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Nanolime for the consolidation of lime mortars: A comparison of three available products

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HIGHLIGHTS

- The consolidation effectiveness of Calosil® and Nanorestore Plus® is reduced with time.
- L'Aquila nanoparticles are more reactive which tend to grow better developed calcites.
- L'Aquila nanolime provides higher durability to dissolution.
- All treatments reduce the porosity in the surface that reduces evaporation rate.
- The whitening effect caused by nanolime treatment is reduced after accelerated weathering.

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ABSTRACT

Nanolime products are one of the most promising consolidation methods for historic calcareous substrates. Whilst the popularity of nanolime has been growing, its consolidation mechanism still needs to be fully understood when applied to highly porous substrates. The aim of this paper is to compare the three available nanolime products in terms of consolidation efficacy on lime mortar specimens. It is shown that repeated applications of a low concentrated nanolime can increase the superficial cohesion and the mechanical strength of the mortar within 1 cm from the surface, while also reducing porosity, number of micro-pores and capillary water absorption coefficient. Nanorestore Plus® yielded the highest short-term consolidation effect. However, L'Aquila nanolime showed a higher durability which was attributed to a better developed crystalline structure.

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1. Introduction

The consolidation of degraded calcareous materials is one of the main challenges in the conservation of historic structures. Calcareous substrates degrade by weathering processes such as crystallisation of salts, biological activity, freeze-thaw action and chemical attack by acid atmospheric pollutants [1–3]. Consolidants can help in recovering the strength of degraded materials as well as decreasing the deterioration rate of the substrate. In general, suitable consolidants must meet the following criteria: i) be physically, mechanically and chemically compatible with the substrate; ii) have a good adhesion to the substrate; iii) increase the substrate's mechanical properties; iv) not induce colour or aesthetic changes to the substrate; v) reduce porosity but not hamper moisture transport through the substrate. The effectiveness of the consolidation depends on the interaction of a number of factors such as the characteristics of the substrate, the properties of the consolidant and its compatibility with the substrate, and the application methods and conditions [4].

Limewater, which is a saturated solution of calcium hydroxide with a maximum of concentration of 1.5 g/L with lime particles in a colloidal suspension [5], has been used over centuries to consolidate deteriorated limestone or plaster. It has the advantage of being durable and compatible with the substrate as it is based on the precipitation of calcium carbonate into the pores of the treated material by reaction of the calcium hydroxide with the atmospheric carbon dioxide (CO_2). The main constraint of the limewater technique is a low consolidation depth due to limited penetration of carbon dioxide into the substrate [6]. Furthermore, the

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application of limewater can cause whitening of the treated surface. The effectiveness of limewater has been greatly discussed in literature. According to Price et al. [7] when using limewater most particles are deposited within 2 mm from the surface yielding an ineffective consolidation. Price's research has led some stone conservators to be skeptical of this treatment. Nonetheless, Brajer [8] demonstrated that a prolonged uninterrupted application produces a noticeable consolidation effect and some authors brought up new perspectives to its practical work such as the use of lime poultices and of an increased number of limewater applications [3,9,10].

During the last century, organic consolidants (i.e. synthetic polymers, such as Paraloid B-72, Mowilith 30 and Primal AC33) were extensively used in restoration treatments for calcareous materials due to their immediate strength enhancement, ease of application and the limitations shown by limewater [11]. These consolidants proved to be effective in the short and medium term for some calcareous substrates. However, the low compatibility with the mineral substrate and their short durability caused more substrate degradation in the long term, particularly in environments where temperatures increase above 40 °C [3,9,11]. Specifically, physical and mechanical incompatibility between organic consolidants and calcareous substrates can cause crack development, aesthetic changes and interference with future treatments [12,13], sometimes with severe consequences [14,15].

Nanolimes were developed to overcome the limitations of the traditional limewater treatment. Their consolidating effect takes place by the same mechanism as for the limewater technique but the smaller size of the lime particles (nanoscale) improves their performance. The advantages of nanolime compared to limewater are: i) nanolimes contain higher amounts of calcium hydroxide particles; ii) lime nanoparticles are more reactive due to their higher specific surface thus increasing the carbonation rate; iii) nanolimes penetrate deeper into the substrate because of their smaller particle size; iv) nanolimes have better colloidal stability due to their smaller particle size and the electrostatic repulsion forces between them; and, v) reduced whitening of the treated surface when nanolimes are used. An overview of nanolime synthetisation methods and use as a consolidant for calcareous substrates can be found elsewhere [16].

The first nanolime to become available on the market was Calosil® (IBZ-Salzchemie GmbH & Co.KR, Germany) in 2006, followed by Nanorestore® (CSGI – University of Florence, Italy) in 2008. Recently, the University of L'Aquila has patented a new method to synthesise nanolime where nanoparticles are produced by an anion exchange process at ambient room conditions in aqueous suspensions [17–19]. Nanolime produced through this method is currently in the process of being commercialised.

Both Calosil and Nanorestore have been extensively used for the conservation of wall paintings and stuccoes, achieving good re-adhesion of detached particles or pigment flakes [5,16,20] and consolidating powdering surfaces [15,20]. However, the effectiveness of the nanolimes decreases when mass consolidation of a porous substrate such as deteriorated stone or mortar is required [21–23]. The effectiveness of nanolime as a consolidant appears to be influenced by several factors: i) multiple applications of low nanolime concentration suspensions (i.e. 5 g/L) reduce the accumulation of consolidating product near the surface and improves the yield of carbonation within the pores [24,25]; ii) the type of alcohol used can influence the nanolime deposition in the pores [26,27] as well as the carbonation process [28], iii) external factors such as relative humidity (RH) and exposure time appear to influence the carbonation rate and precipitation of polymorphs [29,30]; iv) age and storing temperature of the nanolime affects the conversion of $Ca(OH)_2$ particles into Ca-alkoxides which can decrease its effectiveness [31].

The aim of this work is to compare the consolidation effectiveness of the three available nanolime products on lime mortars and investigate their long term performance. The influence of the nanolime treatments on mortar superficial cohesion, water absorption by capillarity, drying rate, drilling resistance, pore structure and aesthetic properties have been investigated.

2. Materials and methods

2.1. Lime and sand

Singleton Birch Ultralime CL90 (≥98% $Ca(OH)_2$, measured by XRD and XRF) and silica sand from Pentney (UK) were used throughout this work for the mortar mixes. The sand grading is shown in Fig. 1. The mineralogical composition of the sand, which was determined by XRD (*PANalytical XPert PRO*) using Rietveld refinements, is 96.3% Quartz (SiO_2, ICSD #00-046-1045) and 3.7% Potassium Feldspar ($KAlSi_3O_8$, ICSD #01-076-0831).

2.2. Nanolime

Three nanolime dispersions were used throughout this work:

- **Nanorestore Plus Propanol 5®** (CSGI Consortium – University of Florence, Italy): 5 g/L calcium hydroxide in 2-propanol. Particle size 100–300 nm. This dispersion is referred to as NAN.
- **Calosil IP5®** (by IBZ Salzchemie GmbH & Co.KG, Germany): 5 g/L calcium hydroxide in 2-propanol. Particle size 50–150 nm. This dispersion is referred to as CAL.
- **Nanolime synthesised through the method developed by Taglieri et al [17] at the University of L'Aquila**: 5 g/L calcium hydroxide in 50–50% water – 2-propanol. Particle size 20–80 nm. This dispersion is referred to as LAQ.

LAQ was synthesized through an anionic exchange process carried out at room temperature and ambient pressure by mixing under moderate stirring an anion exchange resin (Dowex Monosphere 550A OH from Dow Chemical) with an aqueous calcium chloride solution following a methodology described by Taglieri et al. [17,18] and Volpe et al. [19]. When these two components are mixed together, the substitution of OH groups in the resin with chloride ions (Cl^-) in solution leads, in conditions of supersaturation, to the formation of pure $Ca(OH)_2$ nanoparticles, following the reaction:

$$CaCl_2 + 2(R\text{ - }OH) \rightarrow Ca(OH)_2 \downarrow + 2(R\text{ - }Cl)$$

The concentration of chloride was monitored during the process (Vernier Chloride Ion-Selective Electrode CL-BTA) and when this

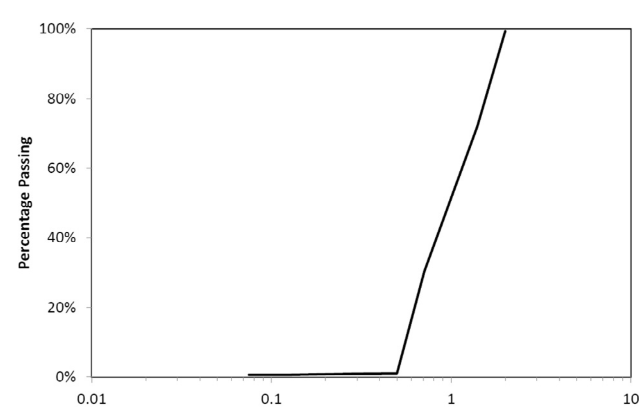

Fig. 1. Sand grading curve.

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