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Experimental Investigation of Multi-Material Reinforcement for Han Dynasty Bricks from the Dingtao Tomb

Shaohan Wang[1], Shuaikang Su[1], Ji-peng Wang[1,*], Jing-yi Shen[2], Ling Wang[3], and Shangqi Ge[1]

[1]*School of Civil Engineering, Shandong University, Jinan, China;*

[2]*School of Archaeology, Shandong University, Jinan, China*

[3] *Dingtao Museum, Heze, China*

Email: ji-peng.wang@sdu.edu.cn, 957799521@qq.com

ABSTRACT

Han dynasty bricks excavated from the Dingtao King's Mausoleum have undergone severe deterioration after long-term burial in a humid underground environment, resulting in reduced mechanical integrity and increased conservation difficulty. In this study, six consolidants, including organic, inorganic, and microbial-based materials, were evaluated for the reinforcement of deteriorated Han bricks. Reinforcement treatment was conducted using the immersion method, and the effectiveness of different materials was assessed through macroscopic tests, including color difference, unconfined compressive strength, and aging resistance, together with microstructural characterization by scanning electron microscopy and CT analysis. The results show that all tested consolidants improved the brick strength to different extents, whereas the one-phase MICP solution and Bioline consolidant exhibited the best overall performance. The one-phase MICP solution provided the most balanced behavior, showing significant mechanical enhancement, relatively small surface color alteration, and more stable durability under freeze-thaw and wet-heat aging conditions. These results demonstrate that the one-phase MICP solution has strong potential for the conservation and reinforcement of deteriorated Han dynasty bricks.

Keywords: Han dynasty bricks; cultural heritage conservation; reinforcement materials; one-phase MICP; aging resistance; microstructural characterization

INTRODUCTION

China has a long history and a rich cultural heritage, preserving a vast number of brick-and-stone cultural relics. However, after centuries of natural weathering, erosion, and anthropogenic disturbance, many brick-and-stone heritage sites have undergone progressive deterioration, which seriously threatens their structural integrity and highlights the urgent need for effective conservation measures.

The Dingtao King's Mausoleum (M2) (Cheng et al., 2020), a royal Han dynasty tomb in China, is one of the most completely preserved and structurally complex mausoleums discovered to date. Rescue excavations of the site began in 2010, yielding a large number of inscribed artifacts (Wu et al., 2019), including numerous Han bricks bearing personal names, place names, and numerals. These materials provide highly valuable physical evidence for the study of Han dynasty history and culture. The geographical location of the Dingtao King's Mausoleum is shown in Figure 1(a). The site is located in southwestern Shandong Province and central Heze City, between 34°57′–35°15′ N and 115°20′–115°47′ E. It lies in the alluvial plain of the middle and lower reaches of the Yellow River, where repeated alluvial deposition has resulted in a relatively shallow groundwater table. As shown in Figure 1(b), the Han bricks in the tomb chamber remained buried for a long time in a dark and humid underground environment (Liu et al., 2017). After excavation, the bricks became extremely soft and fragile, exhibiting multiple forms of deterioration, including powdering, flaking, fracture, and fragmentation (Figure 1(c)). Therefore, urgent conservation and reinforcement treatments are required for Han bricks with different types of damage.

In addition to improving the mechanical strength and structural stability of relics for long-term preservation, reinforcement materials must also be compatible with the original substrate to avoid secondary damage (Donovan, 2011; Rodrigues & Grossi, 2007). Besides conventional organic, inorganic, and emerging nanomaterials currently used in conservation practice, microbially induced calcium carbonate precipitation (MICP) technology (Ortega-Villamagua et al., 2020) has emerged as a highly promising solution for the reinforcement of brick-and-stone cultural relics (Ortega-Morales & Gaylarde, 2021). This technique was initially developed in geotechnical engineering to improve the mechanical properties of soils and has now become an environmentally friendly alternative to traditional chemical consolidation methods. With the addition of soluble calcium salts, carbonate ions react with calcium ions to form calcium carbonate crystals, which fill pores and microcracks within the substrate.

In this study, Han bricks excavated from the Dingtao Han tomb were selected as the research objects. Traditional consolidants and a single-phase MICP solution were applied by the immersion method for reinforcement treatment. For the reinforcement bricks, macroscopic properties, including unconfined compressive strength, color difference, and aging resistance, were systematically evaluated. The results indicate that the single-phase MICP solution can significantly improve the strength of Han bricks and shows the most effective reinforcement performance among the tested materials. Furthermore, the reinforcement mechanism of MICP-treated bricks was investigated through scanning electron microscopy and CT characterization. Further validation under in-situ application conditions and masonry-scale configurations is still required before broader field implementation.

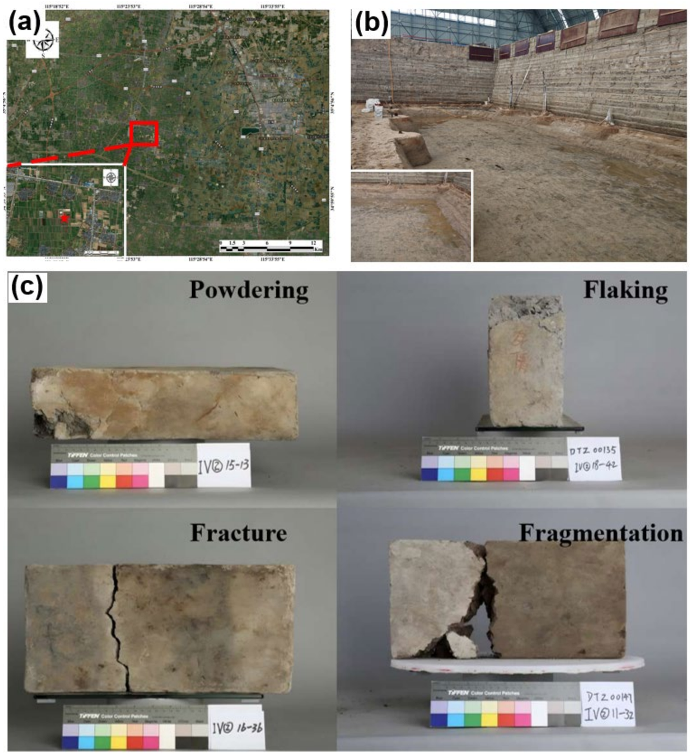

Figure 1. *Overview of the M2 tomb and typical deterioration types of bricks at the Dingtao Han dynasty royal tomb: (a) geographical location; (b) tomb pit; and (c) representative damage types of Han dynasty tomb bricks.*

MATERIALS AND METHODS

Materials

In this study, multiple types of consolidants, including organic, inorganic, and microbial-based materials, were selected for reinforcement treatment of Han dynasty bricks excavated from the Dingtao King's Mausoleum. Table 1 shows the properties of six reinforcement materials.

Among the tested materials, the one-phase MICP solution was prepared based on the low-pH one-phase method proposed by Cheng et al. (2019), in which the bacterial solution and cementation solution are pre-mixed under acidic conditions to delay $CaCO_3$ precipitation and thereby improve penetration and uniformity (Cui et al., 2022; Lai et al., 2023). Sporosarcina pasteurii was used as the bacterial strain. Based on preliminary experiments, the key parameters were determined as follows: $OD_{600} \approx 1$, urease activity of 800-950 μm/(cm·min), initial pH of 6, reaction delay time > 30 min, and cementation solution concentration of 1.5 mol/L.

Table 1. *Properties of reinforcement materials*

Material	Main Chemical Composition	Diluting Solvent	Concentration (Volume Ratio)	Physical State
Bioline	Tetraethyl Orthosilicate	Ethanol	1:2	Colorless transparent liquid
REMMERS 500	Ethyl Silicate	Ethanol	1:1	Colorless transparent liquid
Micro-Nano Lime	$Ca(OH)_2$	Ethanol	1:5	White liquid
Nano-$Ca(OH)_2$	$Ca(OH)_2$	Deionized Water	1:1	Colorless transparent liquid
Nano-$Ba(OH)_2$	$Ba(OH)_2$	Deionized Water	1:2	Colorless transparent liquid
One-phase MICP Solution	Calcium Acetate, Urea	-	-	Pale yellow transparent liquid

Reinforcement procedure

Relatively intact Han dynasty bricks were selected and cut into rectangular laboratory samples measuring 50 mm × 50 mm × 30 mm for tests of physical and mechanical properties as well as durability under aging, as shown in Figure 2(a). To ensure uniform treatment, the samples were consolidated by immersion using different consolidation materials, as shown in Figure 2(b).

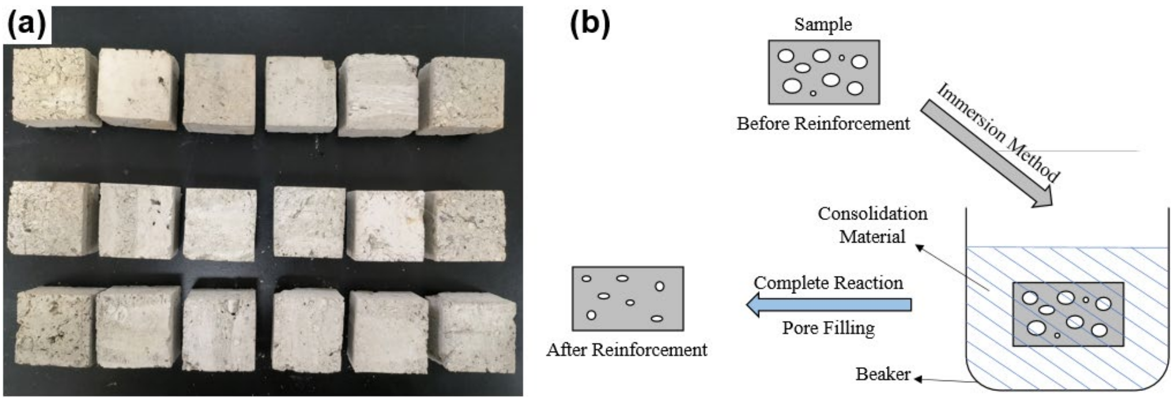

Figure 2. *Han dynasty brick samples and immersion reinforcement procedure: (a) laboratory samples; (b) schematic illustration of the immersion method.*

COMPARISON OF REINFORCEMENT EFFECTS

Color stability

Color difference refers to the change in surface color of cultural relics before and after conservation treatment, and it is commonly used to evaluate the extent of surface color alteration caused by the application of consolidants to bricks. According to relevant conservation requirements, the color variation, expressed as ΔE, should not be excessively large. Based on the CIELAB color system, color measurements were carried out using a CR-10 Plus high-precision colorimeter manufactured by Shenzhen 3nh Technology Co., Ltd., China.

The experimental results indicate that the Han bricks treated with Bioline consolidant and the one-phase MICP solution exhibited the smallest surface color differences, with ΔE values of 0.68 and 1.56, respectively. In contrast, the bricks consolidated with micro-nano lime and nano-$Ca(OH)_2$ showed more pronounced color changes, with ΔE values of 5.47 and 6.42, respectively. The bricks treated with nano-$Ba(OH)_2$ and REMMERS 500 exhibited relatively moderate color differences, with ΔE values of 2.25 and 2.85, respectively. Overall, Bioline consolidant and the one-phase MICP solution caused only minor color alteration, whereas the other reinforcement materials resulted in more noticeable color changes.

In further experiments, the bricks were subjected to multiple applications of Bioline consolidant and the one-phase MICP solution, and the corresponding color differences are presented in Figure 3. The results show that the ΔE values gradually increased with the number of consolidation treatments. After repeated applications, the color differences of the bricks treated with Bioline consolidant and the one-phase MICP solution gradually approached similar levels.

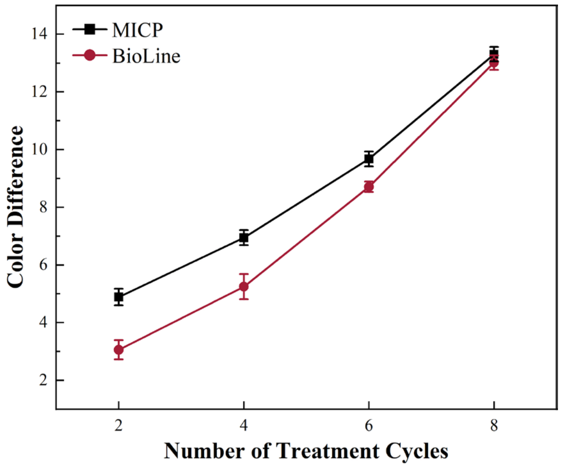

Figure 3. *Variation in color difference with the number of treatment cycles for Bioline consolidant and the one-phase MICP solution.*

Mechanical enhancement

Since the unconfined compressive strength (UCS) test is destructive, bricks with similar mass were selected to ensure the reliability of the results. Each test group consisted of three bricks. The UCS tests were carried out using a YZW-50 multifunctional rock shear and seepage testing machine (Jinan Rock and Mineral Testing Equipment Co., Ltd.), with a rated load of F ≤ 500 kN and an accuracy of 1%. Displacement was recorded by a computerized data acquisition system with a precision of 0.001 mm. Loading was applied through an electro-hydraulic servo system capable of both constant-stress and displacement-controlled modes.

As shown in Figure 4(a), the compressive strength of the Han bricks increased after treatment with all consolidants. The most pronounced improvements were obtained with the one-phase MICP solution and Bioline consolidant, with strength enhancement ratios of 30.30% and 29.95%, respectively. Bricks treated with nano-$Ba(OH)_2$ also showed a notable increase in compressive strength, reaching 21.19%. REMMERS 500 and micro-nano lime resulted in moderate improvements of 18.04% and 14.19%, respectively. In contrast, nano-$Ca(OH)_2$ showed the weakest strengthening effect, with an increase of only 6.48%.

Overall, all tested consolidants improved the compressive strength of the Han bricks to different extents, while the one-phase MICP solution and Bioline consolidant exhibited the best reinforcement performance. These two materials were therefore selected for further comparison. Further UCS tests were conducted on bricks treated with different numbers of applications of the one-phase MICP solution and Bioline consolidant, and the results are presented in Figure 4(b). It can be seen that the compressive strength increased with the number of treatment cycles for both materials. Under the same number of applications, the one-phase MICP solution consistently produced higher brick strength than Bioline consolidant.

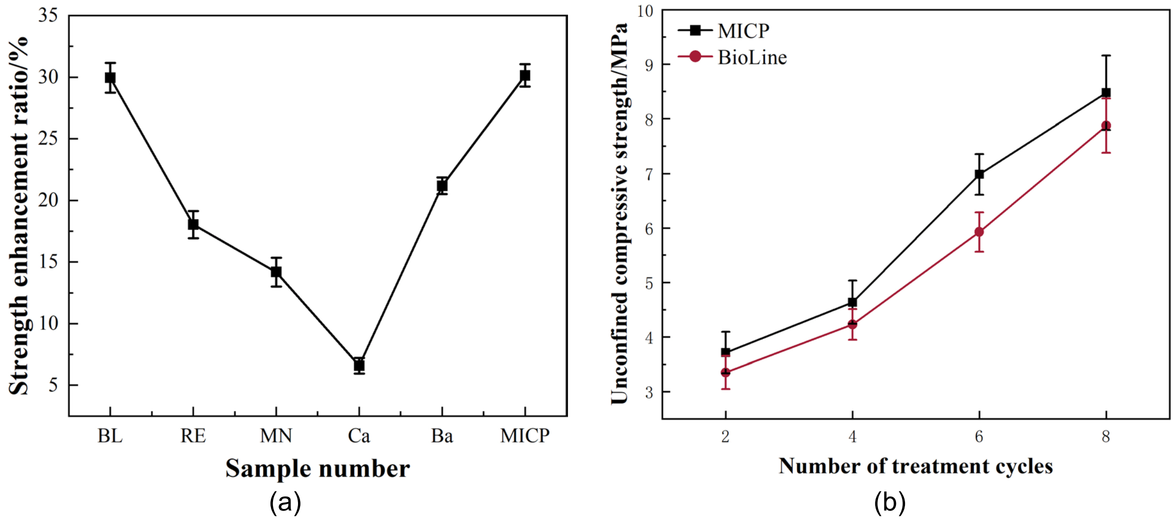

Figure 4. *Unconfined compressive strength results of Han bricks after consolidation: (a) strength enhancement ratio for different consolidants; (b) variation in unconfined compressive strength with the number of treatment cycles for Bioline consolidant and the one-phase MICP solution.*

Aging resistance

The one-phase MICP solution and Bioline consolidant, which showed the best reinforcement performance in the previous tests, were selected for the aging experiments. The unconfined compressive strength, mass, and color difference of the treated Han bricks before and after aging are summarized in Table 2. Overall, both materials exhibited good aging resistance, although their performances varied under different aging conditions, reflecting differences in their intrinsic properties.

Table 2. Mechanical properties, mass change, and color difference of Han bricks treated with the one-phase MICP solution and Bioline consolidant before and after aging

Material	Aging condition	UCS Before Aging /MPa	UCS After Aging /MPa	Residual Strength Ratio	Mass Before Aging /g	Mass After Aging /g	Mass Change Rate	ΔE
MICP	F-T aging	12.004	9.95	82.85%	114.1	110.0	3.55%	13.80
	W-H aging	12.004	11.30	94.12%	113.1	111.7	1.20%	13.29
	Salt aging	12.004	10.62	88.48%	115.9	123.0	-6.05%	5.85
Bioline	F-T aging	12.608	8.86	70.25%	110.9	105.7	4.72%	16.52
	W-H aging	12.608	9.73	77.18%	113.5	111.9	1.36%	15.49
	Salt aging	12.608	9.56	75.85%	113.5	120.3	-6.04%	14.16

Among the three aging tests, freeze-thaw aging had the most significant effect on the mechanical performance of the bricks. After freeze-thaw aging, the strength retention ratios of the bricks treated with the one-phase MICP solution and Bioline consolidant were 82.85% and 70.25%, respectively, indicating that the MICP-treated bricks had better resistance to freeze-thaw damage. Wet-heat aging had a relatively smaller effect, with strength retention ratios of 94.12% for the MICP-treated bricks and 77.18% for the Bioline-treated bricks. In contrast, soluble-salt aging caused only moderate strength reduction, with residual strength ratios of 88.48% and 75.85%, respectively.

The mass change results in Table 2 show that freeze-thaw aging caused the greatest mass loss for both materials, whereas soluble-salt aging resulted in an apparent mass increase due to salt accumulation within the bricks. In terms of appearance, soluble-salt aging produced the smallest color difference for the MICP-treated bricks (ΔE = 5.85), while freeze-thaw aging caused the most pronounced color change for both materials, with ΔE values of 13.80 for the MICP-treated bricks and 16.52 for the Bioline-treated bricks. Overall, both consolidants demonstrated acceptable anti-aging performance, but the one-phase MICP solution showed more stable behavior, particularly under freeze-thaw and wet-heat aging conditions.

MICROSTRUCTURAL CHARACTERIZATION

Scanning electron microscopy experiments

Unreinforced Han bricks, as well as bricks treated with Bioline consolidant and the one-phase MICP solution, were examined by SEM at different magnifications, and the corresponding images are presented in Figure 5.

As shown in Figure 5(a), the unreinforced bricks exhibited a rough and severely weathered surface, characterized by abundant pores and cracks. After treatment with Bioline consolidant, the brick surface was covered by a reaction-generated quartz layer, which formed a relatively hard shell; however, multiple cracks were still observed within this quartz layer, as shown in Figure 5(b). In contrast, treatment with the one-phase MICP solution produced abundant calcium carbonate precipitates, mainly in spherical forms, which accumulated on particle surfaces and along pore edges, thereby effectively filling pre-existing cracks and voids, as shown in Figure 5(c). Compared with Bioline consolidant, the one-phase MICP solution exhibited better crack-sealing capability and more effective microstructural restoration.

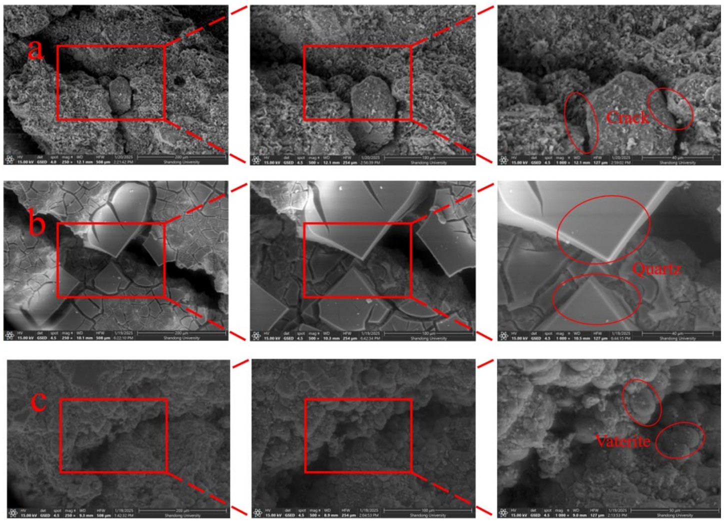

Figure 5. *SEM images of (a) unreinforced Han bricks, (b) Han bricks treated eight times with Bioline consolidant, and (c) Han bricks treated eight times with the one-phase MICP solution, all at 250×, 500×, and 1000× magnifications.*

CT scanning experiments

To investigate the effect of the one-phase MICP solution on the three-dimensional spatial structure of Han bricks, CT scans were performed on the same region of a brick before reinforcement and after eight treatment cycles.

The corresponding gray-value distributions are shown in Figure 6. Two distinct peaks can be clearly identified, representing air and the Han brick, respectively, and their corresponding gray-value ranges remained generally consistent before and after reinforcement. After reinforcement, the overall gray-value distribution shifted slightly to the right, indicating an increase in image brightness, which corresponds to an increase in solid-phase density due to calcium carbonate precipitation. Meanwhile, the gray-value peak associated with air decreased after reinforcement, suggesting that the internal structure of the brick became denser and that the pore volume was reduced. These results provide further microstructural evidence for the effectiveness of the one-phase MICP solution, demonstrating that the generated calcium carbonate can effectively fill pores and significantly enhance the reinforcement of Han bricks.

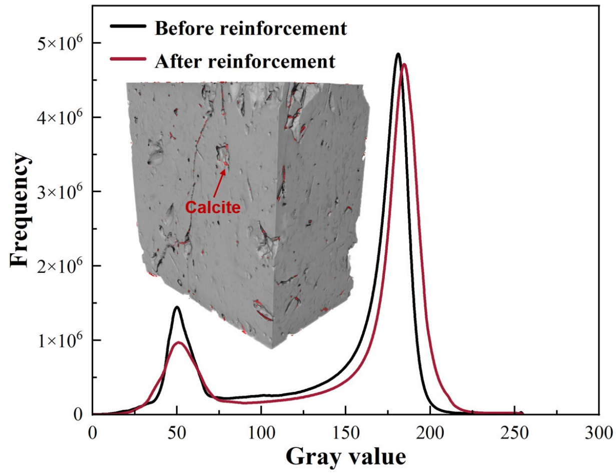

Figure 6. *Comparison of CT-based gray-value distributions of the same region of a Han brick before reinforcement and after eight treatment cycles with the one-phase MICP solution.*

Subsequently, the pore volume in the CT images was quantified to determine the porosity of the bricks before and after reinforcement, defined as the ratio of pore volume to total volume. The porosity decreased from 28.98% before reinforcement to 26.21% after reinforcement, indicating that part of the pore space was filled during the reinforcement process, thereby reducing both the number and volume of pores and making the overall structure denser. In particular, within the small-pore range, the number of pores smaller than 1 mm decreased from approximately 20,000 to about 14,000, suggesting that the one-phase MICP treatment preferentially filled smaller pores, as shown in Figures 7(a) and (b).

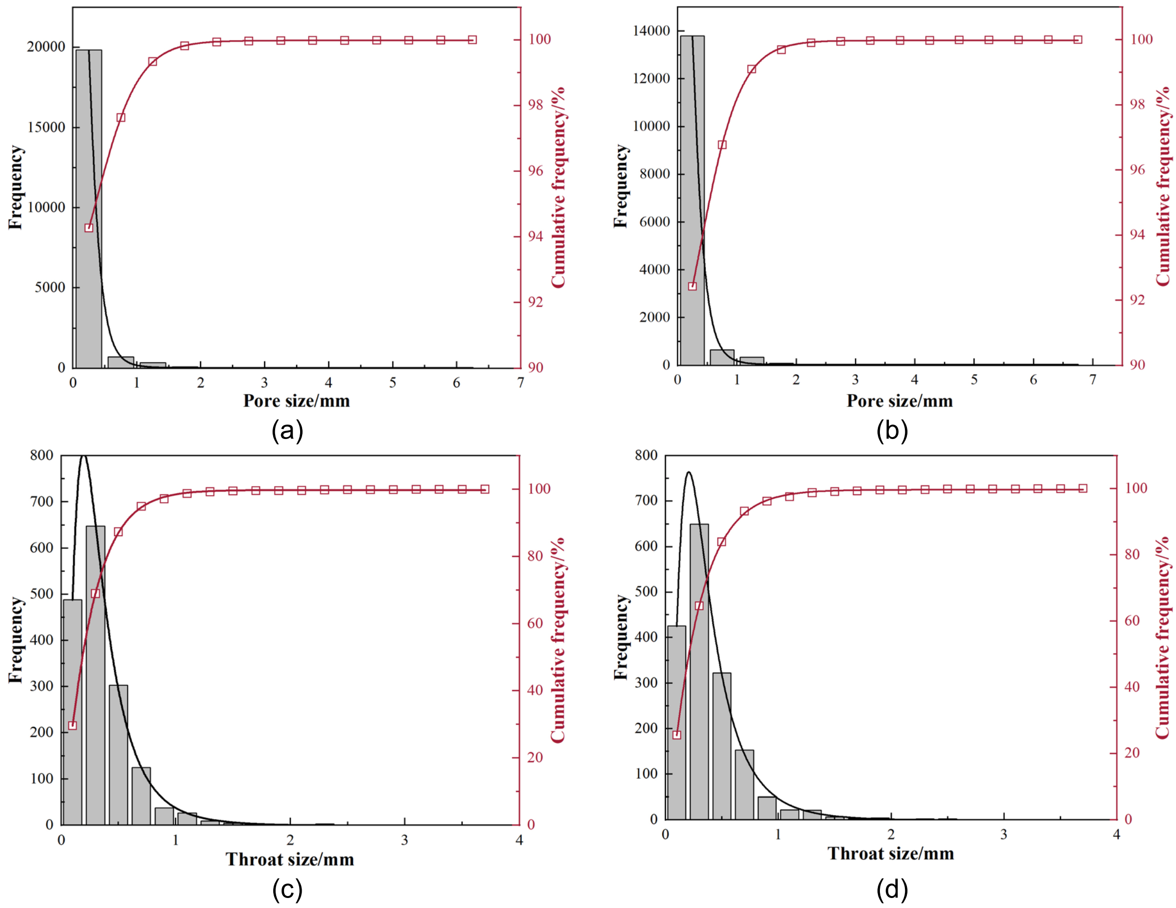

Figure 7. *Pore-size and throat-size distributions of Han bricks before and after reinforcement with the one-phase MICP solution: (a) pore-size distribution before reinforcement; (b) pore-size distribution after reinforcement; (c) throat-size distribution before reinforcement; and (d) throat-size distribution after reinforcement.*

During reinforcement, throat size was noticeably altered by calcium carbonate precipitation. As shown in Figures 7(c) and (d), the number of throats with diameters smaller than 1 mm decreased after reinforcement due to calcium carbonate filling. In particular, the number of the smallest pore throats decreased from about 500 to about 400. Combined with the observed changes in pore-size distribution, throat-size distribution, and overall porosity, these results demonstrate that the one-phase MICP treatment effectively reduced the pore volume of the bricks.

DISCUSSION

The potential applicability of the proposed treatment is closely related to the material characteristics of the heritage substrate. Since the one-phase MICP method relies on calcium carbonate precipitation, it is expected to be more suitable for calcareous brick- and stone-based relics, where calcium-bearing phases are already part of the original material system. Similar conservation contexts can be found in limestone heritage sites such as Göbekli Tepe and in carbonate stone fabrics such as those used in York Minster.

In the present study, the immersion procedure was primarily adopted to ensure treatment uniformity and comparability under laboratory conditions. In real conservation practice, however, complete immersion is often impractical for standing masonry and historic structures. It is worth noting that factors such as pore structure, salt content, weathering degree, and previous conservation history may still influence compatibility and long-term performance. Current work is therefore being extended to spraying and grouting methods, with preliminary grouting tests already showing encouraging results. Future studies will further validate the proposed treatment in wall assemblies or masonry systems and combine it with non-destructive monitoring methods to evaluate penetration, moisture migration, and consolidation uniformity.

CONCLUSIONS

This study experimentally investigated the reinforcement performance of six consolidants for Han bricks excavated from the Dingtao Han tomb, with particular focus on the one-phase MICP solution and Bioline consolidant. Based on the results obtained, the following conclusions can be drawn:

1) All tested consolidants improved the mechanical performance of the Han bricks to different extents. Among them, the one-phase MICP solution and Bioline consolidant showed the best overall reinforcement effects, while also causing relatively small surface color changes.

2) In the aging tests, both the one-phase MICP solution and Bioline consolidant exhibited acceptable durability, whereas the one-phase MICP solution showed more stable performance, particularly under freeze-thaw and wet-heat conditions.

3) SEM and CT results demonstrated that the one-phase MICP treatment effectively filled pores and cracks through calcium carbonate precipitation, reduced pore volume, and densified the internal structure of the bricks. These microstructural improvements explain its superior reinforcement performance.

Overall, the one-phase MICP solution demonstrated the most balanced performance in terms of mechanical enhancement, color stability, aging resistance, and microstructural improvement, indicating strong potential for the conservation and reinforcement of deteriorated Han bricks.

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