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# Weathering and consolidation trials on sandstones used in Irish monuments

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## Abstract

The objective of this study was to assess the efficiency of consolidant treatments in improving the durability of Irish sandstones, particularly sandstones used in national monuments at Cashel, Clonfert and Clonmacnoise. Although not located directly on the coast, these sites have been severely exposed to combinations of wind-driven rain, high levels of moisture from groundwater and salt decay. Samples of the sandstones were characterised and artificially weathered. The majority of salts were washed from the samples and they were then treated with alkoxysilane consolidant treatments.

Consolidant treatments were modified by the addition of solvents and nanoparticles; the former to aid penetration in low porosity samples and reduce film thickness, the latter to increase the gel's elastic modulus, improving cohesion and shrinkage resistance. Further weathering tests were also carried out in order to compare the durability of the treated and untreated samples. Surface-substrate interface stresses due to poor penetration of consolidant were observed.

According to the results of this research, the alkoxysilane consolidants improved cohesion between constituents and the stone's durability to both thermal and crystallisation stresses.

*Keywords:* Accelerated weathering, Consolidants, Durability, Particle modification, Sandstone

## 1. Introduction

Ireland has a wealth of monuments charting the history of Celtic tradition through Early Christian times. The national monuments at Cashel, Clonfert and Clonmacnoise include the seats of High Kings and centres of Christian teaching and scholarship. Their stature and importance were marked by sophisticated and intricate sandstone carvings. The monuments attract thousands of overseas visitors every year and are in the care of the Office of Public Works (OPW).

Located on the western edge of Europe, Ireland's climate is strongly influenced by the Atlantic Ocean. After over 800 years of exposure to the environment the carvings are in need of conservation. Although not located directly on the coast, these sites have been severely exposed to combinations of wind-driven rain, high levels of moisture from groundwater and salt decay. The OPW are endeavouring to consolidate the monuments against continued weathering.

Consolidation of stone is not without its own risks – fine pores may become blocked, exacerbating decay due to trapped moisture, or insufficient penetration of the consolidant can lead to differential thermal stresses and surface scaling. In many previous cases consolidation resulted in only short term benefits, before major losses occurred, often because of treatment incompatibility.

The objective of this study was to assess the efficiency of consolidant treatments in improving the durability of sandstone monuments using laboratory testing. Several attitudes influenced the approach to testing, the key variable being resistance to weathering. The consolidant to be used needed to be well understood and the testing undertaken had to provide enough information to accomplish this. Disparities between laboratory testing and what is achievable on-site were also highlighted as concerns.

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# 2. Experimental program

## 2.1 Materials

Samples of the relevant sandstones were obtained from the Office of Public Works: Drumbane sandstone, Manorhamilton sandstone, Clara Hill sandstone and Killaloe sandstone. The majority were cut into 5cm cubes.

The following products were obtained from standard laboratory suppliers: Sodium sulphate decahydrate salts ($Na_2SO_4.10H_2O$), Butanone solvent, PPE, natural bristle brushes, plastic containers, sponges.

The following products were obtained from specialist suppliers or the manufacturers: Tetraethoxysilane consolidants – Wacker SILRES BS OH100 (from UK supplier) and Tegovakon V100OH (courtesy of Degussa Gmbh), 10nm Silica nanoparticles and 45nm Alumina nanoparticles (from Sigma-Aldrich).

## 2.2 Methods

### 2.2.1 Characterisation

Thin sections were prepared to study the petrographic characteristics of the obtained sandstone samples, based on BS EN 12407:2000. A combination of RILEM PEM-25 and BS EN standards of physical laboratory testing of natural stone were used to determine their apparent density & open porosity (BS EN 1936:1999), capillary absorption & evaporation (RILEM-PEM25 II.5-II.6), and compressive strength (BS EN 1926:1999) and elastic modulus properties.

### 2.2.2 Artificial weathering

The samples were subjected to a series of salt crystallisation tests to assess their response to weathering processes. For 15 cycles, the samples were partially immersed for 2 hours in sodium sulphate decahydrate solution (14% by mass) and then placed in an oven at 105℃, as per RILEM PEM-25 (partial immersion method). By immersing to within 2-3mm of the sample tops, the weathering mechanisms were concentrated on the exposed surface (McMahon *et al* 1992).

The majority of salts were washed from the samples using combinations of surface washing and immersion in containers with constantly flowing water. Their physical properties were determined again to evaluate the changes in the mass structure, moisture transfer and mechanical strength properties.

### 2.2.3 Consolidant treatment

Most of the samples were then treated with alkoxysilane consolidant treatments. Consolidant treatments were modified by the addition of butanone solvents and both silica and alumina nanoparticles. 20% by mass of solvents were added to treatments for application to the more durable/less porous proportion of sandstone samples – 'TEOS+MEK treatment'. Similarly for the less durable/more porous proportion, 10.5% by mass of butanone, 2.0% by mass of 10nm silica and 0.5% by mass of 45mn alumina were added ('Particle Modified Consolidants' – see Miliani *et al* 2003) – 'TEOS PMC treatment, high nanoparticle content' (~25% by volume).

The treatments were applied to the most weathered face of each of the samples, facing upwards, using a natural bristle brush until no more was absorbed 2-3 minutes after application. The excess was removed using a solvent soaked cloth. After 1 month the physical properties of the samples were determined again.

The performance of the consolidant treatments was analysed using relationships between the measured physical properties. Estimations of the depth of penetration that were achieved were also made using the Water drop absorption and Surface wetting methods (Leroux, Verges-Belmin *et al* 2000, Young, Cordiner & Murray 2003). Comparisons of the untreated and treated samples' durabilities were determined by subjecting them to further weathering.

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# 3. Results and discussion

## 3.1 Sandstone composition

The composition and petrographic characteristics of the samples are presented in Table 1:

*Table 1: Composition and petrographic characteristics of sandstone samples*

| Consituent | Drumbane sandstone | Manorhamilton sandstone | Clara Hill sandstone | Killaloe sandstone |
|---|---|---|---|---|
| Quartz | 50-60%, medium-fine, <4% cementing | >70%, medium-v. fine, compact, sparsely cementing | 60-80%, medium-fine, occasional to sparse cementing | 80-85%, medium-fine, polycrystalline, compact, cementing |
| Iron Oxide | 6-15%, secondary, layered | <6%, fine | 7-15%, fine, secondary | 5-10%, secondary |
| Fine matrix (*or other*) | 15-30%, interstitial, cohesive | 5-10%, occasionally cohesive | 10-15%, cohesive | **<5% dolomite and microcline* |
| Pores | 4-10%, medium-fine, evenly distibuted | 10-12%, medium-fine, evenly distibuted | 5-15%, up to 25-30%, fine-coarse, poorly distributed | 1-5%, medium-very fine |

The Drumbane sandstone, present in Cormac's Chapel on the Rock of Cashel, varies from grey to rusty in colour depending on the exposure of iron oxides by weathering of secondary fine matrix.

The Manorhamilton sandstone is white-buff-sandy in colour, and has compact quartz content with little cementation. It has relatively low matrix content in places and high evenly distributed porosity.

The Clara Hill sandstone, present in structures at Clonmacnoise, has weaker cementation and poor distribution of pores. Varies from poorly interconnected low porosity material to coarsely textured and sparsely cemented high porosity material with little or no matrix content.

The Killaloe sandstone, present in St. Brendan's Cathedral at Clonfert, is very dense and strong due to compact polycrystalline structure and generally has low porosity. It is varied in colour due to the minor iron oxide and occasional feldspar contents.

## 3.2 Material lost and decay types caused in sandstone

The type of decay caused in the samples by the salt crystallisation cycles was influenced by their petrographic characteristics. The weathering was observed to reflect the differences in grain size, cementation and packing of fine matrix content of the sandstones. The distribution of material losses is also influenced by the type of decay caused, as shown in Table 2 on the next page. Where significant differences were evident in the samples they have been subdivided.

The Drumbane sandstone in the west side of Cormac's Chapel is exposed to the prevailing south-westerly wind and rain, resulting in differential erosion, as shown in Figure 1. The sheltered east side is damp, resulting in a blistered surface that is flaking, as shown in Figure 2.

The Killaloe sandstone in the doorway of St. Brendan's cathedral has been exposed to high moisture and salt contents for so long that the highly strong and durable stone has become weak and friable. The granular disintegration responsible for the current appearance of the stone was only minutely evident in the samples weathered in the laboratory, as shown in Figure 3.

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*Table 2: Material losses and ranking of decay types evident in samples after artificial weathering*

| Weathering: | Drumbane sandstone | | | Manorhamilton sandstone | Clara Hill sandstone | | Killaloe sandstone | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| Change in mass (%) | -0.7 | -2.2 | -6.0 | -3.5 | -1.5 | -8.5 | -0.1 | -0.3 | -0.3 | -1.7 |
| Change in mass ($g/m^2$) | 210 | 500 | 1320 | 780 | 640 | 1970 | 40 | 80 | 70 | 400 |
| Fracturing | 1 | 2 | 3 | - | 1 | - | - | - | - | - |
| Flaking | 2 | - | - | 1 | 3 | - | 1 | - | 1 | - |
| Granular Disintegration | - | 3 | 2 | - | 2 | 2 | - | - | - | 2 |
| Differential Erosion | - | 1 | 1 | 2 | - | 1 | - | 1 | 2 | 1 |

The fracturing is caused in samples where cohesion is not sufficient to withstand the stresses generated in the pores (Scherer 1999, Flatt 2002). Poorly interconnected pores are quite vulnerable to higher crystallisation pressures. Fracturing was observed to occur in samples that had poor distribution of porosity (as seen in thin sections). Layered structures were also more vulnerable as stresses overcame cohesion along paths through the weaker layers of the material.

The other decay types – flaking, granular disintegration and differential erosion – also reflect the response of combinations of cementation and constituent packing to weathering. In this instance, flaking is limited to the outermost surface and involves the loss of cohesion from grains that are coming away from the stone but remain connected to each other (more localised concentrated loss of cohesion), while granular disintegration can progress deeper because it is due to total loss of cohesion between individual grains. Differential erosion is evident where locally weaker material is removed from the surface (Pavía & Bolton 2002).

*Fig. 1 Differential erosion, Drumbane sandstone*

*Fig. 2 Flaking, Drumbane sandstone*

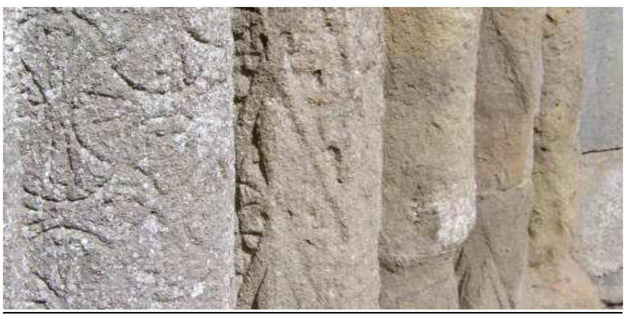

*Fig. 3 Granular disintegration, Killaloe sandstone*

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## 3.3 Performance of consolidant treatments

### 3.3.1 Mass deposited and depth of penetration

The absorption of the consolidants was evaluated based on the bulk open porosity and capillary absorption of the samples before application, the mass of consolidant deposited and the depths of penetration achieved, as shown in Table 3:

*Table 3: Mass of consolidant deposited and estimated depth of penetration in sandstone samples (average for all treatments taken together)*

| Treatment: | Drumbane sandstone | | | Manorhamilton sandstone | Clara Hill sandstone | | Killaloe sandstone | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| Open porosity before (%) | 4.0 | 8.6 | 12.9 | 11.6 | 6.6 | 16.4 | 1.4 | 1.7 | 6.5 | 7.8 |
| Capillary absorption before ($g/m^2/s^{0.5}$) | 11.5 | 42.2 | 50.9 | 9.5 | 31.1 | 38.1 | 2.1 | 3.0 | 3.7 | 16.2 |
| Change in mass (%) | +0.3 | +0.4 | +1.7 | +0.9 | +0.8 | +1.1 | +0.1 | +0.1 | +0.1 | +0.4 |
| Increase in mass/area applied ($g/m^2$) | 370 | 450 | 1060 | 1100 | 820 | 1320 | 120 | 130 | 190 | 510 |
| Depth of penetration (mm, range) | 12<br>9-15 | 11<br>5-18 | | 16<br>6-30 | 11<br>5-20 | 11<br>11-13 | 11<br>5-18 | 12<br>5-20 | 12<br>8-13 | 12<br>5-16 |

Comparison of the open porosity and capillary absorption properties of the samples and the resultant mass changes due to treatment showed no consistent relationship between them. Also, the average depth of penetration was between 11 and 12mm. The spread of the minimum and maximum readings about this average changed, with no obvious relationship to petrographic characteristics, open porosity or capillary absorption. This reflects the variation in the applied consolidant treatments, as outlined in Table 4 (i.e. solvents added in treating less porous samples or PMCs applied to more porous samples, NB: open porosity of samples prior to treatment).

*Table 4: Average mass gain per unit area and open porosity according to treatment type (average for all stone types taken together, equal number of specimens from each type)*

| Sample \ Product applied | Wacker | Wacker +MEK | Wacker PMC | Tegovakon | Tegovakon +MEK | Tegovakon PMC | Average |
|---|---|---|---|---|---|---|---|
| Average mass gain per unit area ($g/m^2$) | 770 | 670 | 500 | 510 | 580 | 680 | 620 |
| Average open porosity (%) | 8.0% | 7.3% | 8.7% | 7.7% | 6.7% | 8.6% | 7.8% |

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### 3.3.2 Resistance to further weathering

On comparison of the material lost by untreated and treated samples after a further series of the salt crystallisation cycles, it was observed that the magnitude of losses was reduced because of consolidant treatment, as shown in Table 5:

*Table 5: Material losses in Untreated and Treated samples after further artificial weathering*

| Further weathering: | Drumbane sandstone | | | Manorhamilton sandstone | Clara Hill sandstone | | Killaloe sandstone | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| Untreated (g/m$^2$) | 540 | 1580 | 9500 | 660 | 2860 | 2980 | -40 | 0 | -10 | 0 |
| Treated (g/m$^2$) | 110 | 300 | 1430 | 140 | 530 | 780 | -60 | -10 | 60 | 280 |

It is obvious from the values reported here that the Untreated samples lost several times more material than the Treated ones did. The negative values in the Killaloe sandstone indicate a net accumulation of salt in the stone.

During the 2$^{nd}$ series the progress of visual decay features seemed to be just as bad as the 1st. The losses reported in Table 5 are for the most part lower than those reported in Table 2 for the 1$^{st}$ series of salt crystallisation cycles however.

To illustrate the differences in treatment type, the average material losses in samples are presented in Table 6 according to the treatment product applied. This also shows the open porosity of the samples both before and after the 2$^{nd}$ series of artificial weathering.

*Table 6: Material losses and open porosity of samples before and after 2$^{nd}$ artificial weathering*

| Product applied / Sample | Wacker | Wacker +MEK | Wacker PMC | Tegovakon | Tegovakon +MEK | Tegovakon PMC | Untreated |
|---|---|---|---|---|---|---|---|
| Average mass loss per unit area (g/m$^2$) | 160 | 129 | 475 | 203 | 230 | 278 | 1679 |
| Average open porosity before (%) | 9.2 | 8.8 | 8.2 | 10.6 | 8.8 | 6.6 | 10.0 |
| Average open porosity after (%) | 8.2 | 7.3 | 10.0 | 7.6 | 8.1 | 9.9 | 9.9 |

The Wacker treatments seemed to be more successful than the Tegovakon ones based on this data but this is inconclusive when relative changes in porosity are considered. Greater losses were observed in untreated samples. The most notable point is that the high content PMC caused more damage than the other treatments, and this was related to significant blockages in the outer pores.

Rupturing in 1 PMC-treated sample was observed during salt rehydration at the treated surface, as blocking pores results in greater stresses. This was an isolated case, but it illustrates how PMC's, and consolidants, affect modulus of elasticity, and can be detrimental in unfavourable conditions.

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## 4. Conclusions and Acknowledgements

The physical properties and weathering behaviour of sandstone reflect their petrographic characteristics, packing and cementation in particular. Particle Modified Consolidant treatments may improve the ability of TEOS and other alkoxysilane consolidants to bridge gaps between particles where weathering is significant.

When treatments are tailored according to the sandstone’s porosity (i.e. additional solvents in less porous/PMCs in more porous material), treatment increases sandstone’s resistance to further weathering.

The nanoparticles in high content PMC’s agglomerate very close to the outer surface, often causing blockages and increasing surface modulus. This may occasionally lead to rupturing or scaling of the surface if exposed to unfavourable conditions, especially thermal differential or crystallisation stresses. Only treatments containing lower nanoparticle content should be used to avoid these problems. Higher nanoparticle size would also reduce the extent of agglomeration. This will be discussed further in a future publication.

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