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Artificial aging route for assessing the potential efficacy of consolidation treatments applied to porous carbonate stones

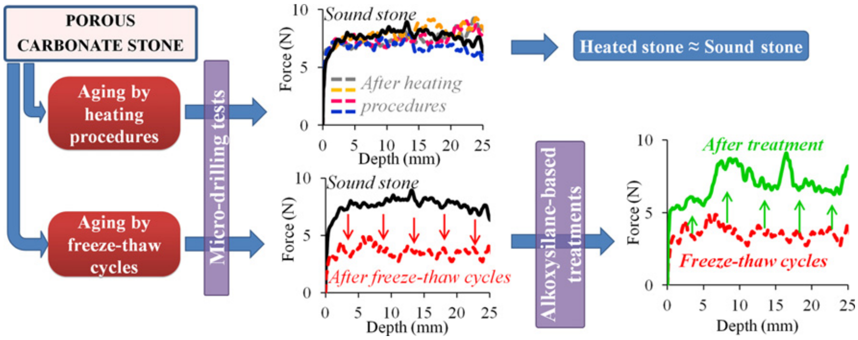
B. Sena da Fonseca [a,*], A.P. Ferreira Pinto [b], S. Piçarra [c,d], M.F. Montemor [a]

[a] *Centro de Química Estrutural-CQE, Instituto Superior Técnico, Universidade de Lisboa, 1049-001 Lisboa, Portugal*
[b] *Department of Civil Engineering, Architecture and Georesources, CERIS, Instituto Superior Técnico, Universidade de Lisboa, 1049-001 Lisboa, Portugal*
[c] *Escola Superior de Tecnologia de Setúbal, Campus do IPS, Estefanilha, 2910-761 Setúbal, Portugal*
[d] *Centro de Química-Física Molecular and Instituto de Nanociência e Nanotecnologia, Instituto Superior Técnico, Universidade de Lisboa, 1049-001 Lisboa, Portugal*

HIGHLIGHTS

- Aging by heating causes none or minor damage in the used porous carbonate stone;
- Freeze-thaw cycles induce significant physical and mechanical modifications in the used porous carbonate stone;
- Drilling resistance is an important tool for the characterization of mechanical damage induced by aging procedures;
- A promising procedure to study the potential efficacy of consolidants in artificially aged porous carbonate stones was used.

GRAPHICAL ABSTRACT



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ABSTRACT

Unsatisfactory behaviors of consolidation treatments applied to porous carbonate stones in the field are frequent, although they had revealed promising performances when applied to sound stones in laboratory. Thus, accurate assessment of the potential efficacy of consolidation treatments in laboratory conditions is of outmost importance to support conservation practice.

This paper compares heating and freeze-thaw as artificial aging procedures for testing consolidants on a selected porous carbonate stone ($CaCO_3 \approx 99.9\%$, porosity $= 16\%$ and average pore radius $= 0.4\ \mu m$). The induced decay was assessed by physical tests and drilling resistance measurement system (DRMS), which is a well-establish method to characterize materials and evaluate the efficacy of consolidation treatments.

Heating caused minor alterations, because the anisotropic thermal expansion of calcite was compensated by the stone porosity, whereas the freeze-thaw cycles produced important alterations.

Sound and pre-aged specimens by freeze-thaw were treated with different alkoxysilane-based consolidants and their effect on the mechanical properties was assessed by DRMS and analyzed using the difference drilling profile method (DDP).

The obtained results highlighted the interest of using pre-aged specimens by freeze-thaw and the usefulness of the procedures adopted for the assessment of the potential efficacy of consolidation treatments applied to porous carbonate stones.

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[*] Corresponding author.
E-mail address: senadafonseca@gmail.com (B. Sena da Fonseca).

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1. Introduction

The consolidation of stone elements is one of the most risky actions in the conservation field due to its non-reversibility and to the possibility of harmful side effects [1]. In addition, successful consolidation has been very difficult to obtain with the available products, especially on porous carbonate stones. Therefore, before any intervention regarding stonework consolidation, it is crucial to guarantee that the treatment will meet all the requirements for which it is being proposed, without any harmful side effect.

The most used, well-established and versatile consolidants are the ones based on alkoxysilanes [2]. However, when used in porous carbonate stones, a few drawbacks constraining their overall performance are commonly highlighted in literature [1,3–5]: lack of strong chemical bonds with the carbonate material and susceptibility to crack during evaporation. Thus, several alternatives to the traditional alkoxysilane-based products are being proposed such as nanolimes [2,6], microbially induced carbonate precipitation [7] or phosphate-based solutions [8,9]. In parallel with these novel alternatives, the versatility of alkoxysilane-based products and their good intrinsic characteristics (capacity of penetration, stability, reduced influence on the vapor characteristics, etc.) justify efforts to design new alkoxysilane-based products, in which the mentioned limitations are suppressed or minimized. Examples are functional alkoxysilanes containing organo-groups [10,11], nanoadditives [12–14] or silicon-based organic polymers [13–16].

The evaluation of the initial efficacy of consolidation treatments and potential harmfulness is commonly studied under laboratorial conditions. However, despite the use of the same or alike lithotypes, dissimilar results in terms of efficacy and harmfulness between laboratory and in situ are often observed when applying alkoxysilane-based treatments. Among other causes, one of the factors contributing to those mismatched results is the use of sound stone specimens in laboratory, whereas in situ stone to be consolidated is generally in an advanced state of decay. Consequently, to better correlate laboratorial and field results, different artificial damaging procedures were recently explored [17–19]. These procedures involve heating the stone specimens (in dry or saturated conditions) during a certain period of time and are intended to induce micro-cracks and grains de-cohesion, simulating decayed stones. In dry conditions, the degradation mechanism varies according to the stone mineralogy [18]. In dry stones composed by different minerals (e.g. granite or sandstones), the different thermal expansion coefficients of stone constituents result into internal stress during heating that causes the opening of micro-cracks, while in stones mainly composed by calcite (e.g. marbles and limestones) the opening of micro-cracks occurs because of the marked anisotropic thermal expansion behavior of calcite. Besides, when saturated specimens are heated, the pressure exerted by water on stone pore walls may be responsible for further damaging [18].

Heating stones up to 400 °C showed to be an effective way to produce microstructural changes and reproducible modifications on the mechanical strength of some lithotypes [18,19]. However, it was reported that in specific porous carbonate stone, the method was less effective because larger volumes of voids allowed free deformation of calcite crystals, without inducing internal stresses [20]. Furthermore, when the stones have secondary minerals that are susceptible to “hot-melt effect” (e.g. clay minerals), chemical-physical transformations may be responsible for a more effective bond of the grains and consequently to strength increases [18]. These are noteworthy drawbacks of the heating procedures, since porous carbonate stones have been quite used as monumental stones, as they are fairly easy to carve due to their softness.

Therefore, an alternative artificial degradation method for porous carbonate stones based on physical decay and with less susceptibility of inducing chemical modifications is required. Some possible ways can be considered, namely freeze-thaw cycles or salt crystallization.

From the perspective of preparing artificially aged specimens to study consolidation treatments, the freeze-thaw action seems to be more appropriate, as there is no risk of having salts blocking the pores or interfering with the consolidation treatment under study, while it is not predictable that it can produce significant and unexpected chemical changes. Furthermore, it is a real decay mechanism that affects porous materials in several countries. Depending on the tensile strength and porous characteristics of the lithotypes, the major drawback of freeze-thaw cycles may be related to the long duration of the procedure. However, a significant degradation degree is expected to occur with a reduced number of cycles in porous soft carbonate stones (porosity > 10%) with a well-connected pore system [21]. The pore size distribution and pore shape also have a prominent role on the decay susceptibility to ice crystallization [22]. In fact, it is known that stones having a significant percentage of pores within the range 0.1 μm and 10 μm are particularly vulnerable to damage due to ice and salt crystallization [21,23].

Regarding the evaluation of the damage produced by aging procedures and the strength gain due to subsequent consolidation treatments, several methods that can hardly discriminate strength variations along the depth have been used (compressive and tensile strength, dynamic and static elastic modulus) [8,9,17,24,25]. However, in most field situations, the physical integrity tends to vary in-depth in naturally decayed stones and in most of the treated stones. Due to this reason, it has been generally recognized that the drilling resistance measurement system (DRMS) is one of the most suitable methods for evaluating the consolidation efficacy, particularly in soft stones [26–29]. The possibility of assessing the variation of material strength in depth in laboratory and in situ is of outmost importance for the characterization of the decay patterns and to assess the treatments efficacy and potential harmfulness [30–32].

According to the gaps that have been identified, the work disclosed henceforth intends to investigate the interest of using artificially aged specimens to assess the potential efficacy of consolidation treatments on porous carbonate stones and to compare heating and freeze-thaw as artificial aging procedures for that purpose. The evaluation of the alterations promoted by the tested aging procedures was based on the comparison of physical and mechanical properties assessed by DRMS. Additional tests were carried out to compare the initial efficacy of alkoxysilanes-based treatments when applied to sound specimens and freeze-thaw pre-aged specimens.

2. Materials and methods

2.1. Stone and consolidant products

The lithotype used in this study was a beige limestone with the commercial trade name of Rosal AR. This stone is under current exploitation in Porto de Mós region (Portugal) and is mainly used for wall cladding and flooring. Fairly similar lithotypes exploited from the same massif were erstwhile used in the construction/reconstruction of some important monumental constructions in Portugal [33,34].

The tested stone can be considered a highly porous limestone, as its open porosity is around 16.0%. It is composed by spar calcite cement as interstitial material and peloids, being less abundant the oolits. The allochems vary in size from 0.1 mm to 1 mm, wherein most of the particles have around 0.5 mm.

The pore size of Rosal AR ranges mainly between 0.02 and 1 μm, with higher percentages of pores with dimensions between 0.1 and 0.6 μm (Fig. 1).

The pore size distribution of Rosal AR stone points out that this lithotype is susceptible to be damaged by freeze-thaw cycles, because the majority of the pores display sizes within the range 0.1 μm and 10 μm [21,23].

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