Science of the Total Environment 575 (2017) 23–32

Contents lists available at ScienceDirect

Science of the Total Environment

journal homepage: www.elsevier.com/locate/scitotenv

Durability assessment to environmental impact of nano-structured consolidants on Carrara marble by field exposure tests

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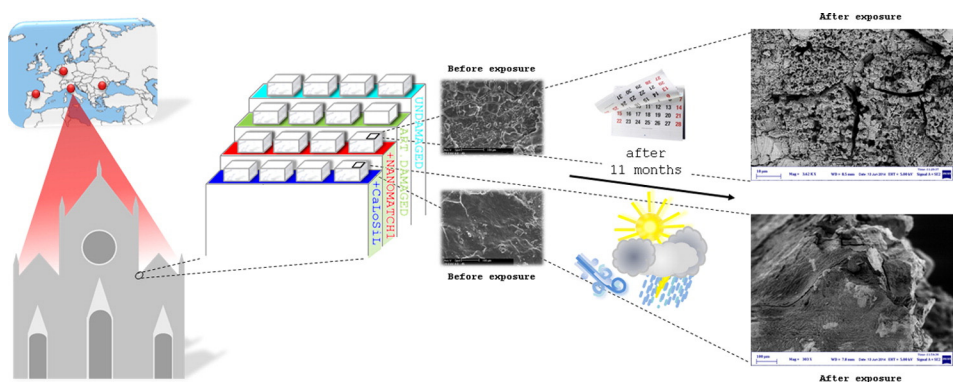
HIGHLIGHTS

- Cultural heritage increasingly at risk due to climate change impact
- Need of compatible and resilient consolidating products for marble
- Carrara marble models (treated and not) exposed at four European sites
- Rain principally affected the durability of $CaCO_3$-precursor consolidants.

GRAPHICAL ABSTRACT



ARTICLE INFO

Article history:
Received 4 August 2016
Received in revised form 30 September 2016
Accepted 1 October 2016
Available online xxxx

Editor: D. Barcelo

Keywords:
Climate change
Durability
Metal alkoxide
Stone conservation
Cultural heritage

ABSTRACT

The EU policy of reducing the emissions of combustion generated pollutants entails climate induced deterioration to become more important. Moreover, products applied to preserve outdoor built heritage and their preliminary performance tests often turn out to be improper. In such context, the paper reports the outcomes of the methodology adopted to assess the durability and efficiency of nano-based consolidating products utilized for the conservation of carbonate artworks, performing field exposure tests on Carrara marble model samples in different sites in the framework of the EC Project NANOMATCH. Surface properties and cohesion, extent and penetration of the conservative products and their interactions with marble substrates and environmental conditions are here examined after outdoor exposure for eleven months in four different European cities and compared with the features of undamaged and of untreated damaged specimens undergoing the same exposure settings.
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1. Introduction

The intensification of climate changes combined with the recent reduction in acidic air pollution in urban areas has made climate conditions to become more influential in the weathering of outdoor built heritage (Grossi et al., 2008). In this regard, research has been specifically devoted in the last decades by the European Union to assess the effects of environmental changes, including climatic ones, on cultural heritage and to develop mitigation strategies aiming at its protection and conservation (Kucera et al., 2005; Grossi et al., 2007; Sabbioni et al., 2007; Watt et al., 2009; Franzen et al., 2011; Bernardi et al., 2012; Sabbioni et al.,

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http://dx.doi.org/10.1016/j.scitotenv.2016.10.004
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2012; Ashley-Smith et al., 2013; Ozga et al., 2014). In particular, available projections on climate change impact on cultural heritage have evidenced that decohesion and fracturing of carbonate stone, widely encountered in archaeological and historic built heritage, are likely to increase in Europe in the near (2010–2039) and the far future (2070–2099) induced mainly by (i) surface recession, (ii) thermal stress and (iii) salts crystallization (Bonazza et al., 2009a, 2009b; Grossi et al., 2011). Several efforts have been made since the 20th century in order to re-establish the original physical-mechanical properties of deteriorated stone and to increase their durability by the extensive use of both organic and inorganic consolidants. It is widely known that the performance and long-term efficiency of organic consolidants have been heavily reconsidered as they frequently undergo irreversibility of the treatments and chemical modifications caused by the interaction with both the environmental conditions and the substrate (Melo et al., 1999; Amoroso, 2002; Favaro et al., 2006, 2007). Although traditional inorganic consolidants as well as innovative nanomaterials (mainly dispersion of barium/calcium/magnesium hydroxide nanoparticles in alcohol) display better physical-chemical compatibility with carbonate stones and good chemical stability to environment, improvements should be introduced in order to ensure higher cohesive effect and better penetration into the porous matrix of damaged substrate (Camaiti, 2000; Giorgi et al., 2000; Amoroso, 2002; Doehne and Price, 2010; Chelazzi et al., 2013; Natali et al., 2014). In addition, possible interactions between the developed material and the outdoor environment should be evaluated, as pollution and weathering factors may act on the new product as well as on the historic substrates. In this regard, most of the studies have been carried out in laboratory, in accelerated and controlled environment. Unfortunately, climate simulation chamber is not designed to accurately replicate the real conditions of outdoor environment failing in the simulation of the synergetic action of environmental parameters as well as their variations at short and long time scale. Higher correspondence to the reality is although achieved by field exposure tests. Hence the need to define the most suitable ageing tests arises whenever resistant and compatible conservative materials are designed. In literature, most of field trials were performed to study damage mechanisms on building materials induced by weathering or pollutants (Zappia et al., 1998; Viles et al., 2002; Urosevic et al., 2012) or to evaluate new products' performance through their application directly on historic damaged substrate, with the risk to be sometimes unsuitable and irreversible applications (Favaro et al., 2005, 2006, 2007; Varas et al., 2007). On the contrary, field tests on model samples allow to evaluate the features of the examined product in response to different environmental conditions and to improve its performance without any damage to the works of art. Nevertheless, still scarce examples of published results on this issue are available (Simon and Snethlage, 1996).

In this paper we discuss the results obtained by performing field tests of model samples in Carrara marble exposed at four European monuments (Santa Croce Basilica in Florence, Cologne Cathedral, Oviedo Cathedral and Stravopoleos Monastery in Bucharest) in order to study the compatibility and environmental durability of the consolidating product developed within the EC Project NANOMATCH (Bernardi et al., 2012). The product's performance is also tested in comparison to CaLoSiL® and its consolidating use in the conservation of cultural heritage discussed. Because of the rising role of climate parameters in causing damage to heritage building materials and the expected increase of surface recession of carbonate stones due to rainfall, the samples under study were placed in unsheltered areas directly exposed to precipitation events. The integration of the results here presented with those carried out in laboratory as part of the EC NANOMATCH Project provided complete information on applicability, compatibility, efficiency and durability of the tested conservative products and allowed to identify and subsequently manage the possible drawbacks before their application on works of art.

2. Materials and methods

2.1. Investigated conservative products

Preliminary tests and application trials were performed to identify the proper alkaline earth alkoxide among those synthesized for consolidating carbonate stones to evaluate the most suitable methodology for its production at industrial scale and to optimize the application parameters (Favaro et al., 2008; Duchêne et al., 2012; Ossola et al., 2012; Favaro et al., 2014; Natali et al., 2015). The best results were obtained with nano-solution of calcium tetrahydrofurfuryloxide (Ca(OTHF)2) (NANOMATCH1) in 1:1 ethanol:ligroin at 20 g/L of calcium, applied by brush on quarried Carrara marble samples for two times on one surface (Natali et al., 2015). In order to compare the performance of the new consolidating agent, CaLoSiL® (IBZ-Salzchemie GmbH & Co.KG), product widely employed for stone consolidation, was selected and applied by brush on other samples. In CaLoSiL®, particles of calcium hydroxide were dispersed in ethanol at 20 g/L of calcium. The consolidating effect of these products is produced by calcium carbonate, obtained by the reaction of calcium hydroxide and calcium alkoxide with atmospheric moisture and carbon dioxide. Since both NANOMATCH1 and CaLoSiL® are precursors for the deposition of calcium carbonate, they could be effectively employed to treat carbonate substrates.

2.2. Field tests

We tested the compatibility and environmental durability of NANOMATCH1 and CaLoSiL® on Carrara marble, a metamorphic stone primarily composed of calcite crystals. Polished marble plaques with dimensions of 10 cm × 10 cm × 5 cm were placed outdoor in an unsheltered area, exposed to rain wash-out, in four European sites characterised by different meteorological and climatological conditions:

- Santa Croce Basilica in Florence (Italy);
- Cologne Cathedral (Germany);
- Oviedo Cathedral (Spain);
- Stavropoleos Monastery in Bucharest (Romania).

A stainless metallic rack, perforated and tilted with 30° slope, supported the samples and allowed the drainage of rainwater (Fig. 1). Field trials lasted for eleven months in each site, from April/May 2013 to March/April 2014.

In order to obtain a stone substrate characterised by a heavy intergranular decohesion, part of samples was previously artificially damaged by thermal stress (i.e. exposed at 600 °C for 1 h), which led to a total structural disintegration of Carrara marble (Bourguignon et al., 2014). Indeed, the anisotropy of calcite in thermal response leads Carrara marble to be affected by thermoclastism and therefore to granular

Fig. 1. Carrara marble model samples exposed on the rack at Santa Croce Basilica, Florence.

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