349-357.
3. FERNANDEZCA,CORREAM,NGUYENMT,et al. Progress and challenges in self-healing cementitious materials[J]. Journal of Materials Science, 2021,56(1):201-230.
4. García Á, Schlangen E, Ven M, et al. Preparation of capsules containing rejuvenators for their use in asphalt concrete[J].Journal of Hazardous Materials,2010,184(1-3):603-611.
5. ZHAO Xiaoyan,GAO Mingliang,ZHOU Jian, et al. Study on the repair of cement-based materials by composite microcapsules and bacillus coriolis[J].Concrete,2021(08):83-87.
6. Sidiq, A., Setunge, S., Gravina, R. J., & Giustozzi, F. Self-repairing cement mortars with microcapsules: A microstructural evaluation approach. Construction and Building Materials,232,117239.
7. Xiuhao Li, Rentai Liu, Shucai Li, et al. Properties and mechanism of self-healing cement paste containing microcapsule under different curing conditions. Construction and Building Materials,357,129410.
8. PU Qi, DENG Jiangliang, LI Longzi, et al. Experimental study of microcapsules in self-healing concrete products [J]. CHINA CONCRETE AND CEMENT PRODUCTS, 2017(12):44-49.
9. WANG Xianfeng, SUN Peipei, XING Feng, et al. Research on Microstructure of Microcapsule Based self-healing Cementitious Composite [J].Journal of Disaster Prevention and Mitigation Engineering,2016,36(1):126-131.
10. JIN Chengyang, YANG Yang, JIN Haidong, et al. Study on the healing efficiency and mechanism of capsule-based self-healing cementitious material [J].New Building Materials, 2019,46(11):108-111+114.
11. WANG Xingang, ZHANG Xuanzhe, ZOU Fubing, et al. Secondary repair characteristics and mechanism of cement matrix intermixed with self-healing microcapsules[J] . Functional Materials, 2023, 54(02):2001-2005.
12. Wan P, Wu S P, Liu Q T, et al. Sustained-release calcium alginate/diatomite capsules for sustainable self-healing asphalt concrete[J]. Journal of Cleaner Production,2022, 372:133639.
13. SUN Jingcheng. Research on the performance and self-healing effect of microcapsule based self-healing cementitious composites [D]. Hangzhou: Zhejiang University,2019.
14. ZHANG Ming. A Study on Microcapsule Based Self-Healing Method and Mechanism for Cementitious Composites[D]. Changsha: Central South University, 2013.
15. SUN Peipei. Research on Strength and Pore Structure of Microcapsule Based Self-healing Cementitious Composites[D]. Shenzhen: Shenzhen University,2016.
16. ZHAO Cha. Study on Fracture Behavior of Microcapsules in Composite Material[D].Tianjin: Tianjin University, 2014.