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# Calcium Hydroxide Nanoparticles for Limestone Conservation: Imbibition and Adhesion

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## Abstract

This paper describes the effects on Portland limestone of colloidal calcium hydroxide nanoparticles (“nanolimes”) dispersed in ethanol, available commercially under the trade name CaLoSiL. The effects of this conservation treatment on water uptake by the stone are discussed along with pore filling by the treatment (because deterioration processes are driven by water flux, understanding the impact on water movement and pore filling is essential). A two-layer model was applied to describe the imbibition behaviour exhibited by our samples. Depth penetration of the nanoparticles was investigated through scratching and drilling techniques, which are novel methods for quantifying consolidation.

In addition, the efficacy of a nanolime-based repair mortar as an adhesive for repairing detached surface features was assessed through lap shear strength of repaired stone.

CaLoSiL penetration depth into unweathered Portland limestone was found to be < 1 mm. Drilling resistance tests predicted little alteration in compressive strength of stone treated with CaLoSiL. Shear strength of the repair mortar was found to increase with time, probably due to carbonation. While the force per unit area generated by the weight of the re-attached fragment is less than the initial strength of the mortar–stone adhesive bond, the use of this adhesive is favourable.

## Titre et Résumé

### **Utilisation de nanoparticules d’hydroxyde de calcium pour la restauration de la pierre calcaire : imbibition et adhérence**

Le présent article traite des effets de l’application de CaLoSiL, un produit commercial à base de nanoparticules colloïdales d’hydroxyde de calcium (des « nanoargiles ») dispersées dans l’éthanol, sur la pierre de Portland. Il comporte aussi une discussion sur les effets de ce traitement de restauration sur l’absorption d’eau par la pierre et sur le phénomène de remplissage des pores (comme les processus de détériorations sont facilités par les courants d’eau, il est essentiel de bien comprendre les incidences du traitement sur l’écoulement de l’eau et le remplissage des pores). La description du comportement d’imbibition des échantillons mis à l’essai dans notre étude a été réalisée en employant un modèle bicouche. La détermination de la profondeur de pénétration des nanoparticules a été réalisée en employant des techniques de rayage et de forage, lesquelles constituent de nouvelles méthodes de mesure quantitative de la consolidation.

De plus, l'efficacité d'un mortier à base de nanoargiles, utilisé comme adhésif pour réparer des éléments superficiels détachés, a été évaluée en faisant subir à la pierre traitée des essais de résistance au cisaillement des joints de recouvrement.

Les résultats indiquent que la profondeur de pénétration du CaLoSiL dans la pierre de Portland n'ayant pas subi de vieillissement atmosphérique est inférieure à 1 mm. Les essais de résistance au forage permettent de prévoir que la résistance à la compression de la pierre traitée au CaLoSiL ne varie que très peu. La résistance au cisaillement du mortier de réparation augmente au fil du temps, probablement grâce au processus de carbonatation. Bien que la force par unité de surface produite par le poids du fragment fixé de nouveau à la pierre est inférieure à la force d'adhérence initiale de l'ensemble mortier-pierre, l'utilisation de l'adhésif étudié donne des résultats avantageux.

## Introduction

Limestone conservation is relevant to both the built environment and museum artefacts (Dei 2001; Bradley 2005; Dei 2006). Processes of deterioration, such as salt crystallisation in pores and freeze-thaw damage, can cause cracking and are linked with water absorption (Hudson 1971 pp. 106-113; Hall and Hoff 2002). A recently developed conservation method aims to consolidate limestone by application of calcium hydroxide nanoparticles (Giorgi 2000, Giorgi 2010) which penetrate into the material and through carbonation form calcite resulting in consolidation and material strengthening (Drdácký 2009).

CaLoSiL, developed for consolidating calcareous historic materials, is composed of lime nanoparticles suspended in alcohol (Ziegenbalg 2008; Drdácký 2009; Ziegenbalg 2010). It is supplied in a range of concentrations and solvents denoted by the identifier after the brand name, e.g. CaLoSiL E5 consists of 5g particles per litre of ethanol. This study investigated ethanol solutions of the minimum, mid-range and maximum concentrations E5, E25 and E50 respectively. Repair mortar, which is calcium hydroxide and calcium carbonate nanoparticles in ethanol, is a lime mortar (Maryniak-Piaszczyński 2010) designed to repair detached surface pieces.

Portland limestone was chosen because of its popularity as a building material in the UK and large fraction of small pores. Portland Base Bed is a coarse bioclastic oolitic grainstone (Hudson 1971) with a bimodal pore size distribution (Dubelaar 2003). Mercury intrusion porosimetry (MIP) data showed two pore throat size peaks characteristic of a bimodal structure with the maximum peak in the range 0.1 – 1.0  $\mu\text{m}$  and a second peak at 2 – 20  $\mu\text{m}$  giving a micro-porosity of around 60% (pores < 1  $\mu\text{m}$ ) as calculated from data published by Dubelaar (2003, figure 5) presented here in Figure 1. The CaLoSiL particles have a diameter range of 0.05 – 0.25  $\mu\text{m}$  which means that according to Figure 1, showing cumulative intruded pore volume against pore diameter, 35% of the pore space is accessible to the largest sphere while 90% is accessible to the smallest sphere. This highlights the high microporosity of Portland Base Bed and the need to achieve very small particles if deep absorption is desirable.

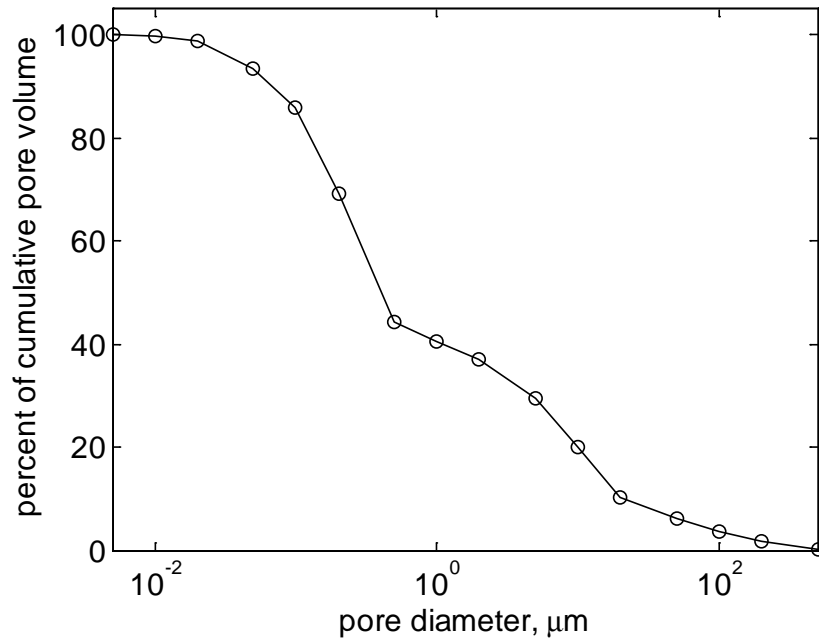


Figure 1: Percentage of cumulative pore volume against pore diameter from MIP data taken from Dubelaar 2003.

## Methods

### Imbibition

Portland stone blocks ( $225 \text{ cm}^3$ ) were dried to constant mass and cooled to room temperature. The sorptivity of each of the 9 blocks used was measured twice, first with ethanol then water uptake for 30 minutes per test and the blocks were oven dried between tests. Sorptivity is the rate at which the imbibing liquid moves through the pore structure and has units  $\text{mm}/\text{min}^{1/2}$ . After measuring the sorptivity of the untreated blocks, they were exposed to CaLoSiL for 30 minutes. The mass of absorbed liquid was measured every 5 minutes to allow determination of CaLoSiL sorptivity, or uptake rate. Blocks were treated 1, 5 and 10 times with CaLoSiL, dried in the oven at  $90^\circ\text{C}$ , then cooled between treatments. After the final CaLoSiL treatment, each block was treated with carbonizing agent, DiLoCarb-E, which reacted with the calcium hydroxide particles to produce calcite faster than reaction with atmospheric  $\text{CO}_2$  alone. A solution was made using a 1:1 ratio of DiLoCarb-E to water and allowed to absorb into the blocks for 4.5 hours to ensure full exposure to the solution. After carbonation with DiLoCarb-E the sorptivity of the blocks was measured again using water and ethanol.

### Penetration depth measurements

A micro-destructive method known as the ‘scratch’ test was used with the aim of determining treatment depth. By tracing several shallow grooves on the surface of the stone the depth penetration of the treatment into the stone could be determined. The normal ( $F_n^c$ ) and tangential ( $F_s^c$ ) forces acting on the cutter as the sample surface was scratched were recorded. Gradually the depth of cut could be increased. The instrument employed for these tests was an Epslog

Engineering WOMBAT Scratch Tool equipped with a 10 mm wide sharp diamond cutter. Data analysis was performed using Epslog Engineering software. The scratches were carved into the stone samples at a depth of 0.10 mm along the entire length of the samples.

Drilling tests were also performed using a Drilling Resistance Measurement System (DRMS) to obtain precise readings. The device, supplied by SINT Technology, was set to operate at 600 rpm using a 5 mm diameter diamond drill bit while the holes were drilled to a depth of 30 mm. This system enables precise drilling resistance measurements on stone, providing a prediction of toughness and uniaxial compressive or flexural strength.

## Modelling

All samples could be considered as two-layer composite materials into which the capillary absorption of water could be analysed using Sharp Front theory (Wilson 1995; Hall and Hoff 2002). The CaLoSiL treated layer and untreated stone were considered two layers, A (of fixed length  $L$ ) and B (indefinite length). Water was applied to the face of A and the wet front progressed through the composite. Known variables were the sorptivity of the composite, sorptivity of the untreated stone and the layer thickness (without carbonation) as determined by optical microscopy. Unknowns were the sorptivity, porosity and permeability of the CaLoSiL layer, which could be estimated from gravimetric data of particle uptake. Knowing this allowed determination of the water transport properties of the CaLoSiL layer itself and then pore filling. Water rise into the sample ( $i$  in mm) is given by equation 1:

$$1) \quad i = S_B(t + L^2 X)^{1/2} - LY$$

where:

$$X = \left(\frac{f_B}{S_B}\right)^2 \left(\frac{K_B}{K_A}\right)^2 - \left(\frac{f_A}{S_A}\right)^2$$

$$Y = f_B \frac{K_B}{K_A} - f_A$$

Here  $S$ ,  $f$ ,  $K$  and  $t$  are the sorptivity, porosity, permeability and time respectively (subscript indicates material) and  $L$  is the length of the CaLoSiL layer.

## Shear test

Repair mortar was used to join two cut Portland stone blocks, each 800 cm<sup>3</sup>. The blocks were painted with a high-contrast speckle pattern to allow the slip displacement across the joint to be assessed using digital image correlation (DIC) (Bisby 2009). The samples were shear tested using an Instron 4505 universal test machine. A gradual force was applied to the dividing plane to generate 0.15 mm per minute crosshead speed whilst the load and crosshead displacement were recorded. Images of the samples under load were taken every two seconds then DIC was performed using GeoPIV software to identify the displacement corresponding to the maximum load at which failure occurred. The crosshead displacement for this test was equal to the movement of the top of the specimen relative to the bottom of the specimen.

## Results and Discussion

### Imbibition

Sorptivity was measured with water and ethanol prior to treatment, CaLoSiL sorptivity was measured during treatment and water sorptivity was measured after treatment and carbonation with DiLoCarb-E. From this data, the mass of CaLoSiL actually in the stone, the expected mass of CaLoSiL in the stone, expected volume percent of pores filled and how far up the stone the CaLoSiL solution or water should travel (height of rise) could be determined. In calculating the expected volume percent of pores filled, an estimate was made of the amount of trapped air in the pores by comparing the capillary moisture content (obtained by imbibition) and total moisture content (obtained by vacuum saturation). Air trapping in Portland Base Bed was 16 – 25% of the total pore space.

The change in composite sorptivity ranged from 3 – 15% varying with treatment type (E5, E25 and E50) and number of treatments as shown in Table 1. From mass measurements before and after treatment (and carbonation) it became clear that fewer CaLoSiL nanoparticles were entering the stone than expected (see Table 1) with roughly 5 – 37% of the expected mass of CaLoSiL nanoparticles actually remaining in the stone. This suggested two possible models for the distribution of particles within the stone: a thin dense layer or a wide sparse layer. Scratching and DRMS could detect no difference in resistance as discussed below therefore further tests were carried out to determine the treatment penetration depth.

Table 1. Measured and calculated property values for CaLoSiL and DiLoCarb-E treated Portland stone. Sw(u) is the sorptivity of the block prior to any treatment. Sw(tc) is the sorptivity of the block after a set number of treatments (1, 5 or 10) with CaLoSiL and then carbonated with DiLoCarb-E. HOR (height of rise) is how far up the block the imbibing liquid travels and was calculated from the final imbibition test with water on the carbonated block. Actual mass of particles in the block was measured gravimetrically after oven drying and between CaLoSiL treatment cycles. The expected mass was calculated from known mass of CaLoSiL solution entering the stone with each cycle and the expected volume percent of particles was calculated from the expected mass.

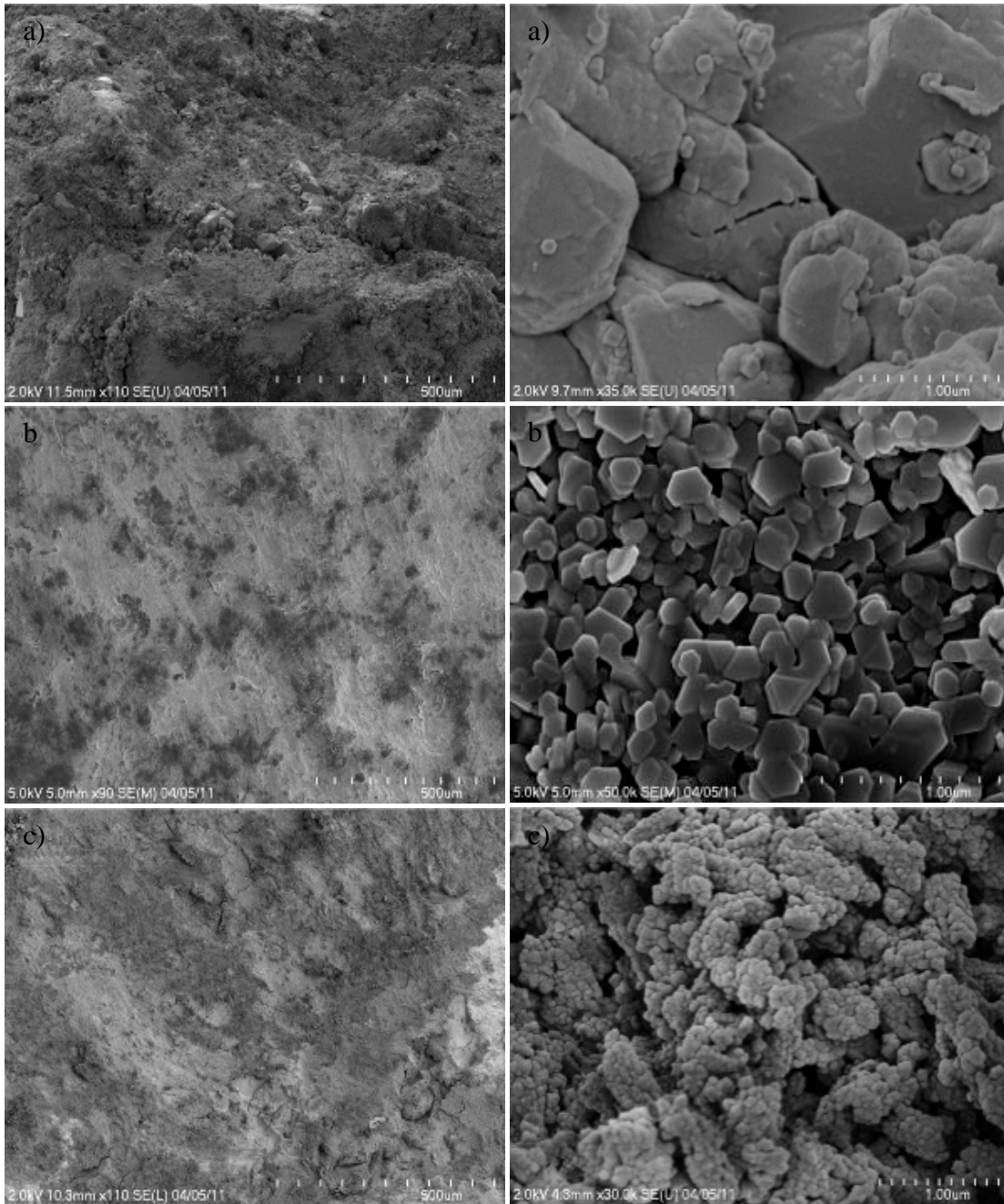
Property	1×E5	5×E5	10×E5	1×E25	5×E25	10×E25	1×E50	5×E50	10×E50
Sw(u) (mm / min <sup>½</sup> )	0.47	0.28	0.42	0.35	0.25	0.34	0.46	0.34	0.26
Sw(tc) (mm / min <sup>½</sup> )	0.43	0.25	0.35	0.32	0.22	0.30	0.45	0.33	0.23
HOR (mm)	13.2	7.5	10.7	9.6	6.6	8.7	13.4	9.6	6.2
expected vol % of pore space filled	0.22	1.1	2.2	1.1	5.5	11.1	2.2	11.1	22.1

expected mass of particles entering (g)	0.04	0.12	0.28	0.2	0.63	1.65	0.41	1.69	2.45
actual mass of particles entering (g)	>0.01	0.04	0.14	>0.01	0.05	0.23	>0.01	0.08	0.23

Test blocks were treated once and five times with E25, cut open and sprayed with phenolphthalein indicator. Depth of penetration after one and five treatments with E25 was 0.4 mm by optical microscopy. The properties of this CaLoSiL layer were determined by modelling as discussed below. It was seen that CaLoSiL did not penetrate far into this stone and as such could only be used to treat surface features. The bimodal pore structure of Portland Base Bed with its high percentage of fine pores is perhaps unusual but should accommodate 35 – 90% the CaLoSiL particles.

Overall, the sorptivity (S) of the composite sample (CaLoSiL layer and bare stone) was reduced by at least half the final reduction observed (after 10 treatments) after the first treatment for E5 and E25. E50 however appeared to reduce the sorptivity by very little until 10 treatments, which is being verified by further work.

High sorptivity reduction, resulting in an impermeable thin layer is not desirable as it may encourage surface flaking from salt crystallisation or freeze-thaw; therefore the smaller reduction in sorptivity produced by CaLoSiL absorption is more likely to avoid these damage mechanisms. The efficacy of CaLoSiL in consolidating stone was examined by Maryniak-Piaszczyński (2008) who found that CaLoSiL with a concentration less than 36 g/L in combination with Funcosil silicic ester successfully consolidated surface flakes, however no quantitative analysis was carried out. Maryniak-Piaszczyński (2008) also found that CaLoSiL concentrations over 36g/L produced a white mist on the stone surface which is in agreement with our experimental observations and can be clearly seen using scanning electron microscopy (SEM). Figure 2 shows SEM images of stone treated with five cycles of E50 before and after carbonation compared to the surface of untreated stone. It can be seen that after treatment with E50 there was a surface layer of hexagonal platelet shaped Ca(OH)<sub>2</sub> which became anhedral after reaction with DiLoCarb-E.



*Figure 2: SEM images showing, at different magnifications  
a) (top, left and right) untreated Portland stone,  
b) (middle, left and right) treated Portland stone, and  
c) (bottom, left and right) treated and carbonated Portland Stone.*

## Penetration depth measurements

WOMBAT and DRMS tests do not detect any real difference in resistance between blank reference and treated samples. This showed that CaLoSiL was not adding resistance to the material through its action which suggested that the treatment was only lining the pores and not acting to block them. Results in Table 1 showing the expected volume percentage of pore space filled is an expression of the maximum possible mass of particles entering the stone only and does not differentiate between particles blocking pores and lining them.

## Modelling

Water transport properties of the CaLoSiL layer and pore filling were investigated using depth information from optical microscopy and by comparing experimental imbibition data (after treatment and carbonation) with predicted imbibition from the sharp front model. Shown in Figure 3 is water uptake in a block treated ten times with E25 and carbonated with DiLoCarb-E. Fitting the experimental data with the model confirmed that CaLoSiL existed as a very thin layer which was known to be c. 0.4 mm thick (from optical microscopy), reducing the porosity from c.19% to c.14% based on the known quantity of CaLoSiL particles in the stone after 10 treatments with E25 (determined gravimetrically). The corresponding estimated permeability reduction was around 40% based on the relationship between permeability and porosity. Sorptivity of the layer was difficult to determine experimentally as the layer was very thin but it was also estimated from the porosity reduction. As shown in Figure 3a, the upwards curve modelled at early time (blue circles) before one minute corresponds to water flow through the thin CaLoSiL layer. The downwards curve in the experimental data (black circles) at later time probably resulted from a band of stone with lower sorptivity. Portland Base Bed is known to have features such as vugs, small to medium sized cavities usually lined with crystals, (Roels 2000) which affect water movement. If the CaLoSiL layer was thicker than say 1 mm, there would be an upwards curve in the sorptivity plot as shown in the lower curve of red stars in Figure 3b which was not observed (upper curve in 3b is a repeat of 3a for direct comparison). This confirmed that the CaLoSiL layer was thin and remained permeable.

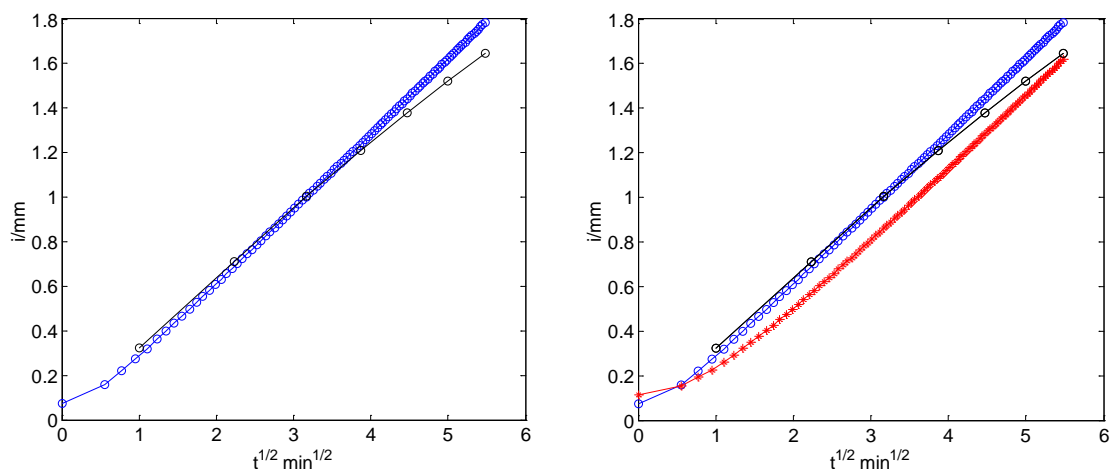


Figure 3a (left) and 3b (right): Model (blue and red) versus experimental (black) water uptake data for Portland stone after ten treatments of CaLoSiL E25 and carbonated. The blue curve is the fit to the data when the treated layer is as measured (0.4 mm) and the red curve is the fit if the layer is thicker (1 mm).

## Shear test

The mortar shear strength increased over time (Figure 4). The mortar was five times as strong after 25 hours as it was after 15 minutes. The rate of strength increase was found to reduce over time. The average adhesive strength from a series of measurements was  $0.020 \text{ N/mm}^2$ ,  $0.034 \text{ N/mm}^2$  and  $0.099 \text{ N/mm}^2$  when tested 15 minutes, 25 minutes and 25 hours respectively after attachment with repair mortar.

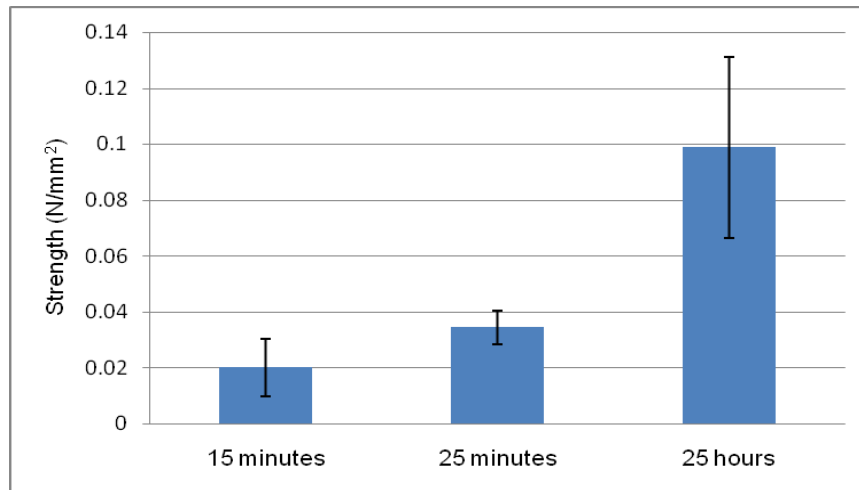


Figure 4: Comparison of adhesive strength with time.

Figure 5 gives an indication of the local joint behaviour under load for samples tested at 25 minutes and 25 hours after forming the adhesive joint. The figure shows the crosshead displacement of the machine (which was the combination of shear displacement across the adhesive joint and “bedding in” of the loading supports into the masonry blocks); however, the difference in stiffness in the two groups of specimens was clear and indicated by the dashed lines on the plot. It was noted that the samples tested after 25 hours required a larger force to cause adhesive failure than for those tested after 25 minutes, thus the adhesive bond was stronger with time. The slip displacement was the distance one of the blocks comprising the sample was able to move before the adhesive bond failed and starts from zero on the x-axis. Negative x-axis values represent sample and loading support “bedding in”. It was observed that the displacement allowed before failure was very small for both sets of samples and reduced with time, therefore reducing the likelihood of being able to reposition stuck items.

It was hoped that DIC could be used to measure the local displacement behaviour across the dividing plane, but this method was not reliable due to the very small displacements involved (as can be seen from the x-axis values in Figure 5).

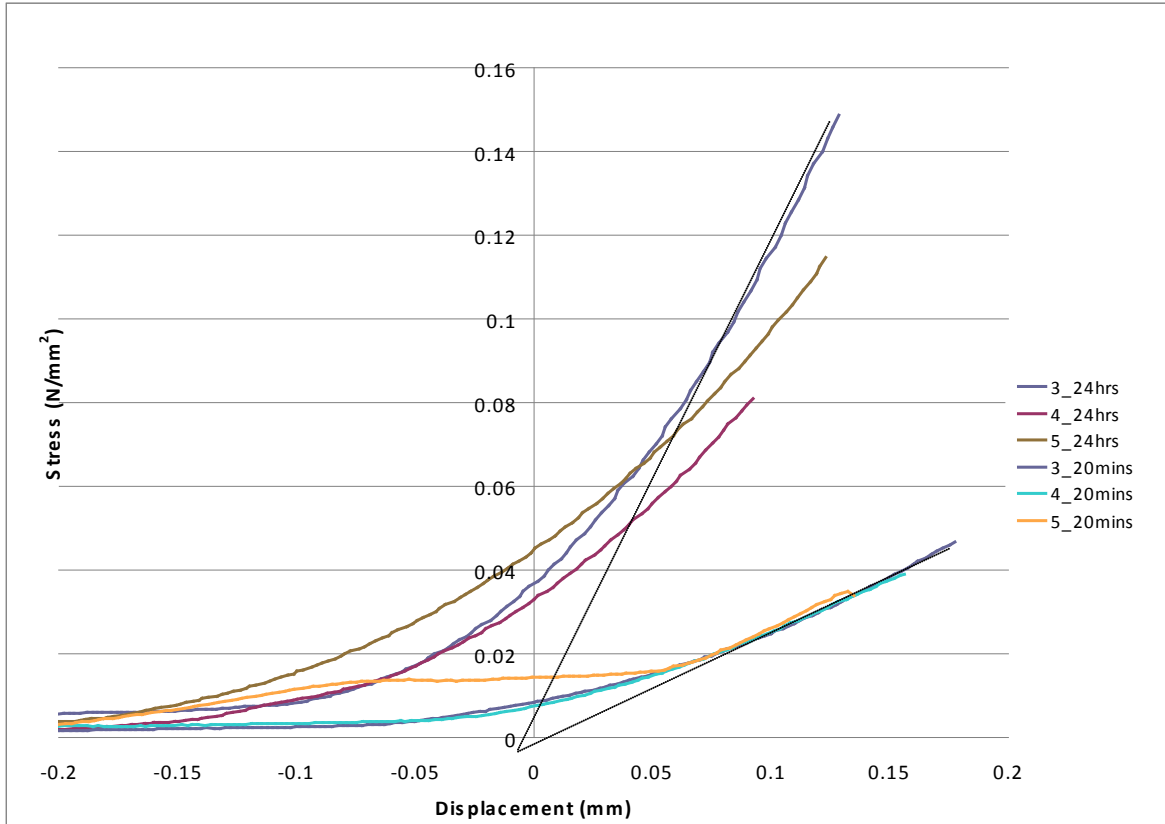


Figure 5: Stress versus crosshead displacement at different times

It can be seen that post-failure the mortar was powdery and that the surface of the glued face differed also with time, appearing less friable after 25 hours compared with the surface after 25 minutes (Figure 6). This suggested natural carbonation was responsible for the strength increase (DiLoCarb-E was not used with repair mortar). Failed samples were tested with phenolphthalein causing uncarbonated material to become pink (Figure 7). It was found that after 25 hours the mortar near the edges of the sample had carbonated whereas after 25 minutes no carbonation had occurred.



Figure 6: Failed surface at 25 minutes (left) and 25 hours (right).



*Figure 7: Phenolphthalein treated failure surfaces exposed by strength testing 25 hours (left) and 25 minutes (right) after adhesion. Pink areas, with higher pH, are uncarbonated.*

## **Conclusion**

Treatment of Portland base bed with CaLoSiL resulted in a 2 – 15% reduction in stone sorptivity varying with number of treatments and CaLoSiL concentration. The first application resulted in the most sizable reduction in sorptivity overall. CaLoSiL was found to exist as a very thin permeable layer (~0.4 mm thickness) and ten treatments of E25 resulted in a porosity reduction from 19% to 14% in the CaLoSiL layer and estimated permeability reduction by ~40%, in this case, in the layer. The compressive strength of CaLoSiL treated stone appeared unaffected as determined by DRMS and scratch tests. Imbibition with CaLoSiL resulted in a very thin layer which did not block pores. Despite the small CaLoSiL particle size, it did not travel far into the pore network in this case. Work is underway to determine what impact pore size and structure have on CaLoSiL uptake and efficacy as a consolidating agent with particular emphasis on weathered stone.

For repair mortar it was found that a displacement shift of < 0.2 mm was sufficient to give adhesive failure. This signified approximately by how much an object stuck with repair mortar could be repositioned. Maximum strength was obtained after 25 hours which probably resulted from carbonation as the tests using phenolphthalein indicator suggested. During the time periods explored the adhesive bond strength was notably less than the tensile strength of the stone itself. Further work is underway to determine strength change over longer time periods for comparison with standard ISO strength requirements for dispersion and cementitious adhesives (Bowman, 1998).

## **Acknowledgements**

PhD studentship funding from the Engineering and Physical Sciences Research Council. We thank Christopher Hall for fruitful discussions, Mike Hall and Christopher Jeffree for technical assistance. We employed GeoPIV courtesy of Andrew Take of Queen's University, Canada.

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## **Materials and Suppliers**

CaLoSiL (all concentrations): Calcium hydroxide, ethanol.

DiLoCarb-E: Diethylcarbonate, ethanol.

Repair Mortar: Calcium hydroxide, calcium carbonate, ethanol.

All above products obtained from:

IBZ-Salzchemie GmbH & Co.KG

Halsbruecker Strasse 34

09599 Freiberg

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