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Original article

Consolidation of carbonate stones: Influence of treatment procedures on the strengthening action of consolidants

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ARTICLE INFO

Article history:

Received 7 October 2010

Accepted 19 July 2011

Available online 19 September 2011

Keywords:

Consolidation treatment
 Carbonate stones
 Assessment method for consolidation
 Effectiveness
 Penetration depth
 Resistance increase
 Microdrilling
 Ultrasonic velocity

ABSTRACT

This article demonstrates the importance of treatment application procedures on the consolidation effectiveness obtained by comparison of the results obtained using three different consolidants on four carbonate stone types, and proposes a general methodology for assessing the potential effectiveness of consolidants in laboratory conditions. It stresses the relevance of taking into account the treatment methodology, given the influence application protocols can have on the overall behaviour of the consolidated material. Several mechanical properties were assessed to demonstrate this influence on the performance of the consolidant. The results demonstrate that the strengthening action achieved with a specific product can only be defined in a strict relation to the treatment protocol used to produce it. The results also contribute towards the definition of standard testing protocols on stone consolidation. The application of a consolidant by direct contact capillary absorption is a reliable procedure and the results are easier to interpret than others obtained by brushing or by full immersion, thus making this procedure a good candidate for an eventual standard laboratory assessment method of the consolidation action of any specific stone/consolidant combination. This study also showed that the best test method to assess the strengthening action of stone consolidants in soft stones is DRMS (Drilling Resistance Measuring System). Moreover, the collection of longitudinal ultrasound velocity profiles determined in stones specimens treated by contact capillary absorption was shown to be a useful non-destructive method to assess the depth of the strengthening action achieved.

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1. Research aim

The aim of this research work was to test the influence of application procedures for common consolidation products by evaluating the changes in certain stone properties before and after treatment. Variations in the final results are always to be expected as a consequence of subtle variations in the testing conditions. Accordingly, a parametric study was followed to demonstrate the importance of the treatment procedure on the consolidation effectiveness, and to identify the best methods for assessing the consolidation action in carbonate stones. From the results of this study a standard test protocol to assess the potential effectiveness of consolidation treatments on carbonate stones is proposed.

2. Introduction

The assessment of consolidation effectiveness is not straightforward, particularly because there is interdependence between the stone and the applied treatment [1–3]. Besides the type of product used, the consolidation action achieved depends on the treatment methodology, which to a large extent can be described using parameters such as product concentration, solvent type, application process, and contact time [4]. The product, solvent, and concentration are in general well managed in research protocols and reported in the published literature. However, the influence of the application process and contact time on the results are much more rarely tackled and the reader is frequently faced with enormous difficulties to compare the results of others with his own research. In fact, results obtained with different testing methodologies make comparison difficult, if not impossible. Consequently, the integration of results from other researchers into one's practice is a hard and mostly unfruitful endeavour.

It is commonly accepted that the evaluation of consolidation products should be carried out primarily through laboratory studies, the main objectives of which being to know their effectiveness and durability as well as their potential harmfulness.

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Literature offers manifold papers that employ different treatment methods, namely, by brushing [5–7], by immersion [8], by capillarity [9,10], and also combined procedures adapted to the parameters to be analysed [11]. In fact, it is with respect to the treatment method that opinions diverge the most regarding standardised procedures [12]. The methods followed are mostly adopted according to personal preference and it is not uncommon to find the same application process used with different application times [2,11–14].

Exactly what is meant by the “effectiveness” of a stone consolidant is not properly defined [2] and despite the intrinsic importance of establishing a suitable definition, this does not appear to concern researchers. However, because the main aim of a consolidation process is to enhance the cohesion and adhesion of stone constituents, it is logical that parameters used to assess this should directly or indirectly try to characterise this increment. The most popular parameters used are: surface hardness [2,15], strength in depth [16,17], bending strength [14,18], compressive strength [3,19,20], ultrasound velocity [11,21], and modulus of elasticity [16,18].

Many studies on consolidation treatments have been carried out and published, and yet very little have been published on the criteria and properties that should be used as laboratory estimators of the potential performance of consolidation products. A few papers [12,16,22] address this theme using multivariate analysis of parameters and open a new road where further research is certainly necessary.

Studies that analyse the modifications induced by the stone consolidant are frequent, but the measured properties are mostly unrelated to the consolidation action, leaving little room to properly assess the effectiveness, harmfulness and durability of the consolidation products appropriately. Changes in porosity and other water related properties are certainly informative, but they do not give direct information about the consolidation action. Accordingly, consensus regarding the assessment parameters and evaluation methods to be used to assess the potential effectiveness of consolidation products would be highly welcome.

The main goal of a consolidation treatment is the improvement of cohesion and increase of mechanical resistance of a degraded stone. Therefore, the assessment of its potential effectiveness should focus on changes in properties that might directly reflect the presence of a consolidation action and the strengthening capacity of the product. The papers referred to above show several trends in this direction.

It is widely accepted that the in depth consolidation is an important parameter to be taken into account when assessing consolidation effectiveness [2,23,24]. Its importance was recognised by Schaffer in 1932 [25], and became a critical parameter when important cases of failure were attributed to the insufficient penetration depth of consolidants [26].

The direct detection of the consolidation product in depth can be achieved through chemical analysis and visual (including coloration) techniques. They include the use of SEM [8,27–30], FTIR and other spectroscopic techniques [31], as well as product specific coloration methods [32]. Summary highlights of these techniques are reported in [33]. Other techniques have been used to detect indirectly the presence of the consolidant, such as ultrasound velocity [34], abrasion loss [35], and properties related to water absorption and water vapour permeability [36]. Besides being used to detect where the product has penetrated, properties such as water absorption, porosity and pore size distribution have been also used to a certain extent to assess the overall consolidation action [7,8,12,19,32,37,38]. However, laboratory studies never reproduce what occurs in a real situation in its full complexity and under such conditions only the potential effectiveness can be evaluated. Furthermore, it is clear that the assessment of effectiveness has to integrate parameters that give direct information

about the mechanical resistance of the stone. In general, when a product is present other stone characteristics are modified, such as water absorption, water vapour permeability, drying behaviour, and colour. Such modifications may provide additional information to help characterise the overall situation, although they are not critical indicators of the consolidation action.

The present research was carried out on four different carbonate stones treated with three consolidating products (ethyl silicate, acrylic, and epoxy resins) applied by brushing, by full immersion and by contact capillary absorption. The potential effectiveness of the consolidation treatments was evaluated in terms of the modification of the mechanical resistance in depth (determined with a microdrilling device), as well as the ultrasonic velocity, flexural resistance, and superficial hardness. The results show that the consolidation action achieved with a specific product can only be defined and assessed within the context of the treatment method followed.

3. Materials and methods

3.1. Stone materials

The laboratory experiments were carried out on four carbonate stones. Ançã and Boiça stones are two very pure calcitic lime-stones formed almost exclusively of calcite with silica as the main, but relatively scarce, accessory mineral. They have quite different porosities (Ançã: 27%; Boiça: 10%), but both present a typically unimodal pore size distribution characterised by the presence of larger pores in Ançã (0.05 to 1 μm) than in Boiça stone (0.01 to 0.2 μm) [1]. Ançã stone is a fine grained and homogenous white stone with a very high porosity. Boiça stone presents a homogeneous micritic matrix with frequent fossil remnants and veinlets of sparitic calcite.

Coimbra and Lisbon stones are two calcitic dolostones and their chemical compositions include silica and alumina, which indicate the possible presence of clay minerals in both stones. Coimbra stone is a heterogeneous brecciated material, which in some cases has a significant number of fissures that are filled with iron oxides and calcite. The heterogeneity of Coimbra stone is so pronounced that in some areas it is possible to visually identify zones where the stone is softer and more porous (porosity of 19%) and others where it is harder and less porous (porosity of 14%). Lisbon stone is a fine-grained micritic stone with frequent vacuoles of diverse forms and dimensions and a porosity of 15%. The borders of the vacuoles are made of well-formed dolomite crystals, frequently occurring with perfect rhombohedral shapes.

3.2. Consolidants

Three different consolidants: ethyl silicate, acrylic, and epoxy resins were applied individually to samples of each stone type [1]. The tested consolidants are all commercially available products.

The ethyl silicate (TG) tested is a ready to use product that contains a pre-polymerised TEOS mixed with white spirit. According to the manufacturer (Goldschmidt), it should be applied until the support is saturated, it being presumed that this situation is achieved when the surface remains wet for 1 minute.

The acrylic consolidant Paraloid B72 is described by the manufacturer (Röhm and Hass) as an ethyl-methacrylate copolymer. It was used in the form of a low concentration solution prepared directly from the solid material according to the following formulation: 0.06:0.61:0.09:0.2 (resin:toluene:xylene:acetone) (by weight) and will be referenced in this paper with the acronym ‘B’.

The epoxy consolidant (EP) is a cycloaliphatic epoxy resin, EP 2101, produced by EUROSTAC, in the form of a 25% solution (by weight) in toluene and isopropanol [40]. The hardener is an

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