New insight into the craftsmanship of sucrose-modified rammed earth-lime materials

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ABSTRACT
In this work, the performance and mechanism of a Chinese traditional sucrose-modified rammed earth-lime were discussed in lab for its potential utilization in the restoration of rammed earth-lime architectural heritages. The results showed that the addition of sucrose would effectively improve compressive strength and wet-dry resistance of rammed earth-lime. The addition of sucrose would markedly increase the concentration of C₅O₃ in pore solution and prolong the duration of high temperature, thereby making the microstructures of the rammed earth-lime much more compact and improve the formation of C-S-H gel. These may be the reasons for the better performance of sucrose-modified rammed earth-lime. In general, the optimal amount of added sucrose was about 1–3 % by weight of the total mortar.

1. Introduction
In ancient China, rammed earth-lime was widely applied to build city walls, fort barbettes, tombs, residences, etc (Zheng et al. 2016). Some of these constructs are still very solid and can be used, represented by the UNESCO world cultural heritage “earthen buildings” in Fujian Province (Figure 1). In ancient times, these earthen structures in round or square shapes functioned as fortresses against foreign enemies. The exterior of these earth buildings is commonly built with stones as the base and rammed earth-lime as walls. Inside the rammed earth-lime walls, the whole building is divided up to hundreds of small rooms by traditional Chinese wooden structures. According to legend, in order to strengthen the external rammed earth-lime wall, ancient Chinese added sticky-rice pulp and brown sugar to the raw earth-lime materials (Zhang et al. 2014). Although these legends have not been confirmed by existing literature or modern scientific analysis, the use of sucrose in ramming the floor in the earth buildings has been confirmed by the owner of Yude building (a private house). This floor is very hard and flat that can be used for many years, and needs a hammer to pry it open (Figure 1c).

Comparing with common organic-modified lime mortars, the preparation of sucrose-modified rammed earth-lime material is different. According to generations of craftsmen, the earth, quicklime, sucrose and the necessary water for hydration of lime should mixed simultaneously, i.e. “hot hydration” at first. Then, the mixtures are maintained for a period of time to make the quicklime completely hydrate before application. Parracha et al. (2020) and Zhang et al. (2020) analyzed the ancient rammed earth-lime materials in Portugal and China respectively and found “hot hydration” producing an improved bond between the earth-lime matrix. And Eires, Camoes, and Jalali (2017) also drew the similar results through simulation experiments of oil-modified earth-lime material. Rodriguez-Navarro et al. (2017) studied the slaking process of quicklime in aqueous nopal juice extract or pectin. Their results showed that these two additives had significant effects on the size, habit (shape), and colloidal stability of Ca(OH)₂ crystals in lime putties prepared following slaking of quicklime, thereby contribute to the high quality and durability of the final lime mortars and plasters. Moreover, Gamrani et al. (2012) studied the properties of rammed earth of a sugar refinery in Morocco which contained organic matter and had greater mortar strength compared to that of a normal rammed earth. The authors assumed that it was related to the craftsmanship, unfortunately, they didn’t analyze it in detail.

Although there are some relevant researches, the preparation process of this kind of Chinese sucrose-modified rammed earth-lime material lacks detailed description, and its underlying principle is unclear, which restricts the use of this kind of material in the protection and restoration of rammed earth-lime constructs in China. Moreover, today, the pollution and energy consumption caused by the production of building materials has seriously affected the sustainable development of society. Comparing with modern
cement mortar, earth-lime materials have better economic and environmental benefits that could potentially fulfil the need for an alternative building material. Thus, in this paper, we intend to understand the special technology, properties and mechanism of this sucrose-modified rammed earth-lime material in lab. And through this work, we hope to provide more insights for the production of rammed earth-lime as an alternative material for both rammed earth-lime architectural heritages and modern constructions.

2. Experimental design

2.1. Raw materials

Earth was gathered from the hill near Zhejiang University (Yuquan campus), which was first sieved to remove plant fibers and rock fragments, then washed with water to deprive soluble salts. After air-drying for 20 days, the earth was crushed and sieved again, and the fraction of particles under 2 mm was used for sample preparation. The composition of the treated earth was listed in Figure 2. It is particularly necessary to point out that this kind of earth is a typical yellow earth widely distributed in southeast China which contains little clay.

Quicklime lumps, hydrated lime powder and sucrose, all in analytical grade were manufactured by Sinopharm Chemical Reagent Co., Ltd. The quicklime lumps were crushed into small particles (<1 mm) before application.

2.2. Earth-lime mixture during slaking process

2.2.1. Temperature variation

80 g treated earth and 20 g crushed quicklime was stirred with 24 ml sucrose solution (0, 1, 3, 5, 10 \%w) for

![Figure 1. The pictures of earthen buildings in Fujian Province, China. (a) the external face of Yuchang (AD1368-1644); (b) the internal face of Chengqi (AD1628-1709); (c) the flooring of Yude (private house, more than 100 years old)](image)

![Figure 2. The composition of the treated earth (a) XRD pattern; (b) EDS result.)](image)
2 minutes, transferred to insulated cup and gently compacted, rapidly. Then an external probe of temperature recorder (Pingyang County Miaoqun Technology Co., Ltd. (type T10R-PT)) was inserted into the center of the earth-lime mixture and measured the temperature variation continuously.

2.3.2. Compositions in bulk of earth-lime mixture
The mineral phases in bulk of earth-lime mixture at different stages were measured by X-ray power diffraction (Ultima IV, Japan). The process as follows: first, 80 g treated earth and 20 g crushed quicklime was stirred with 24 ml sucrose solution (0, 5, 10 %w) for 2 minutes. Second, 1 g mixture was taken out at 0, 15, 30 minutes after stirring and dried at 60 °C for 2 h, respectively. Then, the dried samples were grounded into powder and tested by XRD quickly.

2.3.3. Ions in pore solution of earth-lime mixture
pH and calcium ion in pore solution of earth-lime mixture were analyzed by ion chromatography (Dionex, ICS-2100) and pH meter (PHS-25, INESA Scientific Instrument Co., Ltd). Inside a magnetic stirrer, 0.1 g Ca(OH)2 and 100 ml of a certain concentration of sucrose solution (the sucrose-Ca(OH)2 ratios of 0, 1, 3, 5, 10 w %) was well-mixed together, then left to react for 1 h under constant stirring. After filtering, the Ca2+ concentration and pH value of filtrate were measured.

2.3. Properties and microstructures of rammed earth-lime mixture

2.3.1. Sample processing
First, 400 g treated earth and 100 g crushed quicklime were mixed thoroughly, and 120 ml sucrose solution (0, 1, 3, 5, 10 %w) was added into the above mixture while stirring. Second, the prepared materials were stored in airtight containers for 20 d. Third, the above materials were rammed into cubic blocks (20 × 20 × 20 mm3) using a homemade compaction device. Each sample was rammed 8 times with the same ramming force (500 g hammer weight, 40 cm landing height). These samples were cured in lab condition (T = (22 ± 3)°C, RH = (80 ± 5) %) before mechanical properties and weather resistance tests.

2.3.2. Property tests
2.3.2.1. Compressive strength. Compressive strength test of different rammed earth specimens was performed on an electronic universal testing machine (CMT 5205) after 28 and 60 d of curing. For each test, 3 specimens were tested.

2.3.2.2. Wet-dry resistance. Specimens were subjected to wet-dry cycles to accelerate the deterioration process after 28 d. For each cycle, the specimens were first soaked in water for 24 h, then taken out, wiped dry, and placed in drying oven (60 °C) for 4 h. After 10 cycles, their compressive strengths were measured.

2.3.2.3. Water absorption. The water absorption of different specimens was performed according to BS-EN 1925 (2001).

2.3.2.4. Degree of carbonation. The specimens were cut along the center line to reveal the cross-section, which was then cleaned by ear ball and brush. Phenolphthalein reagent was then applied onto the cross section and photographed. The ratio of colored red area was calculated by ipwin32 software. The degree of carbonization of different specimens was expressed as (A0-A)/A0 × 100 % (A0: total sectional area, A: red area).

2.3.3. Microstructure
The microstructures of the rammed samples were observed by scanning electron microscopy (SEM, FEI SIRION100). First, a fresh section was prepared, then the sample was coated with gold before observation.

3. Results and discussions
3.1. Properties and compositions of earth-lime mixture during slaking process
3.1.1. Temperature variation
According to the temperature curves in Figure 3, the earth-lime mixture without sucrose had the highest slaking rate of all tested samples. Its temperature increased rapidly with the addition of water and already exceeded 100 °C during stirring process, but only 2.5 min after mixing, the temperature dropped below 100 °C. The samples with 1, 3, 5 and 10 % sucrose reached 100 °C about 25, 14, 10 and 6 minutes after stirring, and maintained above such high temperature for 9, 7, 6 and 5 min, respectively. Moreover, for samples with 0, 1, 3, 5 and 10 % sucrose, the temperatures inside the earth-lime mixture maintained above 50 °C for 58, 97, 111, 107 and 114 min, respectively. These results showed that although the temperature of the sample without sucrose increased quickly, its high temperatures lasted the least amount of time. For the samples with sucrose, the duration of temperature above 50 °C increased along with sucrose content, but the duration of temperature above 100 °C decreased as sucrose content increased. At last, although the time needed to reach the highest temperature and the duration of high temperature were different in these five samples, their maximum temperature was basically the same, at 104–106 °C. It was reported by Ciancio, Beckett, and Carraro (2014) that higher temperatures and longer duration inside the earth-lime materials were favorable for the formation of C-S-H gel which could improve mortar properties.


3.1.2. Compositions in bulk of earth-lime mixture

The rates of quicklime slaking in the samples with 0, 5 and 10 % sucrose were calculated by XRD semi-quantitatively. The results of the time evolution of the portlandite/quartz ratio (Figure 4) demonstrated that the hydration rate of the sample without sucrose was always the lowest. The value of portlandite/quartz went from 12.4 % to 15.5 % within 30 minutes after mixing. For the samples with sucrose, the values of portlandite/quartz of the sample with 5 % sucrose were lower than that of the sample with 10 % sucrose within 15 minutes after mixing. But this situation reversed at 30 minutes, when the value of portlandite/quartz of the sample with 5 % sucrose was 27.3 % and it was 1.25 times more than that of sample with 10 % sucrose. It is well known that sucrose is a water reducer for the cement and lime mortar (Fang et al. 2015). Consequently, the quicklime in the mixture with sucrose had more contact with water and facilitated hydration within a certain period. However, on the other hand, sucrose is an inhibitor of quicklime hydration (Banfil 1986; Rodriguez-Navarro et al. 2002). Thus, with the increase of sucrose, its inhibitory effect on hydration of quicklime was also enhanced. Thus, the slaking and heat release rate of the earth-lime mixture (Figure 3) was regulated by the inhibition of quicklime hydration and water reducing effect by sucrose.

3.1.3. pH and calcium ion in the pore solution of earth-lime mixture

Figure 5 showed that the pH values of the lime-saturated water with 0, 1, 3, 5 and 10 % sucrose were 12.32, 12.37, 12.33, 12.35 and 12.34, respectively. Obviously, it seems that the sucrose amount has little effect on the pH, i.e. OH⁻, in the pore solution. On the other hand, Figure 7 showed that calcium ions present in the solution almost linearly increased with the increase of sucrose, probably due to the formation of more calcium sucrose due to lime-sucrose reaction. The Ca²⁺ concentration started at 0.8916 g/L (without sucrose), and the slope of the curve was 0.0197 g/L. The increase of calcium ion dissolution might promote the ion exchange between lime and earth, thereby enhancing the mechanical property of earth-lime material (Ciaccio, Beckett, and Carraro 2014). Chatterji (1979), Chatterji and Claussenkaas (1984) regarded that the high calcium ion concentration in pore solution could prevent the diffusion of active silicon in the aggregate during alkali-silicon reaction, and promote the formation of alkali-silicate gel. Du et al. (2019) found that the addition of Ca in solution tended to

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**Figure 3.** Results of the time (t)-evolution of temperature (T) of earth-lime with different sucrose contents under the condition of water deficiency.

**Figure 4.** The results of the time (t)-evolution of the portlandite/quartz of earth-lime with different sucrose content under the condition of water deficiency.
increase the initial polymerization kinetics of the C-S-H gel although it had a negative effect on the final amount of C-S-H gel. This means that the addition of sucrose might promote the formation of C-S-H gel, consequently affect the performances of this material.

3.1.4. Simulated hydrothermal reaction
The above experimental analysis indicated that the addition of appropriate amount of sucrose could facilitate the formation of C-S-H gel through “hot hydration”. This is one factor that affects the properties of earth-lime materials. Here, we explore this possibility in the pore solution of earth-lime materials through simulated hydrothermal reaction.

The hydrothermal reaction was divided into two groups: group A, 1 g earth, 1 g CaO and 40 ml water; group B, 0.1 g sucrose, 1 g earth, 1 g CaO and 40 ml water. The volume of the hydrothermal reactor was 100 ml and the reaction temperature was 105 °C. After a certain reaction time, an appropriate amount of solid was taken out for XRD and SEM analysis.

Figure 6(a) displayed that the main mineral phases in the earth-lime mixture without sucrose were portlandite, quartz and calcite during the whole hydrothermal reaction experiment. Among them, portlandite was a product of the hydration of quicklime, quartz was the principal constituent of earth, and calcite was formed by carbonation of portlandite. Comparing with the sample without sucrose, in addition to the above phases, calcium oxide was observed in the initial sample with 5 % sucrose of the hydrothermal reaction (Figure 6(b)). As mentioned above, this is caused by the inhibition of sucrose on the hydration of calcium oxide. The enlarged spectrums in Figure 8 showed that two peaks (29.2° and 32.6°, respectively) started to appear in the spectrums of the sample without sucrose after 8 h of hydrothermal reaction, which can be attributed to C-S-H gel (Hu et al. 2018). Moreover, these peaks became more and more obvious as the reaction time increased, showing that the earth could react with lime at 105 °C, and the continuous high temperature caused by addition of sucrose was helpful for the formation of C-S-H gel. Comparing with the sample without sucrose, there was an advance in the appearance of these two peaks in the sample with 5 % sucrose. They started to appear in the spectrums of the sample with 5 % sucrose after 3 h of hydrothermal reaction. These results confirmed that an increase of Ca²⁺ in the solution could help in the formation of C-S-H gel.

The SEM images (Figure 7(a)) demonstrated that there were many dumbbell-shaped and hexagonal structures in the picture of the sample without sucrose after 3 h of hydrothermal reaction. These were the characteristic structures of aragonite (Chavagnac et al., 2013) and calcium hydroxide (Mascolo et al. 2010), respectively. As the reaction time increased, the structure of the materials in this sample changed gradually. After 12 h of hydrothermal reaction, several needle-like structures appeared in the bulk. This was the characteristic structure of calcium silicate (Shi et al. 2019). The SEM pictures of the sample with 5 % sucrose (Figure 7(b)) showed that the needle-like calcium silicate appeared after 3 h of hydrothermal reaction till 8 h. However, after 12 h of hydrothermal reaction, there were a lot of honeycomb structures, which should also be calcium silicate gel (Garcia et al. 2009; Chen et al. 2019). This means that with the increase of hydrothermal reaction time, the amount of calcium silicate gels not only increased, their structures were also changing in the sample with 5 % sucrose. Moreover, the large flake structure in the picture of this sample after 3 h of hydrothermal reaction was the crust of calcium sucrose.

3.2. Properties and microstructures of rammed earth-lime
3.2.1. Compressive strength
The compressive strength of the specimens without sucrose reached 3.40 and 3.80 MPa after curing for 28 and 60 d in lab condition, respectively (Figure 8). With
the sucrose content below 5%, the compressive strength increased with the sucrose content. The specimens with 5% sucrose had the maximum compressive strengths at 28 and 60 d (4.86 and 5.47 MPa). However, the compressive strength decreased rapidly when the sucrose content reached 10%, with values of 2.90 and 2.60 MPa at 28 and 60 d, respectively. Comparing the compressive strength values at 28 and 60 d, the

Figure 6. The XRD patterns of earth-lime mixtures ((a) without sucrose and (b) with 5% sucrose) during the hydrothermal reaction.

Figure 7. The SEM pictures of earth-lime mixtures ((a) without sucrose and (b) with 5% sucrose) during the hydrothermal reaction.
specimens with 1% sucrose had the maximum increase (31%), yet with 10% sucrose addition there was a negative growth. For the remaining groups, the percentage changes in compressive strengths from 28 to 60 d were 12–18%.

3.2.2. Wet-dry resistance
The wet-dry cycle test results (Figure 8) showed that the compressive strength of the specimens without sucrose declined from 3.40 to 2.03 MPa after 10 wet-dry cycles. Similarly, the compressive strength of the specimens with sucrose also decreased to varying degrees 10 wet-dry cycles, and as the sucrose content increased, the compressive strength decreased. Among them, the compressive strength of the specimens containing 1 and 3% sucrose (2.33 and 2.24 MPa) exceeded the compressive strength of the specimens without sucrose (2.03 MPa). However, if the reduction percentage in compressive strength before and after the wet-dry cycle was compared, only the specimens containing 1% sucrose (reduced 28%) was lower than the specimens without sucrose (reduced 40%). Thus, the wet-dry cycle test showed that the specimens with 1% sucrose had the best wet-dry resistance, and above 3% sucrose addition in the rammed earth-lime material, the resistance is drastically reduced.

3.2.3. Water absorption
The water absorption test (Figure 9) showed that the trend of all curves was similar: the absorbed water in the specimens increased rapidly in the first 30 minutes, then became slow and tended to stabilize. However, there were also some differences in these curves. First, when the sucrose content reached 10%, the water absorption of the specimen decreased significantly. By the end of the experiment, it absorbed only about 75% of the water of the other groups. Second, the inserted image displayed that although the water absorption increased within the first few minutes, it then gradually decreased as the increase of sucrose content. This is related to the microstructure of these specimens (see Figure 11).

3.2.4. Carbonation degree
The results of carbonization test (Figure 10) showed that as the sucrose content increased, the 28 d carbonation degree of the rammed earth-lime specimens decreased. After phenolphthalein reagent spraying, the percentages of red area were 59, 56, 66, 78 and 77% when the sucrose content went from 0 to 10%. After 60 d, the coloration of the specimens was much lighter than that of the specimens cured for 28 d. Moreover, the percentage of the areas that turned red decreased to 29, 57, 52, 51 and 33%. In general, for rammed earth-lime specimens with less than 5% sucrose content, the carbonation degree was lower than specimens without sucrose. However, the compressive strength of these specimens (with less than 5% sucrose content) was higher than that of the specimens without sucrose. This means that the compressive strength of these rammed earth-lime specimens was affected by other factors besides lime carbonation. Moreover, there was an obvious Liesegang pattern on the section of the specimen with 1% sucrose. This phenomenon also was found in the lime mortar prepared by aged slaked lime (Rodriguez-Navarro et al. 2002), which may affect the properties of cured lime mortar (Wei et al. 2012).

3.2.5. Microstructure of rammed earth-lime specimens
When observing low magnification SEM images (Figure 11- A1, B1, C1, D1, E1), all specimens were consisted of granular structures with different sizes, and no significant differences among these five specimens can be noted. However, images with high magnification (Figure 11- A2, B2, C2, D2, E2) displayed that the microstructures of rammed earth-lime specimens changed with the sucrose content. Figure 11-A2 showed that the fine particles in the cross section of the specimen without sucrose were loosely bound together and distributed with many pores. As the sucrose content increased from 1 to 10% (Figure 11- B2, C2, D2, E2), the number of pores gradually reduced, and the size of the particles distributed on the cross-section gradually increased and adhered to each other tightly, thereby making the structure become compact. The differences in the microstructure could help to explain the effect of the addition of sucrose on the water absorption and strength of rammed earth-lime specimens.

4. Conclusions
In this paper, an ancient Oriental technology of sucrose modified rammed earth-lime was studied in order to
explore its application in the restoration of rammed earth-lime architectural heritages. Through the performance test, material composition and microstructure analysis of the simulated rammed earth-lime in the lab, the following conclusions can be made:

(1) The properties and compositions of earth-lime mixture during slaking process showed that the addition of sucrose would prolong the duration of high temperature in earth-lime mixture and increase the concentration of Ca\(^{2+}\) in the pore solution. The simulated hydrothermal reaction confirmed that these changes would facilitate the formation of C-S-H gel, thereby affecting the performance of rammed earth-lime.

(2) The tests of compressive strength and wet-dry resistance of the rammed earth-lime specimens showed that the addition of a small amount of sucrose had a positive effect on the improvement of these two properties, but too much sucrose had an negative effect. The carbonation test showed that the carbonation speed of the specimens with 1–5 % sucrose was relatively slower. This means that comparing with the specimens without sucrose and with 10 % sucrose, these specimens had more strength growth space. According to the above analysis, the optimal amount of added sucrose was about 1–3 %.

(3) The SEM results demonstrated that the microstructures of the rammed earth-lime specimens would become more and more compact with the increase of sucrose content. This may be an important factor to reduce water absorption and increase mortar strength of modified rammed earth-lime.

Figure 9. Water absorption of different rammed earth-lime specimens cured for 28 d.

Figure 10. The carbonation degree of different rammed earth-lime specimens maintained 28 d and 60 d.
Figure 11. The SEM pictures of rammed earth-lime specimens with different content of sucrose (A1 and A2. without sucrose (×5000, ×30000); B1 and B2. with 1 % sucrose (×5000, ×30000); C1 and C2. with 3 % sucrose (×5000, ×30000); D1 and D2. with 5 % sucrose (×5000, ×30000); E1 and E2. with 10 % sucrose (×5000, ×30000)).

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