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Effect of hydrophobization treatment on the hydration of repair Roman cement mortars

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HIGHLIGHTS

- ▶ Influence of several water-repellent treatments on the hydration process Roman cement repair mortars.
- ▶ The surface treatment prevented water absorption and the hydration process.
- ▶ For inner hydrophobization agents, the hydration process progressed.
- ▶ Durable and well-hydrated repairs. Improve in the freezing and thawing resistance.

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ABSTRACT

Hydrophobization agents are commonly used in conservation practice to enhance the water-repellent properties of stone and masonry structures such as bricks and restoration mortars. In this project, we have tested and analyzed the effects of various commercially-used water repellents on the hydration process of Roman cement mortars. The necessity to investigate the progress of hydration of these materials in the presence of water-proofing agents arises from the well known fact that Roman cement mortars require adequate times and conditions for curing, which gives the restoration material compatibility with the original substrates. The effects of hydrophobic treatment on the pore size distribution and some physical features, such as water absorption and capillary rise, were investigated on Roman cement mortars treated with polymer-based coatings and inner waterproofing agents. The behavior of mortars in terms of their freeze resistance was also evaluated. The surface treatment using a hydrophobization agent completely prevented water absorption and thereby interrupted the hydration process. In the case of inner water-repellent admixtures, the hydration process progressed in spite of the decrease in water capillary transportation. In such circumstances, unimodal distribution of pore sizes was observed along with a decrease in threshold pore width with increased curing time.

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1. Introduction

Water is one of the most important factors that contributes to the decay of porous building materials such as mortars, stones, or bricks. Water penetrates into the pores of these materials by capillary force and induces several deterioration effects such as chemical dissolution and transportation of soluble compounds, e.g. the calcium carbonate component of stone and mortars, or the introduction of water soluble salts as well as freezing–thawing processes. Thus, the use of various water-repellent products to minimize the rate of decay in porous materials has been widely discussed in recent years [1–4]. The main function of a waterproofing system is to reduce water absorption and penetration into the pore structure of the object. At the same time, they should make the diffusion of water vapor through the structure possible allowing moisture to evaporate. This transfer is a particularly essential requirement when dealing with the conservation of historic buildings. A recent conservation trend is to apply water repellents after any repairing procedure since the new unprotected surfaces are exposed to the weathering mechanisms and accelerate the decay processes.

Recently, there has been increased scientific interest in the study of water repellents for lime- or cement-based mortars [5–7]. However, in the context of porous building materials that should be potentially protected by water repellents, Roman cement mortars have never been discussed. These highly hydraulic binders, known as natural or Roman cements, were key materials for an economical and simple way of manufacturing stuccoes for building exteriors during the nineteenth and early twentieth

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centuries. They were produced by burning naturally occurring deposits of calcium carbonate rich in clay minerals and then grinding the burnt stone to the required fineness. These materials offered a high speed of set, a beautiful texture, and a warm ochre color. Another advantage was their capability of withstanding exterior conditions very effectively. The production and use of Roman cements declined in the first half of the twentieth century with the dominance of the newer Portland cement and the emergence of modern functional architecture. Traditionally, architectural conservation in the late nineteenth and early twentieth centuries has not received the same consideration as other historical periods. This is mainly due to the lack of binder constituents of mortars that would closely match those of the original structure. It is only relatively recently, with the growing interest in European art of the period, that efforts have been undertaken to investigate the use and properties of these historic renders across Europe [8–10]. The ROCEM project, supported by the European Commission 5th Framework Programme, has recently re-established their manufacturing and use in conservation practice [11].

Earlier studies [12,13] have already highlighted that when properly cured, Roman cement repair mortars attain chemical, physical and structural compatibility with the original ones. In general, the compatibility can be broadly defined as the capacity of the repair mortar to interact with the original historic substrate without inducing any decay. One of the main properties responsible for the compatibility is the pore structure of the mortars as it greatly influences water transport, water vapor permeability and strength. In the case of Roman cement mortars, the pore structure strongly depends on the progress of hydration.

The key concept of this research was to investigate the effect of water-repellent agents on the hydration process of repair Roman cement mortars. Two water-repellent treatments were carried out: surface application of a protection layer onto the mortar surface and modification of the hydrophobic properties inside the material. Moreover, four types of commercial water-repellent products were tested and their effects on the repair of Roman cement mortar were determined. In order to check the improvement of Roman cement mortars several properties were evaluated: pore structure through mercury intrusion porosimetry, hydric parameters, and durability assessed by means of freeze–thaw cycles.

2. Materials and methods

2.1. Materials

The formulation of the restoration mortars used in this study replicated some representative groups of historic mortars. Three sets of repair mortars were prepared from Folwark Roman cement (Poland) by mixing the cement with aggregate (a/c = 0.5 by weight) and water (w/c = 0.6). Quartz sand (d = 0.25–0.5 mm) was used as the aggregate. Due to the quick setting of the cement, citric acid was added as a retarder at 0.3 wt.% (relative to dry cement). Mortars were cast into silicone molds as prismatic specimens of 8 × 2 × 2 cm or laid on fired-clay brick substrates with dimensions of 4 × 4 × 2 cm. The materials were then de-molded after setting and stored in humid air at 100% relative humidity (RH) conditions for 3 months to ensure wet curing conditions. The influence of different curing conditions on hydration was previously described [14]. The lack of moisture in the external conditions may result in restricted hydration and affect the microstructure and strength of the mortars. In real world conditions, ideal wet-air curing of mortars is rarely possible in the course of practical work on the façade.

The characteristics of the original marl feedstock, the calcinations conditions, the oxide and mineralogical compositions of the cements, as well as their strength and porosity development in the progress of hydration have already been reported [15].

The first set of mortars was used as reference samples; the remaining samples were treated with water repellents on the surface and on the bulk. For the surface treatment, the following experimental procedure was adopted to simulate the situation occurring on a building's facade during restoration work when freshly applied mortars are treated with water repellents. First, the Roman cement mortar was applied on a brick (WAC = 0.08). After one day of hydration under wet-air curing conditions, the sandwich was dried to stop hydration and to remove water from the mortar–pore system. The specimen's edges were covered with a sealant and the compact surface layer of the mortar was removed by abrasion. The surface was treated with Funcosil® WS water-repellent emulsion, which was applied to the mortar by brush. Immediately after being thoroughly soaked (0.5 l/m^2), the specimen was stored under a 100% RH environment.

Roman cement mortars with internal hydrophobization were prepared as follows. Aida Porenmörtel-Konzentrat (Remmers) and Silres® BS 1306 (Wacker) emulsions were added to the water in 1:20 and 1:9 ratios by volume, respectively. Another preparation method involved the use of 0.5 wt.% Silres® BS Powder A (Wacker) mixed with cement. The composition, codename and way of application of the four selected products are shown in Table 1.

2.2. Methods

The pore structure of the mortar samples was determined using a Poremaster mercury intrusion porosimeter (quantachrome), which allows studying pore sizes in the range of 440–0.0035 μm. After the predetermined curing period, the specimens were immediately soaked in acetone for 24 h to stop the hydration of the cementitious materials [16,17]. Afterwards, they were placed in a rotary vacuum evaporator flask at 20 °C for 4 h to remove the acetone and finally were allowed to dry.

Hydric tests including the determination of the free water absorption (A_F), the saturation coefficient (S), the total water absorption by immersion (n_a) and the water absorption by capillarity (C) were performed to characterize the parameters associated with fluid uptake and transport inside the pores according to the procedure recommended by the European Standard EN1015-18 [18]. The calculations were performed using the following equations:

$$A_F = (M_S - 1) \times 100/M_0 \quad (1)$$

$$S = (M_S - M_0) \times 100/M_0 - M_H \quad (2)$$

$$n_a = (M_S - M_0) \times 100/M_S - M_H \quad (3)$$

$$C = (M_X - M_0) \times 100/A \quad (4)$$

where M_S is the mass of the saturated test sample; M_0 is the mass of the dried test sample; M_H is the hydrostatic weight of the saturated test sample, M_X is the mass of the wet sample (as compared with the dried mass) for a function of time t and A is the basal area of the test sample. Capillarity coefficients were calculated by determining the slope of the curves in the initial linear segment of the graph. For all determinations, an average of three sample measurements was used.

Freeze–thaw cycles were performed according to the procedure given by RILEM MS-TC 127-B.1 [19]. Mortar quarter-prism specimens of 100 ± 20 g were used for testing. Before testing, the samples were cured for 3 months at 100% RH. Prior to each cycle, specimens were immersed in water for 4 h and then weighed. Subsequently, the freeze–thaw cycle was set up as follows: 8 h at a temperature of −15 °C followed by 8 h under room temperature conditions. Specimens were weighed after each cycle and the loss of mass in relation to the initial mass of the specimens was plotted. The damage was visually and photographically monitored. Cycles were repeated until the destruction of the specimens or until a maximum value of 25 sequences was reached. Five specimens of each mortar type were tested.

3. Results and discussion

Porosity and pore size distribution are important parameters used for describing a hydrated cement system when the physicochemical behavior and the durability of repair mortars is considered. The differential mercury intrusion curves corresponding to incremental pore volume intruded as a function of pore diameter for the studied Roman cement mortars are presented in Fig. 1. The reference untreated mortar was studied at three aging points: 1 day, 14 days and 3 months. The observed peaks are sharp and well defined. In the case of fresh mortar, the unimodal peak at 0.7 μm was observed while for the sample cured for 3 months the threshold of pore diameters decreased and remained in the 0.01–0.03 μm range. This shift indicated the progress of the hydration process as the pores were filled with hydration products. Quite similar pore-size regions were observed after studying historic Roman cement mortars with values that were dependent on the initial water to cement ratio (w/c) and the degree of cement hydration. Three categories of pores were found to coexist in the historic Roman cement mortars. The finest pores, with diameters below 0.2 μm were present within the hardened aged Roman cement matrix. Pore diameters shift to smaller values in the case of well-hydrated and matured Roman cement mortars, which were

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