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Original article

The identification of organic additives in traditional lime mortar

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ABSTRACT

Organic-inorganic lime mortars were widely used in many ancient buildings due to their good performance in some fields (such as caking property, water repellency, weatherability, etc.). However, many ancient buildings and sites are suffering from various degrees of damage with the development of the economy and society and appropriate conservation and restoration are needed. The application of traditional construction materials, such as organic-inorganic lime mortars, attracts more and more attention in the conservation and restoration of ancient buildings in the recent years. So, the understanding of the components of original lime mortar which remained in ancient sites is of fundamental significance. In this work a set of analytical procedures to identify the organic additives in lime mortars by classical chemical analysis is proposed. The results show that using iodine-potassium iodide reagent, Benedict's reagent, reduction phenolphthalein reagent, Coomassie brilliant blue and sodium periodate oxidation glycerin acetyl acetone method could effectively detect a small amount of starch, reducing sugar, blood, protein and fatty acid ester that remained in ancient buildings' lime mortars, respectively. These analytical methods are easy to operate with low detection limit, high accuracy and some other advantages.

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1. Introduction and research aims

During the long history, lots of architectural sites were remained. They reflected our ancestor's wisdom and provided valuable materials to study our history for later generations. However, a lot of historical buildings and sites are facing huge threat because of the explosion of modern industrial and traffic activities, man-made destruction and increasing tourism pressure. Therefore, protecting these precious cultural relics is urgent.

Cement paste as modern building material has been used for the restoration of ancient architecture in the past few decades. But the disadvantages of cement, such as too high strength, low porosity, poor compatibility with ancient buildings and the introduction of soluble salts, have been revealed by conservation scientists [1–3]. However, lime mortars, the traditional construction cementing materials had been widely applied around the world. Today, lots of lime mortars which were used in ancient sites are still working. For instance, hydraulic mortar prepared by lime with ash, clay and brick powder [4,5], organic-inorganic lime mortar prepared by lime with sticky-rice, blood, juice of plants, drying oil or egg white [6,7], and reinforced mortar prepared by lime with straw and fiber [8]. Different lime mortars have different compositions, ratios, applications and properties. So, a reliable detection method could help us deeply understand the materials used in different construction sites, and also could provide a guide to select appropriate repair materials.

As far as we know, the studies of inorganic additives in lime mortars are abundant [4,5,9–11], while the research about organic-inorganic lime mortar is scarce. At present, the analytical methods for organic additives in mortars mostly rely on instrument analysis and biological analysis. However, the instrumental analysis may not be feasible for the in situ analysis of mortar in ancient sites because of the limitation of instruments, such as their requirement of professional and complicated analysis, high costs, etc. Even the portable instruments may not be able to detect the tiny amount of additives in complicated mortar mixtures. But a reliable analytical result about the information of additives in mortar is always needed as soon as possible in archaeological field work for the consideration of in situ handling and future conservation. So, a simple and effective method to analyze ancient mortars is important to study and understand the original mortar's content and structure in ancient architectures.

In this work, a simple and effective method to analyze the organic additives in ancient building lime mortars is proposed using classical chemical analysis. This method, combining with other analysis means, could provide the original information about the ancient sites for future conservation and restoration.

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2. Experimental methods

2.1. Reagents

Iodine-potassium iodide reagent: 3 g potassium iodide dissolving in 100 ml distilled water. And add 1 g iodine after the potassium iodide was completely dissolved. This reagent was kept in brown bottle before test.

Benedict's reagent: dissolve 85 g sodium citrate and 50 g anhydrous sodium carbonate in 400 ml distilled water. This solution was marked as reagent 1. Weigh 8.5 g anhydrous cupric sulfate and dissolve into 50 ml hot distilled water. And mark this solution as reagent 2. Add reagent 2 into reagent 1 and stir. Then keep it in bottle after filtered.

Coomassie brilliant blue reagent: weigh 100 mg Coomassie brilliant blue G250 and dissolve in 50 ml 95% ethanol. Add 100 ml 85% phosphoric acid and distilled water to reach 1000 ml. This solution was kept under 4 °C after filtered.

Reducing phenolphthalein reagent: add 100 ml 20% KOH solution, 2 g phenolphthalein and 1 g zinc into 250 ml round bottom flask. Heat the solution until colorless. Add some zinc powder into the colorless solution and keep in brown bottle.

Takayama's reagent: weigh 3 g glucose, 3 ml pyridine, 3 ml 10% NaOH solution and 7 ml distilled water and mix them completely. The solution should be stored for 24 hours before test.

Sodium periodate solution: weigh 1.065 g sodium periodate, 4.8 g and 15.4 g acetic acid ammonium acetate, then add water to reach 100 ml. The concentration of sodium periodate solution is 0.05 mol/L.

Acetyl acetone solution: add 4 ml acetyl acetone into 96 ml isopropyl alcohol and mix them completely.

2.2. Preparation of simulated mortar samples

The preparation of simulated mortar samples refers to the production technology of Chinese traditional mortar [7,12–14].

2.2.1. Sugar lime mortar and egg white lime mortar

Weigh certain amount of brown sugar (or egg white) and make different concentrations of solutions (0.1, 0.25, 0.5, 1.0, 2.0, 4.0, 6.0% (w/w)). Then mix the solution with $Ca(OH)_2$ completely (the ratio of $H_2O/Ca(OH)_2$ is 0.8) and daub the mortar on the glass plate. The samples were stored in laboratory for 3 months before test.

2.2.2. Sticky-rice lime mortar

Weigh 4 g sticky-rice and 94 g distilled water, and boil for 4 hours. During the boiling process add sufficient water to keep the ratio of sticky-rice/water unchanged. The concentration of this sticky-rice slurry is 6%. Use the same method to prepare other sticky-rice slurry with different ratio (0.1, 0.25, 0.5, 1.0, 2.0, 4.0%). Mix the aforesaid sticky-rice slurry with $Ca(OH)_2$ (the ratio of $H_2O/Ca(OH)_2$ is 0.8) and daub the mortar on the glass plate. The samples were stored in laboratory for 3 months before test.

2.2.3. Tung-oil lime mortar

Add certain amount of tung-oil and $Ca(OH)_2$ into iron container. Then grind the mortar repeatedly until the mortar become fine and smooth. The samples should also be stored in laboratory for 3 months before test.

2.2.4. Blood lime mortar

Squeeze the fresh animal blood clot with the help of gauze. Then mix the blood with 17% lime water (the ratio of blood and lime water is 10:7). Place the mixed solution in water bath (30 °C) for 3 hours. At last, mix the above products with appropriate $Ca(OH)_2$ completely. This kind of mortar is called blood lime mortar. The mortar was daubed on the glass plate and stored in laboratory for 3 months before test.

2.3. Methodology

In this paper, the classical chemical analytical methods were selected to detect the organic additives in lime mortar.

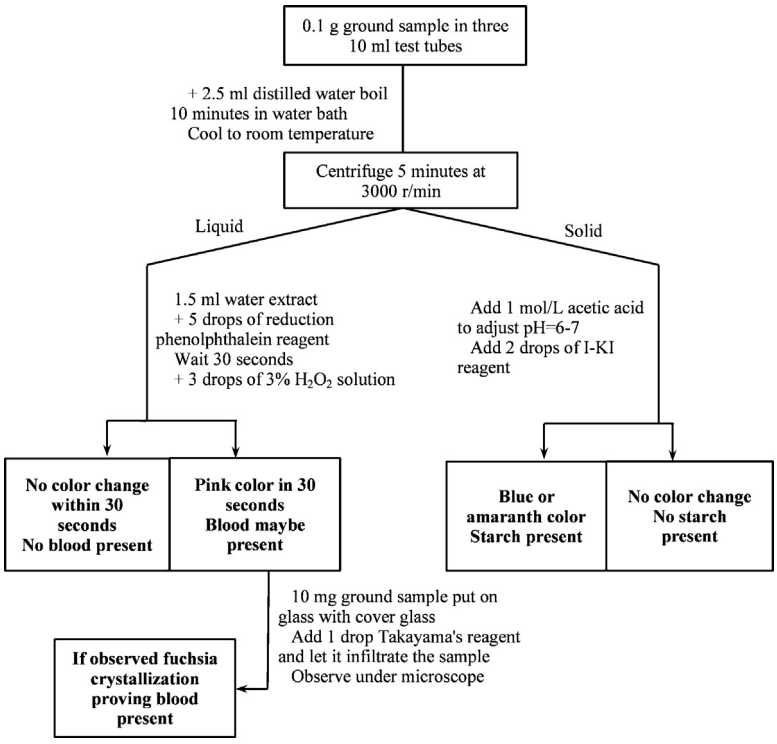

Fig. 1. The analytical flow diagram of blood and starch in lime mortar.

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