



A case study and mechanism investigation of typical mortars used on ancient architecture in China

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ABSTRACT

Mortars sampled from Dutifulness Monument, where typical ancient China mortar formulas and manufacturing processes were used, were analyzed by starch-iodine test, FTIR, DSC-TG, SEM and XRD methods. Several modeling samples were then made according to historical records of Chinese ancient mortar formulas and analyzed with the same techniques. The modeling formulas also were used to consolidate loose specimens. The results show that sticky rice plays a crucial role in the microstructure and the consolidation properties of lime mortars. A possible mechanism was suggested that biomineralization may occur during the carbonation of calcium hydroxide, where the sticky rice functions as a template and controls the growth of calcium carbonate crystal. The organic-inorganic materials formed based on this mechanism will be more favorable for consolidating the loose samples both in strength improvement and durability.

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1. Introduction

Lime-based mortars are most widely used in architectural monuments. Normally, it is safe, durable and harmless to the original materials if necessary repair is done with similar materials and using the correct way of application [1]. Original and replacement mortar materials are, therefore, encountered during the conservation of monuments. However, properties of mortars, especially original mortars, are not fully understood for their complexity and diversity. Several analysis techniques, for example, thermal analysis, including thermogravimetry (TG), derivative thermogravimetry (DTG), differential thermal analysis and differential scanning calorimetry (DSC), X-ray diffractometry (XRD) and Fourier-transform infrared spectroscopy (FTIR) have been proved [2–5] to be the most useful methods for the analytical studies of ancient mortars; microcopy [6], such as polarized light microscopy (PLM) scanning electron microscopy (SEM) and atomic force microscopy (AFM), are becoming more and more popular for their morphology observation. Some reported results indicated that mortars mainly consist of inorganic materials, involving lime, pozzolana, magnesium and barium salt and occasionally some organic adhesive, such as egg white, blood, milk of figs, egg yolk, casein, animal glue, beer, vegetable juices, tannin, urine, etc [7].

Although great efforts have been involved, many works still can be expanded. First, more cases from various regions should be studied for the most cases reported only focus on Europe [8–13] presently. In fact, mortars were used very popularly in ancient architecture of China, for instance, dyke of Songhua river [14], Qiantan river [15], Dutifulness Monument, sticky rice bridge [16] and circumvallation of Jinzhou [17]. These projects had been finished during Ming dynasty (1368–1644 A.D.) and Qing dynasty (1644–1911 A.D.) and still are conserved well after several hundreds years. According to a unique formula recorded in ancient books, the mortars used in those structures were mainly made of lime and sticky rice solution [18]. Second, in the past decades, few reports touched consolidation mechanism of mortars, especially functions of organic substances during setting and hardening processes. These organic additives may play an important role though their proportion in formula is small.

Mortars in this work were sampled from Dutifulness Monument, which locates in Fanpu village, Shaoxin, Zhejiang, China, and was constructed in 1750 A.D. Starch-iodine test was conducted on parts of the samples and others were analyzed with DSC-TG, FTIR, SEM and XRD methods. Then, several modeling samples were fabricated according to formulas of the literature [18], and the same analyses were conducted for further understanding their microstructure. A possible mechanism was suggested how the microstructure of the mortars is affected by sticky rice. Finally, the modeling formulas were applied to reinforce some loose specimens to test their consolidating abilities.

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Fig. 1. The sampling site on Dutifulness Monument.

2. Materials and methods

2.1. Materials

Two historical mortar samples from Dutifulness Monument were taken when the site was being repaired (Fig. 1). The two sample positions are about 5 cm and 10 cm under surface of the original mortar (location labeled on Fig. 1), respectively.

CaCO_3 powders and CaCO_3 particles for strength test specimens preparation are 1.3 μm and 150 μm , respectively. Pure chemicals including CaCl_2 C.P. Grade, $\geq 96.0\%$ assay, $\text{CO}_2 \geq 99.9\%$, KI, I_2 , CH_3COOH and NaOH A.R. Grade, $\geq 99.8\%$ assay were used in this study. Sticky rice, which is a type of rice grown in Southeast and East Asia and mainly composes of amylopectin [19], is commercially available. White marble, which is at the size of 5 cm \times 5 cm \times 2 cm and not weathered, was selected as sample stone for loading mineralization solution.

2.2. Instruments and conditions

The surface morphology of the samples was inspected with scanning electron microscopy (FEI SIRION-100).

For identifying the crystallography of the mortars, XRD analyses were performed with an AXS D8 ADVANCE X-ray diffractometer using $\text{Cu K}\alpha$ radiation ($\lambda = 1.54 \text{ \AA}$), 40 kV power and 40 mA current.

Infrared analysis usually permits the identification of the main molecular groups present in mortars. In this article, a NICOLET 560 Fourier-transform infrared spectrometer was employed, and the mortar samples were analyzed in KBr pellets. The spectra were traced in the range of 4000–400 cm^{-1} (wave number), and the band intensities were expressed in transmittance (%).

The thermal analyses were completed on a simultaneous DSC–TG equipment (TA Instruments, model NETZSCH STA 409 PC/PG). The experimental conditions were: (a) continuous heating from room temperature to 1000 $^\circ\text{C}$ at a heating rate of 20 $^\circ\text{C}/\text{min}$; (b) air–gas dynamic atmosphere (45 cm^3/min); (c) alumina, top-opened crucible; (d) sample mass: 15 mg approximately. DSC and TG curves were collected.

The compression strength test was carried out by using a Yinchi YC-125B tension meter (Shanghai, China), of which compressive

stress can be measured from 0 to 2000 N. The form of specimen is a cylinder, $\Phi 39.8 \text{ mm} \times 80 \text{ mm}$. The surface hardness (Shore hardness) was determined with a Qianzhou LX-A hardness scale (Wuxi, China). The hardness test procedure: fix the specimen on a firm worktable, press pin of the hardness instrument into the surface of the specimen and read the value within 1 s after full touch between the pin and the surface. Five positions (at least 15 mm between two positions) of specimen surface have to be tested and an average value calculated presents the hardness value of the tested specimen.

2.3. Methods

The historical samples were pulverized and dried under 60 $^\circ\text{C}$ for 48 h before the various analyses were conducted.

Starch–iodine test: 10.0 g of the historical samples was ground into grains, diameter of which is about 1–10 μm , and was added into 100 ml water, heated at 80–90 $^\circ\text{C}$ for 10 min with stirring. The formed suspension then was cooled to 25 $^\circ\text{C}$ and its PH was adjusted to 6.0–7.0 with acetic acid. Iodine–KI reagent was made by dissolving 0.2 g iodine in 100 ml water in the presence of 2.0 g KI [20]. Iodine–KI reagent was dropped into the suspension with stirring, and a blue or red brown color could be observed if starch (blue for amylose and red brown for amylopectin) is present [21].

1–4% sticky rice solution was made by cooking mixture of 1–4 g sticky rice and 100 ml distilled water at 100 $^\circ\text{C}$ and 1.0 bar for 30 min, cooling to 25 $^\circ\text{C}$, and adjusting total weight of the mixture to 100 g by adding the water. 0.01 mol l^{-1} super saturated solution of $\text{Ca}(\text{OH})_2$ was prepared with 1.11 g (0.01 mol) CaCl_2 and 0.80 g (0.02 mol) NaOH in 1000 ml standard container.

The loading solutions A and B were made of 100 ml of the $\text{Ca}(\text{OH})_2$ solution mixing with 100 ml and 25 ml of 3% sticky rice solution, respectively; the consolidating solution 1–4 were prepared by mixing 100 ml of the $\text{Ca}(\text{OH})_2$ with a same volume of 1–4% sticky rice solution.

The modeling samples for investigating how sticky rice affects crystal morphology of CaCO_3 were generated by both surface method and solution method. When the solution method was adopted, CO_2 was respectively introduced into 1000 ml of the loading solutions A and B from the bottom of a container. The rate of CO_2 is 20 ml/min and time is 30 min. The formed powder precipitations were labeled MPA and MPB, respectively, which were dried at the same conditions as those of the historical samples before being analyzed by FTIR, XRD and SEM. When the surface method was employed, the modeling sample MPS was processed by coating the loading solution A on the white marble for nine times, and the interval time is about 5 min. The morphology of the MPS sample, the hybrid of sticky rice and CaCO_3 crystal film on white marble, was observed by SEM after 3 days exposure at room temperature and 60–70% humidity.

The specimens for strength test: evenly blended 75 g CaCO_3 powders, 75 g CaCO_3 particles and 15 ml the consolidating solution 1–4 (or water) were added into the cylinder shaped mould of the home-made instrument and the hammer was dropped from the height of 277 mm to compress the samples for 50 times. The compaction work is 1159.42 kJ/m^3 . The columned specimens were dried at room temperature and 60–70% humidity for 7 days before the strength tests.

3. Results and discussion

3.1. Analyses of the historical samples

Only two samples were collected because the repair mainly was done on its surface. Generally, the number is too small to gain repre-

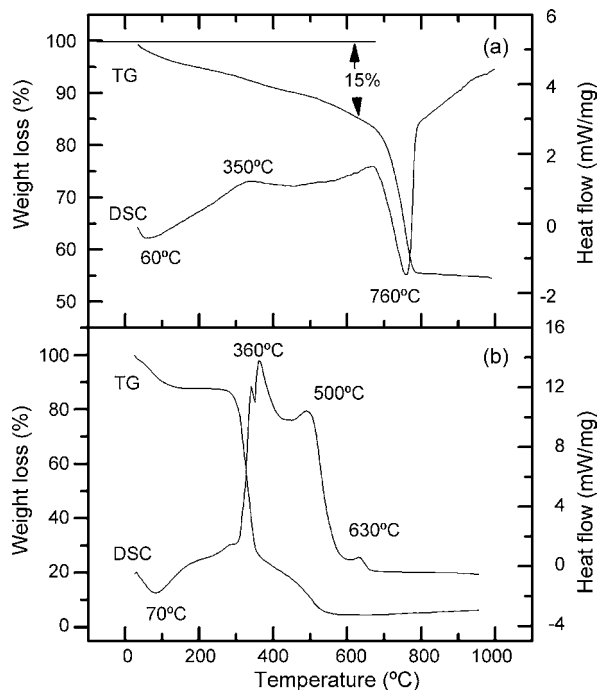


Fig. 2. DSC–TG curves of (a) the historical sample and (b) sticky rice.

sentative information of mortars for its complexities. However, the samples in two positions had not been disturbed by people since the monument constructed, and the results will be valuable if they are analyzed carefully.

Starch–iodine test shows that red brown was very evident after added into 3–5 drops of iodine–KI reagent in the 100 ml mortar suspension at room temperature. The reason is that amylopectin in sticky rice coordinates with iodine and form brown iodine–amylopectin complex [21] in the test. This complex, however, is very easy to be absorbed on surface of the mortar grains and it will deposit on bottom of the container when placed hours without being stirred. It conveys that some sticky rice might still exist in the historical mortars.

According to test outcome of the samples at two positions, the IR, TG and XRD of two samples are nearly equivalent, therefore, only a set of results will be discussed in this section.

Fig. 2 shows the DSC–TG curves of the historical samples and pure sticky rice. As seen in TG curve of Fig. 2a, weight of the samples begins to decrease slowly at 30 °C, and about 15% of the weight is lost before 630 °C, where the curve drops down rapidly and another 30% of the weight vanished between 630 and 790 °C. In the DSC curve of Fig. 2a, two endothermic peaks at 60 and 760 °C, and an exothermic peak around 350 °C can be observed. It suggests that the free water in the samples begins to vaporize at 60 °C, organic materials start to decompose in the oxygen atmosphere around 350 °C, and the CaCO_3 decomposes at 630 °C. The organic materials, if compared to DSC–TG curves of Fig. 2b, might be sticky rice for the same decomposed temperature though another exothermic peak around 500 °C on DSC curves of Fig. 2b cannot be found on that of Fig. 2a, which may occur when sticky rice content is very low in the mortar samples. The content of CaCO_3 ($W_{\text{CaCO}_3}\%$), according to the percentage of carbon dioxide ($W_{\text{CO}_2}\%$) release, can be calculated by the formula:

$$W_{\text{CaCO}_3}\% = \frac{M_{\text{CaCO}_3}}{M_{\text{CO}_2}} W_{\text{CO}_2}\%$$

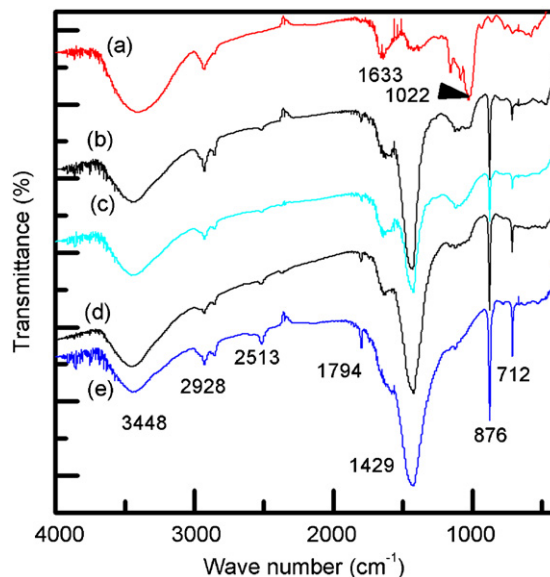


Fig. 3. Infrared spectra of (a) the pure sticky rice, (b) the historical sample, (c) the sample MPA, (d) the sample MPB and (e) calcite. MPA (MPB): the precipitation of 1 volume (4 volumes) 0.01 mol l^{-1} calcium hydroxide solution mixing with 1 volume 3% sticky rice solution reacting with CO_2 .

where M_{CaCO_3} is molecular weight of CaCO_3 and M_{CO_2} is that of CO_2 . The calculated result of CaCO_3 is about 70% in the samples. The content of sticky rice is about 3%, estimated by TG curves of Fig. 2a between 200 and 500 °C, where it is degraded (according to Fig. 2b).

The FTIR results of the sticky rice (a), the historical samples (b) and calcite (e) are given in Fig. 3. The wave number bands at 712, 876, 1429, 1794 and 2513 cm^{-1} are assigned to the calcite, which all can be found on the curve of the sample (b), where absorption bands at 3448 and 1633 cm^{-1} brought by the –OH of water or that of the sticky rice also can be observed. In addition, the adsorption between 1000 and 1100 cm^{-1} could be from the sticky rice [22] when comparing curve (b) with (a). These results indicate that the inorganic components of the samples mainly consist of calcite, and the organic components might contain part of the sticky rice, which still is not decomposed.

In order to further testify the crystal of the calcium carbonate in the historical samples, powder diffraction was carried out and the results are presented in Fig. 4. It is easily to be observed that all the peaks in Fig. 4a can be found in Fig. 4b, which is the XRD results of the pure calcite. However, the intensity of the diffraction peaks of the historical sample is very weak when comparing with those of the pure calcite, which suggests that there is a great amount of amorphous calcium carbonate in the sample besides the calcite. The result also can be seen in the SEM results of the sample (Fig. 5) because almost no perfect crystal of the calcite could be found.

According to the analyses above, a conclusion can be drawn that the historical sample mainly composes of two forms of CaCO_3 , i.e. calcite and amorphous body, and organic materials mainly formed by sticky rice and some of its degraded products.

3.2. Analyses and tests of the modeling samples

In order to take a deeper look at the historical mortars, several modeling samples were fabricated by the solution method and the surface method. Two methods aimed at investigating how sticky rice affects microstructure of CaCO_3 in the mortars. Different from previous works [23], we adopted $\text{Ca}(\text{OH})_2$ super saturated solution

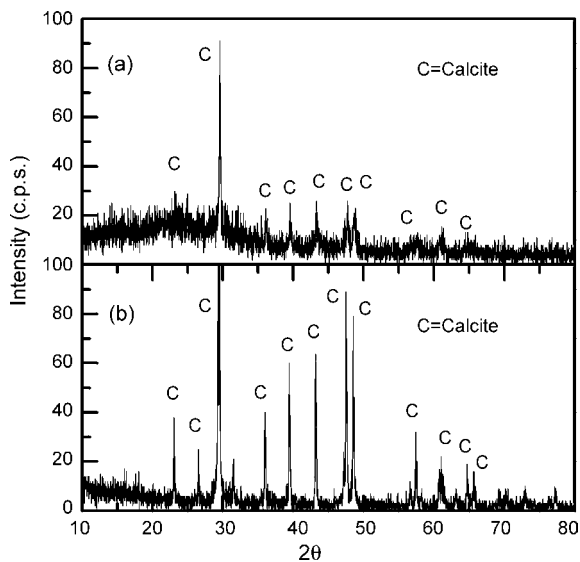


Fig. 4. XRD spectrum of (a) the historical sample and (b) calcite.

instead of suspension in the two methods, because $\text{Ca}(\text{OH})_2$ grains in the suspension will be difficult to react with CO_2 after parts of them on grain surface have been converted into CaCO_3 . The XRD results show that carbonation has finished after 30 min for solution method and 3 days for surface method.

The FTIR analysis was firstly conducted on sample MPA and sample MPB, which were made by the solution method, and the results were illustrated in Fig. 3c and d. It is found that the shapes of the two curves are very similar. Both of them possess all the characteristic peaks of calcite and the sticky rice, but the intensity of which is different, and the peaks of the calcite are lower when the ratio of the sticky rice during the modeling sample preparation is higher (the sample MPA). To present more information of crystal in the modeling samples, the XRD analyses were carried out and the results were given in Fig. 6. It is clear that all the calcium hydroxide was converted into calcium carbonate because only the characteristic peaks of calcite were found in Fig. 6a and b. Just as presented in

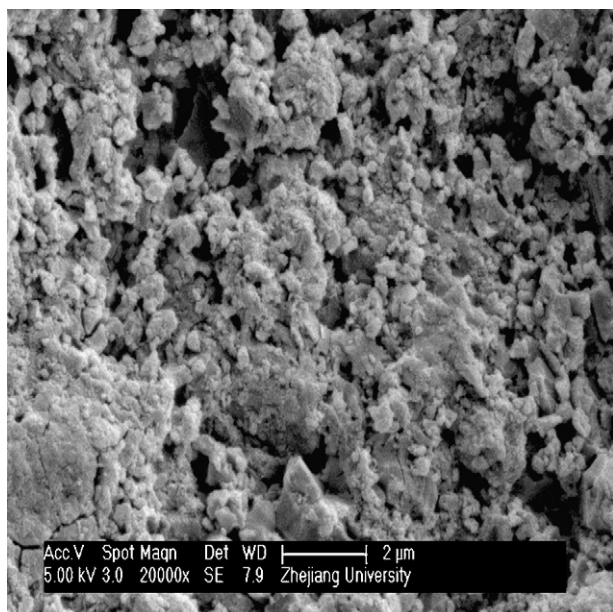


Fig. 5. SEM image of the historical sample.

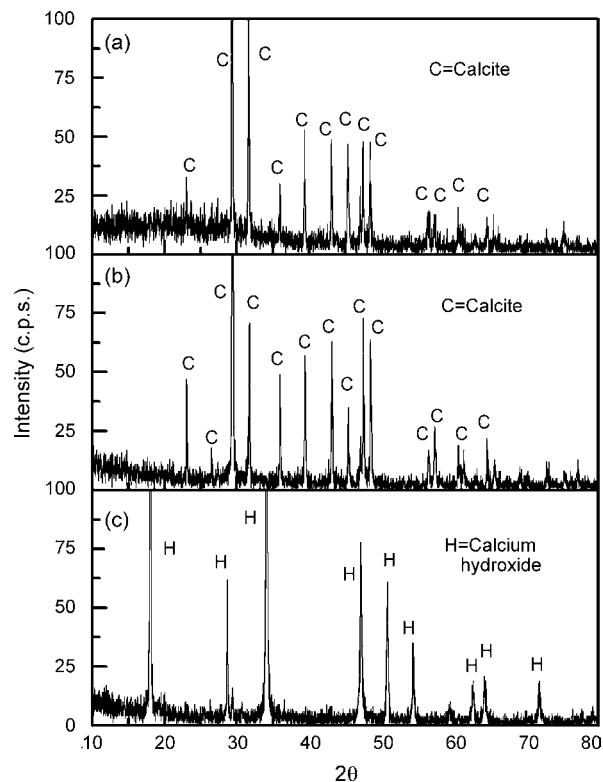


Fig. 6. XRD spectra of (a) the sample MPA, (b) the sample MPB and (c) calcium hydroxide.

the FTIR results, the sample MPA, which has more sticky rice in the crystal, shows lower intensity of the calcite peaks.

Fig. 7 gives the SEM results of the solution method samples. As can be seen from Fig. 7a, where the modeling sample MPA was prepared with higher sticky rice concentration, the morphology of precipitation is very irregular and few perfect calcite crystals can be detected. However, when the sticky rice solution was adjusted to a lower concentration, the morphology of the calcium carbonate precipitation (Fig. 7b) mostly presents cubic shape and dimension of the crystal becomes larger, which is very similar to that of the calcite, where an interesting sandwich and step structure is observed. It implies that the morphology of the calcium carbonate crystal can be controlled by changing the concentration of the sticky rice, which denotes the characteristics of biomineralization [24]. Actually, the morphology of the crystal in sample MPA is very similar to that of the historical sample when comparing Fig. 5 with Fig. 7a. It also suggests that the microstructure of the modeling samples is very close to that of the real traditional samples.

Fig. 8 presents the SEM results of the surface method. It is found that the marble surface becomes very smooth and is covered with some strip shaped agglomeration (Fig. 8a), the dimension of which is about $0.5 \mu\text{m} \times 1.5 \mu\text{m}$. In a much finer SEM image (Fig. 8b), the smaller crystal growing under the sticky rice can be detected. The results are different from the growth of calcium carbonate without additives, where the crystal is generally in the calcite shape.

From Fig. 8b, it is clear that sticky rice embeds among the crystals in the surface method based samples, which cannot be detected in the solution method sample figure (Fig. 7a). The difference is due to the difference mechanisms of the formation of the two types of samples. When the surface method is used, with the evaporation of water, $\text{Ca}(\text{OH})_2$ and the sticky rice deposit easily on the surface of the marble to form smooth surface; then, $\text{Ca}(\text{OH})_2$ in the mixture reacts with CO_2 from the air to produce CaCO_3 crystals from the

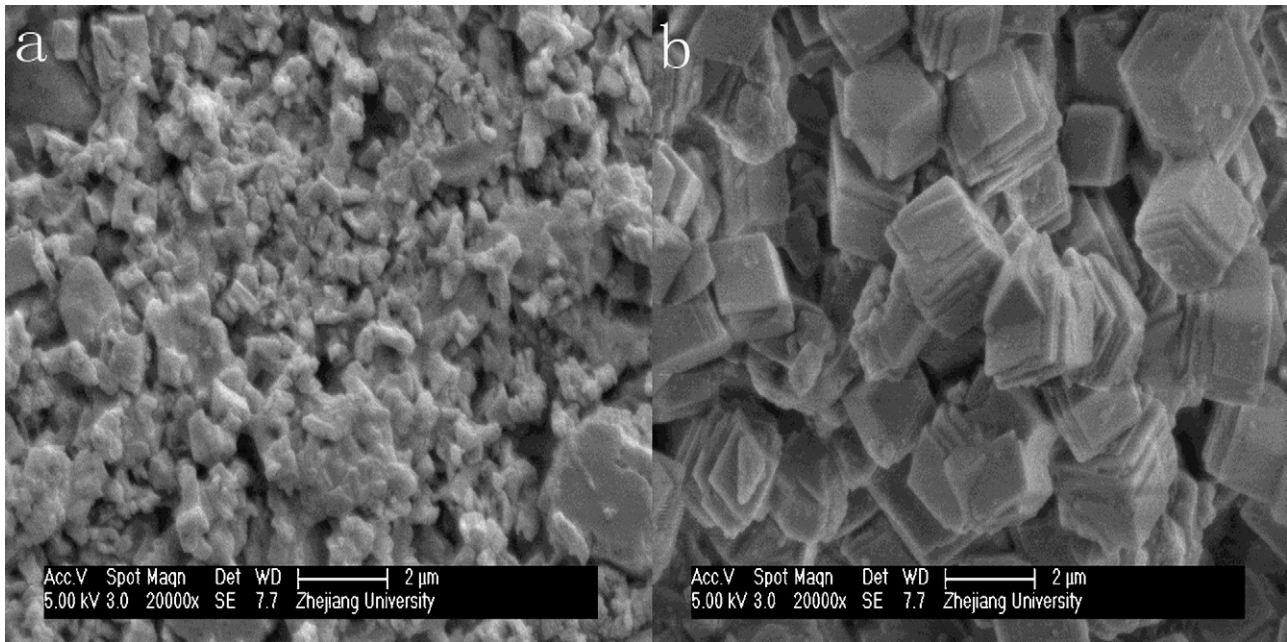


Fig. 7. SEM image of the solution method samples: (a) the sample MPA and (b) the sample MPB.

undersurface of the smooth surface, the morphology of the surface in Fig. 8b, therefore, appears. When the solution method is adopted, CO_2 is introduced directly into $\text{Ca}(\text{OH})_2$ solution to generate CaCO_3 ; during the deposition of the CaCO_3 , part of the sticky rice in the solution can only be adsorbed on the surface of the CaCO_3 particles to form very thin enrapping. As a result, the surface just looks like what presents in Fig. 7a. It also indicates, in the surface method, that the sticky rice influences the morphology of the calcium carbonate crystal during the carbonation of the lime mortars.

Application of the modeling formulas also was tested besides the investigation of their microstructure in this work. Fig. 9 gives the consolidation results of the 0.01 mol l^{-1} calcium hydroxide

solution mixing with different concentrations of the sticky rice solution at a 1:1 volume ratio. It is disclosed that the compression strength of the blank loose samples is only 0.04 MPa, while the value increases to 0.95 MPa after the samples are consolidated with the 0.01 mol l^{-1} calcium hydroxide solution mixed with 3% sticky rice solution. Meanwhile, the surface hardness of the consolidated samples increases from 80 HA to 200 HA. In Fig. 9, it is also shown that sticky rice has a favorable concentration at 3% for specimen consolidation, and this characteristic is very similar to that of styrene–butadiene (7.5%) used in mortars [25]. The results indicate that the modeling samples not only possess the similar microstructure as that of the historical samples, but also can enhance the

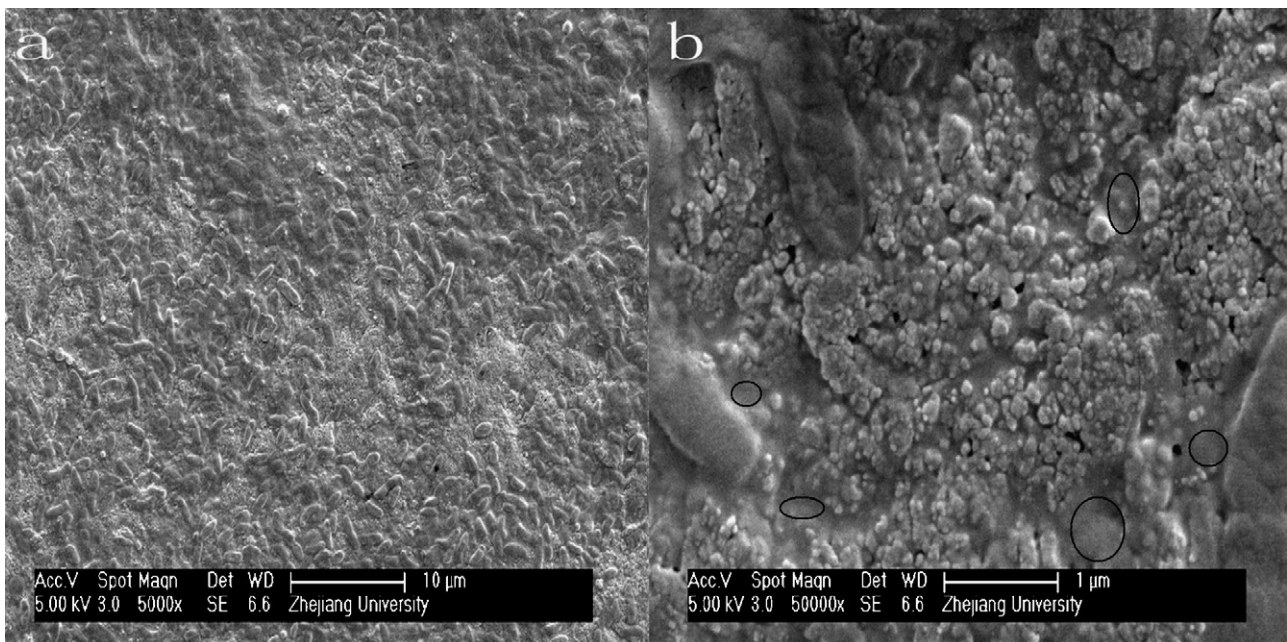


Fig. 8. SEM image of the sample MPS prepared by the surface method: (a) 5000 \times and (b) 50,000 \times . Sample MPS: the white marble surface covered with 0.01 mol l^{-1} calcium hydroxide solution and 3% sticky rice solution mixed with the same volumes.

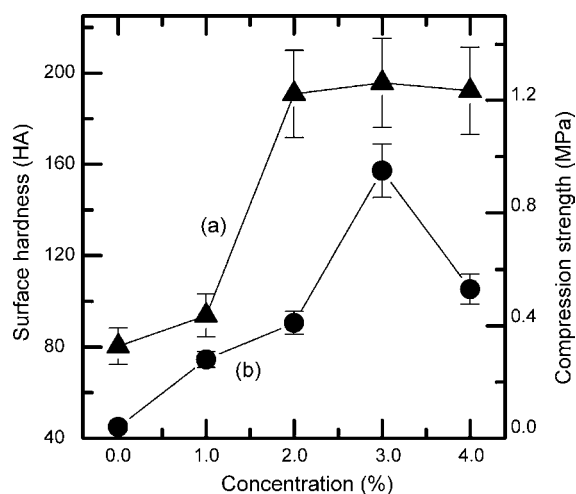


Fig. 9. Strength test results of different concentrations sticky rice consolidated specimens: (a) surface hardness; (b) compression strength.

strength of the loose samples, like the ancient mortars do. In addition, the consolidated samples were immersed into water to test their water resistance abilities. It is discovered that the shape of the consolidated loose samples keeps intact within 2 months in water while that of the blank samples just collapse at once when submerged by water. It conveys that the modeling formulas are helpful to resist water corrosions.

4. Conclusions

The XRD, SEM, FTIR and DSC–TG analyses can help to draw a conclusion that the historical samples from Dutifulness Monument are mainly consisted of 70% calcium carbonate and 15% organic materials, and the organic materials contain sticky rice because red brown amylopectin-complex has been found in iodine–starch test. It is consistent with the historical record of traditional mortars, which normally are the mixtures of products of calcium hydroxide carbonation and sticky rice. Further analysis will be helpful to test this consistence if more samples can be gained in the next repair of the monument for limitation of sample numbers in the work. The same analysis techniques also were conducted on the modeling samples prepared by both the solution method and the surface method. The results indicate that the morphology of the mortars can be controlled through adjusting the volume of the sticky rice solution added in the calcium hydrogen solution. The microstructure of the modeling samples is very alike to that of traditional samples when the volume ratio of the added 3% sticky rice solution and the 0.01 mol l^{-1} calcium hydroxide is 1:1. The characteristics of these processes can often be found during biomineralization researches, where the morphology of inorganic crystals is controlled by organic additives. The consolidation tests suggest that the modeling formulas also can be used to improve the strength of loose samples and the water resistance ability of the consolidated samples is much better than the untreated samples. The investigations on both the microstructure and the application of the mortars in this article could be helpful to understand the properties of ancient China mortar formulas and to investigate the

proper usages of the mortars in loose historical stone consolidations.

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