Pore-structure and water transport properties of surface-treated building stones

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ABSTRACT

This paper describes the details of a laboratory investigation aimed at ascertaining the effect of penetrating surface treatments on the micro-porosity and water transport properties of three building stones used in the construction of historic buildings and monuments within Europe. The pore-structure and morphology of treated and untreated stone specimens were assessed using a combination of helium autopycnometry, mercury intrusion porosimetry and scanning electron microscopy techniques. Water repellence and transport properties were evaluated using the Karsten tube and magnetic resonance imaging techniques respectively.

It was found that surface penetrating treatments have a pore-refining effect on the stone matrix, the extent of internal alteration being dependent on the initial pore-structure. A treatment efficiency index has been established for evaluating the performance of treated surfaces.

RESUME

Cet article présente d'une manière détaillée une étude en laboratoire avec pour objectif d'identifier l'effet des traitements de surfaces sur la micro-porosité et les propriétés de transport de l'eau de trois principales sortes de pierre de construction utilisées dans la réalisation d'édifices historiques et de monuments en Europe.

La structure des pores et la morphologie d'échantillons de pierre traitées et non-traitées ont été évaluées au moyen d'une combinaison d'hélium auto-pycnométrie, de porosimétrie de mercure et de techniques de microscopie par balayage d'électrons. Les propriétés de transport-hydrophobiques ont été évaluées en utilisant la méthode du tube Karsten et la technique de l'image de résonance magnétique.

Les résultats de l'étude ont montré que les traitements aux surface pénétrantes ont un effet de raffinement des pores sur la matrice de la pierre mais que la mesure de changement intérieur dépend de la structure initiale des pores. Un index d'efficacité de traitement a été établi pour l'évaluation de la performance des surfaces traitées.

1. INTRODUCTION

An understanding of both the internal changes likely to occur in building stones after penetrating treatment and the performance limits of a given treatment are essential to enable a correct diagnosis of the condition of the treatment and to reach an informed decision on expected level of protection enjoyed by a treated structural surface. Whilst there is a rich body of published literature on surface treatments in general, detailed data on the micro-structural alteration caused by surface treatment is scarce. There is also insufficient data on how damage mechanisms may be influenced by the change in micro-porosity. For instance, consolidating treatment may result in very fine pores which may not necessarily improve durability to frost attack or salt crystallisation in certain situations [1]. In addition, there are conflicting reports regarding the effect which penetrating sealers have on the micro-structure and transport properties of treated stone faces. Some researchers have reported that hydrophobic treatments have no effect on the micro-porosity [2]. Others have found that such treatments reduced the micro-porosity of the test specimens [3].

The objectives of this investigation were (a) to establish the extent of micro-structural modification which occurs when stone faces are treated either with a hydrophobe or a consolidant; (b) to establish the limitations of the effectiveness of surface treatment; and (c) to provide a method to enable a quick but reliable interpretation of test results.
2. MATERIALS USED AND THEIR CHARACTERISTICS

2.1 Stone specimens

The three types of stone used in this study were Ebenheider red sandstone, Rüthener green sandstone and Krensheimer limestone, samples of which were obtained from quarries in Germany. These stones were selected to represent some of the types frequently encountered throughout Europe. Specimens of each stone type were supplied in the form of prisms of dimensions $50 \times 50 \times 100$ mm, and were coated along their length with epoxy resin. The total porosity and density of the untreated stone samples were determined using helium autopycnometry and mercury intrusion porosimetry, Table 1.

<table>
<thead>
<tr>
<th>Stone Type</th>
<th>Total Porosity (%)</th>
<th>Density (g/cm$^3$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ebenheider red sandstone</td>
<td>17.70</td>
<td>2.66</td>
</tr>
<tr>
<td>Rüthener green sandstone</td>
<td>25.20</td>
<td>2.73</td>
</tr>
<tr>
<td>Krensheimer limestone</td>
<td>9.63</td>
<td>2.72</td>
</tr>
</tbody>
</table>

2.2 Treatment and conditioning

2.2.1 Treatment materials

The stone specimens were treated at one end with one of two materials. These were a commercial hydrophobic treatment based on an oligomeric alkyl alkoxysilane (treatment 'H'), and a consolidating treatment based on a silicic acid ethyl ester (treatment 'S'). Treatment 'H' was diluted with white spirit to a ratio of 1:1.5 prior to application while treatment 'S' was used as-received from the manufacturer and without the addition of a diluent.

2.2.2 Treatment application

Treatment was applied by capillary suction into the samples in accordance with the IBAC method [4]. This involved weighing the untreated test specimens in air; immersing the top $5$ mm of one face of each specimen into a bath containing the impregnation material, Fig. 1, for a period of four hours and then reweighing the specimens. The measured increase in weight was then used to calculate the weight of treatment uptake per unit area, Fig. 2(a).

The highly porous Rüthener green sandstone absorbed 12.4 kg/m$^2$ of treatment 'H' compared to 3.1 kg/m$^2$ absorbed by the Ebenheider red sandstone. The Krensheimer limestone absorbed only 0.5 kg/m$^2$ of treatment 'H' over the same period reflecting its lower total porosity. It should be noted that only the Ebenheider red sandstone was treated with the consolidant treatment 'S'. It absorbed the equivalent of 2.1 kg/m$^2$.

The specimens were cured at 50±5% RH and 23±2°C for a period of 28 days after which the specimens were retreated following the same procedure, Fig. 2(b). The presence of the initial treatment restricted the quantity of treatment 'H' that could be absorbed into the samples to between 15 and 30% of that observed in the untreated material. The repeated application of the consolidant treatment 'S' to the Ebenheider red sandstone produced an uptake of 30% of that observed in the untreated stone. These values of uptake were considered to be quite high and so after the second application the stones were cured for a further 5 days at 50°C to help promote the full cure of the two treatments. As a check on the efficiency of this curing process the stones were