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RESEARCH

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Surface crack treatment of concrete via nano-modified microbial carbonate precipitation

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Abstract

As a new concrete crack patching technology, microbial self-healing slurries offer favourable characteristics including non-pollution, ecological sustainability and good compatibility with concrete. In this paper, a nano-sio_2-modified microbial bacteria liquid, combined with sodium alginate and polyvinyl alcohol, was used to prepare a nano-modified microbial self-healing slurry. This slurry was used to coat concrete under negative pressure in order to verify its restoration effect, and the micromorphology of the resulting microbial mineralization products was observed. The results revealed that patching the concrete using the nano-modified microbial slurry significantly improved its permeability, and increased its carbonization resistance by three times in comparison with the control group. Through a combination of Scanning electron microscopy (SEM) and X-ray diffraction (XRD) observation, it was determined that the microbial mineralization reaction products were mainly calcite crystals, which, integrated with the nano-sio_2, sodium alginate and polyvinyl alcohol at the microscopic level, filled the internal pores of concrete, thus improving its durability.

Highlights

- Surface crack treatment of concrete using a nano-modified microbial slurry was investigated.
- Patching concrete using nano-microbial slurry clearly improved its chloride penetration.
- The carbonization of the concrete was three times in comparision with the control group.
- The main product of the microbial mineralization reaction was calcite crystal.

Keywords Nano-modified microbial, Carbonate precipitation, Chloride penetration, Carbonation, Microstructure, Concrete crack patching

Introduction

It is widely acknowledged that micro-cracks and fissures are inherent to concrete and will inevitably be present. Due to the low tensile strength of concrete, cracking is certain occur during its service, especially cracking of the concrete surface layer. The durability of concrete is reduced due to a series of matrix degradation processes caused by incoming water and harmful chemicals, and the corrosion of embedded steel bars [1]. It is estimated that the direct cost of maintaining and repairing concrete highway bridges in the United States is $4 billion a year (FHWA-RD-01–156, 2001). While the need for more sustainable cement-based products has been recognized for decades, the development of sustainable maintenance and repair methods for concrete structures remains in its infancy. Thus the development of safer, more environmentally friendly

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methods for repairing surface cracks in concrete remains a pressing need.

Numerous restoration methods for patching concrete cracks exist worldwide. Traditional restoration methods mainly use inorganic or organic polymers for surface sealing, pressure grouting and plugging [2, 3]. Inorganic materials have the advantages of good compatibility with concrete and strong anti-aging performance, but suffer from poor adhesion. Organic materials offer good adhesion and excellent chemical corrosion resistance. However, they are expensive, and for the most part, contain hazardous substances prone to cause environmental pollution and harm to humans.

Given the high cost and poor aging of traditional methods, microbial concrete crack self-healing technology has gradually emerged as the research focus in recent years [4–8]. Gollapudi et al. [9] made the first attempt to repair structural leakage in concrete via mineralization of bacterial deposits in 1995. Qian et al. [10, 11] further analyzed the mechanism of microbial mineralization deposition, identifying that this lead to partial consolidation of loose particles. Rodriguez-Navarro et al. [12] discovered that microbial mineralization deposition lead not only to the adherence of loose particles, but also the filling with mineralized sediments of tiny cracks in limestone surfaces, patching these cracks. Hill et al. [13] and Dick et al. [14] used microbial mineralized deposits to fill gaps in fractured rocks, improving their impermeability. At present, microbial self-healing slurry finds widespread application, with good reinforcement efficacy, in sand reinforcement [15]. Ma et al. [16] applied high-concentration microbial slurry for coating concrete, obtaining results indicating a reduction in the capillary water absorption coefficient at the concrete surface of over 90%, and remarkable restoration efficacy.

Recently, technologies such as nanotechnology have also been used to enhance the properties of concrete. Research has shown that nano-sio_2 has high activity, and has potential to form a new calcium silicate hydrate gel phase in combination with cement hydration products, filling internal pores in concrete. Meanwhile, nano-sio_2 can partially consume $Ca(OH)_2$, promoting the hydration of cement and improving the mechanical properties of concrete. Mohammed et al. [17] studied the effect of nano-sio_2 on the performance of pervious concrete, finding improved compressive strength in the nano-sio_2 modified version, although its porosity and permeability were not greatly improved. Jalal et al. [18] studied the influence of nano-sio_2 content on the mechanical properties of high-performance self-compacting concrete from the perspective of microstructure. The pore structure of concrete prepared with nano-sio_2 admixture was more refined and compact, especially at a higher ages, and its strength was improved.

Extant research worldwide has confirmed the compatibility of microbial slurries and nano-sio_2 with concrete, but has yet to exhaustively study the optimal specific ratios for use in nano-microbial slurries. Therefore, in this study, the optimal mix proportions for a nano-modified microbial slurry were determined, using fluidity as the evaluation index, and its restorative effect on concrete surface cracks tested. The nano-modified microbial mineralization products were subsequently analyzed using scanning electron microscopy (SEM) and X-ray diffraction (XRD), and the results reported.

Materials and experiments

Selection and culture of bacteria

Sporpsarcina pasteurii was selected as the target bacteria in this paper, strain number 1.3687 sourced from the China General Microbial Species Preservation and Management Center (CGMCC). *S. pasteurii* is aerobic, alkali-resistant and gram-positive, with cells characterized by high-temperature resistance, rapid recovery and strong enzyme-secreting ability. Supplier data mentioned that the cells were 1–2 μm in diameter and about 2–3 μm long, while the spores were round and 0.4–1.2 μm in diameter.

The bacteria used in this paper were obtained in the form of vacuum freeze-dried powders, which required activation recovery culture followed by expanded culture. The specific process was as follows:

(1) Culture medium: The culture medium was prepared according to CGMCC formula No. 907 (detailed composition shown in Table 1). 800 mL ultra-pure water was poured into an enamel cup and heated in an induction cooker. The pharmaceutical reagent was added after the water boiled, while stirring with a glass rod. When the reagent had completely dissolved, it was then sub-packaged into conical bottles, sealed with sealing film, and placed in a sterilization pot for sterilization.

(2) Sterilization treatment: The test equipment and culture medium were placed in a high-pressure steam sterilization pot for high-temperature sterilization at 121 °C for 20 min.

(3) Ampoule opening: the ampoule was first sterilized with UV light for 15 min, before its surface was wiped with cotton dampened with 75% alcohol. The tip of the ampoule was placed over a flame and

Table 1 Culture medium compositions, Formula No. 907

Compositions	Peptone (g)	Meat extract (g)	Urea (g)	Distilled water (L)
Content	5.0	3.0	20.0	1.0

heated for half a minute, and then dipped into a small amount of distilled water, and the tip cracked with tweezers until it was open.

(4) Activation culture: approximately 0.3 mL of medium was aspirated using a pipetting gun and dispensed into ampoules and shaken to dissolve the lyophilized powder into suspension. All of the bacterial solution was then aspirated via pipette, inoculated into a centrifuge tube of sterilized medium, and incubated for 24 h at a constant 30 °C temperature to obtain the 0th generation, or original, strain (Fig. 1). This was then refrigerated at -70 °C in a glycerol tube for future study.

(5) Expanded culture: the bacteria solution obtained in step (4) was inoculated at a concentration of 1‰ into the sterilized medium, and incubated in a constant temperature shaking bed for 24 h (30 °C, 150 rpm). The first-generation working bacteria solution was obtained when turbidity became obvious (Fig. 2). The cultured strains were centrifuged and stored at 4 °C.

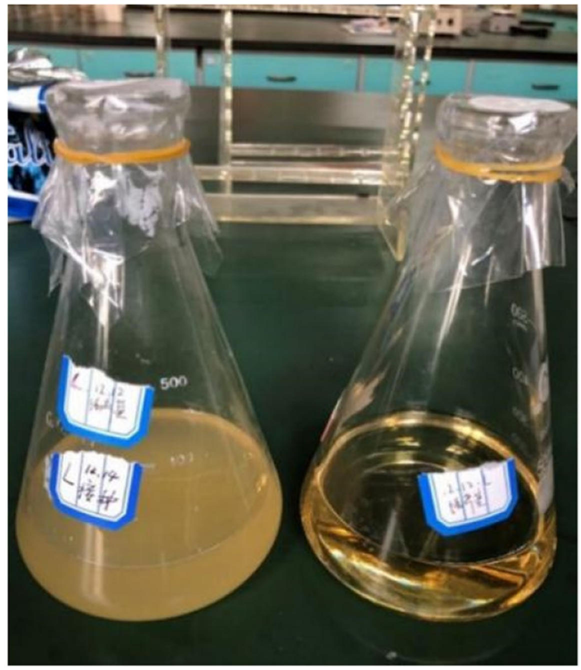


Fig. 2 Expanded culture

Nano-modified microbial slurry

Nano-modified material

The main constituents of the nano-modified microbial self-healing slurry were sodium alginate, polyvinyl alcohol, nano SiO_2 and microbial liquid. The Nano SiO_2 used was an amorphous white powder that is non-toxic, tasteless, and insoluble in water, model SP30, with particle size of 30 ± 5 nm. The sodium alginate was an odorless, tasteless white powder that dissolved easily in water and formed a viscous liquid in solution. The polyvinyl alcohol used in this study was a white, odorless powder easily soluble in water above 95 °C. The preparation process for the nano-modified microbial self-healing slurry was as follows:

Fig. 1 Original strain

(1) Sodium alginate and polyvinyl alcohol were each dissolved separately in a water bath at 95 °C and then cooled to room temperature for testing.
(2) The mixture was prepared according to the mix proportions in Table 2, and then stirred with a magnetic mixer for 15 min.
(3) Li [19] showed that microbial activity was optimal in the pH range 8–11. Considering the strongly alkaline internal environment of concrete [16], pH = 11 was selected for the bacterial solution. The mixed solution obtained from (2) was therefore adjusted to pH = 11 using 1 mol/L NaOH solution.

Fluidity of nano-modified microbial self-healing slurry

To prevent the sodium alginate in the slurry from prematurely reacting with Ca^{2+}, calcium lactate was added to the slurry at 0.03 mol/L. The slurry was thoroughly mixed with the calcium lactate using a magnetic mixer, and became calcified and sticky (as shown in Fig. 3). After the slurry

Table 2 Nano-modified microbial self-healing slurry mix proportions

No	Working bacteria solution (mL)	Distilled water (mL)	Sodium alginate (g)	Polyvinyl alcohol (g)	Nano SiO_2 (g)
A	300	300	0	6	12
B	300	300	6	6	12
C	300	300	12	6	12
D	300	300	18	6	12
E	300	300	24	6	12
F	300	300	30	6	12

Fig. 4 Cement mortar fluidity tester

Fig. 3 Calcified slurry

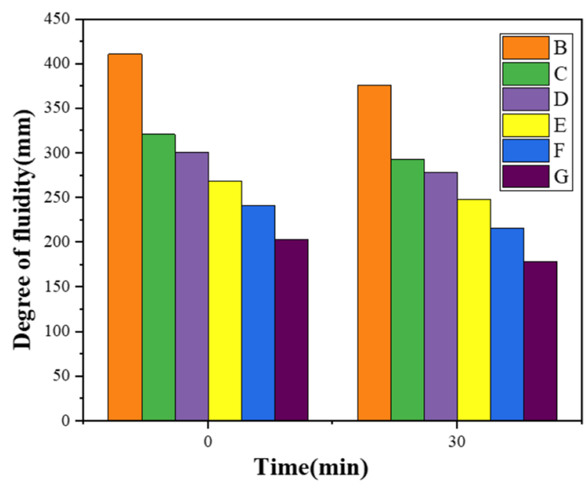


Fig. 5 Effect of sodium alginate on slurry fluidity

had become calcified, its fluidity was tested in accordance with Chinese standard GB/T 2419–2005 (see Fig. 4). In this paper, fluidity at 0 min and 30 min were used as indices for evaluation of the engineering applicability of the slurry [20].

The results of the fluidity test (shown in Fig. 5) indicate that the fluidity of the slurry in each group decreased slightly over time. In addition, the fluidity of the slurry decreased as its sodium alginate content increased. The fluidity of the slurry ranged between roughly 320–200 mm at 0 min and 300–180 at 30 min after the addition of sodium alginate. Research indicated that this was within a range acceptable for the requirements of brushing [21]. Taking into account the requirements of the negative pressure process, group B was considered to have the most appropriate proportion of sodium alginate.

Concrete constituents

Cement: P.O42.5 ordinary cement was used in this test, with physical properties presented in Table 3.

Aggregates: Coarse aggregates with particle sizes of 9.5–13.2, 13.2–16, and 16–19 mm continuous gradation were used. The fine aggregate used was medium sand with a particle size of 0.075-2 mm. The main properties of the coarse and fine aggregates were measured in accordance with Chinese standard JGJ 52–2006.

Water: The water used in the test was tap water, which fulfilled the requirements of the Water Standard for Concrete (JGJ 63–2006).

Table 4 summarizes the detailed mix proportions of the concrete.

Test methods

Chloride penetration into the concrete

Measurement of the penetration of chloride ions into the concrete was performed using coulomb electric flux and RCM methods conforming with Chinese standard GB/T 50082-2009. . In accordance with Chinese standard GB/T 50082-2009, a cylindrical concrete specimens, diameter of 100±1mm, height of 50±2mm, were used. The specimens

Table 3 Cement physical properties

Compression strength (MPa)		Flexural strength (MPa)		Setting time (min)		Specific surface area (m^2/kg)
3 d	28 d	3 d	28 d	Initial condensation	Final condensation	
19.2	46.6	3.8	6.7	246	305	352

Table 4 Concrete mix proportions (Kg·m^{-3})

Water	Cement	Fine aggregates	Coarse aggregates
168	432	558	1242

were subjected to vacuum saturation water treatment using an intelligent concrete vacuum saturation machine before testing. Slurry with mix proportions given in Table 5 was then applied evenly to the concrete surface using a brush (shown in Fig. 6a), and sprayed evenly with 5% calcium lactate (shown in Fig. 6b). After spraying, the specimen was left to stand for 15min. The coating was then repeated 5 times to form a multi-layer gel protective layer, and the specimen was left for 2d. Data were then collected automatically using a concrete chlorine circuit electric flux tester.

Carbonation of the concrete

The carbonation of the concrete was tested by measuring the specimens' carbonation length in accordance with Chinese standard GB/T 50082–2009. Immediately after one side of the concrete specimens (40×40×160 mm prisms), had been coated with slurry, the remaining sides were sealed with paraffin wax, and the specimens were cured under the following conditions: a CO_2 concentration of (20 ± 3)%, temperature of (20 ± 2)°C and humidity of (70 ± 5)%.

Table 5 Slurry mix proportions

No	Working bacteria solution(mL)	Distilled water (mL)	Sodium alginate (g)	Polyvinyl alcohol (g)	Nano SiO_2 (g)
I	0	600	0	0	0
II	0	600	6	6	12
III	300	300	6	6	12

SEM and XRD tests

Calcium lactate was added to the microbial liquid prepared in Sect. "Nano-modified microbial slurry" to 0.03 mol/L and cultured for 48 h in a laboratory environment at 30 °C and 150 r/min. After the sample was fully mineralized, it was put into a centrifuge tube for centrifugation at 10 °C and 8000 r/min. Once the excess bacterial liquid had been poured out, the slurry mineralization product remained in the centrifuge tube as a solid precipitate (Fig. 7). After drying and gold spraying, its micromorphology was observed via SEM.

In preparation for XRD analysis, the powder was dried at 50 °C and sieved through a 75 μm sieve. XRD analysis was performed for $2\theta = 10–80°$ in increments of 0.02°.

Results and discussion

Concrete chloride penetration results

Figure 8a shows test results for the chloride ion migration coefficient of the concrete. For the control group, the chloride ion migration coefficient was $17.69 \times 10^{-13} m^2/s$, while for the sterile group it was $14.75 \times 10^{-13} m^2/s$. The

(a) Brushing slurry (b) Spraying calcium source

Fig. 6 Nano-modified self-healing slurry brushing process

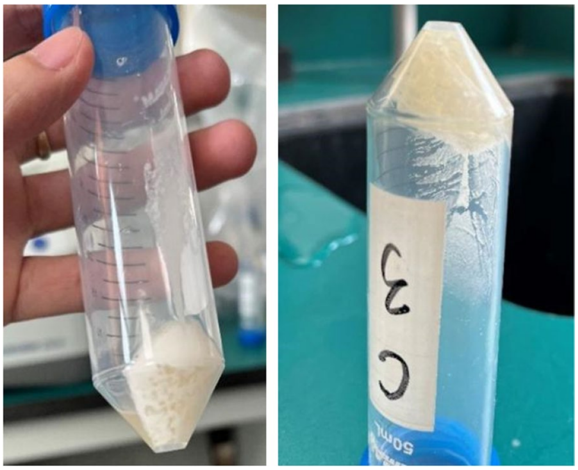


Fig. 7 Nano-modified microbial self-healing slurry mineralization products

reason for this result may be that the slurry applied to the surface of the concrete penetrated deeply into it, partially filling its pores and thus reducing the chloride ion migration coefficient. The chloride ion migration coefficient of the nano-modified slurry group was $8.82\times10^{-13}m^2/s$, significantly lower compared with groups I and II. This indicates that microorganism-produced calcium carbonate crystals were the main reason for the decrease in the coefficient. In combination with nano-SiO_2C the calcium carbonate deposited by microorganisms created a micro-aggregate effect [22], densely filling micro-cracks in the concrete surface [23], improving the internal pore structure of the concrete and thus improving its durability.

Coulomb electric flux results for the concrete are presented in Fig. 8b. As can be seen from Fig. 8, the electric flux of the three groups of samples was 1730.04C, 1229.68C, and 397.59C, respectively. The electric flux of the concrete treated with the nano-modified microbial restoration solution was thus also significantly reduced.

Concrete carbonation results

Figure 9 shows the carbonation depth of the three groups of concrete at different ages. Compared with the control group, the 14 day carbonation depth of the concrete remediated with the nano-modified microbial self-healing slurry was reduced by half, indicating that this treatment greatly improved the carbonation resistance of the concrete. This may be because calcium carbonate crystals produced by microbial mineralization on the surface of the concrete reduced the ingress of carbon dioxide.

The carbonation depth of group II at 3 and 5 days was also reduced in comparison with the control group, but, as the concrete aged, gradually reverted toward the same level. This may reflect short-term blockage of concrete surface pores by the hydrogel generated by the polyvinyl alcohol-sodium alginate—Ca^{2+} reaction, giving rise to a protective effect [24]. However, at increased age, the carbonation resistance of group II was not clearly improved relative to the control group, suggesting that the presence

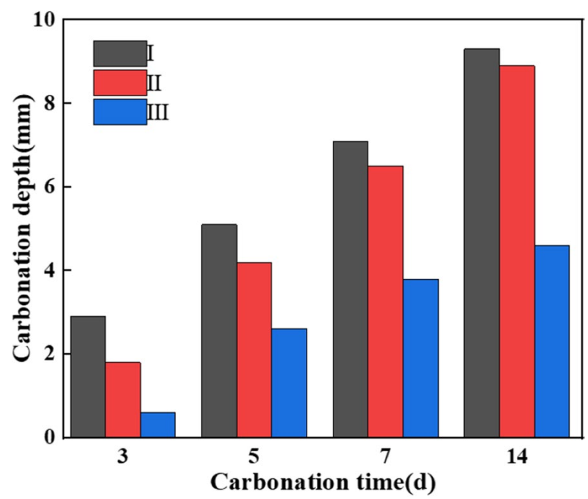
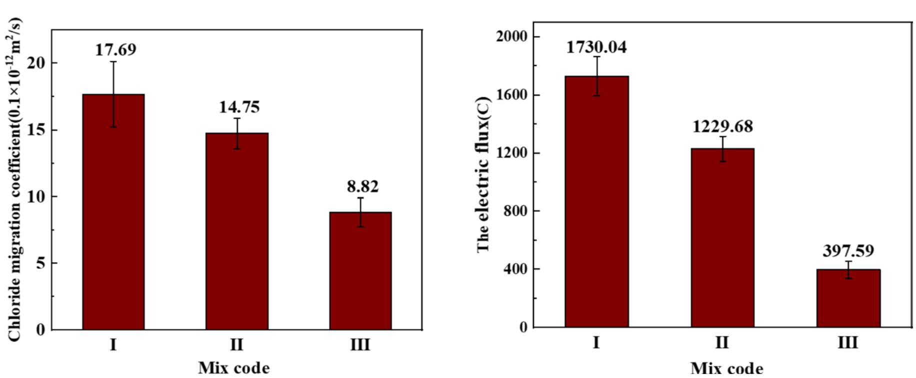


Fig. 9 Concrete carbonation



(a) Chloride migration coefficient (b) Coulomb electric flux

Fig. 8 Concrete chloride penetration results of microorganisms was the key factor underlying the observed improvements in durability.

Characterization of microstructure

Figure 10 shows the results of XRD analysis of the microbial mineralization reaction products, indicating that this is mainly created calcite crystal. The highest calcite crystal peak intensity obtained for the specimen was at 2θ value of 30°; the smaller peak at 2θ of about 40° is also indicative of calcite crystals.

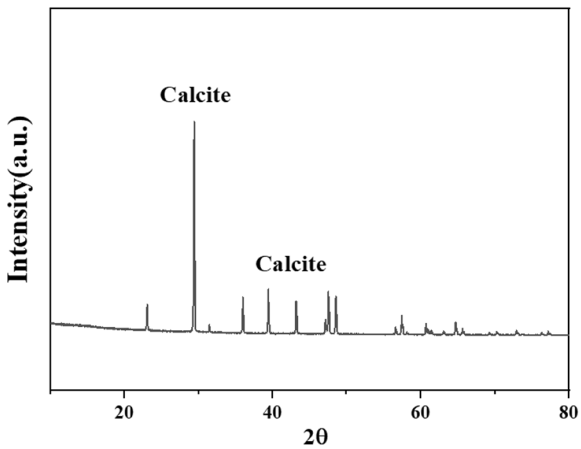


Fig. 10 XRD analysis of microbial mineralization reaction products

The microstructure was further observed by SEM, with results shown in Fig. 11. Studies have shown that the mineralized crystallization of Bacillus pasteurii produces massive calcite type calcium carbonate crystals 20-80 μm in diameter [19], with bacterial indentations about 2 μm long created during the crystallization process [25]. The SEM results showed that *Bacillus pasteurii* energy mineralization produced massive calcite-type calcium carbonate crystals with indentations about 2 μm in length on some crystal surfaces, as shown in Figs. 11a and b. In addition, calcium carbonate crystals accumulated around pores created by the bacteria, as shown in Fig. 11c. Sodium alginate reacted with Ca^{2+} to form calcium alginate gumballs around 3 μm in diameter. Filled with calcium carbonate crystals generated by microorganisms, these bonded into masses, as shown in Fig. 11d. Rong et al. [22] showed that the composition of microbial mineralization products is not affected by calcium alginate gel, but that the formation of large-sized calcium carbonate crystals is inhibited. This is significant because the small-sized calcium carbonate crystals generated by microbial mineralization can better fill the tiny pores present in concrete, improving its structural compactness and thus enhancing its durability [26–28].

Conclusions

In this paper, the effect of nano-modified microorganisms on the performance of concrete crack-patching slurry was investigated. The following conclusions can be drawn from the experimental results:

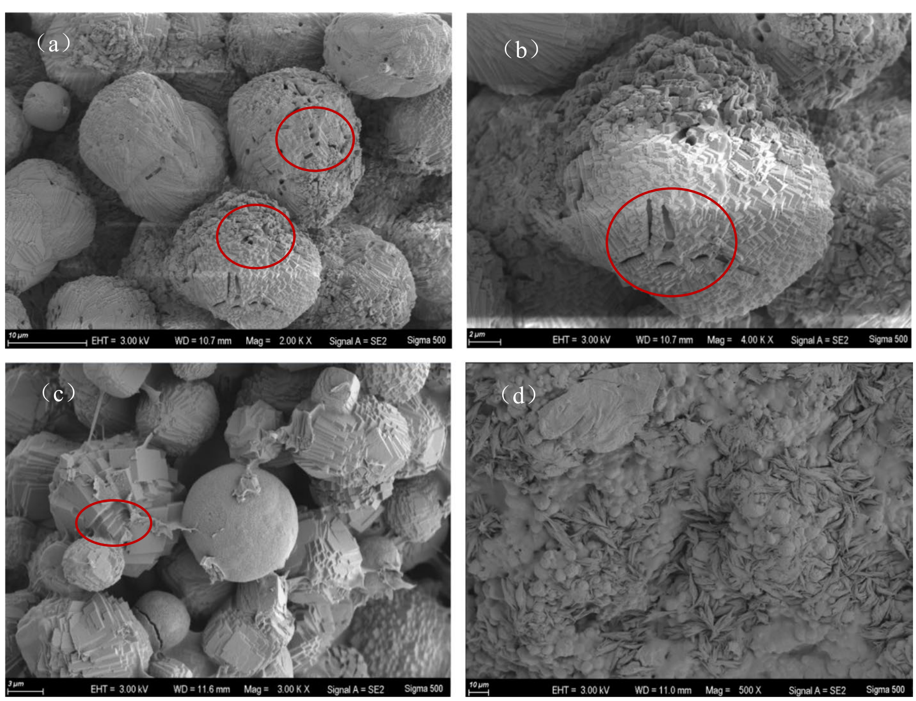


Fig. 11 SEM images of microbial mineralization reaction products

(1) The chloride ion resistance of concrete patched with nano-microbial slurry was more than doubled compared with the control group and the sterile group. The sterile group had a small decrease compared with the control group, but this was not significant, indicating that calcium carbonate crystals generated by microbial mineralization were the main factor in enhancing the durability of the concrete.
(2) The carbonation resistance of the concrete patched by the nano-microbial slurry was over three times higher than that of the control group and the sterile group. The carbonization depth of the sterile group was decreased in the short term, but after 7 days was almost the same as that of the control group. Nano-microorganism self-healing slurry is capable of effectively patching cracks in concrete, and, amongst the slurry components, microorganisms are the main factor in determining its efficacy.
(3) The chemical morphology of calcium carbonate crystals is dependent on the microbial mineralization reaction, which integrates a whole system comprising nano-sio_2, sodium alginate, and polyvinyl alcohol. When the concrete pores are infilled with nano-modified microbial self-healing slurry via vacuum absorption, this effectively patches defects and cracks in the interior of the concrete, optimizing the characteristics and structure of its internal structure, and thus improving its durability.

Authors' contributions
Tao Li: Data curation, Methodology, Writing - Original Draft.Xiaohui Yan: Conceptualization, Methodology, Writing - Review & Editing.Hanqing Yang: Supervision, Writing - review & editing.Maolin He: Data curation, Formal analysis. Haojie Gu: Data Curation, Investigation.LiMing Yu: Data Curation, Investigation.

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Availability of data and materials
No datasets were generated or analysed during the current study.

Declarations

Ethics approval and consent to participate
Not applicable.

Competing interests
The authors declare no competing interests.

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