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Original article

A new method for making artificially weathered stone specimens for testing of conservation treatments

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ABSTRACT

The application of new consolidating products on the surface of weathered materials is a common intervention technique in conservation practice. Due to the difficulty of producing artificially weathered substrates in a reproducible way, the effect of consolidating products in laboratory is generally assessed on sound substrates. However, the properties of a weathered substrate largely differ from that of the original sound material; this might make the results of laboratory tests unreliable or hamper their interpretation. In this research, a new method for the production of weathered specimens in a reproducible way has been developed and validated on three types of limestone with different total porosity, pore size and petrographical characteristics: Maastricht, Savonnières and Euville. The aim was to develop a substrate on which the effectiveness, compatibility and durability of consolidating products can be tested in laboratory in a more reliable way than when using fresh stone. The method consists of grinding and sieving the stones in a grain size largely similar to that of the sound material and re-aggregating the particles by the use of air lime: a lean "mortar" is obtained which is applied as a layer on the sound stone to simulate the decayed surface of a material showing granular disintegration. The grain size and the binder to aggregate ratio are chosen in such a way as to reproduce those characteristics typical of weathered stones showing loss of cohesion (i.e. sanding or powdering): i.e. increased pore size and open porosity and lower cohesion and strength in comparison to the sound substrate. The properties of the obtained weathered substrates have been studied in comparison to that of the fresh stone: pore size and pore size distribution have been measured by Mercury Intrusion Porosimetry; Polarized and Fluorescence Microscopy has been carried out to study the petrographical characteristics of the assemblage sound stone/re-aggregated layer; the water absorption behavior and hardness (by means of Drilling Resistance Measurement System, [DRMS]) have been measured as well. The results of the research show that with this method it is possible to obtain specimens reproducing the higher and coarser porosity and lower mechanical strength, typical of stones suffering loss of cohesion.

1. Introduction

Porous building materials exposed to the environment may suffer from different damage mechanisms, such as salt crystallization, frost-thaw and biological growth. Damage may manifest itself as different decay patterns, like delamination, loss of cohesion or cracking of the material. When decay occurs in the form of loss of cohesion (sanding or powdering) of the material surface, consolidating products (e.g. ethyl silicate) can be applied on the surface to re-establish the original properties of the sound material.

Most common consolidating treatments are irreversible; therefore, testing their effectiveness and durability in laboratory prior to application on historic surfaces should be common practice. A consolidating treatment can be considered as effective when it improves the cohesion and strength of the decayed surface layer without surpassing the strength of the underlying sound stone. Moreover, the physical properties (water absorption, thermal and hydric dilation) of the consolidated layer should not differ too much from that of the original stone, in order not to enhance the decay [1,2]. From the above reported considerations, it is clear that evaluating the effectiveness of a consolidating product by applying it on a sound substrate (as often done in laboratory research, e.g. [3–5]) has large limitations. It might give unreliable results or hamper

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their interpretation. In order to obtain more reliable results, significant for practice, consolidating products should be tested on weathered substrates. These might be either sampled from the field or reproduced in laboratory. The first option [6] is generally not realistic in the field of conservation: it is in fact generally not allowed nor desirable to sample significant amounts of material from monumental buildings and/or objects. Moreover, differences in properties (as e.g. salt content, strength, degree and intensity of damage) amongst the specimens often occur. The second solution has been attempted by different researchers. For example, consolidating products have been tested on substrates weathered by accelerated salt crystallization test and subsequently desalinated by poulticing prior to the application of the treatment [7]. This procedure has the advantage to reproduce loss of cohesion of the surface layer of the substrate, a type of decay for which the application of consolidating treatment is effective and recommended. However, this method is very laborious and time consuming (several months might be necessary to obtain decay) and it might still result in significant differences in decay and salt content among the specimens to be treated. Recently, a method for weathering stones in a reproducible way, has been developed by Franzoni et al. [8] and applied in the assessment of consolidating products [9]: the method consists of heating up the stones to high temperature in order to cause anisotropic thermal expansion of the calcite grains and, consequently, inter-granular disjunction. However, the effectiveness of the method varies depending on the nature and microstructure of the original stone.

2. Research aims

This research aimed at the development of a substrate on which the effectiveness, compatibility and durability of consolidating treatments can be tested in laboratory in a more reliable way than when using fresh stone. The method starts from the assumption, confirmed by the practice (see for example Fig. 1), that a weathered stone showing loss of cohesion in the form of sanding or powdering has, in the phase preceding the loss of material, a higher total porosity and coarser pores than the original, sound stone, factors resulting in a lower mechanical strength [8,10].

The method for obtaining weathered specimens is described in Section 3; its validation, carried out by means of different methods and techniques, is discussed in Section 4.

3. Material and methods

3.1. Limestone

In this research, three types of stone have been used: Maastricht, Euville and Savonnières limestone. These lithotypes have been selected because of multiple reasons:

- they are limestones, thus constituted by particles cemented by a calcium carbonate binder;
- they are building stones often used in the Netherlands and Belgium and, in the case of the latter two lithotypes, also in other parts of Europe;
- they have different petrographic characteristics, porosity and pore size distribution.

Maastricht limestone is a soft (most recurrent values of compressive strength vary between 3 and 5 MPa [11], yellow colored limestone constituted of calcium carbonate bioclasts of about 300–500 μm diameter, poorly cemented by a sparite binder (Fig. 2left). It has a high $CaCO_3$ content, up to 94–98 wt% [12]. Maastricht limestone has been widely used for building purposes in the southern part of the Netherlands and adjacent Belgium, since Roman times.

Table 1
Grain sizes of the "aggregate" and binder/aggregate ratio used.

Stone	Grain size selected	Binder/aggregate ratio (in bulk vol)
Maastricht	Between 300 and 500 μm	1:4 and 1:6
Savonnières	Between 350 and 700 μm	1:4 and 1:6
Euville	Between 500 and 2000 μm	1:4 and 1:6

Savonnières limestone is a greyish yellow colored oolithic limestone, deposited in the Tithonian (Jura) and quarried in the Dept. Meuse, France. This stone is used as a building material in the Netherlands since the mid 19th century, with a few rare older examples. [13,14] It is constituted of large part by cemented oolites, i.e. small, more or less round calcium carbonate particles, concentrically grown around a detrital core. The size of the oolites in the studied thin sections vary between 350 and 700 μm. Besides oolites, shell fossils are commonly present in this stone (Fig. 2middle). The carbonate content of Savonnières stone can be up to 99%. In the studied thin sections, regions with different density have been observed, suggesting that this stone is not fully homogenous. Its compressive strength may vary, depending on the variety, between 6.4 and 27.2 MPa [15].

Euville limestone is a light brown to beige colored crinoid limestone, deposited in the Upper Oxfordien (Jura) and quarried in the Dept. Meuse, France. As many other French limestones, Euville has been commonly used in the Netherlands as replacement stone since the second half of the 19th century [13,14]. It is constituted by cemented crinoid fossils (Fig. 2right), the size of which varies between 500 and 2000 μm. Its compressive strength may vary between 12.4 and 38.3 MPa [15] and its porosity between 6.7 and 17.9 vol%, probably due to differences in original porosity, diagenesis and compaction [16].

3.2. Procedure for preparing artificially weathered stone specimens

The stones were ground and sieved to select the particle sizes most recurrent in the original sound stone, as visually assessed by petrographic observations (Table 1). The grains were then mixed with air lime powder (Supercalco 90 by Carmeuse) in ratios 1:4 and 1:6 (by bulk volume). The aim was to obtain, after grinding and sieving, particles of the same size as those observed in fresh stone and to re-aggregate the particles using as less as possible binder, in order to simulate a substrate suffering by loss of cohesion. The ratios 1:4 and 1:6 are a compromise between the need of giving sufficient cohesion to the re-aggregated layer and creating at the same time an artificial material as much as possible similar to a decayed stone in terms of chemical (same composition as sound stone), physical (higher porosity and coarser pores with respect to sound stone) and mechanical (lower strength and hardness than the sound stone) properties.

In the case of the Maastricht limestone, both the 1:4 and 1:6 mixes have a sufficient workability. The workability is here meant as the possibility of working with the mortar and applying it in a layer on the top of the stone, keeping the binder in between the grains to give cohesion. In the case of Savonnières and Euville limestones, the 1:6 mixes were not workable and, once hardened, too weak to be handled. Therefore, the 1:6 mixes of Savonnières and Euville were not further studied.

The obtained "mortar" was applied in a layer of about 10 mm on both cylinders (diameter 45 mm, height 40 mm) and blocks ($150 \times 150 \times 100\,mm^3$) of fresh stone (Fig. 3), after pre-wetting of the substrate. The cylinders and blocks of fresh stone were cut in